5th Meeting on

Chemistry & Life

BRNO, Czech Republic
September 14-16, 2011

Organized by Faculty of Chemistry
Brno University of Technology

The Fifth Meeting on Chemistry & Life was held in the beautiful city of Brno which is the educational and cultural centre of the South Moravian part of the Czech Republic. It draws upon the tradition of four annual meetings providing a forum for exchange of ideas on recent advances in research and development in chemistry, biotechnology, materials science and environmental technology for people from industry, research and academia. The conference was held as a part of events organised on the occasion of the 100th anniversary of the estabilishment of Faculty of Chemistry under the auspices of the rector of Brno University of Technology Prof. Karel Rais, MBA.

THE SUBJECT SESSIONS

1. Physical & Applied Chemistry (head of the session: Miloslav Pekař)
2. Materials Chemistry (head of the session: Josef Jančář)
3. Environmental Chemistry & Technology (head of the session: Josef Čáslavský)
4. Food Chemistry & Biotechnology (head of the session: Jiřina Omelková)

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TABLE OF CONTENTS

PREFACE

Dear participants of the Chemistry and Life 2011 conference, like three years ago, this year a special issue of the Chemické listy Journal with contributions presented at this 5^{th} successive conference Chemistry and Life has been delivered to you again Since 1999 the conference Chemistry and Life has regularly been organized by Faculty of Chemistry of Brno University of Technology (BUT), it has become one of the most significant items in the field of science and research activities implemented by the faculty. Specialists have shown increasing interest in the conference and the present Organizing Committee has received more than 300 contributions that will be presented in four sessions. Besides the traditional attendance of Czech and Slovak chemists we also appreciate increasing number of contributing scientists and researchers from abroad.

Fruitful cooperation with industry enables implementation of the conference in broad extent designed by the organizers. The conference committees are pleased to offer accompanying social programme that might contribute to successful course of the conference.

Dear participants of the 5th Chemistry and Life conference, on behalf of both the Organizing and Scientific Committees I feel honoured to welcome you to the grounds of Faculty of Chemistry of BUT. At the same time let me express the hope that the conference outcomes will contribute to development of knowledge in various branches of chemistry.

Prof. Jaromír Havlica,
Dean of Faculty of Chemistry of BUT
Chairman of the Scientific Committee
of the Chemistry and Life 2011 conference

PL1

METABOLOMICS: A CHALLENGING TOOL FOR THE ASSESSMENT OF THE ENVIRONMENTAL IMACTS ON FOOD CHAINS

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At the time of its emergence, metabolomics was mainly viewed as an advanced, specialised tool of analytical biochemistry enabling innovative research on plants and other organisms. Recently, this "omics" strategy centred around detection of the broadest possible range of small molecules (<1,500 Da) in complex biological matrices using a single or small number of analyses has also been introduced into various fields environmental sciences. Metabolomics may be used either for "fingerprinting" of samples to perform e.g. comparative analyses aimed at detection of differences or for "profiling" in which individual, differential sample components (markers) are identified for further investigation.

A lot of scientific effort has been spent to develop rapid, reliable, and cost effective analytical approaches applicable for effective fingerprinting/profiling examinations. Besides of spectroscopic techniques employing nuclear magnetic resonance (NMR), Raman, or infrared spectra, a wide range of methods based on gas chromatography–mass spectrometry (GC–MS), and/or high-performance liquid chromatography (HPLC) hyphenated to MS with atmospheric pressure ionisation (API) have been implemented for this purpose.

Over the few recent years, a large number of novel ambient desorption ionisation techniques, have become available. When coupled with high resolution MS, chromatographic sepatration can be omitted. The main advantages of ambient MS involve: (*i*) easy method development and optimisation, (*ii*) significantly reduced workload and, consequently (*iii*) increased laboratory throughput. One of the most challenging ionization techniques in this field has become Direct Analysis in Real Time (DART), which was used in our study for metabolomic fingerprinting[1]. The generated mass spectra were used to assess an extent and nature of metabolome responses in carp (*Cyprinus carpio*) under various stress conditions occurring in the aquatic environment (samples obtained within the COST 867 project[2]). Generally, it should be noted, that concerns on stress issues are not only associated with welfare of farmed fish but also distinct relationship between aquaculture practices and quality of fish has to be taken into consideration. Interestingly, several earlier studies documented the influence of feeding history on the start of stress response demonstrated by changes in levels of various markers in plasma; the symptoms were more pronounced in well fed fish compared to those rather starving prior to sampling.

In our experiments we separately examined nonpolar and polar fraction of the extracts obtained from various experimental fish. An example of positive and negative mass spectra obtained by DART–TOFMS technology is shown in Figures 1A and 1B. Partial least squares–linear discrimination analysis (PLS-LDA) was employed for markers processing. In Figure 2, LDA separation of 4 carp groups grown under different feeding conditions (farming: natural pond/fish pond *vs.* natural feed (benthos)/supplementary feed (triticale) is shown. In this case, a prediction ability of 100% was obtained using negative DART–TOFMS spectra.

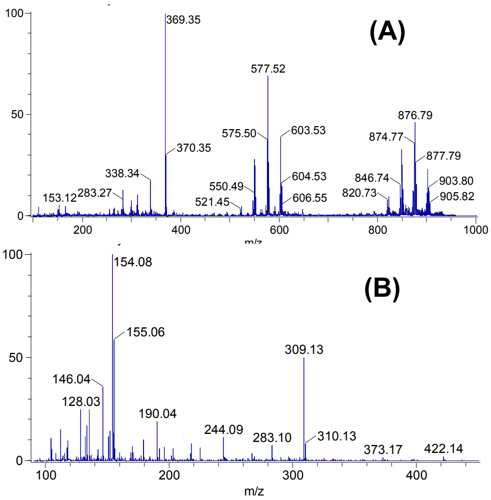

Figure 1. DART–TOFMS mass spectra of (A) nonpolar and (B) polar fish extracts.

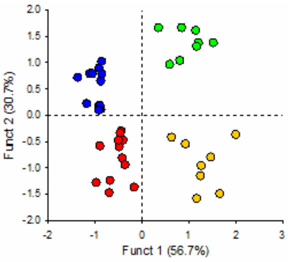

Figure 2. Score plot of two discriminant functions of LDA calculated from DART–TOFMS spectral data of carp samples fed under different conditions: natural pond/natural feed (left-down), natural pond/supplementary feed (left-up), fish pond/natural feed (right-down), fish pond/supplementary feed (right-up)

This work was supported by the Ministry of Education, Youth and Sports of the Czech Republic (projects COST 867–OC09063 and MSM 6046137305).

REFERENCES

1. Hajslova J., Cajka T., Vaclavik L.: Trend Anal. Chem. *30*, 204 (2011).
2. http://w3.cost.esf.org/index.php?id=181&action_number=867 [cit. 2011-11-05].

PL2

ENGINEERING ADVANCED MATERIALS THROUGH PRE-STRESSED DOUBLE NETWORK POLYMER SYSTEMS

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This presentation illustrate how the application of an external pre-stress can alter the fracture mechanisms and significantly increase the fracture toughness of glassy polymer systems under specific pre-stress states. Innovative process methods are introduced to fabricate pre-stressed double network glasses over a range of crosslink densities of each network. Basic physical and mechanical properties of these systems are evaluated as well. Finally, the concept of pre-stressed double networks is also extended to elastomeric systems and hydrogels and results are presented showing how this process method affects basic thermo-elastic and hygrothermal behavior of these systems.

PL3

WATER IN THE CELL

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Everyone knows some of the properties of liquid water. Often they think of these properties as typical of liquids in general; for example, most people mistakenly believe most liquids dissolve gasses less well at higher temperatures. However liquid water only behaves similar to most other liquids at very high temperatures (i.e. when superheated) and atypically behaves strangely at low temperatures. Overall, liquid water can be considered as an intimate mixture of two miscible liquid phases, one predominant at lower temperatures and the other predominant at higher temperatures[1]. Hydrogen bonding is generally said to be the cause of these phenomena but confusion still exists over what 'hydrogen bonding' in water entails. No longer should we simply describe (or model) liquid water in terms of individual water (H_2O) molecules or describe water's hydrogen bond as simple electrostatic interactions between discrete molecules. We must consider both proton quantum effects and extensive electron delocalization[2] within network(s) of water molecules (i.e. neither water's protons nor its electrons are pinned to individual molecules).

Within the cell, the structuring of liquid water is intimately linked with the surface properties of the biological molecules. Biomolecules affect both the localized and less-localized clustering of the water molecules as well as pathways of electron and proton delocalisation. However in processes of at least equal importance, the water molecules affect the three-dimensional structure and surface topography of the biomolecules[3]. Also affecting the water structuring are the concentration and electrical charge distribution on the lower molecular weight solutes present and the formation or dissolution of larger biomolecular structural complexes. Changes in the localized water structuring can extend to affect other biomolecular structuring at considerable distance, in molecular terms, thus acting as a rapid signalling mechanism outpacing metabolite diffusion.

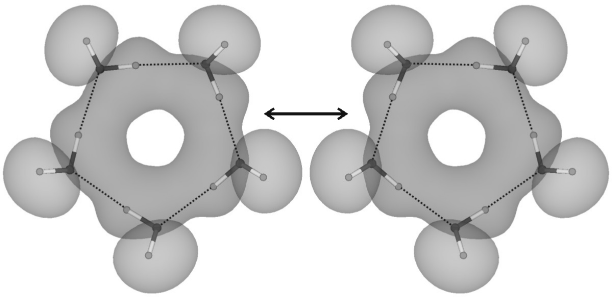

Figure 1. Electron and proton delocalization in a water pentamer $(H_2O)_5$. Shown is molecular orbital eleven out of twenty-five showing the electron overlap possible for extended hydrogen bonding.

Most of these concepts run counter to the commonly held belief that liquid water may be treated as other liquids. This is particularly true when referring to the water inside live cells. Ignoring these recent advances is an obstructive philosophy that runs counter to experimental science and holds up progress in the life sciences. This lecture attempts to put the record straight and describe the true picture of the function of liquid water within cells.

REFERENCES
1. http://www.lsbu.ac.uk/water/index2.html [cit.. 2011-22-05].
2. Del Giudice E., Fuchs E. C., Vitiello G.: Water *2*, 69 (2010).
3. Chaplin M. F., Opinion: Nature Rev. Mol. Cell Biol. *7*, 861 (2006).

PL4

THE DEVELOPMENT OF SILICATE MATERIALS FOR BIOMEDICAL APPLICATIONS

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The silicate materials are the largest worldwide available materials, representing approximately 90 percent of the Earth´s crust and the most used in different branches from traditional to advanced ceramics, cements, glass, electronics, semiconductors, composites and biomaterials. Contrary to carbon compounds that can be found in gaseous, liquid or solid state forming macromolecules (polymers), silica or silicium oxide exists generally in solid state as tetraeder forming different channels and structures. Silica is chemically bond with different oxides (Al_2O_3, CaO, ZrO_2, MgO) to form compounds with specific properties. Apart from metals and their alloys, polymer and silicate based materials have found applications in biomedicine as implants to replace or to repair damaged hard and soft tissues.

There are a large range of silicate materials that have been explored to be used as implants or part of biocomposite mate-rials. Calcium silicate based materials known as wollostonite ($CaSiO_3$) were considered as a potential bioactive material for bone tissue regeneration due to their osseointegration properties[1]. The mechanism of biomineralisation in tissue regene-ration process is dependent on functional groups Si-OH (formed on the surface of wollastonite after immersion in simulated blood fluid) which provides the bonding interface with tissues. These functional groups act as nucleation center for the precipitation of hydroxyapatite phase on the surface of implants. Unfortunately, wollastonite is highly soluble and hence its application alone enhances the pH of biological environment and affects the activities of cells. In order to retard the solubility of wollastonite, glass and glass ceramics in CaO-SiO_2-Na_2O with addition of CaO-P_2O_5 were developed, commercialized and applied. The strongest composite mostly used as bioactive material is phosphate-silicate-apatite glass and related glass-ceramics denoted as A/W (apatite-wollastonite) containing a dispersion of tetragonal Zirconia[2] Attempt has been undertaken to make biomaterials from silicate white cement rigid paste after full hydration. However, such ideas have not received positive echo, as the solubility of implant may enhance the pH of biological environment[3].

In the last years, much attention is paid to the development of biomaterials based on lithium silicate as glass and glass ceramics. Though the primary ideas of the development of LS2 glass and glass ceramics was to verify the Classic Nucleation Theory (CNT), today multicomponent lithium disilicate glass and glass ceramics have found usage in dental application as crowns or bridges due to their mechanical, optical, thermal and chemical properties.

The development of lithium disilicate glass ceramics with high mechanical strength is based on control of volume nucleation by phase separation of the base glasses. Translucent, high-strength and pressable lithium disilicate glass ceramics were prepared by Schweiger et al. in the system with the composition of (57 - 80) % SiO_2, (0 - 5) % Al_2O_3, (0,1 - 6) % La_2O_3, (0 -5) % MgO, (0-8) % ZnO, (0-13) % K_2O, (11 - 19) % Li_2O, (0,5 - 11) % P_2O_5, (0 - 6) % additives and (0 - 8) % coloring substances (wt. %)[3]. Flexural strength of this material achieves the value of (300 - 400) MPa. High-strength and machinable glass ceramics were formed from the ZnO-free system with the composition of (64 - 73) % SiO_2, (13 - 17) % Li_2O, (0,5 - 5) % Al_2O_3, (2 - 5) % K_2O, (2 - 5) % P_2O_5 (wt. %)[4].

Our recent work was aimed at the development of lithium disilicate glass and glass ceramics with addition of different amount of CaO, P_2O_5, F to form different amount of fluorapatite[5-8].

The optical properties of these samples were investigated via method developed by Majling based on optical transparency (or optical opacity) due the nucleation and crystal growth as consequence of heat treatment[9].

Besides the appropriate mechanical properties (hardness), the glasses and glass ceramics based on LS2 have been tested to demonstrate bioactive properties according the P_2O_5 content, temperature treatment, static and dynamic regime and medium[9].

The formation of hydroxyl carbonate apatite (HCA) layer on the surface of glass and glass ceramics under different conditions has been proved in simulated body fluid (SBF) by SEM, FTIR and EPMA methods.

This work was supported by the Slovak Academy of Sciences VEGA, grant No. 1/0934/11.

REFERENCES

1. Stookey S. D.: Ind. Eng. Chem. *51*, 805 (1959).
2. Durschang B. R., Carl G., Rüssel C., Roeder E.: Glastech. Ber. Glass Sci. Technol. *67*, 171 (1994).
3. Schweiger M., Höland W., Frank M., Drescher H., Rheinberger V.: Quint. Dent. Technol. *22*, 143 (1999).
4. Kuzielova E., Kovar V., Palou M.: J. Therm. Anal. Calorim. *94*, 849 (2008).
5. Cerruti M., Sahai N.: Rev. Mineral. Geochem. *64*, 283 (2006).
6. Su J. C., Wang Z. W., Yan Y. G., Wu Y. F., Cao L. H., Ma Y. H., Yu B. Q., Li M.: J. Nanomater. *2010*, 5 (2010).
7. Ramaswamy Y., Wu C. T., Zreiqat H.: J. Bone Joint Surg. Br. *91*, 346 (2009).
8. Kuželová E., Hrubá J., Palou M., Smrčkova E.: Ceramics-Silikáty *50*, 159 (2006).
9. Kuželová E., Palou M., Lokaj J., Kozánková J.: Adv. Appl. Ceram. *107*, 203 (2008).

PL5
OXIDATION OF MARINE PHOSHOLIPIDS

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The beneficial effects of long chain polyunsaturated fatty acids (especially EPA and DHA) on human health are well documented. Therefore marine lipids is an important ingredient in foods. However, due to the high content of polyunsaturated fatty acids, marine n-3 fatty acids are highly susceptible to oxidation, which leads to formation of off-flavours and in some cases even toxic compounds. In order to be able to prevent oxidation of marine lipids, more knowledge on the kinetics of lipid oxidation is needed. Studying lipid oxidation is complicated as the the number of products formed is very large and there is a lack of reliable methods for determination of oxidation products.

We have developed a model system that enables us to follow oxidation of marine lipids continuously. The system is based on measuring consumption of dissolved oxygen by polyunsaturated fatty acids that is used to quantify the oxidation in terms of oxygen uptake rate. This method is fast and makes it possible to study the effect of different prooxidants, such as haemoglobin and iron, as well as antioxidants on lipid oxidation. The effect of physical and chemical parameters such as: temperature, pH, and concentration of anions and cations, can also be studied. This has led to development of mathematical models, showing that it is possible to model the rate of oxidation in liposomes. The study of oxidation kinetics has also led to a better understanding of oxidation mechanism of long chain polyunsaturated fatty acids. The system was also successfully used to study the effect of different antioxidants (chelators and phenolic compounds) on lipid oxidation. Antioxidant studies showed that in order to select a proper antioxidant, the type of prooxidant in the system should be known.

Reducing the pH of food will increase microbial stability, but our study also shows that this can also reduce oxidative stability of marine lipids. Due to this, the effect of the physicochemical environment such as pH on the effect of prooxidants and antioxidants and thereby on oxidative stability of marine lipids should be well understood in order to maintain good oxidative quality of mairne lipids.

Reducing the pH of food will increase microbial stability. Our study shows that this can also reduce oxidative stability of marine lipids. The slowest oxidation occurs near neutral pH. Our study shows that both effect of prooxidants and antioxidants can be changed by changing pH. The solubility of iron increases when the pH decreases. However, a decrease in pH also leads to increased concentration of positive ions near the negative liposome surface, resulting in a reduced attraction of positive Fe^{2+} to the surface where oxidation occurs. Those two factors (solubility and attraction) have opposite influence on oxidation making it difficult to predict. The highest oxidation occurs at pH between 4 and 5.

Chelating agents can contribute to reduce iron induced oxidation. Among the tested chelators, EDTA was the most effective one. However, when decreasing the pH (pH<3,8), the lipid oxidation increased due to the reduced ability of EDTA to bind iron.

Also a phenolic antioxidant propyl galate, affects iron-mediated oxidation differently, being a pro-oxidant at pH <3,5. Due to this, other strategies to reduce oxidation at pH<3,8 should be sought. Proteins are also known to be good antioxidative compounds. Casein showed an extremely good inhibiting effect on oxidation. However, the effect of the studied proteins was reduced by reducing the pH.

PL6
NEW TRENDS IN ALUMINIUM ELECTROLYSIS

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Aluminium, the third most abundant element in the earth´s crust was first time introduced to the public at the Paris Exposition of 1855. Sir Humphry Davy gave aluminium its name in 1808. It took 17 years when Danish chemist Hans Christian Oersted finally produced a sample of aluminium, albeit very impure, by chemical way. Over next 20 years Friedrich Wöhler improved this process by using metallic potassium. Henri Sainte-Claire Deville substituted potassium with less expensive sodium in 1854 and was able to produced enough aluminium for display at the Paris Exposition of 1855. At that time, pure aluminium was valued at 80 € per pound, more expensive than gold.

Aluminium has a very high affinity for oxygen and never occurs in its metallic form in nature. It is made from bauxite, a reddish-brown rock discovered in Lex Baux, France, in 1821. But it was not until 1886 that chemists finally discovered an economical way to separate pure aluminium from its ore.

In 1886, two young chemists Charles Martin Hall of the US and Paul L.T. Héroult of France, both of age 22, independently discovered the way to produce aluminium economically. Industrial production of aluminium is carried out in alumina (Al_2O_3) reduction cells by the process named after its inventors Hall-Héroult process[1]. Aluminium oxide is dissolved in molten cryolite (Na_3AlF_6). The electrolyte is modified by addition of aluminium fluoride (AlF_3), calcium fluoride(CaF_2) and in some cases also by other additives (mainly fluorides). A strong electric current passes through the electrolyte and removes the oxygen, leaving deposits of nearly pure liquid aluminium on the bottom of the cell (t~950 °C). The oxygen reacts further with the carbon anodes and thus gradually consumes them by the formation of gaseous carbon dioxide (CO_2). The overall chemical reaction of dissolved alumina with carbon to form liquid aluminium and gaseous carbon dioxide may then be written:

$$Al_2O_3 + 3\ C = 2\ Al + 3\ CO_2 \qquad (1)$$

An important progress has been made on industrial cells since 1980 in current efficiency; cell size; higher amperage (Fig. 1); longer cell lives; health, environment and safety; and modernisation of old cells.

Fig. 2 represents world primary aluminium production in 2010. Development in fluoride emissions from aluminium smelters can be expressed as kg fluoride per tonne of aluminium produced: 1st Generation Plants (1940-1955) 12 - 15 kg per tonne; 2nd Generation Plants (1955-1975) 2 - 6 kg per tonne; 3rd Generation Plants (1975-1995) 0,3 – 1,3 kg per tonne; 4th Generation Plants (1995-today) 0,1 – 0,7 kg per tonne.

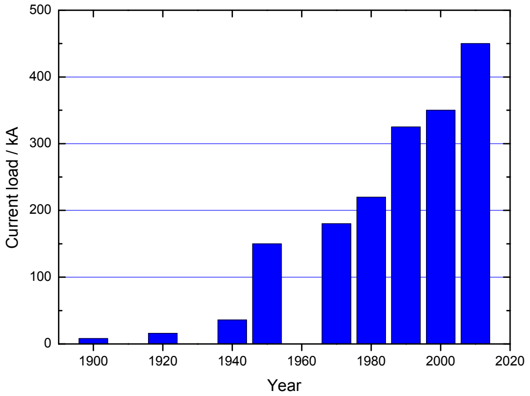

Figure 1. Increases in operating current of the electrolytic cells since the discovery of the Hall-Héroult process in 1886.

There is no doubt that fundamental research has made a significant contribution to the recent advances in aluminium electrolysis technology. However, at present there are few universities involved in fundamental research on the Hall-Héroult process. Listed alphabetically, the universities in Auckland, Bratislava, Shenyang, and Trondheim appear to be most active in this field.

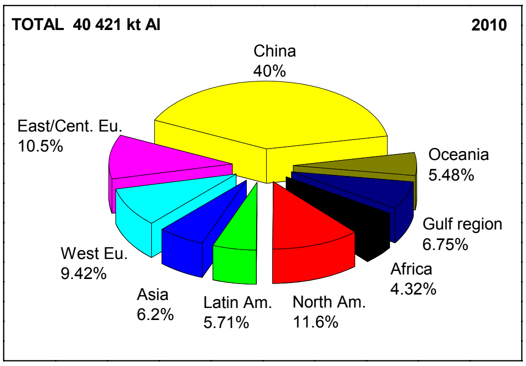

Figure 2. Primary Al production in 2010.

This work was supported by MŠVVŠ SR, grant No. 1/0579/10.

REFERENCES

1. Thonstad J., Fellner P., Haarberg G. M., Híveš J., Kvande H., Sterten Å., in the book: *Aluminium Electrolysis-Fundamentals of the Hall-Héroult Process*, 3-rd Edition, Aluminium-Verlag, Düsseldorf, Germany, 2001.

1-IL

CHARGE TRANSER MASS SPECTROMETRY: FROM PROTON TRANSER (PTR-MS) TO ELECTRON TRANSFER (ET-MS) IONIZATION MASS SPECTROMETRY AND THEIR APPLICATIONS

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Proton Transfer Reaction- Mass Spectrometry (PTR-MS) is by now a well established technique in trace gas analysis. It offers many advantages, including such as real-time analysis, no sample preparation, very low detection limits, high selectivity and very short response time. These many advantages have made it an ideal tool for many applications ranging from atmospheric chemistry, food science, biological research, process monitoring and quality control, biotechnological questions, all the way to medical applications.

Here we will present several recent in instrumental developments in PTR-MS, including the following: (i) the improvement of the detection limit allowing now for measuring trace gas compounds in a concentration range from several ppmv down to the ppqv (parts-per-quadrillion) region with a typical response time well below 100 ms, (ii) a mass resolution up to m/Δm = 8000 for the latest PTR-TOF-MS instrument, (iii) the development of the direct aqueous injection (DAI) technique, which allows the direct measurement of trace compounds even in liquid samples[1], and finally (iv) the so-called "switchable reagent ions (SRI)" feature, i.e. the possibility to switch between H_3O^+, NO^+ and O_2^+ as reagent ions allowing now to detect compounds with PA values below that of the water molecule. This feature has very recently been extended to other reagent ions and in general takes PTR-MS into the wider field of charge exchange mass spectrometry as these additional reagent ions react via electron transfer (ET) and other ion molecule reactions.

In a typical PTR-MS instrument[2,3] water vapor from a distilled water reservoir is converted into hydronium (H_3O^+) in a high performance hollow cathode ion source. This source is designed in a way that the purity of H_3O^+ ions extracted out of this primary ion source reaches values of up to 99%, thus making a signal-diminishing mass filter (e.g. quadrupole ms as used in instruments based on technologies similar to PTR-MS, e.g. SIFT-MS) for purifying the primary ions obsolete. Subsequently the H_3O^+ ions are injected into a drift tube together with the air sample to be analysed. Whenever a substance present in the air sample has a proton affinity (PA) that is larger than the PA of water, proton transfer takes place resulting in neutral H_2O and the protonated substance molecule. By chance the PAs of all common air constituents (N_2, O_2, Ar, CO_2, etc.) are much lower than the PA of water, so the air itself can act as a buffer gas and only the trace compounds in the air sample will get ionized.

Following the drift tube a mass spectrometer analyzes the product ions. As a result of this technique and its well known parameters, one gets highly accurate concentration readings in real-time (about 100 ms reaction time) without the need of sample preparation down to a typical detection limit below the single digit pptv region.

While utilizing a quadrupole mass filter has its advantages, the limited mass resolution makes the identification of unknown substances somehow difficult. Therefore we recently coupled our well established PTR ion source with a high resolution (m/Δm up to 8000) time-of-flight (TOF) mass analyzer. This so-called PTR-TOF 8000[4] and PTR-TOF 2000[5] is due to its high mass resolution capable of separating isobars (e.g. ketene and propene) and full spectra are acquired in split-seconds, while still achieving a detection limit of below 10 pptv.

Another "limitation" of PTR-MS so far, was that only H_3O^+ could be used as primary ions. Therefore, we recently developed the so-called "switchable reagent ions" (SRI) source[6]. It is now possible to choose from H_3O^+, NO^+ and O_2^+ as reagent ion with a switching time below 10 s. While all advantages of PTR ionization are preserved with H_3O^+, electron transfer ionization extends the number of substances that can be analyzed (for instance the very important molecules ethylene and acetylene, which possess lower PAs than water). NO^+ (which is produced in high purity from normal air without the need of a NO cylinder) as a primary ion leads to a different benefit because NO^+ interactions with aldehydes follow the reaction: $NO^+ + XH \rightarrow X^+ + NOH$ whereas ketones follow: $NO^+ + XH \rightarrow XH^+ + NO$ (and clustering). This means that we can even detect isomeric compounds on different nominal masses and can identify them unambiguously.

Moreover, here we will demonstrate proof-of-principle investigations about all common solid explosives, several chemical warfare agent (CWA) analogues and in addition illicit and controlled prescription drugs. It will be shown that not only the sensitivity of PTR-MS and ET-MS instruments is sufficient to detect explosives with their rather low vapor pressures via direct headspace sampling at room temperature, but the techique also provides a selectivity that allows for unambiguous identification and therefore avoids false positives or false negatives.

REFERENCES

1. Juerschik S., Tani A., Sulzer P., Haidacher S., Jordan A., Schottkowsky R., Hartungen E., Hanel G., Seehauser H., Maerk L., Maerk T. D.: Int. J. Mass Spectr. *289*, 173 (2010).
2. Lindinger W., Hansel A., Jordan A.: Int. J. Mass Spectr. *173*, 191 (1998).
3. Blake R. S., Monks P. S., Ellis A. M.: Chem. Rev. *109*, 861 (2009).
4. Jordan A., Haidacher S., Hanel G., Hartungen E., Maerk L., Seehauser H., Schottkowsky R., Sulzer P., Maerk T. D.: Int. J. Mass Spectr. *286*, 122 (2009).

5. Mayhew C. A., Sulzer P., Petersson F., Haidacher S., Jordan A., Maerk L., Watts P., Maerk T. D.: Int. J. Mass Spectr. *289*, 58 (2010).
6. Jordan A., Haidacher S., Hanel G., Hartungen E., Maerk L., Seehauser H., Schottkowsky R., Sulzer P., Maerk T. D.: Int. J. Mass Spectr. *286*, 32 (2009).

1-KL

QUANTUM MODEL OF HYDROGEN ATOM

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Fractals seem to be very powerful in describing natural objects on all scales. Fractal dimension and fractal measure are crucial parameters for such description. Many natural objects have self-similarity or partial-self-similarity of the whole object and its part[1].

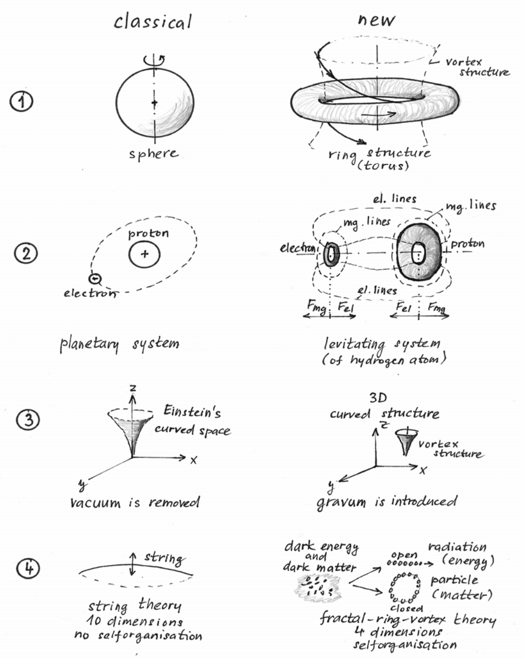

Figure 1. Main ideas and four differences between a classical and the vortex-ring-fractal models

The new model of the hydrogen atom with a levitating electron was introduced in the previous work[2]. There is attractive (electric) force F+ and (magnetic) repellent force F- :

$$F_n = F_+ - F_- = \frac{e^2}{4\pi\varepsilon_o}\left(\frac{1}{d^2} - \frac{n^2 d_{on}}{d^4}\right) = \frac{e^2}{4\pi\varepsilon_o}\left(\frac{1}{d^2} - \frac{n^4 d_o^2}{d^4}\right) = \frac{e^2}{4\pi\varepsilon_o}\frac{1}{d^2}\left(1 - \frac{n^4 d_o^2}{d^4}\right) \tag{1}$$

where n is quantum number, d_o is distance between the electron and the proton for $n=1$.

The electron structure is a semi-fractal-ring structure with a vortex bond between rings. The proton structure is a semi-fractal-coil structure. The proton is created from electron subsubrings e^{-2} and positron subsubrings υ^{-2} which can create quarks u and d. This theory can be called shortly "ring" theory. It is similar name like string theory. Differences are shown in Fig.1 and fractal structurs in Fig.2.

In the covalent bond pair of electrons oscillate and rotate around a common axis. There are two arrangements of hydrogen: with a left and a right side orientation of the electron in their structure. Very important is symmetry and self-organization of real ring structures.

The exact analysis of real physical problems is usually quite complicated, and any particular physical situation may be too complicated to analyze directly by solving the differential equations or wave functions. Ideas as the field lines (magnetic and electric lines) are for such purposes very useful. A physical understanding is a completely nonmathematical, imprecise, and inexact, but it is absolutely necessary for a physicist[1]. It is necessary combine an imagination with a calculation in the iteration process. Our approach is given by developing gradually the physical ideas – by starting with simple situations and going on more and more complicated situations. But the subject of physics has been developed over the past 200 years by some very ingenious people, and it is not easy to add something new that is not in discrepancy with them.

Most of our knowledge of the electronic structure of atoms has been obtained by the study of the light given out by atoms when they are exited. The light that is emitted by atoms of given substance can be refracted or diffracted into a distinctive pattern of lines of certain frequencies and create the line spectrum of the atom[3].

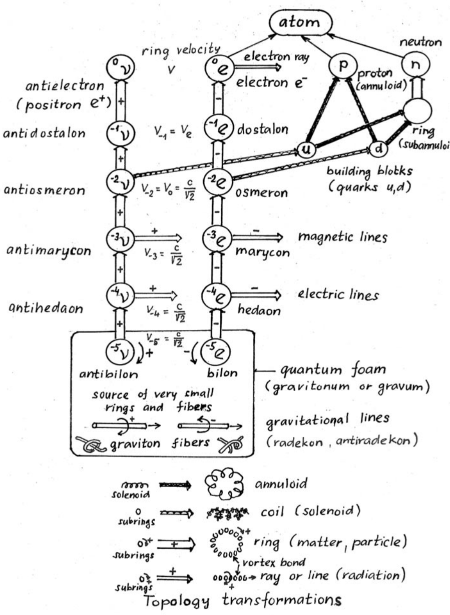

Figure 2. The fractal structure of basic particles (topology transformations)

This work was supported by EPI Kunovice

REFERENCES

1. Pauling L.: *General Chemistry*, Dover publication, Inc, New York, 1988.
2. Ošmera, P.: *Proceedings of the 16th International Conference on Soft Computing MENDEL2010,* p.146 - 153, Brno University of Technology, 2010.
3. www.pavelosmera.cz [cit. 2011-21-05]

1-L1

ELECTROPHORETIC DEPOSITION OF THIN ORGANIC FILMS FOR SOLAR ENERGY CONVERSION PURPOSE

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Electrophoretic deposition (EPD) have been long years used for coating of industrial products such as automobiles[1]. Recently successful EPD of thin organic films with microelectronic application was reported[2].

Unlike the spin coating, where most of the solution dropped on the surface is blown away during the spinning of the substrate, EPD uses the precursor materials effectively, depositing films of several hundred nanometers from low suspension concentrations[3]. It is an important advantage when thin films of low soluble conjugated polymers should be prepared. The essential stage of the EPD process consists of solving the material under the interest in a proper solvent, then adding a precipitator to form and charge the suspension particles.

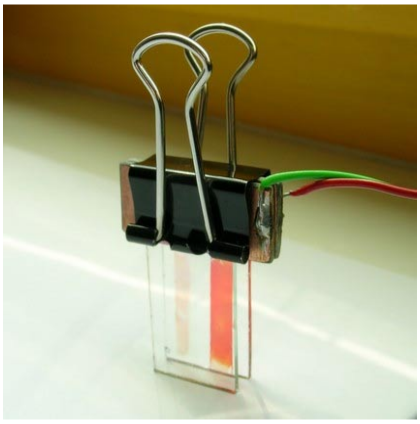

Figure 1. Electrophoretic cell module with two fixed at 3 mm ITO plate electrodes. The red colored area presents the deposited on the ITO electrode MDMO-PPV film

The film structure strongly depends on the suspension particle size and charge, which could be controlled by the proper choice and mixing of the solvent and precipitator[4].

This work aims to find optimal conditions for a suspension preparation and deposition of thin polymer films for solar energy conversion purpose. Effective photoconductive polymers as MDMO-PPV and high T_g-PPV were investigated.

The properties of the suspensions and the solid state films prepared were estimated by UV-VIS and fluorescence spectroscopy. It could be seen from the spectra that the increased precipitator concentration leads to a broadening of the characteristic absorption and fluorescence peaks. This is an evidence of the increased solid matter and should be related to the suspension particle size. A detail study of the precursor influence was carried out estimating the first derivatives of the spectra. UV-VIS spectra from solid state samples were also measured by exciting the sample with picosecond pulses.

On Scheme 1 a photograph image of the constructed EPD cell is presented. The simple holder construction provides a mutual parallel position of the electrode plates, which improves the substrate covering. The film deposition was carried out on a preliminary structured ITO electrode controlling the voltage by Keithley 2410 SourceMeter. The dependence of the film quality on the precipitator concentration for both MDMO-PPV and high T_g-PPV was studied. It was found that stable MDMO-PPV films could be obtained in a wide range of precipitator concentrations from 40 to 90%. On the contrary the precursor range for a quality preparation of T_g-PPV films is 40-50%.

Sandwich type ITO|MDMO-PPV|Al samples were subsequently prepared and photoelectrical measurements was performed by Keithley 6517A electrometer. Parallelly, same experiments were carried out on spin-coated samples with similar MDMO-PPV film thickness.

It was found that while structures with EPD films show clear diode behaviors; the spin coated film ones exhibit more symmetrical characteristics. Dependencies of the photo generated current on the light intensity and spectral dependencies of the photocurrent was also measured. It could be concluded that the structures with EPD MDMO-PPV films could be utilized for solar energy conversion purpose. More investigations have to be carried out to optimize the performance of the samples and increase the efficiency.

This work was supported by South Moravian Region and 7th Framework Programme for Research and Development (grant SIGA 885).

REFERENCES

1. Van der Biest O., Vandeperre L.: Annu. Rev. Mater. Sci. *29*, 327 (1999).
2. Tada K., Onoda M.: Thin Solid Films *477*, 187 (2005).
3. Tada K., Onoda M.: Adv. Funct. Mater. *12*, 420 (2002).
4. Tada K., Onoda M: Thin Solid Films *518*, 711 (2009).

1-L2

FADING OF INKJET PRINTED DIGITAL PHOTOGRAPHS AND METHODS FOR ITS EVALUATION

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Inkjet printing technology became a popular technology for printing digital photographs in the last decade. The stability of printouts is affected by many factors, such as ink-receiving layer, ink composition, UV and visible light intensity and airborne pollutants concentration in the environment.

Interactions of radiation and pollutants with dyes in receiving layers of inkjet printed digital photographs were studied. Relations between spectral properties of printed areas and quantity of delivered ink were studied for selected combinations of ink and receiving medium.

The long-term lightfastness tests of selected photo papers printed with both dye-based and pigment inks were started at typical university indoor conditions. Simultaneously, the selected printed target were aged by accelerated way and exposed to ozone. The kinetic studies of dye degradation were based on VIS spectra measurement and ICC profile and gamut volume calculations. According to obtained results the new test targets were prepared to better understand of dye-based inks catalytic fading.

The main results of this study are data about stability of digital photographs made by inkjet printing technology exposed to UV and VIS radiation and to simultaneous effect of ozone. It is possible to forecast printed image stability and to visualize a simulation of image deterioration caused by exposition with UV radiation and ozone.

Colorimetric criteria as colour difference, lightness difference, gamut volume, combination of colour coordinates differences or combination of all mentioned quantities can not fully describe the colour changes in prints during their fading. Our measurements showed that the time dependent changes of relative colour gamut volumes were found to be of great informational value. These colour gamut volume changes corresponded to dye degradation kinetics.

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1-L3

EPR STUDY OF ANTIOXIDATIVE EFFECT OF MELATONIN IN VIVO

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This work deals with the monitoring of free radicals content and singlet oxygen concentrations in the tissues of laboratory rats by EPR spectroscopy "in vivo". The aim of this work was to determine the antioxidant effects of melatonin and its dosage. The experiments are part of a broader, longer term project[1].

The free radicals were identified by EPR spectroscopy and establish a spin trapping method. Used EPR spectrometer: Bruker Biospin Elexsys, type E-540, operating in the X-band, with rectangular resonator. Recording and evaluation of spectral parameters was carried out through programmes of Bruker (Linux) and a graphics program Origin. The measurement was carried out at room temperature. Spin trap was used DMPO (5,5-Dimethyl-1-pyrroline N-Oxide, Sigma), Melatonin (Sigma) and usually laboratory chemicals. The special injections were used: spin traps (DMPO, PBN), detector of singlet oxygen (2, 2, 6, 6-Tetramethylpiperidine), antioxidants (special mixture of ascorbic acid, Tocopherol, Selene, polyphenolic antioxidants). The dosage of melatonin, spin trap DMPO and narcosis substances was carried out by injection into the muscle of the animal. The animal was placed in the special chamber between the magnets of EPR spectrometer and its tail was inserted into the resonator. The animal was under narcosis (injection). The calculations of spectral parameters were carried out using a computer connected to the spectrometer.

A high level of the radicals was measured at hyperthyreosy, which is a common illness in human medicine. In the literature it can be found that the free radicals are the cause of a variety of organ and tissue damage for these diseases. At the same time, we have shown that levels of hydroxyl radicals can reduce chronic administration of very high doses of melatonin, which is an antioxidant and scavenger of free radicals.

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REFERENCES:

1. Fricova J., Vejražka M., Stopka P., Křížová J., Běláček J., Rokyta R.: Arch. Med. Sci. *6*, 764 (2010).

1-L4

STUDY ON HYALURONAN INTERACTIONS WITH L-LYSINE AND 6-AMINOCAPROIC ACID

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This paper reports the results over the investigation of *hyaluronan* (HA) interactions with some aminoacids, particularly *L-Lysine* and *6-Aminocaproic acid* in different kinds of aqueous environment.

Hyaluronan, a sodium salt of hyaluronic acid, as a ubiquituous, natural and linear polysacharide composed by repeating disacharide unit consisting of *D-glucuronic acid* and *N-acetyl-D-gucosamine* linked with *β-1,3* and *β-1,4* glycosidic bonds has become a promissing biopolymer for variety of pharmaceutical, and cosmetic applications, e.g. drug delivery[1,2].

Utilization of the native HA as a carrier of poorly water-soluble bio-active agents is fairly unfavorable due to its strong hydophilicity. Therefore, some kind of modification of HA needs to be carried out in order to support its interaction with the agents[2]. One of the ways is a chemical modification[3,4] of HA, e.g. by grafting it with a hydrophobic chain[4,5], or a "*physical modification*", which would keep the properties of the native HA, e.g. by means of HA physical (electrostatic) interactions with cationic surfactants[6] or aminogoups-containing compounds, e.g. aminoacids.

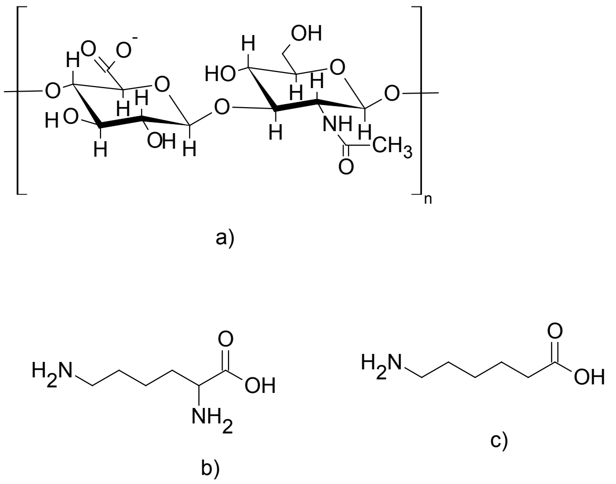
Under appropriate conditions, protonated aminogroups of aminoacids and negatively charged carboxylic groups of HA are able to interact with each other in terms of forming a complex capable of carrying a drug. Such aminoacids ought to associate with each other into aggregates, thus they need to be amphiphilic.

The presented work brings the first overview on the HA interactions with model aminocids *L-Lysine* and *6-Aminocaproic acid (6AcA)*, by means of rheometry, viscometry, pH and conductivity measurements performed in aqueous conditions with altering ionic strength, pH and a form of aminoacids.

The utilized instrumental methods proved electrostatic interactions of HA with the aminoacids by means of a decrease in the system viscosity, analogically with the results gained from the studies of HA interactions with cationic surfactants[7], and a decrease in relative conductivity within the same regime of the aminoacid concentrations, namely for the system containing high-molar mass HA and L-Lysine. The study of HA and 6-AcA interactions showed moderate decrease in system viscosity and in the presence of the low-molar mass HA were nearly negligible.

The obtained results also exhibit a strong sensitivity of the HA-aminoacids interactions against ionic strength. Addition of NaCl above 40 mM into the system efficiently screens the HA-aminoacid interactions. Phosphate buffer (pH = 6) also screens the HA-aminoacids interactions.

In order to support and strengthen the HA interactions with aminoacids, L-Lysine and 6-AcA were protonated with a certain amount of HCl beyond their isoelectric point for a complete protonization of the amnigroups. All methods reveal the fortification of the interactions even for HA–L-Lysine system; however the mechanism of HA–6-AcA interactions seems to be more complicated than that of the former one.



Scheme 1. a) Structure formula of hyaluronan, b) formula of L-Lysine and c) formula of 6-Aminocaproic acid

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REFERENCES
1. Vercruysse K. P., Prestwich G. D.: Crit. Rev. Ther. Drug *15*, 513 (1998).
2. Jaracz S., Chen J., Kuznetsova L. V., Ojima I.: Bioorg. Med. Chem. *13*, 5048 (2005).
3. Prehm P., Vandomme E. J., De Baets S., Steinbückel A.: in the book: Biopolymers, *Polysaccharides I: polysaccharides from prokaryots*, p. 379, Vol. 5, , Wiley–VCH, Weinheim 2002.
4. Creuzet C., Kadi S., Rinaudo M., Auzély-Velty R.: Polymer *47*, 2706 (2006).
5. Mlčochová P, Hájková V., Steiner B., Bystrický S., Koóš M., Medová M., Velebný V.: Carb. Pol. *69*, 344 (2007).
6. Thalberg K., Lindman B.: J. Phys. Chem. *93*, 1478 (1989).
7. Heslöf Å., Sundenlöf L. O., Edsman, K.: J. Phys. Chem. *96*, 2345 (1992).

1-L5

OPTICAL PROPERTIES OF DIKETO-PYRROLO-PYRROLES FOR ORGANIC ELECTRONICS APPLICATIONS

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Derivatives of 3,6-diphenyl-2,5-dihydro-pyrrolo[3,4-c] pyrrole-1,4-dione, commonly referred to as DPPs, constitute recent industrially important class of high-performance pigments[1-6] (see the parent molecule in Figure 1). They are endowed with brilliant shades (ranging from yellow-orange to red-violet) and exhibit exceptional chemical, heat, light, and weather fastness. Furthermore, some of their physical properties such as high melting points are exceptional in view of the low molecular weight relative to pigment standards. It has been shown that the DPP units introduced into various materials e.g. polymers, dendrimers, polymer-surfactant complexes, and oligomers results in deeply coloured, highly photoluminescent and electroluminescent materials. Due to their interesting properties, there is wide range of possible applications which have been already investigated covering for example latent pigment, charge generating materials for laser printers and information storage systems, solid-state dye lasers or gas detectors etc.

In order to tune the DPPs properties, we modified the basic structure by introduction of electron donating and/or withdrawing groups. Furthermore, solubilising groups (*N*-alkylation) were attached to enable solution based depostition techniques, see Figure 1. The influence on absorption and fluorescence is discussed and faced with the results obtained by quantum chemical calculations.

Introduction of electron-donating groups increased the molar absorption coefficient (ε) and was accompanied with strong bathochromic shift. This behaviour implies that charge separation occurs via electron delocalization leading to creation of permanent dipole moment. Blurring of vibration structure in absorption spectra of mono substituted derrivatives imply interaction with polar dimethylsulfoxide and shows polar character of the mono substituted DPPs.

The effects of electron-donor (piperidino) and electron-acceptor (chloro) groups on the electronic spectra were also investigated theoretically. It was found, that in general, electron withdrawing group stabilizes both phenyl molecular orbitals, while electron donating group destabilizes them (and even to a greater extent)[7]. An electron-donor substituent increases the electron density on the phenyl group to which it is attached, and on acceptor C=O group of the second pyrrolinone ring in HOMO, while in LUMO further CT is observed to the opposite phenyl group. This indicates the electron-acceptor character of the whole central dipyrrolinone mainly localized on keto groups.

Introduction of the *N*-alkylation led to the decrease of the ε and hypsochromic shift. First *N*-alkylation causes only small change, whereas second alkylation lead to the value of ε almoust similar to the parent, non *N*-substituted, DPP. This decrease is acompanied by the hypsochromic shift and loss of vibrational structure of the absorption. We proposed the same mechanism as for the *N*-alkylated only derivatives[3]: the *N*-alkylation causes rotation of the phenyls (see the angles α and β in Figure 1) and consequently breaks the molecule symmetry. This causes decrease of the effective conjugation and increases the polarity.

The fluorescence spectra of DPPs usually show small Stokes shifts, which are significantly increased by N-substitution (e.g. alkylation) inducing higher degree of nonplanarity[8]. Thus, the *N*-substituted derivatives are promising with respect to applications like OLED, laser, etc. The Stokes shift between 0-0 vibronic bands in absorption and fluorescence spectra is higher for all derivatives with electon donating or withdrawing substituents than that for parent compound which further supports the explanation given above.

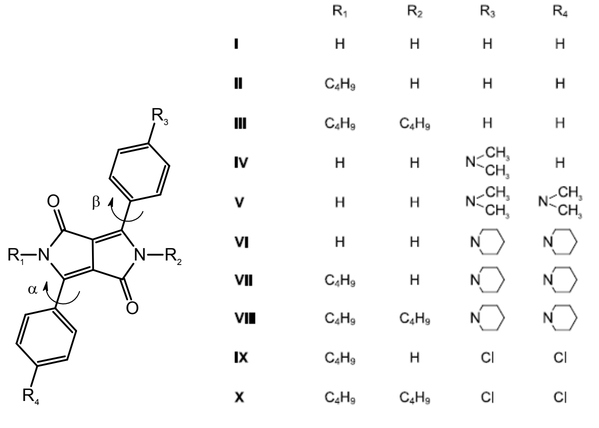

Figure 1. The basic structure of 3,6-diphenyl-2,5-dihydropyrrolo[3,4-c]pyrrole-1,4 dione, also known as DPP (structure I) and the discussed derrivatives.

This work was supported by the project "Centre for Materials Research at FCH BUT" No. CZ.1.05/2.1.00/01.0012 from ERDF and by IGA of the BUT via project No. FCH/FEKT-S-11-2.

REFERENCES

1. Rochat A. C., Cassar L., Iqbal A.: EP 94911 (1983).
2. Iqbal A., Pfenninger J., Rochat A. C., Babler F.: EP 181290 (1989).
3. Pfenninger J., Iqbal A., Rochat A. C., Wallquist O.: USP 4778899 (1986).
4. Surber W., Iqbal A., Stern C.: EP 302018 (1989),
5. Wooden G., Schloeder I., Wallquist O.: EP 672729 (1995).
6. Hendi S. B.: EP 962499 (1999).
7. Luňák, S., Vyňuchal, J., Vala, M., Havel, L., Hrdina, R.: Dyes Pigments *89*, 102 (2009).
8. Vala, M., Weiter, M., Vyňuchal, J., Toman, P., Luňák, S.: J. Fluoresc. *18*, 1181 (2008).

1-L6

RADICAL PRODUCTS GENERATED BY THE OXIDATION OF SOME SELECTED TYPES OF SECONDARY AMINES

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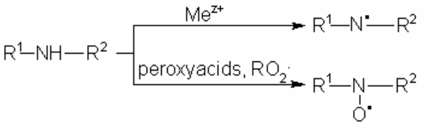
The oxidation processes occuring on the –NH- group of secondary amines mostly result in the formation of *N*-centred radical intermediates. On this principle, *e.g.* the action of aminic antioxidants is based[1]. As the oxidation agents the ions of some metals (Pb^{4+}, Mn^{4+}, Ag^{+}), peroxy compounds (peroxy acids, hydroperoxides, diperoxides), peroxy radicals and others can be used. Generally, two types of *N*-centred radicals, aminyl and aminoxyl radicals, can be generated (Scheme 1). For their identification EPR spectroscopy is the most convenient method.

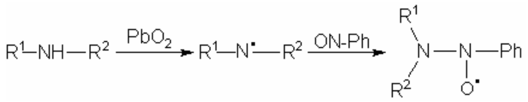


Scheme 1: *Oxidation of bifunctional secondary amines.*

Aminyl radicals R^1-$N^•$–R^2 are very reactive and exhibit the tendency towards the dimerization to hydrazines[2]. For their detection the application of special techniques (e.g. flow method, photolysis of hydrazines) is mostly inevitable. It is worth to mention that till now the indirect detection of aminyl radicals using spin trapping method was not practically employed. Characteristic feature of aminoxyl radicals R^1–$NO^•$- R^2 is their substantially higher stability, stemming from the specific structure of – $NO^•$ – fragment. Aminoxyls can be formed from the aminyl radicals by the reaction with $RO_2^•$ radicals[3]. Within this contribution the application of spin trapping technique for the detection of aminyl radicals from some selected types of alkyl-aryl amines is reported. The attention is also focused on some specific radical reactions, where the alkyl substituent is attacked by the oxidation agent. By the EPR study of the radical products formed by the oxidation of bifunctional secondary amines the problem of the preferentially oxidizable -NH- group is discussed.

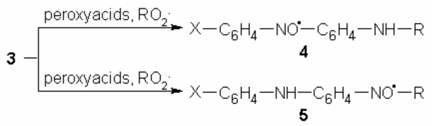
Alkyl-aryl amines R^1-NH-R^2, 1-anilino-1-phenylpentane-3-ones (**1,** R^1= C_2H_5-CO-CH_2-CH(Ph), R_2 = C_6H_4-X) and *N*-alkylanilines (**2**, R^1 = CH_3, C_2H_5, C_6H_5-CH_2, R^2 = C_6H_5) were easily oxidized to the corresponding aminoxyl radicals by the employment of peroxy acids. By the oxidation with PbO_2 the generated aminyl radicals were indirectly evidenced by the spin trapping method using nitrosobenzene as spin trap, according to Scheme 2.

Replacing PbO_2 by $Pb(CH_3COO)_4$ the substantially different reaction mechanism was observed with alkylanilines (**2**) having -CH_2– group in the vicinity of -NH-group (R^1 = C_2H_5, C_6H_5-CH_2, R^2 = C_6H_5). In this case the transformations of the alkyl substituents leading to the formation of C-radicals were confirmed by the application of spin trapping technique. Specific behavior of alkyl-anilines (**2**) with -CH_2– fragment was observed also by their RO_2• oxidation. The gradual degradation of methylene hydrogene atoms proceeding under transient formation of nitrones was confirmed by the analysis of EPR spectra.



Scheme 2: *Spin trapping of aminyl radicals from alkyl-aryl amines.*

By the oxidation of bifunctional amines X-C_6H_4-NH-C_6H_4-NH-R (**3**, R = alkyl) with peroxy acids or RO_2• radicals the question arises, which NH-group is preferentially attacked. Theoretically, two types of aminoxyl radicals (**4**, **5**) can be produced (Scheme 3)



Scheme 3: *Radical transformations on alkyl substituent*

The EPR experiments confirmed the equilibrium between these radicals, which is influenced by the solvent and substituent X.

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REFERENCES:
1. Pospíšil J.: Advan. Polym. Sci. *124*, 89 (1995).
2. Danen W. C., Neugebauer F. A.: *Angew. Chem. 87*, 823 (1975).
3. Thomas J.: J. Am. Chem. Soc. *82*, 5955 (1960).

1-L7

ANALOGICAL MODELING AND NUMERICAL SIMULATION OF STATE PARAMETERS FOR A THERMAL EXPLOSION

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The present work deals with the problem of extending some notions and methods specific to systems theory, in the domain of the numerical modeling and simulation of a thermal explosion[1-3].

The time evolution of the three state parameters: pressure, temperature and concentration, based on experimental results, are approximated by periodical exponential functions of the form:

$$y_{AN}(t) = \left(1 - \frac{T_1}{T_1 - T_2}\varepsilon^{-t/T_1} - \frac{T_2}{T_2 - T_1}\varepsilon^{-t/T_2}\right)\cdot K \cdot u \qquad (1)$$

for the increasing evolutions of pressure and temperature, respectively of the form:

$$y_{AN}(t) = \left(\frac{T_1}{T_1 - T_2}\varepsilon^{-t/T_1} + \frac{T_2}{T_2 - T_1}\varepsilon^{-t/T_2}\right)\cdot K \cdot u \qquad (2)$$

for the decreasing evolution of concentration. The input signal u_0 = constant, necessary to induce the explosion, and (K) is a proportionality coefficient. The two time constants (T_1) and (T_2) are specific for each state parameter, respectively: $T_2 = \lambda \cdot T_1$ and the final time $t_f = 10^{-5}$ s is approximated by:

$$t_f = \mu \cdot (T_1 + T_2) = \mu \cdot (1 + \lambda) \cdot T_1 = \mu \cdot \left(1 + \frac{1}{\lambda}\right) \cdot T_2 \qquad (3)$$

where is considered μ=5 and λ=2.

The inflexion point of these evolutions results at the time:

$$t_i = \frac{T_1 T_2}{T_2 - T_1}\cdot \ln\left(\frac{T_1}{T_2}\right) = \mu \cdot \frac{\lambda + 1}{\lambda - 1}\cdot \ln\lambda \cdot t_f \cdot \qquad (4)$$

The analogical model associated to the function (1) and (2) can be expressed through the ordinary differential equation:

$$a_0 y + a_1 \frac{dy}{dt} + a_2 \frac{d^2 y}{dt^2} = a_0 y_0 + a_1 y_1 + a_2 y_2 = K \cdot u_0 \qquad (5)$$

where a_0 = 1; $a_1 = T_1 + T_2$ and $a_2 = T_1 \cdot T_2$.

The initial conditions considered at t = t_0 = 0 are: i) for pressure: y_{0IC} = 1; y_{1IC} = 0; u_0 = 1; ii) for temperature: y_{0IC} =

272,96; $y_{1IC} = 0$; $u_0 = 0{,}04$; iii) for concentration: $y_{0IC} = 0$; $y_{1IC} = 0$; $u_0 = 0{,}03$.

With these preliminaries is elaborated the software in order to obtain the numerical simulation, which operates with the following algorithm, based on Taylor Series:

$$\begin{aligned}
&y_0 = y_{0IC}\\
&y_1 = y_{1IC}\\
&y_2 = \frac{1}{a_2}\left[Ku_0 - \left(a_0 y_0 + a_1 y_1\right)\right]\\
&y_3 = \frac{1}{a_2}\left[Ku_1 - \left(a_0 y_1 + a_1 y_2\right)\right]\\
&y_4 = \frac{1}{a_2}\left[Ku_2 - \left(a_0 y_2 + a_1 y_3\right)\right]\\
&y_5 = \frac{1}{a_2}\left[Ku_3 - \left(a_0 y_3 + a_1 y_4\right)\right] \qquad (6)
\end{aligned}$$

where $K = 1$, $u_0 = 1$; $u_1 = 0$; $u_2 = 0$ and $u_3 = 0$.

The above iterations continue with two Taylor Series, which will replace the first two equations from the algorithm:

$$y_{0K} = y_0 + \frac{\Delta t}{1!} y_1 + \frac{\Delta t^2}{2!} y_2 + \frac{\Delta t^3}{3!} y_3 + \frac{\Delta t^4}{4!} y_4 + \frac{\Delta t^5}{5!} y_5 \qquad (7)$$

and

$$y_{1K} = y_1 + \frac{\Delta t}{1!} y_2 + \frac{\Delta t^2}{2!} y_3 + \frac{\Delta t^3}{3!} y_4 + \frac{\Delta t^4}{4!} y_5 \qquad (8)$$

where the integration step $\Delta t = t_f/100$.

The performance indicator for this numerical integration is expressed by "the relative error cumulated in percentages", denoted with:

$$\text{crep } y_0 = 100 \cdot \frac{\sum\limits_{K=0}^{K_f} \Delta y_{0K}}{\sum\limits_{K=0}^{K_f} y_{AN,K}} \qquad (9)$$

where the initial and final sequences of calculus are $K_0=0$, respectively $K_f = t_f/\Delta t$ and the sum $\sum\limits_{k=0}^{K_f} \Delta y_{ok}$ is the difference between the experimental and analytical results. This indicator is established at each iteration. The final values ($t=t_f$) are of the order (10^{-4})%, which proves the remarkable precision of this method of numerical integration.

The results of the method are sinthetised as follows: i) the integration step $\Delta t = 10^{-9}$; ii) the time constants: $T_1 = 6{,}67 \cdot 10^{-7}$; $T_2=1{,}33 \cdot 10^{-6}$; iii) the coefficients of the equation (5): $a_0 = 1$; $a = 2 \cdot 10^{-6}$; $a_2 = 8{,}88 \cdot 10^{-7}$; iii) the inflexion point $t_i = 9{,}24 \cdot 10^{7}$; iv) crep y_0 is of the order 10^{-4}.

It can be concluded that the presented algorithm contribute to a good phenomenon interpretation of the extremely small period of the explosion, studied from the point of view of time evolution of pressure, temperature and concentration, in steady state and transient too.

We would like thank prof. dr. eng. Colosi Tiberiu for the guidance they gave us.

REFERENCES

1. Oppenheim A. K., *Chemical Kinetic Aspects, Dynamics of Combustion Systems*, Edition 2, p. 81-113, Springer Berlin Heidelberg Publisher 2008.
2. Kotoyori T., *Critical Temperatures for the Thermal Explosion of Chemicals*, p. 406, Hardbound 2005.
3. Chan C. Y., Kong P. C.: Appl. Math. Comput., *71*, 201 (1995).

1-L8

DIFFUSION OF ORGANIC DYES IN AQUEOUS SOLUTIONS AND AGAROSE GELS STUDIED BY METHOD OF HORIZONTAL DIFFUSION CELLS

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Hydrogels represent a state of matter of substantially growing research interest. Natural hydrogels can be found everywhere – in non-living parts of the environment (such as soils and sediments) as well as in the bodies of living organisms. In recent decades, remarkable research attention was paid to a utilization of hydrogels in a production of controlled release systems for agricultural, pharmaceutical and sanitary use[1,2]. Drug carriers in a form of hydrogels allow delivering desired material over long pathway and period of time; the rate of release can be governed by both internal and external conditions. In last decades, substantial research aimed at explanation and prediction of swelling and mechanical response of gels under various conditions[3]. Nevertheless, such a deep knowledge is still missing in the case of diffusion of different solutes in the utilized gels, although it would be of the great interest in both preparation and action of the corresponding controlled release systems.

In the presented work, the method of horizontal diffusion cells[4] was utilized in a standard way in determination of diffusion coefficient of several acidic and basic dyes in an aqueous solution. Commercial diffusion cells apparatus (PermeGear, Inc.) was used in combination with polycarbonate membrane with the uniform pore size of 2 µm. The apparatus was pre–calibrated using 1M KCl according to standard procedure[5]. The metod was consequently modified in order to provide a simple laboratory technique allowing determination of diffusion coefficient of the model solute in a hydrogel media.

Table I shows the list of utilized organic dyes with corresponding values of diffusion coefficient in the aqueous solution at 25°C as determined by method of diffusion cells. All dyes were of an ionic nature – methylene blue (Basic blue 9) represents model basic diffusion probe while all the others have an acidic structure.

As was expected, a correlation between diffusion coefficient and molecular weight of a diffusing dye can be found. In general, as the molecular weight increases, the diffusivity decreases. The only exception among the studied compounds is represented by Direct blue 106, were unusually low diffusivity value was obtained. This anomaly is difficult to explain and should be taken as a scope of oncoming experiments.

Table I, Determined values of the diffusion coefficients of studied organic dyes in aqeous solutions

Organic dye	Molecular weight [Da]	D_w [$\times 10^{-10}$ m^2 s^{-1}]
C.I. Basic blue 9	319	8,89
C.I. Acid blue 41	488	8,44
C.I. Direct Blue 106	744	3,07
C.I. Direct Blue 71	1034	4,92
C.I. Direct Green 26	1446	3,57

The agarose gels, utilized in subsequent diffusion experiments, were prepared by routine thermal gelation procedure[6]. Methylene blue was chosen as a model diffusion probe. In Graph 1, values of diffusion coefficient of methylene blue in agarose gels are shown for varying agarose content of the gel and for 25°C and 40°C, respectively.

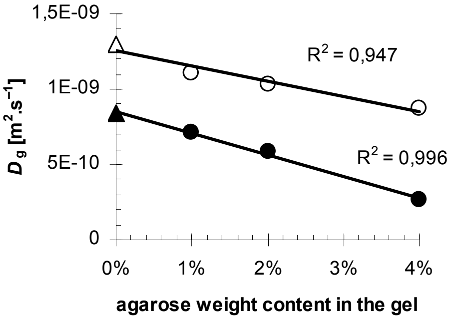

Figure 1. Variation of diffusion coefficient of methylene blue with the content of agarose in the gel at 25°C (black) and 40 °C (white).

The strong correlation can be found between concentration of agarose in the gels and the resulting diffusivity of the dye. For completness, the corresponding values of diffusivity in aqueous solutions were added in the graph (triangles).

As the presented results have confirmed, method of horizontal diffusion cells is suitable for studies on diffusion characteristics of hydrogels. Other effects have recently been studied, such as a content of reactive polymer in the gel.

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REFERENCES

1. Park H., Park K.: In *ACS Symposium Series 627, Hydrogels and Biodegradable Polymers for Bioapplications*, p. 2, 1996.
2. Hamidi M., Azadi, A., Rafiei P.: Adv. Drug Deliver. Rev. *60*, 1638 (2008).
3. Oppermann W.: In *ACS Symposium Series, Polyelectrolyte Gels: Properties, Preparation and Applications* p. 159, 1991.
4. Falk B., Garramone S., Shivkumar S.: Mater. Lett., *60*, 3261 (2004).
5. Stokes R: J. Am. Chem. Soc., *73*, 3527 (1951).
6. Narayanan J. Xiong J. Y., Liu X. Y.: J. Phys. Conf. Ser. *28*, 83 (2006).

1-L9

ON THE THERMODYNAMIC CONSISTENCY OF RATE CONSTANTS IN MASS ACTION RATE EQUATIONS

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Thermodynamics is usually thought to tell nothing about rates of chemical transformations. Rates of chemical reactions belong to the domain of chemical kinetics. However, as thermodynamics gives some restriction on the course of chemical reactions, similar restrictions on their rates are also looked for. Relationships between chemical thermodynamics and kinetics traditionally emerge from the ways that both disciplines use to describe equilibrium state of chemical reactions. Combining these two views various results on compatibility between thermodynamics and kinetics were published. The main idea can be illustrated on the trivial example of decomposition reaction AB = A + B with rate (kinetic) equation 1.

$$r = \overrightarrow{k}c_{\mathrm{AB}} - \overleftarrow{k}c_{\mathrm{A}}c_{\mathrm{B}} \tag{1}$$

In equilibrium, the reaction rate is zero, consequently we get equation 2

$$\overrightarrow{k} / \overleftarrow{k} = \left(c_{\mathrm{A}}c_{\mathrm{B}} / c_{\mathrm{AB}}\right)_{\mathrm{eq}} \tag{2}$$

Because the right hand side corresponds to the thermodynamic equilibrium constant (*K*) it is concluded that

$$K = \overrightarrow{k} / \overleftarrow{k} \tag{3}$$

This approach is also the basis for finding Wegscheider conditions[1,2], i.e. restrictions on ratios of forward and reversed rate constants or their products in a reaction scheme. However, this is simplified approach not taking into account conceptual differences between the true thermodynamic equilibrium constant and the ratio of rate constants that is called here the kinetic equilibrium constant. Using simple example of carbon dioxide decomposition, 2 CO = CO_2 + C, it will be shown that thermodynamic equilibrium constant need not be equal to kinetic equilibrium constant even in ideal systems. The main cause lies in that kinetic equations are formulated in concentra-tions whereas thermodynamic equations are formulated in activities and transformation of activities to concentrations is not straightforward to say nothing about the dependence of activities on the selection of standard state. Further, it will be demonstrated that Wegscheider conditions are probably consequences of dependence among individual reactions forming reaction scheme. Finally, rational thermo-dynamic approach[3-5] to resolving the problem of thermo-dynamic consistency of rate constants will be illustrated on the same example. This, in fact, means that backward rate constants are unnecessary and "thermodynamic consistency" of forward rate constants is naturally embedded in rate equations resulting from this approach.

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REFERENCES

1. Ederer M., Gilles E.D. Biophys. J. *92*, 1846 (2007).
2. Vlad M.O., Ross J. WIREs Syst. Biol. Med. *1*, 348 (2009).
3. Samohýl I.: *Rational Thermodynamics of Chemically Reacting Mixtures*, Academia, Prague 1982 (in Czech).
4. Pekař M.: Collect. Czech. Chem. Commun. *74*, 1375 (2009).
5. Pekař M.: React. Kinet. Mech. Catal. *99*, 29 (2010).

1-L10

SOLUTION PROCESSED TITANIA COATINGS FABRICATED BY INKJET MATERIAL DEPOSITION

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Photocatalysis on TiO_2 has received much attention during the last two decades. If TiO_2 absorbs a quantum of UV radiation of sufficient energy, an electron is excited into the conduction band and an electron-hole pair is created. The potentials of electron and hole are strong enough to oxidize water to hydroxyl radicals and reduce molecular O_2. Resulting reactive oxygen species (ROS) are very powerful oxidizing agents and readily attack any organic matter in their proximity until it is totally cleaved to CO_2 and water. Numerous applications utilizing this process for water purification, toxic waste treatment, air purification and deodorizing have been proposed and some of them already successfully marketed. The same process can be applied for the design of self-cleaning and self-disinfecting surfaces. Moreover, the oxygen vacancy creation and subsequent photo-corrosion on irradiated surfaces of TiO_2 convert the surface nature to superhydrophilic one, which further enhances its self-cleaning ability.

So far, the sol-gel process definitely proved its benefits and potential. However, in order to produce titania layers, the liquid sol formulation must be coated onto a substrate. Many different coating techniques have been used for this purpose, such as dip-, spin- or spray-coating, doctor blade, roller etc.[1] While all these techniques proved to be useful, they are buried by some inherent limitations. These include, but are not limited to: sensitivity to surface defects, limited coating area, ambient humidity interference, efficiency of precursor use. Fortunately, there has been a major breakthrough of a new promising deposition technique recently. The novel approach is usually termed inkjet material deposition or shortly material printing. The technique shares the basic principles with conventional inkjet printing, i.e. tiny droplets of a low-viscosity liquid are precisely deposited onto a substrate by means of thermal or piezoelectric printhead. In the case of material printing, the ink is a specially formulated liquid used for transporting a functional component onto the substrate surface.

The presented work outlines the recent research activities on solution processed titania coatings fabricated by inkjet material deposition performed at the Laboratory of photochemistry, Faculty of chemistry, Brno university of technology.

The authors started their work with a conventional sol based on tetraisopropoxy titanate and acetylacetone[2] deposited by a modified office inkjet printer equipped with piezoelectric print head (Epson R220)[3]. This work was targeted to the fabrication of smooth compact films applicable as self-cleaning surfaces. Samples up to 25 cm^2 area were conveniently printed. The structure of prepared layers was strongly depending on the printing conditions: If the sol ejection rate is faster then solvent evaporation, a smooth compact layer is produced. On the other hand, when the solvent evaporation rate is grater than sol ejection, we obtain optically rough and highly structured layer.

Further study focused on the preparation of the TiO_2 thin films from alkoxide solutions containing polyethylene glycol (PEG) as an anticracking agent by the sol–gel method on soda–lime glass plates[4]. Sol application was again carried out by inkjet printing using a modified office inkjet printer equipped with piezoelectric print head. We were able to prepare transparent thin layers of TiO_2 with varying thickness and surface morphology, while PEG proved to be an efficient agent suppressing the formation of cracks in all cases.

Another project worth mentioning dealt with the adaptation and optimization of previously reported[5] reverse micelles sol-gel compositions to make them suitable for inkjet printing. The solvent choice and viscosity adjustment issues crucial for reliable jetting performance were thoroughly investigated. The optimized sol formulation was deposited by a dedicated material printer Fujifilm Dimatix, in contrary to the previously discussed projects.

Apart from smooth compact coatings, mesoporous layers have also been successfully fabricated in our lab. Sol-gel compositions utilizing various templating agents and reactive solvent systems were again printed by Dimatix.

However, not only sol-gel compositions can be used for the preparation of titania films. The other approach utilizes a stabilized suspension of titania nanocrystals dispersed into suitable liquid vehicle[6]. Such nanosuspension can be used for the fabrication of titania coating on heat sensitive substrates, because the need for thermal calcination is eliminated. Both Dimatix and modified Epson printers were used for the deposition of titania nanosuspension. While Dimatix proved to be optimal for small precise items, larger samples (A4) were conveniently and quickly printed by the Epson printer.

To conclude, inkjet printing proved to be an elegant method for sol delivery to substrate. It provides a complete control over the deposition process parameters together with an excellent efficiency of precursor use. Moreover, the possibility of precise patterning and the ease of up-scaling make this type of deposition very appealing for the production of sensors, solar cells etc.

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REFERENCES

1. http://www.solgel.com/articles/Nov00/coating.htm [cit. 2011-11-05].

2. Heller, A.: WO 97/07069.
3. Dzik P., Veselý M., Chomoucká J.: J. Adv. Ox. Tech., *13*, 172 (2010).
4. Cerna M., Vesely M., Dzik P.: Cat. Tod., *161*, 97 (2011).
5. H. Bartkova et al.: Thin Solid Films *515*, 8455 (2007).
6. Černigoj U., Lavrenčič Štangar U.: SI 22672 A.

1-L11

FRACTIONAL MODEL OF THE (^{13}C) ISOTOPE SEPARATION COLUMN

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A great number of chemical elements are mixtures of various isotopes. For instance, (^{12}C) is the basic carbon element, with a concentration of 98,9%, while (^{13}C) is a “heavier” isotope, with the natural abundance of 1,1%. If some chemical compounds with higher abundance of (^{13}C) are available, detection of compounds with higher concentration of (^{13}C) allows valuable qualitative and quantitative measurements, very important in scientific research and industrial applications. The rising of heavier isotope concentration is known as isotope separation process, based on various technologies.

The column for cryogenic (^{13}C) isotope separation is the case studied in this paper, using the equipment belonging to the National Institute of Research and Development for Isotopes and Molecular Technologies (INCDTIM), Cluj Napoca.

Unfortunately, for control design purposes control techniques and their implementation are directly proportional to the complexity of proposed control models[1]. Then, it is essential a noncomplex and simple control model that represents in a precise way the process behavior. However, this type of systems corresponds to long distributed systems with complex dynamics. Furthermore, these systems involve mass energy transport phenomena which behave as intrinsically distributed parameter systems, and their characteristics are very complex such as the variation of parameters with operation points, large delays that vary with operation point, and numerous interactions between different consecutive subsystems and strong nonlinearity. Their complete dynamics is represented by nonlinear partial differential equations, which depend on the time as well as the spatial coordinates: Cohen’s equations. This equation system has unknown analytical solution in real geometry and it has to be solved numerically (for example collocation method). The results are time consuming simulation models, suitable for scientific purposes but they are too complex for on-line applications and control needs. Distributed parameters systems, considered as systems with a very large number of states could be approximated with low-order linear time invariant (LTI) models in order to use classical linear control design tools, as an usual practice in control engineering. There are two main approaches that can be followed to obtain a linear model for separation column: the use of linearized Cohen equations or the use of identification methods. Normally, classical identification methods are used to obtain LTI discrete models which describe dynamics of process. However, in such systems LTI models lose information about these characteristics (nonlinearity, dynamics, parameters changing over operation time in a wide range, etc.). Then, a simplified control model structure – that still preserves their information – is needed. Such a structure can be provided identifying fractional order model from frequency response of the system[2] or from time response[3]. The used method, developed by Duarte *et al.*[3] is an extension of the integer case, developed to be used when conceiving second-generation Crone controllers. The resulting model is a fractional transfer function, with a commensurate order α:

$$G(s) = G_0 \frac{\prod_{k-1}^{m} 1 + \frac{s^{\alpha}}{b_k}}{\prod_{k-1}^{n} 1 + \frac{s^{\alpha}}{a_k}}, \; G_0>0, \; m+n=M \qquad (1)$$

where M is the total number of zeros and poles.

Model validation is the core of the identification problem because it makes possible to evaluate the model quality, that is, if the method fits the measured experimental data with accuracy enough, if it is valid for its purpose, and if the model describes correctly the real process.

Using the model described in the previous section, the authors simulated the model output for step input, highlighting the performance of fractional order models in comparison with the integer model and experimental data of the column. Globally, it can be appreciated that fractional order model track better measured values than integer models.

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REFERENCES

1. Dulf E. H., Festila C., Dulf F. V.: Chem. Listy *102*, 1075 (2008).
2. Valério D, Sá da Costa J. Identification of fractional models from frequency data. In: *Machado JAT, Sabatier J, Agrawal O, editors. Fractional calculus: theoretical developments and applications in physics and engineering*. Dordrecht: Springer (2007).
3. Duarte V., Sá da Costa J.: Commun. Nonlinear Sci. Numer. Simulat. *15,* 911 (2010).

1-P1

INVESTIGATIONS OF THERMOCHEMICAL PARAMETERS OF COMPLEXES OF THE FIRST ROW TRANSITION METAL HALIDES WITH N-DONOR LIGANDS

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Thermochemical parameters are important tools to determine the energies involved in the formation of metal complexes[1]. The present research work is concerned with the synthesis, thermal studies and calorimetric determination of heats of formations and heats of Lewis acid/base reactions of the complexes formed by metal bromides with the ligand, *3-Cyanopyridine*. The complexes $MBr_2.nL$ where (*M* is *Zn, Cu, Ni, Co, Fe* or *Mn*; *L* is *3-cyanopyridine*; n=2 or 4) were synthesized and characterized by elemental analysis, melting points, IR, UV and thermal analysis. Thermochemical parameters involved in the formation of complexes were determined by solution-reaction calorimetry.

All the complexes were solids at room temperature. The yields range from 35 to 78 %. The experimental analytical data is in good agreement with the proposed general formulas. The more important IR bands of the compounds are similar to that of the free ligand however, the spectra show shift of several bands after coordinating with respect to the free ligand. These variations in some bands intensity indicate the bonding of ligands to the central metal ions[2-3].

The ligand field parameters investigated from electronic spectra and absorption bands of the complexes[4-6]. These parameters demonstrate the pseudo-octahedral local symmetry for the complexes *Mn(II)*, *Fe(II)*, *Co(II)* and *Ni(II)* and tetrahedral geometry for *Cu(II)* and *Zn(II)* complexes. The TG/DTG data shows the loss of the ligands in several steps of mass loss followed by the loss of bromine *Mn(II)*, *Fe(II)*, *Co(II)* and *Ni(II)* complexes or complete loss of the ligand and part of the bromine *Cu(II)* complexes. All the complexes left a residue which is part of the metals *Zn(II)* and *Cu(II)* complexes or metal and bromide (Mn, Fe, Co and Ni) complexes. The DSC curves show few endothermic and exothermic peaks due to melting and dissociation of the complexes, consistent with the TGA data. The relative decrease in thermal stabilities order of the complexes of the transition metals, based on the data obtained from TG/DTG curves is $CuBr_2 > ZnBr_2 > NiBr_2 > FeBr_2 > CoBr_2 > MnBr_2$.

The standard enthalpies of Lewis acid/base reactions ($\Delta_r H^\theta$) of the complexes were determined by solution-reaction calorimetry. Several thermochemical cycles were used to calculate the standard enthalpies of formation ($\Delta_f H^\theta$) of the complexes. The calorimetric data obtained shows the interaction of the ligand with the metal bromides. Based on the values of ($\Delta_r H^\theta$) the acidity order of the salts in the synthesized complexes is $ZnBr_2 > CuBr_2 > FeBr_2 > MnBr_2$.

1-P2

THE CALCULATION OF FRAGMENTATION ENERGY OF ORGANOMETALLIC COMPOUNDS BY USING AB INITIO METHOD

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Fragmentation energy is the property of molecular structure, and it is defined as the required energy for separation of several chemical bonds of the large molecule and getting smaller particles (fragments). The fragmentation energy has a relation to reactivity of the compound. Particularly, in this case, the fragmentation of the propane molecule near the catalyst surface was investigated. The fragmentation energy can be calculated by *ab initio* methods used in quantum chemistry, which are based on Born Oppenheimer Approximation.

In the paper, the data from literature review and some preliminary simulations are presented. According to the literature, the *ab initio* approach is realized by several methods such as Hartree-Fock (HF or RHF), which is also known as the Self-Consistent Field (SCF), Møller-Plesset (MPn) perturbation theory, Density Functional Theory (DFT), and Becke-3-Lee-Yang-Parr method (B3LYP). The input properties are atomic topologies of molecule and basis set. The topology defines atoms which are connected to one another through chemical bonds. Basis set is a set of functions used to create the molecular orbitals. They include split valence, polarization basis function, and diffuse basis set. In general, *ab initio* methods have significant sensitivity to basis set, and thus fragmentation energy is different according to type of used basis set[1].

Primary results of calculations are coordinates of atoms, the potential energy curves of the molecule, its vibrational frequencies, thermodynamic properties, and the shapes of its molecular orbitals.

The *ab initio* method can be applied also for predictions of physical properties of compounds. In the literature, prediction of infrared spectra[2] show very good agreement with experimental data, also prediction of heat capacities[3], enthalpies of formation[3], and entropies[3] of gas-phase energetic materials used in explosives and propellants. The resulting data, currently not available in the literature, are critical for modeling reaction mechanisms and combustion-wave structures of these materials.

The calculation of fragmentation energy was presented in the literature[4]. It was calculated for higher fullerenes C_{80} and C_{82} was calculated by *ab initio* method (MP2, DFT), the result was in the case of the most probable Stone-Wales pathway of C_2 fragmentation of C_{80}, the calculated $D_0(C_{80}^+)$ agree well with experimental data, whereas in the case of C_{82} fragmentation, the calculated $D_0(C_{82}^+)$ exceed by up to 1,2 eV the experimental data[2]. Fragmentation energies can be measured (experimentally) by methods such as kinetic-energy-release-distribution (KERD) and metastable-fraction (MF) measurements.

The preliminary simulations were performed using MOLPRO software for propane molecule, which the task was geometry optimization. Input file must contains information about geometry specification, basis set specification, and a method of calculation. The output file will contain information about potential energy, atomic coordinates, and bond lengths and angles. In future, the data will be calculated by different methods (HF, MPn, DFT, B3LYP) and basis sets and compared to the experimental data.

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REFERENCES

1. Boese D. A., Scuseria G. E.: Chem. Phys. Lett. *294*, 233 (1998).
2. Kalaichelvan S., Sundaraganesan N., Joshua D. B.: Ind. J. Chem. *47A*, 1632 (2008).
3. Osmont A., Catoire L., Gokalp I., Yang V. : Combust. Flame. *151*, 262 (2007).
4. Dolgonos A. G., Peslherbe G. H. : J. Mol. Model. *13*, 981 (2007).

1-P3

CHARACTERISATION OF FATTY ACIDS IN HUMIC ACIDS EXTRACTED FROM PHYSICALLY MODIFIED SOUTH-MORAVIAN LIGNITE

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Humic substances are the most widely distributed matter occurring on the Earth playing fundamental roles in the soil, e.g. as a plant nutrient transporter in rhizosphere or as one of the essential member of carbon cycle. Their addition to soil is considered as a way to increase soil "quality" and support processes vital for soil fertility. Processes occurring in rhizosphere, i.e. in the interface layer between roots and soil body are important from the physical-chemical point of view since the exudates released by plant roots (typically small organic acids) induce the reconformation of humic matter structure associated with a transport of nutrients through the root cell walls. Together with inorganic part, also a part of humic acids is transported and incorporated into the plant biological cycles. This is recognized as biological activity or hormone-like activity of humic acids. The simulation of those processes during the humic acids production was the motivation of this research. Humic acids (HA) were extracted from South Moravian lignite which was first pretreated by several small organic acids (e.g. formic acid, acetic acid, etc.) and a line of samples with different physical structures was obtained. The objective of this study was to focus on the composition of fatty acids presented in those samples using chemical degradations such as thermochemolysis[1] using TMAH (tetramethylamonium hydroxide). This approach supports the release of extractable hydrophobic compounds such as fatty acids which are consequently detected and analyzed by Gas Chromatography coupled with Mass Spectrometry (GC-MS).

Fatty acids as methyl esters (FAMEs) mostly ranging from the C16 to C32 were observed dominated by the C28 and C30 members what refers to higher plants wax origin[2]. Up to 24 methylated lignin derivates were indentified in obtained extracts. The total lignin yield decreased after parental lignite modification. Less pronounced decrease was observed in case of samples pre-treated by benzoic and phenylacetic acids.

Knoweledge of molecular structure of HA is inevitable for understanding the biological activity of extracted humic acids and their agricultural application.

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REFERENCES

1. Válková D., Grasset L., Amblès A.: Fuel *88*, 2113 (2009).
2. Kolattukudy P.E., in the book: *The Biochemistry of plant lipids structure and function*, p. 571. Academic Press, New York 1980.

1-P4

INSIGHTS INTO LIGNITE STRUCTURE FROM CHEMICAL DEGRADATION METHODS

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Lignite is a heterogeneous material with a very complex structure and difficult chemical composition. For example, knowledge of coal structure may be important for explication of sorption properties of heavy metals. The most common view is that coal formed mostly from the altered biopolymers found in plants. Degradation methods can be used as one of the approaches for determining the chemical structure. The aim of this work was to investigate the South Moravian lignite degradation by three methods; (i) oxidation by cupric oxide, (ii) thermochemolysis with tetramethylammonium hydroxide and tetraethylammonium acetate and (iii) DFRC (derivative followed by reductive cleavage). Tetramethylammonium hydroxide and tetraethylammonium acetate was used to distinguish between free fatty acids tightly trapped within the lignite and esterified acids. The degradation products were fatty acids and aromatic molecules. The work also showed that the studied lignite contains intact lignin monomers. Comparison of thermochemolysis data with the results of the traditional cupric oxide oxidation method and DFRC indicated that thermochemolysis produces significantly proportion of fatty acids.

1-P5

DEVELOPMENT ORGANIC SOLAR CELLS BASED ON CONJUGATED POLYMERS

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This work is focused on a preparation and study of flexible polymeric solar cells. Three different methods of a thin layer deposition from solution were used: spin-coating, ink-jet printing[1] and electrophoretic deposition[2]. Properties of prepared solar cells on rigid (glass) and flexible (PET foil, textile) substrates were studied by electrical and optical measurements.

Solar cells were prepared from a solution mixture of a electron donor polymer - high Tg-PPV (copolymer of poly-(phenylene vinylene) derivates with high glass transition temperature)[3] and a electron acceptor - fullerene PCBM (phenyl-C61-butyric-acid-methyl ester). The chemical structures of the used materials are shown on Figure 1.

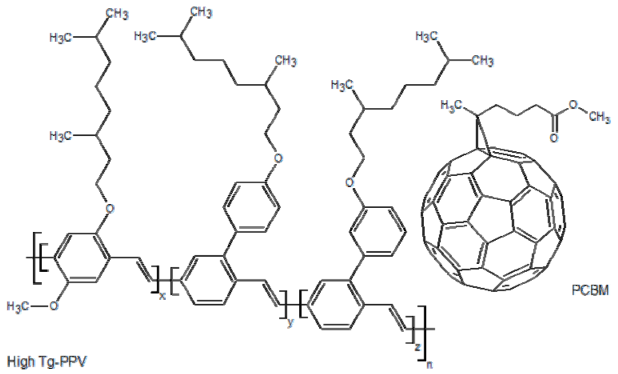

Figure 1. Structures of used materials: electron donor polymer high Tg-PPV and electron acceptor fullerene PCBM.

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REFERENCES

1. Krebs F. C.: Sol. Energ. Mat. Sol. C. *93,* 394 (2009).
2. Tada K., Onoda M.: Thin Solid Films *477*, 187 (2005).
3. Vandewal K., Gadisa A., Oosterbaan W. D., Bertho S., Banishoeib F. Severen I., Lutsen L., Cleij T. J., Vanderzande D., Manca J. V.: Ads. Funct. Mater. *18*, 2064 (2008).

1-P6

INFLUENCE OF THE MODIFICATION OF HUMIC ACIDS ON THEIR REACTIVITY

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Humic acids are natural compounds, which can be found in soils, coal, water and sediments. The main function of humic acids in soils and sediments is to impact the porosity and to act as a sorbent and reservoir of water and different kind of chemicals[1]. High affinity of humic acids towards other compounds (i. e. heavy metals, hydrophobic species, tenzides) is object of many studies in present days.

Carboxylic functional groups of humic acids are responsible for creation of the interactions with heavy metals. Used humic acids were modified by the methylation of reactive carboxylic group. Non-modified and modified humic acids were mixed in different ratios. We obtained humic acids with different amounts of free non-methylated carboxylic groups. From these mixtures were prepared humic hydrogels[2]. The interactions between heavy metals (cooper (II) ions) and humic acids were studied in diffusion experiments. Diffusion coefficients of copper (II) ions in different hydrogels were calculated.

The results showed, that with increasing amount of methylated humic acids in the mixtures, the values of diffusion coefficient are decreasing. This is due to the fact, that with increasing of the ratio of modified humic acids in the mixtures the amounts of carboxylic groups are decreasing (due to methylation), which results in the decrease of the formation of complexes with copper (II) ions.

Presented involvement of modified humic acids in easy diffusion experiment showed to be suitable for deeper study of reactivity of humic acids and the formation of the interactions, which between heavy metals and humic acids occur in the nature.

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REFERENCES

1. Jansen S. A., Malaty M., Nwabara S., Johnson E., Ghabbour E., Davies, G.: Mat. Sci. Eng. *4*, 3, (1996).
2. Sedláček, P., Klučáková, M.: Geoderma. *153*, 286, (2009).

1-P7

DENSITOMETRY OF HYALURONAN AND HYALURONAN WITH CTAB

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Hyaluronan is a biocompatible, bidegradable polymer produced even in human bodies. Due to its biological properties and physiological functions it among ideal candidates for fomulation of systems for targeted delivery of biologically active substances. Many of these substances are hydrophobic and cannot be loaded on highly hydrated hyaluronan chain. One possibility to overcome this obstacle is forming complexes between hyaluronan and oppositely charged surfactant.

In this work hyaluronan was complexed with cetyl trimethylammonium bromide. Properties of both hyaluronan and formed aqueous colloids were investigated by means of densitometry using DMA 4500 densitometer (Anton Paar).

Density of aqueous solutions of hyaluronan of various molecular weights were measured in dependence on concentration at 25 °C. After carefull dissolution of the biopolymer (for about 48 hours) and conservation against microbial degradation (using sodium azide) reproducible results can be obtained that are stable during 14 days of storage of solutions, at least. Regardless the molecular weight the density is a linear function of hyaluronan concentration except very dilute solutions. At very low hyaluronan concentrations the solution density is less sensitive to the presence of the biopolymer which could be a result of disentangling hyaluronan chains.

Addition of increasing amount of CTAB surfactant to hyaluronan solution containing constant hyaluronan concentration resulted in increasing density with increasing CTAB concentration with at least one break point indicating formation of polymer-surfactant complexes.

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1-P8

STABILITY OF METAL-HUMIC COMPLEXES

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This work is focused on comparison of thermo-oxidative and chemical stability of metal complexes with humic acids. Three elements representing the group of heavy metals were selected for these experiments (cobalt, copper, nickel). The utilized humic acid was extracted from South-Moravian lignite. Differently concentrated solutions of metals were used for complexes preparation, in order to observe the influence of their initial concentration on both studied stabilities of prepared complexes. Chemical stability of metal complexes was assessed in term of metal ions release from the humic acid structure into two different extraction agents. Thermo-oxidative behaviour was investigated employing methods of thermal analysis, differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) in particular.

The results of this work suggest that the incorporation of metal ions influences not only the thermo-oxidative stability of humic acid but its thermo-oxidative behaviour in general. All metal ions bound to HA structure implicate the decrease of starting and ending temperature of HA thermo-oxidative degradation. The most substantial changes in the degradation process of HA were produced by the presence of Cu^{2+} in HA structure. On the contrary, records of samples with Ni and Co ions exert very comparable course, which is probably due to similar properties based on close atomic masses. All samples were subjected to extraction by two different agents, $MgCl_2$ and HCl. Extraction of metal ions with HCl was entirely successful. Besides the extraction of metal ions, HCl influences also the structure of HA, since there is almost zero content of ash and the thermogravimetric degradation step is very slow and gradual. From our results we also infer the application of Langmuir isotherm only for concentrations below 0,05 M.

The conclusions bring deeper insight into the realm of metal interaction with the humified part of soil organic matter; and at the same time try to shed light on the fate of metal pollutants in the environment and help in the desirable knowledge of treatments such as soil decontamination and remediation.

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1-P9

CONFORMATIONAL CHANGES IN AQUEOUS SOLUTIONS OF HUMIC ACIDS

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Conformational changes of humic acids in two different aqueous solutions (NaCl and NaOH) are studied by means of High Resolution Ultrasound Spectrometry. It is based on the measurement of parameters of ultrasonic waves propagating through the sample. Attenuation describes the decay of the amplitude of the ultrasonic wave with distance traveled. Ultrasonic velocity is the speed of this wave and is related to the wavelength and the frequency of oscillation of the deformation. It is determined by the density and elasticity of the sample, which is strongly influenced by the molecular arrangement. The elastic response is generally dominant.

The minimum of ultrasonic velocity was observed for humic content of 1 g dm^{-3} in 0,1M NaCl. Compressibility was practically the same up to this concentration and then they decreased. This confirms that the organization of humic particles in diluted and concentrated humic sols is different. The decrease in compressibility showed the forming of more rigid structures which could cause the decrease in humic binding ability. This conformational change was confirmed also by the strong increase in ultrasound attenuation for humic sols in NaCl. On the other hand, no minimum but two breaks were observed, if humic acids were dissolved in 0,1M NaOH. First one was measured at 0,1 g dm^{-3}, second one at 1 g dm^{-3}. Increase in attenation was lower for humic solution in NaOH comparing with NaCl.

Our results confurmed that dissolved humic acids may be micelle-like, which are supramolecular assemblies of small entities[1-3]. If micelle-like structures form at high concentrations, a portion of humic reactive functional groups may be less accessible for e.g. metal ions as they might be entrapped in the more hydrophobic interior[4].

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REFERENCES

1. Piccolo A., Nardi S., Concheri G.: Chemosphere *33*, 595 (1996).
2. Conte P., Piccolo A.: Environ. Sci. Technol. *33*, 1682 (1999).
3. Simpson A. J., Kingery W. L., Hayes M. H. B., Spraul M., Humpfer E., Dvortsak P., Kerssebaum R., Godejohann M., Hofman M.: Naturwissenschaften *89*, 84 (2002).
4. Christl I., Metzger A., Heidmann I., Kretzschmar R.: Environ. Sci. Technol. *39*, 5319 (2005).

1-P10
UTILIZATION OF IN-ELECTRODE COULOMETRIC TITRATION IN HUMIC RESEARCH

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In this work, the in-electrode coulomeric titration is used for analysis of humic acids in aqueous solutions. The method is based on the galvanostatical reduction of splitting off hydrogen ions at a suitable potential. The method was valited using simple organic acids (acetic acid, benzoic acid, citric acid, oxalic acid, phthalic acid and salicylic acid). It was found that efficiency of the method depends on acid concentration and strength. The limits are concentration of ~20 mmol.dm^{-3} and pK_a ~ 5.

Humic acids are analysed as extract in solution of sodium sulfate (1,5 % wt.). The efficiency of galvanostatic reduction depended on concentration of humic acids as in previous case. The limit obtained for used sample was 20 g of humic acids per litre. It is well-known that humic acids are soluble in neutral aqueous solutions only partially[1-2]. Their carboxylic groups have various strength which influences their solubility and activity in natural systems. Humic fractions rich with carboxylic groups are much more soluble. The composition of dissolved fraction is the dependent on initial ratio between humic acids and aqueous medium. Solubility of humic acids in water and aqueous solutions is thus far from simple as might be expected from their solubility-based definition and a multi-mechanism of their dissolution and dissociation[1-2].

In contrast to measurement of pH, which deteremines the amount of dissociated hydrogen ions, the in-electrode coulomeric titration gives results corresponding with total content of COOH groups in humic extract. The combination of these two methods can be utilized for determination of pK_a of dissolved humic acids without titration. Regarding gradual dissolving of various humic fractions with increasing humic content we can compute mean value of pK_a in dependence on initial ratio between humic acids and aqueous medium.

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REFERENCES

1. Klucakova M., Pekar M.: Colloid Surface A *252*, 157 (2005).
2. Klucakova M., Pekar M.: Colloid Surface A *318*, 106 (2008).

1-P11
EFFECT OF COMPOST APPLICATION ON PROPERTIES OF SOIL HUMIC SUBSTANCES

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Humic acids represent very important part of soil organic matter. Object of our study was Luvihaplic Chernozem (locality Praha-Ruzyně). Three different amounts were added in soil in 2008: 124, 239 and 478 t ha^{-1}. Soil humic substances were extracted one year after compost application into the soil and analysed by means of fluorescence spectrometry, infrared spectrometry, ultraviolet-visible spectrometry a titration methods. EEM spectra obtained using fluorescence spectrometry showed that compost contains well humified fractions of humic substances. Relative intensity of fluorescence of all three main peaks is lower in comparison with soil humic substances due to content of „young“ less condensed aromatic structures. Infrared spectra confirmed that increasing added amount of compost caused the increase of content of carboxylic groups and ether bridges in soil humic substances. Humification index $E_{4/6}$, determined on the basis of measured UV/VIS spectra, decreased with desresing amount of added compost. Its values were higher than 4 for all extracted humic samples, which show on brown humic acids with lower molecular mass. The high value of $E_{4/6}$ for compost confirmed the presence of „young“ less condensed aromatic structures detected by fluorescence spectrometry. Because the index decreases with depth of soil horizon, we can say that lower soil layer content „older“ more humified humic substances. Total acidities computed from titration curves increased with increasing amount of added compost which corresponds with results of other used methods.

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1-P12
EFFECT OF METHYLATION ON HUMIC ACIDS

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Humic acids (HAs) play an important role in carbon cycle, sustaining plant growth, stabilizing soil structure, and protection from environmental contamination. Despite their importance, their structure remains largely unknown. Their heterogenity and extreme complexity render structural information often difficult and controversial. In this present study, the effect of methylated HAs and untreated HAs by methylation was investigated. Obtained extracts were fractionated by liquid chromatography. Insoluble residues after treatment of humic acids were submitted hydrolysis reactions. For experiments were used four variously treated lignite humic acids and one humate. All products were analyzed by chromatography-mass spectrometry (GC-MS). The main families present were linear hydrocarbons, fatty acids and aromatic molecules. Analyses of hydrolysis products were carried out to gain further insight into the structure of HAs. Hydrolysis products were rich in aromatic molecules. It was found that the methylation of carboxylic groups responsible of non-covalent linkages (such as hydrogen bonds) permits the release of organo-soluble compounds. Then, weak forces are strongly implicated in the layout of these HAs confirming partly the macromolecular concept of humic substances.

1-P13
DYNAMIC TENSIOMETRY OF HYALURONAN-SURFACTANT SYSTEMS

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The interactions between a natural negatively charged polysaccharide hyaluronan[1] and cationic surfactants tetradecyltrimethylammonium bromide (TTAB) and hexadecyltrimethylammonium bromide (CTAB) were studied by a dynamic tensiometric method – maximum bubble pressure method.

Hyaluronan is a very important biopolymer in living organisms, it occurs mainly in tissues and extracellular matrix of vertebrates where it plays a role of a moisturiser and lubricant due to its strong hydrophilic character[2]. Hyaluronan is also one of the possible carriers in the targeted drug delivery. Together with the suitable hydrophobic domain the solubility of nonpolar drugs is guaranteed. For this purpose, cationic surfactants were chosen and the interactions between the opposite charge of the surfactant head group and hyaluronan carboxylic group were studied.

The aim of the present work was to study the expected electrostatic interactions using the dynamic tensiometry because of the task of the properties of the forming aggregates in the bulk. The experiments were performed both in water and sodium chloride solution and with two molecular weights of hyaluronan.

The results are compared from the point of the surfactant carbon chain length, molecular weight of hyaluronan and ionic strength. The added hyaluronan decreased the interfacial tension of both surfactants in water, the influence of its molecular weight was not observed. In addition, the longer the carbon chain of surfactant the faster stabilization of interfacial tension in both water and sodium chloride environment.

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REFERENCES
1. Lapčík L. Jr., Lapčík L., De Smedt S., Demeester J., Chabreček P.: Chem. Rev. *8*, 2663 (1998).
2. Ribeiro W., Orfao M., Mata J. L., Saramago B.: J. Colloid Interface Sci. *317*, 536 (2008).

1-P14
DETERMINATION OF CARBOHYDRATES AND VOLATILE COMPOUNDS AT ASH WOOD PREHYDROLYSIS

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Water prehydrolysis appears as a perspective pretreatment for lignocellulosic biomass conversion on different chemical stocks[1,2].

Ash wood (*Fraxinus exelsior,* L.) chips were subjected to hydrothermal pretreatment at a liquor to wood ratio 4:1 (ml/g) and temperatures within the range of 160-200°C. The time treatment was carried out from 30 to 240 min.

The released saccharides (D-xylose, L-arabinose, L-rhamnose, D -glucose, D-mannose and D-galactose) and volatile compounds (acetic acid, 2-furaldehyde, methanol and propionic acid) were determined in hydrolyzates by gas chromatography method (GC)[3,4].

The water pretreatments of lignocellulosic materials have got a different effect on their main components, depending on the operational conditions.

The maximum concentration of the released sugars from wood in monosaccharide form was found at the temperature 180 °C and at the time 60 min. The presence of oligosaccharides and low-molecular polysaccharides in hydrolysates was approximately five times higher than monosaccharides. The maximum concentration of mono- and oligosaccharides sum was found at the temperature 160 °C and at the time 120 min.

From volatile compounds acetic acid and 2-furaldehyde were dominate in hydrolyzates. At the mild conditions of hydrolysis the acetic acid and 2-furaldehyde concentration increases due to the saccharides deacetylation and dehydration, respectively. The amount of 2-furaldehyde was decreased at 200 °C due to its participation in the condensation reactions.

FTIR spectrum of treated wood confirmed decay of hemicelluloses portion.

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REFERENCES

1. McKendry P.: Biores. Technol. *83*, 37 (2002).
2. Geffertová J.: Acta Facultatis Xylologiae *51*, 63 (2009).
3. Kačík F., Kačíková D.: Folia Forest. Polon. *40*, 61 (2009).
4. Kačík F., Výbohová E., Kačíková D.: Acta Facultatis Xylologiae *49*, 39 (2007).

1-P15
MONITORING OF HISTORICAL BOOKS PROPERTIES

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Crucial influence on the paper quality is a method of pulp production. If the pulp to produce high-quality paper was prepared from natural raw materials, i.e. of rags, it was made the first step to their longevity. Thus, even 300-400 years old papers have very good properties. On the other hand, papers produced after 1850 have significantly poorer quality because of its pulp production are used and the sizing is started using aluminium sulphate (acid sizing)[1].

An important factor causing the decrease in paper strength properties is acid catalysed hydrolysis. In addition to the cellulose hydrolysis can take place other reactions - oxidation, cross-linking and thermal degradation[2-4].

Samples of various historical books (3 samples from XVIII. century, 3 samples from XIX. century and 1 sample from XXI. century) were studied using ATR–FTIR spectroscopy, carbohydrates by HPLC according standard ASTM E 1758-01.

The predominant sugar in all samples was D-glucose, mainly in the older ones (approx. 96-98%). In the samples after 1850 the relative content of D-glucose was diminished to approx. 82-86%, amount of D-xylose raised to 4-5%

FT-IR spectra show the products of cellulose oxidation peaks in range 1500 -1800 cm^{-1} (carboxyl, aldehyde, carbonyl, enolic, β-diketones and conjugated carbonyl groups)[5]. Some inorganic and organic compounds (e.g. $CaCO_3$, clay) used for glue sizing were detected, too.

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REFERENCES

1. Bukovský, V., Trnková, M., in the book: *Ochrana knižných fondov I.*, p. 212, SNK, Martin 2006.
2. Zervos, S., Moropoulou, A.: Cellulose *12*, 485 (2005).
3. Bansa, H.: Restaurator 23 (2002).
4. Kačík, F., Kačíková, D., Jablonský, M., Katuščák, S.: Polym. Degrad. Stab. *94*, 1509 (2009).
5. Brandt N. N., Chikishev A. Yu., Itoh K., Rebrikova N.
6. L.: Laser Physics *19*, 483 (2009).

1-P16
HYDROLYSIS OF RECYCLED FIBERS

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This theses deal with the water and acid hydrolysis (1 % H_2SO_4) sample of pulp from recycled fibres. The main aim of this research was to determine optimal conditions (temperature, time) by which is given out the maximum of the saccharides extracts. By the determination of the saccharides was used gas chromatography (GC)[1]. The results of this work confirmed the effect of the temperature (140, 160, 180 °C) on the recovery of the saccharides during the time of use 30, 60, 90 a 120 min. The sample was hydrolysed in water and in 1 % H_2SO_4 [hydromodule 1:40 (g ml^{-1})]. To the creation of hydrolyzed products became early at the tempetarure of 140 °C. After the GC was increasing in time gap the concen-tration of the saccharides and after the acid hydrolysis (1 % H_2SO_4), the concentration of the saccharides was decre-asing in time gap. The maximas of the recovery of mono-saccharides after the GC were spotted at the temperature of 160 °C in the time period of 120 min., after the use of acid hydrolysis at the temperature 140 °C in the period of time of 30 minutes. The maximas of the recovery of overall sacchari-des after water hydrolysis were recorded at the temperature 180 °C (120 min.) and after acid hydrolysis also at temperature 180 °C but after 30 min of hydrolysis. Volatiles like acetic acid, propionic acid, methanol, ethanol and 2-furaldehyd were specified by the gas chromatography[2]. Their production was in progress simultaneously with the making of saccharides. The maximum amount of the acetic acid after the water hydrolysis was recorded at the temperature of 140 °C during which time the 2-furaldehyd was not present in the hydrolysates. After the acid hydrolysis was recorded the biggest amount of acetic acid at the temperature of 140 °C and 2-furaldehydu at the temperature 180 °C. Specified vola-tiles in hydrolysates relate with the presence of sacchari-des and upright effect their recovery after the hydrolysis. In the hydrolysates after the water and acid hydrolysis was with the use of gravimetric method stated the amount 2,4-dinitro-fenylhydrazones (2,4-DNPH). The top amount 2,4-DNPH was noticed after the acid hydrolysis at the tempereture of 180 °C and time of use 90 minutes. Besides this had been also carried out Seamans hydrolysis and was determined the amount saccharides with the method of GC. The total extract of saccharides was 65 %. The sample of the recycled fibers contained 14,25 % of lignin stated by the ASTM D 1106 (2001).

This contribution is the result of the project implementation: Centre of Excellence „Adaptive Forest Ecosystems“, ITMS: 26220120006, supported by the Research & Development Operational Programme funded by the ERDF.

REFERENCES

1. Kačík F., Kačíková D.: Folia Forest. Polon., *40*, 61 (2009).
2. Kačík F., Výbohová E., Kačíková D.: Acta Facultatis Xylologiae *49*, 39 (2007).

1-P17

PHOTOCATALYTIC INACTIVATION OF THE YEAST ON TITANIUM DIOXIDE THIN LAYERS

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Photocatalytic inactivation of microorganisms by titanium dioxide has been widely studied since 1985 when Matsunaga and co-workers reported that titanium dioxide could demage bacterial cells. When photocatalyst titanium dioxide (TiO_2) is illuminated by near-UV light, TiO_2 demonstrates excellent microbicidal activity. Reactive oxygen species (hydroxyl radical, hydrogen peroxide and superoxide) which are generated during photocatalytic process can harm various cells.

Photocatalytic inactivation of yeast Candida glabrata has been investigated using imobilized thin layer of TiO_2 on the glass support. The deposition of TiO_2 on the glass plates was accomplished in an innovative way, print with device FUJI Dimatix. Epi-fluorescent microscopy was used as a suitable method for analysis of cell viability.

1-P18

A STUDY OF THE CHEMISTRY OF PLASMA JET INTERACTION WITH SOLUTIONS

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Plasma, the so-called fourth state of matter, has become a very interesting and important subject for study through the last century. Plasma conditions can produce a wide range of unique chemical reactions that are not possible under thermodynamic equilibrium. Thus plasma is now widely used in various technologies such as surface modification, thin film coatings for various purposes, in the production of microelectronic devices etc[1]. There is also increasing interest in plasma applications in environmental chemistry, e.g. for the removal of pollutants from air and water[1].

Within the last two decades there has been an increasing focus on plasma chemistry in gas discharges interacting with various liquids or even generated directly in the liquid phase[2,3]. A number of papers discuss plasma chemistry in different areas of biology (extermination of microorganisms)[4], ecology, electrochemistry (degradation of dangerous toxic compounds in water)[5] and medicine (e.g. in surgery)[6]. The physics of such discharges has also been examined and has found a range of applications (e.g. destruction of kidney stones by shock waves)[7].

Here we present an atmospheric pressure plasma jet interacting with a liquid surface. The plasma is generated above the surface of a solution and thus the effect on the chemical properties of the solution are explored. Distilled water both with and without dissolved compounds is used for the experiments. A DC voltage of 2 kV was applied to the plasma jet which uses He as the supporting gas. Optical emission spectroscopy of the plasma jet provided information about the plasma parameters (density and temperature) and plasma chemistry.

While the spectra is dominated by N_2 emission, which can be used to determine the gas temperature, there is also evidence of the dissociation of water molecules reflected in the presence of OH radical emission. H and O emission are also observed. The OH radicals are H_2O_2 precursors. OH radicals are characterized by high reactivity with organic compounds and high redox potential (2,80V), guaranteeing non-selective behaviour in oxidative reactions with any organic substances which are presented in solution, and therefore OH radicals belong to the most important particles which are generated by electric discharge because of their high chemical efficiency[8,9]. The detailed study of these reactions and their efficiency will be a subject of further studies. Within the liquid environment we anticipate that the dominant long-lived species created will be H_2O_2. We are therefore determining H_2O_2 production efficiency in the present system and these results will be reported.

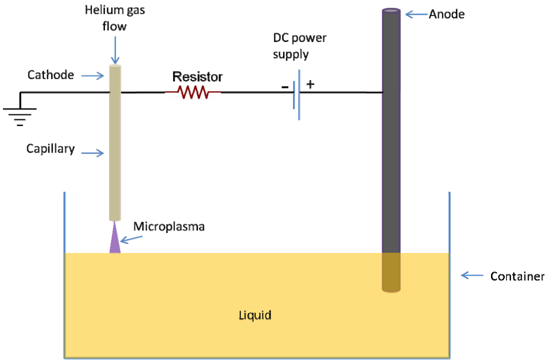

Figure 1. Diagram of Plasma Jet Setup

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REFERENCES

1. Fridman A. in the book: *Plasma Chemistry*. Cambridge University Press, Cambridge 2008.
2. Nikiforov A., Leys Ch., Němcová L., Krčma F.: Plasma Sources Sci. Technol. *20*, doi. 10.1088/0963-0252/20/3/034008 (2011).
3. Krčma F., Stará Z., Procházková J.: J. Phys. Conf. Ser. *207*, 6 (2010).
4. Anpilov A. M, Barkhudarov E. M., Christofi N., Kop'ev V. A., Kossyi I. A., Taktakishvili M. I., Zadiraka Y.: Lett. Appl. Microbiol. *35*, 90 (2002).
5. Brablec A., Slavíček P., Sťahel P., Čimžmár T., Trunec D.: Czech. J. Phys. *52*, 491 (2002).
6. Stalder K. R., McMillen D. F., Woloszko J.: J. Phys. D., *38*, 1728 (2005).
7. Chaussy Ch., Brendel W., Schmiedt E.: Lancet *2*, 1265 (1980).
8. Němcová L., Krčma F., Nikiforov A., Leys Ch., in the book: *HAKONNE XII-Book of Contributed Papers*, p. 398, Comenius University, Bratislava (2010).
9. Němcová L., Nikiforov A., Leys Ch., Krčma F.: IEEE Trans. Plasma Sci. *39*, 865 (2011).

1-P19

EDXS AND FTIR SRATR SPECTROSCOPIC CHARACTERIZATION OF SOIL HUMIC SUBSTANCES

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Soil humic substances and their predominant fraction humic acids are very active in interacting with organic and inorganic chemicals and can act as scavengers for various kinds of contaminants. For this reason humic acids were isolated from different soil types (*Luvic Chernozem, Leptic Cambisol and Haplic Luvisol)* and characterized by EDX-ray spectroscopy and FTIR SRATR (single reflection attenuated total reflectance) spectroscopy. Elemental composition was determined by the standard methods and PE2400 CHNS/O elementary analyzer was used. FTIR SRATR spectra were measured by Shimadzu 8700. Results showed bands indicative of aliphatic groups (C-H at 2925 cm^{-1}); aromatic groups (C=C at 1620 cm^{-1}); phenolic groups (at 1404 - 1419 cm^{-1}); and carboxyl and carbonyl groups (at 1225 – 1223 cm^{-1}) were similar in all samples. More aromatic groups were found in humic acids isolated from *Luvic Chernozem*. More aliphatic groups were found in humic acids isolated from *Leptic Cambisol*. Differences were found in the finger print region 1700 – 1000 cm^{-1}. Reactivity of humic acids was given by O/R ratio calculated from FTIR SRATR spectra according to Wander & Train (1996). EDX-ray spectra were recorded by energy–dispersive X-ray spectrofluorimeter XEPOS. Results showed elements occur *in situ* in HA molecule. All samples contained Fe, Cu, Zn, Ti, Ca, K, S, Ph, Si and Br.

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1-P20

PULSE MODE IN PLASMA POLYMERIZATION OF HEXAMETHYLDISILOXANE

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The interest of experts from thin film branch has already been focused on chemical vapour deposition (CVD) techniques for more than twenty years. Especially plasma enhanced CVD brought new options to this field. Thin films produced this way – plasma polymers – have excellent adhesion to the substrate, are resistant to most chemicals and improve mechanical properties of the substrate[1,2].

Variety of monomers offers additional advantages. Using organosilicons, e.g. hexamethyldisiloxane, or tetravinylsilane, is a modern trend in PECVD. Thanks to silicon atom in its structure the layer can bind to glass and organic groups provide many possibilities of modifications of thin film properties. In this manner we can produce SiO_2 thin films[3,4] used in electronic engineering.

The aim of our experiment was a study of properties of hexamethyldisiloxane plasma during thin film deposition in pulsed regime. The experiment was held first at constant power and variable duty cycle, second at constant mean power. In this case the distribution of energy is quite different[5] and may be useful to obtain thin layers with new properties. Experiment shows that dependence of fragment population on duty cycle was increasing with increasing duty cycle in the first case, however, there was a maximum detected between 30 and 40 % in the second case. This point might be an optimal setup for the deposition of SiO_2 thin films from hexamethyldisiloxane.

REFERENCES
1. Inoue Y., Sugimura H., Takai O.: Thin Solid Films *345*, 90 (1999).
2. Ingaki N., Kondo S., Hirata M., Urushibata H.: J. Appl. Polym. Sci. *30*, 3385 (1985).
3. Goujon M., Belmonte T., Henrion G.: Surf. Coat. Technol. *188*, 756 (2004).
4. Bang S.B., Chung T.H., Kim Y.: Thin solid films *444*, 125 (2003).
5. Förch R.: Plasma Polymerization. Max-Planck-Institute für Polymerforschung. Mainz 27th January 2005.

1-P21

PLASMA TREATMENT OF CORROSION LAYERS FROM IRON IN RF LOW PRESSURE HYDROGEN PLASMA

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An effective and fast way of restoration and preservation of archeological artifacts is described [1]. The process is based on using RF low-pressure hydrogen plasma in which the artifacts are treated for several tens of minutes, usually in more steps[2,3]. This method offers significant advantages such as preservation of the quality of the object surface and time savings [1].

The optical emission spectroscopy of OH radical was used for the process monitoring. Atomic hydrogen reacts with oxygen from corrosion layers and forms OH radical (in excited state). This OH radical emits in UV area of spectrum (305–325 nm). Its integral intensity was used for the quantitative analysis of oxygen removal from the corrosion layer. Plasma treatment stoped when value of relative intensity OH radicals reached one tenth of maximum OH radicals [4].

Another monitored magnitude was temperature of samples. Sample temperature is very significant indicator for protection sample against metallographic changes of metal bulk material and therefore damage of samples [5]. Temperature is depends on using regime continual or pulsed. Our results show that pulsed regime can be effectively used in the corrosion removal process. The main advantage of the pulsed regime is lower heating of sample. However, process runs longer time.

This work was supported by NAKI, grant No.202/07/P371.

REFERENCES
1. de Graaf M. J., Severens R. J., van Iyendoorn L. J., Munnik F., Meijers H. J. M., Kars H., van de Sanden M. C. M. , Shram D. C.: Surf. Coat. Tech. *351*, 74 (1995).
2. Veprek S., Patscheider J., Elmer J.: Plasma Chem. Plasma P. *5*, 201 (1985).
3. Veprek S., Elmer J., Eckmann Ch., Jurcik-Rajman M.: J. Electrochem. Soc. *134*, 2398 (1987).
4. Cihlář M., Diploma thesis, Diagnostics of plasma during reduction of corrosion layers from copper, Brno, 2005.
5. Sázavská V., Řádková L., Krčma F., Zmrzlý M., Přikryl R., Zahoran M.: Plasma Treatment of Corrosion Layers from Brass, *Proceedings of 18th Symposium on Application of Plasma Processes,* p. 266-271, 2011.

1-P22

CHARACTERIZATION OF PHTHALOCYANINE DERIVATES FOR APPLICATION IN ORGANIC PHOTOVOLATIC

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Phthalocyanines exhibit very high extinction coefficients around 700 nm, where the maximum of the solar photon flux occurs for efficient photon harvesting, and many other features that make them especially suitable for integration in light energy conversion systems[1]. Phthalocyanines also have excellent stability, a rich redox chemistry, p-type and also n-type semiconducting properties, high LUMO energy level, and relative high hole mobility[2]. Therefore, the aim of our work is to characterize the various derivatives metallophtha-locyanine for their potential applications in organic solar cells.

The general structure of our metallophthalocyanines (MPc) is shown in Figure 1. Optical and electrical properties of various derivates of MPc with different central metal atom M and miscellaneous types of substituents were studied in solutions and thin layers. Spincoating, microdisperzing printing and vacuum evaporation were used for thin layers preparation. The various type of transport layer (PEDOT:PSS, TiO_2) and electrodes were used to finalized the org. solar cells structure.

The materials and prepared thin layers structures were characterized by optical (UV-VIS, fluorescence and quenching measurement), electrical (voltage current measurement) and optoelectrical methods (transient photocontuctivity measurement). The relationship between the derivative molecular structure and their respective properties were determined. Based on these findings derivatives suitable for application in organic photovoltaic were identified and optimized.

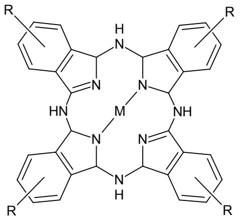

Figure 1. The general structure of metallophthalocyanines

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REFERENCES

1. Koepp, R.; Sariciftci, N. S.: Appl. Phys. Lett. *87*, 244102 (2005).
2. Claessens Ch. G., Hahn U., Torres T.: The Chem. Rec. *8*, 75 (2008).

1-P23

THE INFLUENCE OF SUBSTRATE AND PRIMER LAYER ON VARIOUS CHARACTERISTICS OF PRINTED CONDUCTIVE COMPONENTS

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Electrical properties of printed conductive layers relates to print quality and they are affected by many parameters: reological properties of ink, surface tension of ink, characteristic properties of substrate (e.g. roughness, porosity, SFE) or printing conditions (e.g. used kind of mesh, mesh tension, squezze pressure, angle deflection of squezze, edge sharpness of squezze, squezze rigidity).

Special testcharts are used for evaluation influence of printing conditions to print quality and electric characteristics. These testcharts often contain some patterns from real aplications (e.g. RFID antenna and electrode systems), but also include elements for estimating usual print quality indicators (e.g. lines of different frequencies, solid areas, objects situated toward or vertically to print direction). Printed electroconductive layers can be characterized from different point of view by several parameters (electrical, geometrical or mechanical parameters). RLC bridge can be used for measurement of electrical properties of printed layers. The image analysis can be used for determination of geometric parameters of prints. Profilometer can be used for thickness detection of printed layers. Cross hatch test is suitable for evaluation of printed layers adhesion.

This study deals about printing of conductive layers based on conductive ink by using screen printing technique. Experimental part of paper is focused on monitoring the interaction of Ag particles based conductive ink with different types of printing substrates or with primer dielectric layer, which was printed on these printing substrates first. The influence of printing substrates and primer layer by means of print quality evaluation from printed testcharts and measuring of electrical properties of selected ROIs of testchart was evaluated and compared. For evaluation were prepared sets of prints printed under same conditions on following substrates: APCO, Melinex, Pretex, Synaps with and without primer layer. Print samples were made with semiautomatic screen printing machine. Prints were investigated by image analysis methods from microscopy images. The electrical properties of printed patterns were characterized by resistance measurement as a function of printing substrate.

The results confirm, that print quality and electrical properties of printed conductive layers, are dependent on properties of printing substrates.

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1-P24

INFLUENCE OF PH ON SYSTEM CONTAINING ANIONIC POLYELECTROLYTE AND CATIONIC OR NONIONIC SURFACTANT

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The aim of this work was examination of pH influence on aggregation process and properties of aggregates in system of polyelectrolyte and surfactant. As anionicpolyelectrolyte, hyaluronan[1,2]with different molecular weights, was used. Mixtures of hyaluronan and cationic surfactant, cetyltrimetylammonium bromide (CTAB) or nonionic surfactant polyoxyethylene 20 (TWEEN 20), were studied by fluorescence spectroscopy[3], using pyrene[4,5] as fluorescent probe.

First, the influence of pH on critical micelle concentration of surfactant was studied. CMC was determined in acetate buffer (pH 4,6) and Bates and Bower buffer with $Na_2B_4O_7$–NaOH (pH 10). Results showed lower value of CMC for CTAB atlower pH and conversely. Influence of pH on TWEEN CMC was not evident. Further, mixtures of surfactantsin two concentrations (near CMC and hundredfold CMC) and hyaluronan (106 kDa and 1,36 MDa) in fixed concentration 1 $g.l^{-1}$ were prepared. Thein pH was changed from acidic to alkalic region with HCl respektive NaOH solutions. Results showed no influence of pH on properties of hydrophobic domain in aggregates polyelectrolyte-surfactant. That meansthat these systems are stable against changes in observed pH region.

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REFERENCES

1. Cowman M., Matsuoka S.:Carbohydr. Res. *340*, 791 (2005).
2. Lee Y., Spicer A.: Curr. Opin. Struc. Biol. *12*, 581 (2000).
3. De S., Girigoswami A., Das S.: J. Colloid. Interf. Sci. *285*, 562 (2005).
4. Zhou W., Zhu L.: J. Hazard. Mater. *B109*, 213 (2004).
5. Aguiar J., Carpena P., Molina-Bolívar J., Ruiz C.: J. Colloid. Interf. Sci. *258*, 116 (2003).

1-P25

DYNAMICS OF HYALURONAN WATER SOLUTIONS AS AFFECTED BY MOLECULAR SIZE

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Effect of different molecular sized hyaluronansystems on water structure was investigated by ^{1}H T_1 fast field cycling (FFC) NMR relaxometry.

FFC-NMR relaxometry probes the molecular dynamics of complex systems by measurement of longitudinal (T_1) relaxation times. In particular, the technique appears to be very sensitive to water molecules inaqueous systems, due to its ability in monitoring solute-solvent interactions. In fact, water mobility becomes slower as water is involved in H-bonds with solute molecules. For this reason homonuclear^{1}H-^{1}H dipolar interactions become stronger and faster longitudinal relaxation rates (i.e. short T_1 values) are achieved[1].

Hyaluronan (HYA) is an anionic, unbranched, non-sulfated glycosaminoglycan. It is ubiquitous as it occurs, for example, in the extracellular matrix of connective, epithelial, and neural tissues.In addition, it is also the main component of the synovial fluid which lubricates and maintains the cartilage. HYA has a unique water binding capacity. That is the reason why studies on the interactions between HYA and water are carried out in many laboratories around the world.

Previous results revealed that three different water-structural systems surround the molecule of hyaluronan in water solution[2]. In addition, backbone fluctuations were identified which allowed to recognise that the structure of hyaluronan goesfrom intra-molecular hydrogen-bonded organization to inter-molecular hydrogen-bonded structure where watermolecules can bridge carboxyl and amido groups ofadjacent saccharide units of HYA chains.

In the present study, different molecular sized HYA molecules dissolved in water were investigated. Results showed that mobility of water molecules become more restricted as HYA molecular size increases. This suggests that no bulk water is surrounds large molecular sized HYA systems. Conversely, as molecular size decreases the three hydration shells evidenced in our previous study[2] become more evident. Model free analysis approach was used.

The aforementioned results showed the great potential of FFC-NMR relaxometry in revealing water nature in polysaccharide water solutions and the possibility for future applications on complex biological systems.

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REFERENCES

1. Kimmich A., Anoardo E.:Prog.Nucl. Mag. Res. Sp. *44*, 257 (2004).
2. Průšová A., Conte P., Kučerík J., Alonzo G.: Anal. Bioanal. Chem. *397*, 3023 (2010).

1-P26

DECOMPOSITION OF VOC IN SURFACE DIELECTRIC BARRIER DISCHARGES COMBINED WITH IN SITU PHOTOCATALYZIS

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The VOCs (volatile organic compounds) are organic chemical compounds that have vapor pressures under normal conditions high enough to significantly evaporate and enter the atmosphere. They are sometimes accidentally released from the industrial processes into the environment, where they can damage soil and groundwater. Vapors of VOCs escaping into the air contribute significantly to the air pollution (e.g. they cause photochemical smog and also contribute to the global warming). In recent years, their influence on the quality of indoor environment has started to be a serious task, too. Common artificial sources of VOCs include paint thinners, wood preservatives or cleaning solvents. Regarding the medical risks, it is known that many VOCs are toxic, several of them are known as human carcinogens[1].

The problem of VOCs is one of the urgent tasks in contemporary research. Besides the classical techniques, the plasma assisted combustion methods are widely developed. The contemporary experiment was focused on the VOC decomposition in planar surface dielectric barrier discharges using alumina ceramics. The photocatalytic TiO_2 was prepared by various techniques based on ink-jet printing on inner side of discharge ceramic plates. The exhaust gas analyzis was carried out by simple gas analyzer Testo 350-XL that was used for the detection of simple low molecular weight discharge products as CO, NO_2, NO, H_2, etc. The analyses of discharge exhaust gas showed the high concentration of CO in decomposition of xylene. For measurements, the different layers of TiO_2photocatyzer were used. The higher concentrations of all low weight discharge products was observed then smoother layer was used and a strong frequency dependence of its efficiency was observed if cyclohexane was used.

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REFERENCES

1. Jones S. P.: Atmos. Environ. *33*, 4533 (1999).
2. Kim H. H., Ogata A., Futamura S.: Appl. Catal. B *79*, 356 (2008).

1-P27

CHEMICAL CROSSLINKING OF LIGNITE HUMIC ACIDS

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Humic acids (HA) are important fraction of the natural organic matter playing a significant role in environmental and ecological processes such as for example transport of various elements and compounds in environment, water holding capacity or biological activity[1].

Humic acids represent remarkable materials with great physical-chemical properties that could allow their wider application in industry, agriculture and ecology. Nevertheless some of their properties such as low mechanical strength, swellability, long wetting time and partial solubility in water can complicate some of their applications. With this respect, a suitable modification of humic acids can enhance their reactivity, thermal and chemical resistance, and sorption properties and can strongly affect their solubility[2].

The aim of this work was the modification of South Moravian lignite humic acids in order to obtain cross-linked materials with improved water retention capacity. The modification by formaldehyde was used and it is assumed that the reaction mechanism is similar to that by which phenol-formaldehyde oligomer is formed[3].

Affinity to water, kinetics of hydration and water retention capacity of obtained products were studied by thermal analysis TGA (thermogravimetric analysis) and DSC (differential scanning calorimetric). Sorption of water was carried out by exposing the sample to the specific atmosphere while water holding capacity and kinetics of hydration were assessed after addition of water directly to the sample. It was observed that all the modified samples of humic acids had higher retention capacity in comparison with the parental humic material which means that the supramolecular structure of humic acids was cross-linked and the hydrogel system was formed.

This work was supported by Ministry of Education, Youth and Sport of the Czech Republic project No. 0021630501.

REFERENCES

1. Stevenson F. J., in the book: *Humus Chemistry. Genesis, Composition, reactions*, John Wiley, New York 1994.
2. Lyubchenko V. I., Dumbai I. N., Gubanova E. N.: Khim. Tverd . Topl., *2* (1999).
3. Ryabova N., Mustafina G. A.: Zh. Prikl. Khim. (Russ. J. Appl. Chem.) *76*, 2 (2003).

1-P28

LIGHT FASTNESS OF HISTORICAL TRANSPARENCIES

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The lightfastness of transparencies was studied in this work. The study of permanence was realized on five different materials. The samples were prepared by two printing technologies: by pigment-based inkjet printing technology and by laser light exposure of silver halide photopaper followed by chemical procesing (standard process RA-4).

There are several ways to determine lightfastness of photographs or prints. The transparencies were exposed to accelerated ageing in Q-SUN test chamber. The transparencies were tested for 340 hours. They were measured by densitometer after exposure. The degradation of inks was expressed by the decrease of optical density, which was evaluated according to ISO standard 18909 in dependence on the exposure dose. Exposure needed to reach the failure criterion was recalculated to equivalent lifetime of photographs or prints[1-3].

This work was supported by MŠMT ČR, grant No. OC09069.

REFERENCES

1. ISO 18909 Photography – Processed photographic colour films and paper prints and paper prints – Methods for measuring image stability, Switzerland, 2006.
2. Wilhelm H.: *International Conference on Digital Printing Technologies,New Orleans, Sept 28 – Oct. 3 2003*, Final Program and Proceedings, p.444 (2003).
3. Feller L. R., in the book: *Accelerated agging: photochemical and thermal aspects*, chapt. Appendix A, p. 200. The J. Paul Getty Trust, Michigan 1994.

1-P29

EPR STUDY OF N-CENTERED RADICALS PRODUCED BY THE OXIDATION OF SOME MONO-, BI- AND TRIFUNCTIONAL SECONDARY AMINES

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Monofunctional (MSA), bifunctional (BSA) and trifunctional (TSA) secondary amines represent the important group of industrial antioxidants. Their action is based on the reaction of -NH- group with RO_2• radicals, which results in the formation of aminoxyl radicals. Aminoxyl radicals can also be produced by the oxidation of secondary amino group with peroxy compounds, Therefore, in monofunctional secondary amines is the generation of aminoxyls the dominating process and only in some specific cases the simultaneous attack of oxidizing agent on other groups has to be taken into account (*e.g.* HO- and NH_2- substituted diaryl amines). In the case of bifunctional secondary amines (**1**) the presence of two –NH- group in the molecule initiates the problem of the preferential attack of oxidizing agent, which can lead to two different types of aminoxyl radicals (**2**,**3**) (Figure 1)

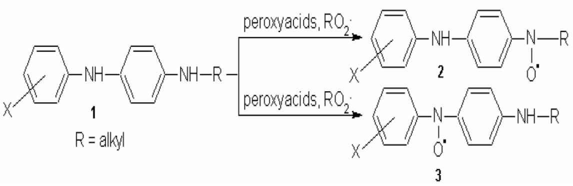

Figure 1.

EPR investigation of the series of 7 bifunctional secondary amines X-C_6H_4-NH-C_6H_4-NH-R (X, R = alkyl substituent) has shown that both types of aminoxyl radicals are in equilibrium, which position is influenced by the solvent and substitution. Under specific experimental conditions pure EPR spectra of both types of aminoxyls can be registered. The formation of corresponding aminoxyl radicals was confirmed also in the case of trifunctional secondary amines.

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1-P30
THE STUDY OF CHEMICAL REACTIONS BETWEEN NITROGEN AND METHANE BY USING ATMOSPHERIC PRESSURE DC GLOW DISCHARGE

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Recent space missions have revolutionized our knowledge of planetary atmospheres in the solar system, most notably those of Mars and Saturn moon Titan. Simultaneously, laboratory plasmas have been used to mimic the physical and chemical processes within such planetary atmospheres both to benchmark physico-chemical models and to interpret observations e.g. by providing plausible candidates for both spectral and mass spectrometric studies[1].

In this contribution we report the products formed in an atmospheric pressure glow discharge fed by a different N_2-CH_4 gas mixtures which mimics Titan's atmosphere. The discharge with Gliding Arc electrode shape with inter-electrode distance of 1 mm) was powered by a non-pulsed DC HV source. The discharge was ignited when voltage of 5500 V was applied on the electrodes then the voltage drop reached a value 400 V. Gas samples from the discharge exhaust were analyzed by GC-MS (Gas Chromatograph-Mass Spectrometer). The formation of various stable products and their dependence on the flow rate and discharge power was investigated during the experiments.

The main products formed in our atmospheric glow discharge were found to be C_2H_2, HCN, and CH_3CN. The yields of these compounds are such that HCN > C_2H_2 > CH_3CN. Minor products detected were: ethane, ethene, cyanogen, propene, propane, propyne, 1,2-propadiene, 1-butene-3-yne, 1,3-butadiene, 1,3-butadiyne, 2-propenenitrile, 2-propanenitril, 2-methylpropanenitrile, 2-methylpropane-nitrile, benzene, and toluene.

These same molecules have been detected in Titan's atmosphere[2] and the present experiments may provide some insights into the plasma chemical mechanisms prevalent in Titan's atmosphere.

This work has been supported by the Grant UK/140/2010, Slovak Research and Development Agency VEGA 1/0051/08, APVV 0365-07, ESF COST Actions CM0601, CM0805, EUROPLANET TNA2, and Czech Science Foundation, project No. 104/09/H080.

REFERENCES

1. Aplin K. L.: Surv. Geophys. *27*, 63 (2006).
2. Vinatier S., Bezard B., Fouchet T., Teanby N. A., de Kok R., Irwin P. G. J., Conrath B. J., Nixon C. A., Romani P. N., Flasar F. M., Coustenis A.: Icarus *188*, 120 (2007).

1-P31
INFLUENCE OF VARNISHES ON INKJET PRINT LIGHTFASTNESS

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Prints change upon the exposed to light is a generally known fact[1]. The UV part of electromagnetic spectrum is richer in energy, so it should induce more severe colour changes than the visible light. Protection of prints and study of protection measure is therefore needed. When varnishes are applied, they should restrict the access of pollutants and UV radiation to the prints. But not always UV absorbers are present.

This work focuses on the testing of protective measures of various varnishes on samples produced by dye-based inkjet prints. Various varnishes were applied on the samples in two different layer thicknesses and on Pyrex glass plates as well. This was done in order to determine the layer transmittance. Data obtained from the spectral measurements show which varnishes absorb in the UV part of the spectrum. The accelerated light fastness test was carried out in a xenon test chamber, where samples were exposed for 120 hours. Changes in colour were followed by the drop of optical density calculated from spectral measurements[2,3].

This work was supported by MŠMT ČR, grant No. OC09069.

REFERENCES
1. Panák J., Čeppan M., Dvonka V.: *Polygrafické minimum*, Bratislava (2008).
2. ASTM International Standard Practice for Determining Relative Lightfastness of Ink Jet Prints Exposed to Windows Filtered Daylight Using a Xenon Arc Light Apparatus; Designation: F 2366 – 05; USA, 2008.
3. ISO 18909 *Photography – Processed photographic colour films and paper prints and paper prints – Methods for measuring image stability*, 2006, Switzerland.

1-P32

FREE RADICALS IN THE NAILS AFFECTED BY FUNGI MYCOSIS (ONYCHOMYCOSIS)

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Fungus of the nails, onychomycosis, is action of microorganisms on the nail bed and nail. We studied fungal disease of the nails in order to detect and identify the generated free radicals, if they are present in the affected nails. Free radicals were measured by EPR spectroscopy. The EPR spectrometer was from Bruker Biospin Elexsys, type E-540. Recording and evaluation of spectral parameters was carried out through programmes of Bruker (Linux) and graphic programme Origin. The measurement was carried out at room temperature. Samples of the nails from inches were left leg (struck nail) and right hand man (healthy nail). The nails samples (oldest part of the nails, strips of nails at the end of the nail beds) were placed in a quartz tubes. Measurements were performed in the time intervals, to find out the time course changes of free radicals concentrations.

We found that the affected nails contain free radicals, whose concentration varies depending on certain factors. Our results could be applied in the treatment of nail out fungi. Our idea is to find relations between nail mycosis of human nails and the presence of free radicals.

We have found that the affected nails showing significant EPR signal. This signal virtually disappear after 14 days, free radicals are no longer present. In healthy nails no free radicals were detected. Affected nails have been treated by drug “Terfimed”.

Monitoring of free radicals in the samples of the nails and changes in their concentrations might i) speed up and clarify the diagnosis of nail mycoses, ii) monitor the progress of the treatment of this objectively relatively rapid method and estimate prognosis treatment more precisely, iii) contribute to the acquisition of additional knowledge about this disease. Another measurement of free radicals may determine more accurately the context and the relationship between nail mycosis and skin mycosis.

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REFERENCES:
1. Plonka P. M.: Exper. Dermatol. *18*, 472 (2009).
2. Halliwell B., Gutteridge J. M. C., in the book: *Free Radicals in Biology and Medicine*, chapt. 2, p.48. Oxford Univ. Press, New York 2000.

1P-33

SYNTHESIS, SPECTROSCOPIC AND THERMODYNAMIC STUDIES OF THE ADDUCTS OF FIRST ROW TRANSITION METAL(II) BROMIDES WITH 1,2,3,4-TETRAHYDROQUINOLINE

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Quinoline and its derivatives are known to form complexes with transition metal(II) halides[1-2]. Thermodynamic studies related to the transition metal(II)-nitrogen coordinated bonds are not found in literature. The present work is concerned with the synthesis, thermal studies and calorimetric determination of thermochemical parameters of the adducts formed by metal(II) bromides with the ligand 1,2,3,4-Tetrahydroquinoline.

The adducts $MBr_2.nL$ where (M is *Zn, Cu, Ni, Co or Fe; L* is; 1,2,3,4-*Tetrahydroquinoline*(*THQ*); n=2, 3, 3/2 or 3/4) were synthesized and characterized by elemental analysis, melting points, IR, UV and thermal analysis. Thermochemical parameters involved in the formation of adducts were determined by solution-reaction calorimetry.

The analytical data is in close agreement with the proposed stoichometry of the adducts. The yields range from 22 to 60%. The pattern of the IR spectra of the adducts is similar to that of the ligands, although slight dislocation, splitting and variation in some band intensity is observed, indicating the bonding of ligands to the central metal ion[3-4]. The ligand field parameters for cobalt adduct have been calculated according to Lever[5]. Considering the number and position of the bands[6-7] and according with the magnitude of the crystal field parameters as compared with that of Bolster[6], it is concluded that two nitrogen atoms from two ligand molecules and two bromides ion pseudo-tetrahedrally surround *Co*(II). The ligand field parameters for the *Ni*(II) demonstrate the same results as for cobalt having pseudo-tetrahedral geometry but one of the bromide ions is attached to other *Ni*(II) in dimeric structure. For the cu*(II)* adduct, the electronic spectra shows a rather broad assymetrical band with maxima at 10528 cm^{-1}. Its intensity and position corresponds observe for pseudo-octahedral compounds. The adduct of *Fe*(II) has pseudo-octahedral symmetry. The TG/DTG data shows the loss of the ligands in several steps of mass loss followed by the loss of bromine or complete loss of the ligand and part of the bromine. All the adducts left a residue which is part of the metals or metal and bromide. DSC curves show few endothermic peaks due to melting and decomposition of the adducts, consistent with the TGA data.

The standard enthalpies of Lewis acid/base reaction ($_{r}H^{0}$) of the adducts were determined using solution reaction calorimetry. The standard enthalpies of formation ($\Delta_f H^\theta$), the standard enthalpies of decomposition($\Delta_D H^\theta$), the standard lattice enthalpies ($\Delta_M H^\theta$) and the standard enthalpies of Lewis acid/base reactions in gaseous phase ($\Delta_r H^\theta_{(g)}$) were calculated by several thermochemical cycles. The standard enthalpies of M-N bonds, being equal to $D_{(M-N)} = - \Delta_r H^\theta_{(g)}/n$. Based on the values for $\Delta_r H^\theta$ the acidity order of the salts for the adducts of the same stochiometry obtained is: $CoBr_2 > CuBr_2 > ZnBr_2$. Using the $D_{(M-N)}$ values the acidity order is $CoBr_2 > ZnBr_2 > CuBr_2$.

REFERENCES:

1. Garriba E., Micera G., Sanna D., Lodyga-Chruscinska E.: Inorg. Chim. Acta *348*, 97 (2003).
2. Dunstan P. O.: Thermochim. Acta *468*, 21 (2008).
3. Dunstan P. O: J. Chem. Eng. Data *55*, 4995 (2010).
4. da Silva Jr. U. G., de Oliveira O. A., de Farias R. F., Thermochim. Acta *450*, 2 (2006).
5. Lever A. B. P., J. Chem. Educ. *45*, 711 (1968).
6. West D. X. Severen J. C., Transit. Met. Chem. *13*, 45 (1968).
7. Brown D. H., Kenyon D., Sharp D. W., J. Chem. Soc. A *14*, 1474 (1969).

2-IL

TiO_2 THICK FILMS SUPPORTED ON RETICULATED MACROPOROUS Al_2O_3 FOAMS AND THEIR PHOTOACALALYTIC ACTIVITY IN WATER PURIFICATION

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In recent time much interest has been devoted to studies of photocatalytic reactions, which can be used for purification of aqueous or atmospheric environment. The development of eco-friendly methods for complete destroying of organic pollutants became an imperative task. The research has been centered mainly on advanced oxidation processes for the destruction of organic species. Titanium dioxide is the most commonly used semiconductor photocatalyst since it exhibits high effectivity and stability together with low toxicity and adequate price[1]. The main disadvantage of using suspensions of nanopowders, which frequently show high photoactivity, is that the recycling of the nanocrystalline catalyst from the treated water is cumbersome and uneconomical, especially due to waste water. Therefore it is highly advantageous to immobilize the nanocrystalline TiO_2 powder catalyst on an appropriate support. Structured catalysts and reactors are gaining increasing attention since in practical applications often a shaping of the catalyst is required. Reticulated macroporous ceramic foams are well established for industrial applications as structured catalyst supports since they exhibit a high porosity and low flow resistance. The open three dimensional reticulated structure of macroporous ceramic foams is capable of homogenizing non-uniform flows as well the concentration of species in the flow and is well suited for substrate of photocatalytic layers[2,3].

This contribution is devoted to studies on TiO_2 thick films deposited on ceramic reticulated foam substrates. Commercial Aeroxide® P25 nanopowder, TiO_2 powders synthesized by coprecipitation[4] and TiO_2 powders doped with Ce(IV) and Zr(IV) were deposited on macroporous foams by dip-coating from suspensions of powders and subsequently annealed up to 750°C. Two sorts of alumina foams were employed to support photocatalyst coatings - foams prepared in laboratory by the replica method with pore density of 10 *ppi* and 15 *ppi* (pores per inch) and commercial foam ceramics (Vukopor® A) with pore density of 15 *ppi*. These two types of supports differ mainly in their microstructure. The commercial foams have thicker struts and their surface is fairly rough in comparison with the rather smooth surface of the laboratory prepared supports. The photochemical mineralization of aqueous phenol solutions catalyzed by TiO_2 films was investigated by determination of total organic carbon (TOC) under UVA light emitting lamp as a source of irradiation.

Immobilization of titania powder precursors on cera-mic foams leads to thick films with sufficiently good stability and adhesion of the catalyst to the substrate. The micro-structure of titania coatings is highly influenced by the surface morphology of the support and the size of particles in suspensions used for deposition. Incompact and highly porous thick films with variable thickness up to 10 μm were formed using less homogeneous suspensions containing agglomerates of the precipitated titania catalyst. These coatings are highly cracked but no significant influence of the cracks on coating stability was observed. Prolonged homogenization changes the particle size distribution in suspensions considerably. More stable suspensions contain smaller particles and the coatings they form are more compact. The thickness of these coatings varies in the range of 1 μm - 20 μm. From Aeroxide® P25 nanopowder, which forms highly homogeneous suspensions with small particle size, coherent porous almost crackless thick films were prepared. The more rough commercial supports are coated with slightly thinner films than the laboratory made substrates.

In the investigations of photocatalytic activity linear TOC decrease with reaction time was observed - under applied conditions the phenol photomineralization obeys the zero order kinetic law. The best photocatalytic activity is found for films prepared from Aeroxide® P25 and annealed at 600 °C. The photoactivity of precipitated titania coatings is lower in comparison with Aeroxide® P25 films. The difference in activities of these catalysts originates most likely in higher photoactivity of Aeroxide® P25 nanopowder and also in the higher amount of deposited titania in comparison with coatings prepared from precipitated titania. The thinner titania coatings supported on commercial foam show slightly lower photoactivity comparing with the thicker films on substrates prepared by replica technique.

Cerium(IV) doped TiO_2 films show comparable or lower photocatalytic activity than the activity of undoped titania coatings. On the other hand photomineralization rate constants obtained for the Zr(IV)/TiO_2 thick films show higher values than the rate constants calculated for immobilized non-doped titania.

Macroporous ceramic foam supported titania thick films prepared by immobilization of powder precursors are effective photocatalysts in aqueous solutions. They can even completely photomineralize organic compounds, e.g. phenol. The photocatalytic activity of the ceramic foam supported coatings is well comparable to the photoactivity of precursor powder suspension with the same mass of titania. Photo-catalytic coatings show about 75 % of the photocatalytic acti-vity of corresponding powder in suspension. Since the repeated use of the foam supported titania thick films does not influence remarkably the photocatalytic activity, these structu-red photocatalysts represent highly promising materials for environmental applications.

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REFERENCES

1. Fujishima A., Zhang X., Tryk D. A.: Surf. Sci. Rep. *63*, 515 (2008).
2. Rodriguez P., Meille V., Pallier S., Al Sawah M. A.: Appl. Catal. A *360*, 154 (2009).
3. Plesch G., Gorbár M., Vogt U. F., Jesenák K., Vargová M.: Mater. Let. *63*, 461 (2009).
4. Bakardijeva S., Šubrt J., Štengl V., Dianez M. J., Sayagues M. J.: Appl. Catal. B *58*, 193 (2005).

2-L1

DISTRIBUTED PARAMETER MODEL OF THE 2-ETHYL-HEXENAL HYDROGENATION PROCESS

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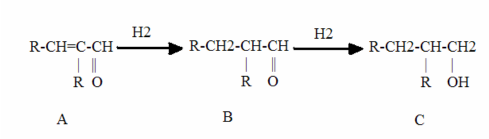
The industrial synthesis of 2-ethyl-hexanol (plasticizer alcohol) is based on the hydrogenation process of 2-ethyl-hexenal in a catalytic trickle bed three-phase hydrogenation reactor. At present time, 2-ethyl-hexanol and n-butanol represent around 70% of the global consumption of plasticizers alcohols. The hydrogenation process can occur in liquid phase and in gas phase. This paper will focus on the liquid phase hydrogenation process preffered in chemical industries, due to it's advantages. The reaction pathway is presented in scheme 1:



Scheme 1. Reaction pathway

where A= 2-ethyl-hexenal (reactant), B= 2-ethyl-hexanal (intermediate product), and C= 2 ethyl-hexanol (final product).

A dynamic model of the hydrogenation process can be used to evaluate the operational challenges and to understand the processes developed in the reactor. The actual literature reported some kinetic studies[1] of the hydrogenation reaction but no mathematical model of the process is given.

The distributed parameters model developed in this paper is based on the mass and energy conservation principles. It consists of balance equations for gas and liquid phases. Reaction rate equations[2], transport models and mass balances are coupled to generalized heterogeneous models which were solved with respect to time and space with algorithms suitable for partial differential equations. The importance of mass transfer resistance inside the catalyst pellets as well as the dynamics of the different phases being present in the reactor is presented.

The basic assumptions about the hydrogenation reaction are: i) model parameters are constant in the radial cross section of the reactor (piston-type flow); ii) heat transfer by conduction and radiation are neglected in the axial direction; iii) both gas and liquid velocities are taken constant; iv) the hydrogenation reactor is adiabatic operated; v) both phases are considered ideal mixtures; vi) the chemical reaction takes place only on the catalyst surface.

Model validation is the core of the identification problem because it makes possible to evaluate the model quality. For model validation eight different cases were chosen to compare simulation results with plant data acquired from functional hydrogenation reactor at S.C. Oltchim S.A, Ramnicu Valcea (Romania). These cases cover a specific domain of reactor loading. The comparison of simulation results with plant data indicates a good prediction.

A dynamic behavior evaluation study of the developed model was also performed considering three different scenarios. From the dynamic point of view, the system behaves as an element with a large time constant and a large time delay.

Hydrogenation multiphase catalytic reactors have complex behavior due to heat, mass and kinetic interactions, which are dependent upon the operating conditions, as well as on the trajectory from one state to another. From this point of view, the use of advanced control strategies together with on-line optimization techniques appears to be a suitable procedure to deal with the problem of operating at high level of performance and safety and respresent a future task.

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REFERENCES

1. Smelder G.: Canadian J. of Chem. Eng. *67*, 51 (1989).
2. Silva J. D., Lima F. R. A., Abreu C. A. M., Knoechelmann A.: Braz. J. Chem. Eng. *20*, 375 (2003).

2-L2

SOL-GEL PHASE TRANSITIONS OF TEMPERATURE-SENSITIVE BIODEGRADABLE COPOLYMERS

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Temperature-sensitive polymers based on hydrophilic poly(ethylene glycol) (PEG) and hydrophobic poly[(lactic acid)-*co*-(glycolic acid)] (PLGA) copolymer are being investigated extensively for medical applications (e.g. injectable polymer drug delivery systems, tissue implants and resorbable adhesives) because of their nontoxicity, biocompa-tibility and bioresorbability.

The PLGA-PEG-PLGA triblock copolymers with PLGA/PEG weight ratio equal to 1,5; 2,0; 2,5; 3,0 and LA/GA molar ratio equal to 2,4; 3,0 and 3,4 were prepared via ring opening polymerization (ROP). Prepared copolymers were subsequently modified in "one pot" by itaconic anhydride (ITA) in order to functionalize both ends with carboxylic acid groups and reactive double bonds. Chemical structure was characterized by means of gel permeation chromatography and nuclear magnetic resonance.

These copolymers are soluble in water forming free-flowing solution (sol) that spontaneously gels as the temperature increases generating a water-insoluble physical hydrogel. Sol-gel phase transitions of both modified and unmodified triblock copolymers were studied by test tube inverting method. It was found that PLGA/PEG ratio affects the critical gel temperature (CGT), whereas LA/GA ratio has influence on the critical gel temperature (CGT) of copolymers. Both unmodified and modified copolymers with PLGA/PEG weight ratio equal to 3,0 were water insoluble due to the high hydrophobicity. When PLGA/PEG ratio was reduced to 2,0 the copolymers displayed clear gel at temperature from about 31 °C to 40 °C followed by the white gel up to the approx. 50 °C. Farther reducing PLGA/PEG ratio increased the CGT. When the PLGA/PEG weight ratio is lower than 1,5; copolymers are too hydrophilic to form gel or the CGT is very high (more than 45 °C). The PLGA-PEG-PLGA and ITA/PLGA-PEG-PLGA/ITA copolymers with the same PLGA/PEG ratios display similar CGT. However, ITA functionalization improved sol-gel characteristics of original PLGA-PEG-PLGA copolymer by approaching gel phase to body temperature.

As a result, aqueous solution of ITA/PLGA-PEG-PLGA/ITA having LA/GA = 3,0 and PLGA/PEG = 2,0 with concentration higher than 6 wt% might be suitable material for biomedical applications as injectable temporary implants.

This work was supported by the Czech Ministry of Education, Youth and Sports under the research project no. MSM 0021630501 and the program NPV II 2B06130.

2-L3

TOWARDS THE DEVELOPMENT OF NEW MATERIALS BASED ON HYALURONIC ACID

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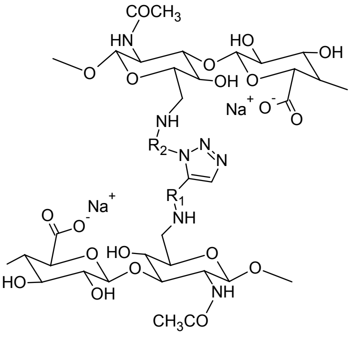
The use of hyaluronic acid (HA) in pharmaceutical compositions has demonstrated enhanced effectiveness in the prevention or treatment of connective tissue damage. However, this polysaccharide degradates enzymatically in the body. The use of chemically modified and crosslinked derivatives of hyaluronic acid increases the stability of the implant. Therefore, new suitable crosslinking reactions which employ mild conditions and which are compatible with sensitive biomolecules should be developed. In this work, HA was chemically modified using a process of oxidation and reductive amination with primary amines containing either a terminal azido or alkynyl terminal group. The crosslinking reaction of these derivatives via click chemistry[1], led to the formation of insoluble networks of type (I).



(I)

The values of diffusion exponent (n) according to Korsmeyer-Peppas equation were calculated for evaluation of release mechanism. Therefore, it was discovered that transport into the network was dominated by swelling and the delivery of model drugs was controlled due to pH and ionic strenght of the environment. Thermal analyses of the modified polysaccharide as well as the crosslinked materials were evaluated in order to find out possible structural changes after chemical modification. We noticed that thermal stability of the derivatives had effectively increased with the partial modification. Incorporated secondary amines within the network had provided stronger interactions. As a result, stability increased after crosslinking. Moreover, the introduction of pH-sensitive groups into the network allow tuning of properties at the molecular level and a reversible switching from shrink to swollen state as a response to changes of pH.

The properties of the crosslinked material can be modified varying experimental parameters such as molecular weight of hyaluronan, degree of substitution and gelation time.

According to scanning electronic microscopy (SEM), the materials prepared in this fashion shows good wall interconnectivity and porosity. Furthermore, the materials were tested as not citotoxic.

The study of those properties has allowed us to believe that the material may be potentially used for drug delivery applications[2], as a matrix for biosensors or as permeation switches[3].

This work was supported by TIP FR-TI1/150 from the Ministry of industry and trade of the Czech Republic.

REFERENCES

1. Rostovtsev V. V., Green L. G., Fokin, V. V., Sharpless K. B.: Angew. Chem. Int. Edit. *41*, 2596 (2002).
2. Qiu Y., Park K.: Adv. Drug Deliver. Rev. *53*, 321 (2001).
3. Hoffman A. S.: Artif. Organs *19*, 458 (1995).

2-L4

POLYMORPHISM OF 3,6-BIS-(4'-PYRIDYL)-2,5-DIHYDRO-PYRROLO[3,4-C]PYRROLE-1,4-DIONE , MATERIAL FOR HYDROGEN SENSOR

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The 3,6-Diphenyl-2,5-dihydro-pyrrolo[3,4-c]pyrrole-1,4-dione (BPPB) and its analogues diketo-pyrrolo-pyrroles (DPPs) are industrially important organic pigments[1,2]. By the substitution of the phenyl group by the pyridyl group we get 3,6-bis-(4'-Pyridyl)-2,5-dihydro-Pyrrolo[3,4-C]Pyrrole-1,4-dione (4PyPP4Py). This brings affinity to protons at the pyridyl nitrogen, firmer crystalline structure due to intermolecular hydrogen bonds, and change of the molecular dipole momentum and conductivity inversion into n-type[3]. The sensing layers prepared by vacuum evaporation and from the solution of so called latent pigment (Figure 1) were studied with respect to polymorphism and compared with the native and purified powder.

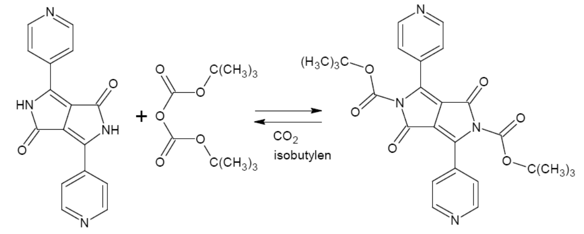

Figure 1: Transition of the 4PyPP4Py to its latent soluble form.

Native as synthesized substance contained a lot of impurities, which can be removed by conditioning (boiling in dimethylformamide). It achieves a bright red colour. Its purity was verified by the thermogravimetric analysis as well as the resistance to the decomposition. Its IR spectrum shows a movement of dominant peak to 1649 cm^{-1} of oxygen stretching oscillations where situated only a small shoulder before (Figure 2). The evaporated from native substance thin film shows the same peak position although a wide shoulder occurred on site of bigger wavenumber (1674 cm^{-1}). It was concluded that this peak is not a new chemical bond but it is a shift as a result of intermolecular interactions due to crystallographic arrangement and creating new intermolecular hydrogen bridge bonds. In compliance with Mizuguchi et al.[4] and Lunak et al.[5] it is anticipated that there exist two phases differing by bonding of pyridyl nitrogen to pyrrolinone oxygen of the next molecule. These bonds are absent in phase I where the intermolecular interactions are in between lactam groups while pyridyl nitrogen is free and able to attract a proton. In phase II the pyridyl nitrogen atoms are saturated by the hydrogen bond to the next molecule lactam.

Phase II is present in native substance. It is transferred to the phase I by conditioning or condensation from vapours at evaporation. Annealing converts it back again to phase II, as it is apparent from IR spectra of by step annealed latent pigment or evaporated layer at temperatures above 170 °C. The effect was confirmed by the x-ray structural analysis.

The hydrogen sensor based on 4PyPP4Py thin film with catalytic Pd interlayer for hydrogen dissociation was fabricated and tested. Evaporated layer of phase I is able to bond hydrogen atom produced by Pd catalytic dissociation. Its electron is attracted to pyrrolinon oxygen and can contribute to n-type conductivity increase (confirmed by field effect conductivity of the layer) by orders from typical $1\cdot10^{-12}\,\Sigma^{-1}\text{cm}^{-1}$ to $1\cdot10^{-6}\,\Sigma^{-1}\text{cm}^{-1}$ in 100 % hydrogen.

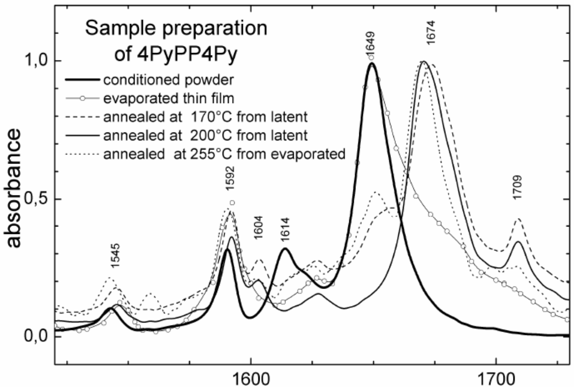

Figure 2. Detail of the IR spectra development of various sample forms and treatment effect.

This work has been supported by the Czech Science Foundation in the projects GACR 203/08/1594.

REFERENCES

1. Iqbal, A., Cassar, L.: EP 98808, Ciba-Geigy, 1982.
2. Morton, C., Macdonald Smith, D., Kabal, A.: Patent No.: US 7,041,235 B2, May 9, 2006.
3. Takahashi H., Mizuguchi J.: J. Electrochem. Soc., *152*, H69 (2005).
4. Mizuguchi J., Imoda T., Takahashi H.: Acta Crystallogr. *E61*, 500, (2005).
5. Luňák S., Vyňuchal J., Horáčková P., Frumarová B., Žák Z., Kučerík J., Salyk O.: J. Mol. Struct. *983*, 39 (2010).

2-L5

CHANGES OF MORPHOLOGY OF COPOLYMER POWDER PP-IC AT 210°C AND AT VARIOUS ANNEALING TIMES

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Copolymer powder PP-IC is prepared in two step process. Homopolymer matrix with high number of pores[1] is prepared in the first step. Copolymer prepared in the second step fills the pores[1] and surface of the homopolymer powder.

The aim of this work is to state the morphology of PP-IC powder at constant temperature and at various annealing times.

Typic copolymer powder with 15 vol. % of EPR was chosen (M_n = 69 500 g.mol^{-1}; MFR = 3,5 g/10 min). Samples were prepared at 210°C and with annealing times 2, 20 and 200 minutes in closed iron chamber after annealing was cooled down to laboratory temperature and then was conditionated for 24 hrs at 23°C. Middle part of the sample was used for AFM, Figure1, and SEM microscopy, Figure 2.

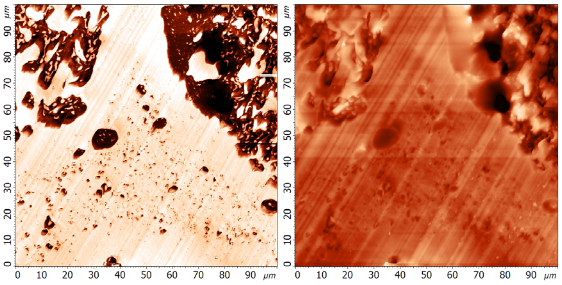

Figure 1. AFM pictures of sample annealed at 210°C for 200 minutes. On the left there is picture with phase contrast, on the right side there is topography picture. The EPR phase is dark on the left pisture and on the right picture is bright. Space among the particles and border parts of particles are well seen on these two pictures.

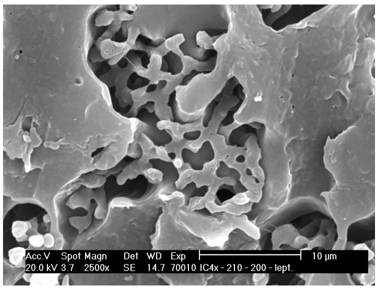

Figure 2. SEM scan of sample with diluted copolymer phase. Great holes that were created by coalescence of copolymer (EPR) phase were created in the particles after 200 minutes of annealing at 210°C.

Annealing influences morphology of PP-IC copolymer and join together EPR domains in the particles. The dimensions of domains increase with increasing annealing time.

This study was attached to changes of homopolymer and copolymer phases. A part of homopolymer phase flow out from the particles and create compact sample. The rest of the homopolymer rest inside of the particle and create the relative fixed spatial arragement of the particle. It preserves the shape and dimensions of the particle even at long annealing times. Copolymer agglomerates[1] inside of the particles.

Authors are thankful to Ministry of Industry and Trade of Czech Republic for financial support, grant FR-TI1/272.

REFERENCES

1. Yong, Ch., Ye, Ch., Wei, Ch., Decai, Y.: Eur. Polym. J. *43*, 2999 (2007).

2-L6

OPTIMIZATION OF CROSS METATHESIS REACTION OF OLEOCHEMICALS WITH ETHYL ACRYLATE

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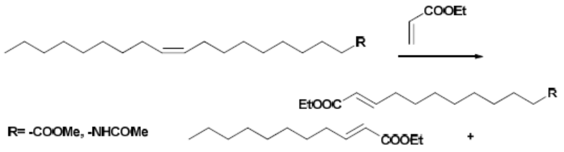
In the last few years olefin metathesis of renewable resources has gained much attention for the synthesis of numerable products in Organic and Polymer Chemistry.[1] In particular the cross-metathesis with acrylates is preferred over ethenolysis due to ease of reaction and for synthesis of new α-ω-bifunctional products.[2] Previously the solvent free reaction conditions using Hoveyda type 2nd generation initiators have been used for this type of reactions.[3] Furthermore, optimized reaction conditions using series of initiators has been reported by our group for the cross metathesis of terminal olefins with ethyl acrylate.[4] So the scope of this investigation is expanded to the valued raw materials from renewable resources especially for cross metatheis of oleylamine.



Scheme 1. Cross-metathesis of oleochemicals with ethyl acrylate

In the latter case proper protective groups provide entry into metathesis reactions.[5] Various protecting groups are tried for this purpose and high yields were only obtained with the acetyl group. Environmentally benign solvent free reaction conditions with commercially available Hoveyda 2nd generation catalyst, more economical **M51** and with ester type *cis-*

dichloro catalysts[6] such as **1** in the conversion of oleochemicals will be presented.

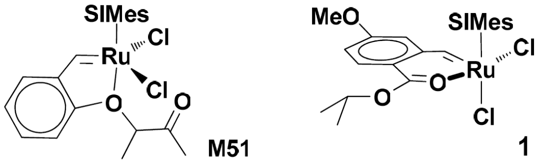


Fig. 1. Initiators used for cross-metathesis

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REFERENCES

1. Biermann, U., Bornscheuer, U., Meier, M. A. R., Metzger, J. O., Schäfer, H. J.: Angew. Chem. *123*, 3938 (2011).
2. Rybak, A., Fokouand, P. A., Meier, M. A. R.: Eur. J. Lipid Sci.Technol. *110*, 797 (2008).
3. Rybak A., Meier M. A. R.: Green Chem. *10,* 1099 (2008).
4. Abbas, M., Slugovc C.: Tetrahedron Lett. *52*, 2560 (2011).
5. Nussbaumer P., Ettmayer P., Peters, C., Rosenbeiger D., Hogenauer K.: Chem. Commun. *41*, 5086 (2005).
6. Zirngast M., Pump E., Leitgeb A., Albering J. H., Slugovc C.: Chem. Commun. *47*, 2261 (2011).

2-L7

DEVELOPMENT AND CHARACTERIZATION OF NOVEL SOLAR CELLS FOR ORGANIC PHOTOVOLTAICS

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Organic solar cells represent an interesting alternative to current photovoltaic sources. Although it is assumed that crystalline silicon will dominate the market for at least 5 years, nowadays the first commercial installations of organic photovoltaics appear. Several industries such as Konarka Technologies, Heliatek, Plextronics, SCHOTT Solar AG, Merck KgaA, Greatcells and others now intensivelly work on development and manufacture of organic solar cells and modules. At the Faculty of chemistry BUT we focus on the development and characterization of new materials for organic photovoltaics and development of new organic photovoltaic structures such as textile solar cells.

New materials based on diketopyrrolopyrrole and phthalocyanine derivatives were synthetized and applied in organic photovoltaics. As a reference, pi-conjugated polymers based on polythiophene and polyphenylenevinylene were used. The optical and electrical properties of new materials were studied in details. The special attention was also paid to the film-forming properties of the materials and to the morphology of the prepared thin films. The thermal, thermo-oxidative and light stability of the materials and prepared multilayered systems was characterized. The results show that low molecular weight materials (especially some of the DPP derivatives) exhibit significantly higher stability than polymeric one. On the contrary, polymeric materials exhibit better film-forming properties and more stable morphology of bulk heterojunctions.

Based on these results, organic solar cells with planar and bulk heterojunctions were prepared, both in the standard and inverse structure. The above mentioned polymer materials were also used for preparation of textile solar cells, various strategies for their preparation will be presented in the contribution. In both cases the aim was to prepare the cells without the use of high-tech procedures (such as vacuum technology, clean room, boxes with an inert atmosphere), to be as much as possible closer to industrial conditions of production. Therefore the solar cells were prepared on flexible PET substrates and printing methods were used for the deposition of the whole photovoltaic structures. Results and cell efficiency were compared with cells that are prepared by standard procedures. The overall efficiency of the prepared structures do not exceed 2%, however, they demonstrate the possibility of preparing large-scale photovoltaic structures using inexpensive technological processes allowing a low-cost high-volume commercial production.

This work was supported by the FP7 project 7E09061 „Developement of Photovoltaic textiles based on novel fibres“, project MPO TIP FR-TI1/144 "Multicomponent electronic systems based on organic materials", project No. P205/10/2280 from Czech Scince Foundation, project "Centre for Materials Research at FCH BUT" No. CZ.1.05/2.1.00/01.0012. supported by ERDF and IGA Brno University of Technology via project FCH/FEKT-S-11-2.

2-L8

STRUCTURE ANALYSIS OF NANOMATERIALS ON A LABORATORY SAXS SYSTEM

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The Small-and Wide Angle X-ray Scattering (SAXS) System "SAXSess mc^2" is a measuring instrument for advanced nanotechnology laboratories.

It is used to study the size, shape and internal structure of macromolecules, the structure of surfaces as well as the surface-to-volume ratio (specific) and the crystalinity of nanocomposites presented in many substances ranging from polymers to biomaterials.

In the standard SAXS configuration (transmission mode), the SAXSess mc^2 acquires scattering data continuously from 0° to 40° scattering angle.

Surface studies (reflection mode) can be performed with the highest precision available in a lab (GISAXS, XRR).

In the given lecture, some of the many applications of this laboratory instrument are demonstrated. Experimental results from a selection of dispersions and polymer systems, applications in the fields of bio-chemistry, drug-delivery systems and quality control are discussed.

2-L9

DEVELOPMENT OF SANDWICH STRUCTURE OF CO-EXTRUDED POLYPROPYLENE SHEETS

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The development of co-extruded polypropylene (PP) sheets was focused to decrease their weight and to preserve their high stiffness, good strength and sufficient toughness as well. To maintain these properties, the high molecular weight impact copolymer PP (Mosten EH501, UNIPETROL, CR) was used.

It is known that the most effective way how to decrease the specific weight of a sheet is to foam the polypropylene melt during the processing. Two technological ways of foaming are discussed in this contribution, namely the chemical one based on a decomposition of inorganic salts and the physical one based on an evaporating of hydrocarbons encapsulated into some soft microcells or a combination of both, respectively. The degree of foaming Φ up to 30 % was reached. A typical structure of such foamed co-extruded PP sheet is given in Fig. 1.

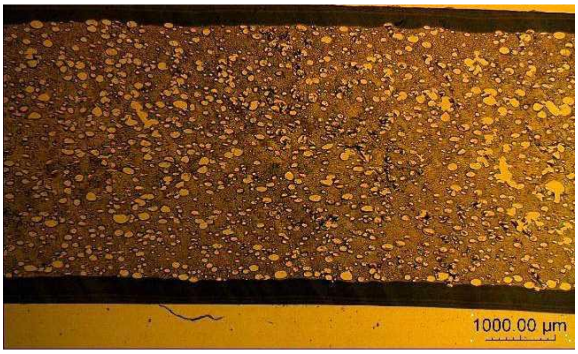

Figure 1. An example of foamed PP structure (microtome slice).

The modulus E_F (stiffness) of the foamed sheet decreases with respect to the PP according to the equation 1 [1]:

$$E_F = E_{PP} \cdot \frac{1-\Phi}{1+\Phi\cdot(q-1)} \qquad (1)$$

where E_{PP} is the modulus of PP and q is a geometrical factor equals nearly 2.

To compensate the decrease of stiffness of the foamed sheet, especially in bending mode, the sandwich structure A-B-A of sheet was taken into account [2]. It was shown that the stiffness of PP for a sandwich with layers A made from standard filled PP (fillers: talcum, kaolin) can be reached for cases when relative thickness of outer layers A is greater then 0.1 with respect to the thickness of the whole sheet.

Some filled polypropylenes were prepared and properties of thin extruded sheets (thickness about 1 mm) were tested (modulus of elasticity, tensile strength, toughness). It was concluded that it is necessary to use impact PP copolymer as a matrix and that the optimal filler mass fraction is in the range about 20 %. The modulus of the materials was high enough and the rheological properties were satisfactory for sandwich production.

Mean diameter and the dispersion of dimensions of bubbles are very important for foamed materials. The influence of these was computed (FEM). The results show that the increase of stress concentration depends on the finite ratio of the diameter of bubbles and sheet thickness, i.e. for larger bubbles decreases the stiffness and also the strength of sheet (Fig. 2).

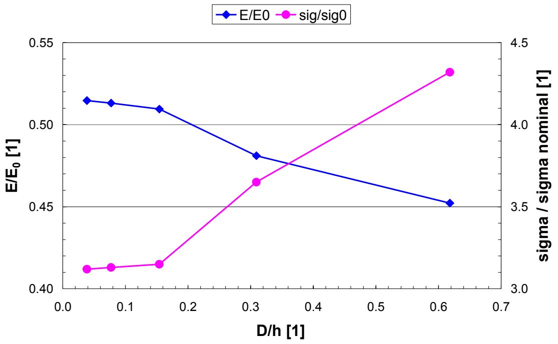

Figure 2. Dependences of modulus of elasticity and stress concentration on the ratio of the diameter of bubbles and the sheet thickness.

The properties of sheet sandwiches with foamed PP core layer were tested. The toughness was determined by the dart test (CEAST DARTVIS equipment), the strength was tested by standard tensile test (INSTRON 4466 tester) and stiffness was evaluated as the effective modulus at tensile and bending tests (INSTRON 4302 tester). The experimental results confirmed relatively well the theoretical relations.

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REFERENCES

1. Nielsen L. E., in the book: *Mechanical Properties of Polymers and Composites*, p. 226 and 242. Chimija, Moskva 1978.
2. Beer F. P., Johnston E. R. Jr., in the book: *Mechanics of Materials*, chapt. 4.7., p. 204. McGraw-Hill, Inc., 2nd edition, New York 1992.

2-L10

EVALUATION OF MECHANICAL PROPERTIES OF PLASMA POLYMER FILMS USING CONVENTIONAL AND CYCLIC NANOINDENTATION TECHNIQUES

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Plasma polymer films are important materials with applications in many areas including electronics, magnetic media, optical devices and protective layers of engineering components[1]. In such developments, good understanding of mechanical properties is essential[2]. Among other techniques available, nanoindentation is very suitable method for determining mechanical properties of thin films, such as elastic modulus, hardness or stiffness, at submicron levels.

In the conventional approach to nanoindentation method, measuring cycle consists of loading segment followed by dwell time (hold time) at peak load, and unloading segment at last. Loading causes both elastic and plastic deformation, while during unloading, mostly recovery of elastic deformation occurs. In nanoindentation experiments, variation of loading rate, hold time or strain rate can modify the time dependent deformation of material depending on its visco-elastic or elasto-plastic properties[2]. Enhancement of this classical approach just described above is cyclic nanoindentation, where the sample is loaded to higher loads immediately after previous cycle was performed. This can be used quite effectively when depth profile of mechanical properties (modulus/hardness in our case) needs to be investigated. The reloading path should not overlap with previous cycle, thus resulting in hysteresis loops[2]. This technique was previously used by some researchers for study of phase transitions of heterogeneous structures of materials[3].

In the study here presented, we investigated the influence of experimental parameters, namely hold time, loading rate and applied load during conventional nanoindentation experiment and we also used cyclic nanoindentation tests to construct depth profiles of samples prepared at different powers of plasma discharge. We were able to compare classical and cyclic nanoindentation in order to demonstrate, how the drift rate and substrate affect the measured values of elastic modulus and hardness. Values of reduced modulus and hardness were evaluated from upper portion of the unloading segment by fitting it to a power law according to Oliver-Pharr method.

For the purpose of our tests, samples of plasma polymer films were prepared on squared silicon wafers under steady-state conditions by plasma-enhanced chemical vapor deposition (PECVD) working in pulse mode. The monomer used was tetravinylsilane (TVS). The thickness of films was determined by in-situ spectroscopic ellipsometry after deposition process. At last, for investigation of mechanical proper-ties, nanoindentation head equipped with hard diamond tip combined with scanning probe microscope was used.

The study found an influence of dwell time and loading/unloading times greater than 5 s, producing progressive increase of tip displacement due to the system drift not correctly subtracted at prolonged measurement times[2]. As a consequence of this fact, values of elastic modulus and hardness were underestimated.

From the depth profiles obtained by both conventional and cyclic indentation under a diminished drift, we observed different mechanical properties of plasma polymer thin films prepared at different powers (0,1; 2,5 and 10 W), most probably as a consequence of different polymer cross-linking. There is a general rule of thumb applied to the problem of evaluation of nanoindentation data, which says that values of mechanical properties are not influenced by substrate of the film until 10 % of the film thickness[4]. Our results proved that the rule can be used for hardness, but in the case of modulus the application is limited especially for stiffer films.

Based on the comparison of conventional and cyclic nanoindentation, we found out that both techniques give very similar and reproducible values of mechanical properties. Thus we could admit that cyclic indentation is reliable method for fast construction of depth profiles of modulus/hardness.

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REFERENCES

1. Helvaci F., Cho J.: MRS Symp. Proc. *841*, 375 (2005).
2. Čech V., Trivedi R.: Surf. Coat. Technol. *205*, 286 (2010).
3. Saraswati T., Sritharan T., Mhaisalkar S., Breach C. D., Wulff F.: J. Mat. Sci. Eng *A423*, 14 (2006).
4. Čech V., Trivedi R., Hoferek L.: Surf. Coat. Technol. (2011) (in press).

2-L11

KINETICS OF FORMATION OF SELF-ASSEMBLED MONOLAYERS

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Although a molecular monolayer is only a few nanometers thick, it can completely change the chemical and physical properties of a surface. Molecular monolayers can be readily prepared using chemisorptions on a variety of surfaces. The self-assembled monolayers (SAMs) are highly ordered two-dimensional structures that form spontaneously by the immersion of an appropriate substrate into a solution of an active surfactant in an organic solvent. The most common adsorbate/substrate combinations are alkylsilanes on oxide surfaces.

There are several types of SA methods that yield organic monolayers. These include alkanethiols and alkanedithiols on gold[1], silver, and copper; dialkyl sulfides and dialkyl-disulfides on gold; alcohols and amines on platinum; carboxylic acids on aluminum oxide and silver; and organo-silanes on hydroxylated surfaces (SiO_2 on Si, Al_2O_3 on Al, or glass), i.e., epoxysilane on silicon wafer or glass[2], octa-decyltrichlorosilane[3] (OTS) and aminosilane[4] on silicon substrate.

SAMs of alkanthiols are used as a dielectic layer in organic thin film transistors[5] (OTFT). SAMs of silanes continue to find use in a multitude of applications in biotechnology and nanotechnology, both as model substrates to study interfacial interactions and as a strategy to chemically graft bioactive molecules[6]. A variety of silanes (mostly trialkoxysilanes) have been applied as coupling agents in the natural fibre polymer composites (NFPCs) to promote interfacial adhesion and improve the properties of compo-sites[7].

Interfacial interactions and interphases play a key role in all multicomponent materials irrespectively of the number and type of their components or their actual structure. Although the role and importance of interfaces and interphases are the same for all multicomponent materials, surface modification must be always selected according to the objectives targeted, as well as to the characteristics of the particular system[8].

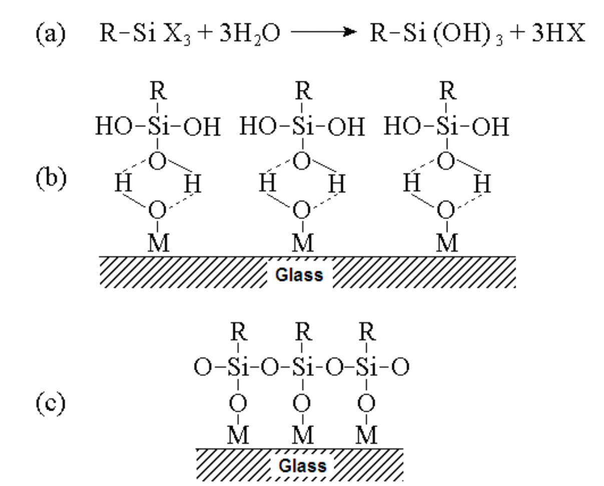

Figure 1. Organosilicon on hydroxylated surfaces: (a) hydrolysis, (b) chemisorption and (c) condensation.

Our study was aimed at SAM deposition using vinyltriethoxysilane (VTES) or vinyltrichlorosilane (VTCS) molecules chemisorbed on silicon oxide surfaces. The VTES monomer molecule is a multifunctional one, which reacts at one end with the glass surface, and the other end is a functional group (Fig. 1). The ethoxy group (X= $-O-CH_2-CH_3$) represents a hydrolyzable unit and so the silane is hydrolyzed to the corresponding silanol (Fig. 1a) in the aqueous solution to which the glass substrate is exposed. These silanol molecules compete with water molecules to form hydrogen bonds with the hydroxyl groups present at the glass surface (Fig. 1b). When the glass substrate is dried, the free water is driven off and condensation reactions occur, both at the silanol/glass interface and between neighboring silanol molecules (Fig. 1c). The result is a SA monolayer bonded to the glass surface, presenting an array of vinyl groups (R= $-CH{=}CH_2$) oriented outwards. The kinetics of formation of VTES or VTCS monolayers on planar glass substrate or silicon wafer with native silicon dioxide layer was characterized by contact angle measurements. The surface free energy and its components were evaluated using the Owens-Wendt-Kaelble geometric mean method and the Wu harmonic mean method. Spectroscopic ellipsometry was used to determine the thickness of deposited films. Detailed information on the elemental composition of SAMs was obtained by X-ray photoelectron spectroscopy (XPS). Surface morpho-logy of deposited films was investigated by atomic force microscopy (AFM).

This work was supported by GACR, grant No. P106/11/0738.

REFERENCES

1. Hamoudi H., Dablemont C., Esaulov V. A.: Surf. Sci. *605*, 116 (2011).
2. Tsukruk V. V., Luzinov I., Jultholgpiput D.: Langmuir *15*, 3029 (1999).
3. Dong J., Wang A., Simon Ng K. Y., Mao G.: Thin Solid Films *515*, 2116 (2006).
4. Yang S. R., Kolbesen B. O.: Appl. Surf. Sci. *255*, 1726 (2008).
5. Bao Z.: Material Matters, Sigma-Aldrich, *1*, 11 (2006).
6. Lee M. H., Boettiger D., Ducheyne P., Composto R. J.: *Silanes and Other Coupling Agents*, Brill Academic Publishers, Leiden 2007.
7. Xie Y., Hill C. A. S., Xiao Z., Militz H., Mai C.: Compos. Part A: Appl. Sci. Manuf. *41*, 806 (2010).
8. Pukanszky B.: Europ. Polym. J. *41*, 645 (2005).

2-L12

THE SOL-GEL PROCES IN MANUFACTURE OF REFRACTORY MATERIALS

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Refractory materials are characterized by the ability resist to enhanced temperatures. This feature is conventionally described by numerical value refractoriness ≥150 which is temperature equal or higher than 1500°C. However, serviceability and properties of refractory materials depends on wider array of factors, e.g. classification temperature, refractoriness under load, chemical resistance and durability, thermal shock resistance, permanent change in dimensions on heating, that are derived from their structure that is affected by applied raw materials and process of manufacture[1,2].

Refractory products can be dividing from several viewpoints such as type of material (oxide and non-oxide ceramics, fused cast refractory products, refractory castables, refractory metals...), chemical composition (oxides, nitrides, carbides, silicon carbides, carbon refractory products...), consistence (shaped and unshaped refractory products), etc. The increasing interest in utilisation of unshaped type of refractory materials, such as gunning materials, dry, tap hole and injectory mixtures, refractory sealants, coatings and castables, etc., hand in hand with tailoring (prepairing special materials with properties for the specific applications) can be observed at the present time.

This work is focused on the aplication of sol-gel methods in production of refractory materials. The colloidal silica with particle size from 5 to 20 nm is mostly used as the setting agent in refractory castables to form coagulative structures (coagulation bonding) in material. The silica sol was introduced into dry castable with water to form a working mixture with appropriate consistence. With the view to advanced ceramic technology is this method classified as the sol-gel process, i.e. one of wet chemical synthesis method.

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REFERENCES

1. Hanykýř V., Kutzendörfer J.: *Technologie keramiky*, Vega, Praha 2002.
2. Bláha J.: *Výkladový slovník z oborů keramiky, žárovzdorných materiálů, skla a maltovin*, ČSVTS – Silikátová společnost České republiky, Praha 2002.

2-L13

RESORBABLE IMPLANTS FOR HARD TISSUE REGENERATION

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Bone tissue is capable of regeneration, however the natural bone healing process is in some cases insufficient (e.g. in excessive loss of bone due to trauma or tumour resection, non-healing fractures, metabolic diseases, arthrodesis etc.) Likewise, the ability of spontaneous regeneration of articular cartilage is rather low with no effective healing of the defect[1]. An important role in the regeneration of demaged tissues of the musculoskeletal system, such as the bone or cartilage play mesenchymal stem cells (MSCs) due to their ability of differentiating into osteoblasts or chondrocytes, respectively[2].

The main aim of this work was to devoloped suitable 3D biopolymer-based composite materials (scaffolds) biocompatible with bone and cartilage tissue. By combining these scaffolds with MSCs via tissue engineering technique the new temporary implants for bone and cartilage tissue regeneration were investigated.

New 3D resorbable porous scaffolds based on the collagen matrix modified by bioactive filler such as hydroxyapatite nanoparticles or chitosan nanofibres (depending on the application) have been prepared. These materials have excellent biocompatibility with hard tissues, high bioactivity, and they are neither antigenic nor cytotoxic[3]. Porous scaffolds were additionaly chemically cross-linked in order to improve mechanical properties and degradation stability. Consequently, scaffolds were seeded with MSCs prior the testing on animals. Prepared scaffolds were investigated with a view of chemical composition, pore size and porosity, biodegradation, biomechanics, proliferenciation of MSCs and their differentciation into bone or cartillage cells. Based on the results, the pore size is affected more by concentration of collagen than filler addition, whereas porosity remained almost similar at all samples. As for MSCs adhesion, the optimal pore size was found to be aproximatelly 400 μm corresponding to 0,5% collagen involving 50 wt% of additive[4].

At preclinical testing bone regeneration, porous composite scaffold of collagen/hydroxyapatite was seeded with allogeneic MSCs and implanted into segmental femoral defect in miniature pigs[5]. The MSCs seeded scaffold initiates tissue regeneration at the deffect and starts to resorb in the body at predefined time. Radiological and histological examination and computed tomography of bone defect locations in 16th weeks after transplantation confirmed positive and faster regeneration of hard tissue into new bone.

Additionaly, the collagen/chitosan scaffold with MSCs was used for the healing iatrogenic defect of articular cartilage of the femur condyle also in miniature pigs[6]. Based on the histological and immunohistochemical evaluation it was proved that using this type of scaffold new hyaline-like cartilage grow-up. In contrary, using only plane scaffold or MSCs alone, only fibrous cartillage in the form of scar arised.

Based on these positive results, projects for clinical testing of these materials on human patients have been presented.

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REFERENCES

1. Mankin H. J.: N. Engl. J. Med. *291*, 1285 (1974).
2. Arinzeh T. L.: Foot Ankle Clin. *10*, 651 (2005).
3. Burg K. J. L., Porter S., Kellam J. F.: Biomaterials *21*, 2347 (2000).
4. Prosecká E., Rampichová M., Vojtová L., Tvrdík D., Melčáková Š., Juhasová J., Plencner M., Jakubová R., Jančář J., Nečas A., Kochová P., Klepáček J., Tonar Z., Amler E.: J. Biomed. Mat. Res. A (2011), in press.
5. Crha M., Nečas A., Srnec R., Janovec J., Stehlík L., Raušer P., Urbanová L., Plánka L., Jančář J., Amler E.: Acta Vet. Brno *78,* 635 (2009).

6. Nečas A., Plánka L., Srnec R., Crha M., Hlučilová J., Klíma J., Starý D., Křen L., Amler E., Vojtová L., Jančář J., Gál P.: Physiol. Res. *59*, 605 (2010).

2-L14

DEVELOPMENT OF FIBRE-REINFORCED GEOTHERMAL CEMENT COMPOSITES FOR APPLICATION IN PRETENTIOUS HYDROTHERMAL CONDITIONS

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Fiber-reinforced cement composites were developed for potential geothermal well cementing applications due to their high performance to wisthand aggressive hydrothermal conditions where temperatures may exceed 400°C. Nowadays, growing interest is paid to the development of geothermal energy as alternative to energy based on fuel due to its advantage in the savings of natural resources, energy and CO_2 emission. During the exploitation of geothermal energy, steel casings are protected from severe hydrothermal environment by cementitious sealant that helps to maintain its structural integrity. Though the minimum compressive strength of cement required for geothermal applications, according to API (American Petroleum Institute) Task Group is 6,9 MPa, this criterion cannot however, meet all scenarios, such as pressure, temperatures, carbonation etc. that may exist in geothermal wells. Fiber-reinforced cement systems were investigated in autoclave (under hydrothermal conditions like in geothermal wells) in order to identify composites offering the best improvement, not only with regard to cement compressive strength, but also tensile strength, porosity, microstructure and durability. For this propose, basalt fibers were used to reinforce the cement matrix composed of Portland cement, calcium aluminate cement, phosphate reactant, retarder, stabilizer and superplascizer.

Four different mixtures were investigated and their strength development during autoclaving at pressures of 4, 12 and 20 bars were studied. The time of hydrothermal treatment – 2, 24 and 72 hours were used. On test specimens compressive and tensile strengths were determined, SEM and mercury porosimetry were used to characterize the pore structure of cured samples. X-Ray diffraction and thermal analysis method were also used in this investigation to identify hydrated products and to characterize their thermal stability.

This article has been produced with the financial assistance of the European Regional Development Fund (ERDF) under the Operational Programme Research and Development/Measure 4.2 Transfer of knowledge and technology from research and development into practice in the Bratislava region /Project Centre for applied research of composite materials for deep geothermy.

2-L15

MOLECULAR MODELLING – STRUCTURE AND PROPERTIES OF (UN)SATURATED N-HETEROCYCLIC CARBENES

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Thermally stable heterocyclic diaminocarbenes (NHCs) were synthesized as izolable crystalline solids by Arduengo in 1991[1]. NHCs can be employed in several types of reactions, including multicomponent couplings and cycloadditions, as reactants. Moreover, NHCs are viable catalysts for a variety of reactions, mainly various acyl anion additions.

Because of difficulty in manipulation of these compounds which are moisture (oxygen) sensitive, molecular modelling represents a powerful tool enabling to predict particular properties and subsequently to intepret experimental data. Moreover, the prediction of their behaviour as for instance the catalysts could be performed as well.

Our study was focused on a 1,3-di-*terc*-butylimidazolium chloride[2] (***1***) and 1,3-di-*terc*-butylimidazol-2-ylidene[3] (***2***) as free carbene and their saturated analogues; 1,3-di-*terc*-butyl-imidazolinium chloride (***3***), and 1,3-di-*terc*-butylimidazolin-2-ylidene (***4***)[4]. Since generally, both the symmetry and the energy of HOMO and LUMO (highest occupied and lowest unoccupied molecular orbitals) have a significant influence on the mechanisms of reactions of molecules, the energies of molecular orbitals of NHCs ***1-4*** were initially computed. The visualization of selected molecular orbitals and electrostatic potential-mapped electron density surface was made using ArgusLab (Fig. 1)[5]. Both the studied carbenes ***2,4*** are due to the small band gap very reactive. Further, the electron density on the carbene centre was bigger onto carbenes ***2,4*** than onto corresponding chloride salts ***1,3***[6,7]. Bond distances and bond angles of NHCs ***1-4*** under investigation were calculated by using PC GAMESS/Firefly[8]. The best results were obtained at B3LYP/6-31G(d) level.

In this work, the prediction of the structures of selected NHCs ***1-4*** was performed. The calculations were extended by different DFT funtionals with 6-31G(d) basis set. Data of already characterized compounds with similar structure, were obtained from CCDC (Cambridge Crystallographic Data Centre) and used for comparison with calculated data.

Well corelation between experimental and theoretical data was found out for ***2*** by using O3LYP functional, whereas for ***4*** by using B3PW91 functional.

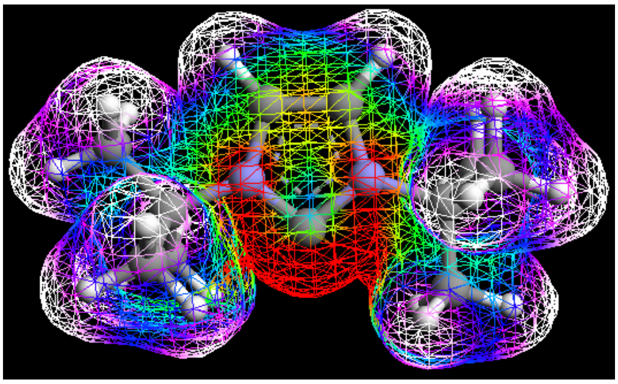

Fig. 1: The electrostatic potential-mapped electron density surface of 1,3-di-*terc*-butylimidazol-2-ylidene in ArgusLab (optimized structure at O3LYP/6-31G* level in PC GAMESS)

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REFERENCES

1. Arduengo III A. J., Harlow R. L., Kline M.: J. Am. Chem. Soc., *113*, 361 (1991).
2. Arduengo III A. J.: US Pat. 5077414, Wilmington, DE, 1991.
3. Arduengo III A. J., Bock H., Chen H., Denk M. K., Dixon D. A., Green J. C., Herrman W. A., Jones N. L., Wagner M., West R.: J. Am. Chem. Soc., *116*, 6641 (1994).
4. Denk M. K., Thadani T., Hatano K. Lough A. J: Angew. Chem., *109*, 2719 (1997).
5. *ArgusLab 4.0.1.* [computer program]. 1996 [ref. 2009-04-24]. Available from: http://www.arguslab.com/index.htm.
6. Kulovaná E., Richtera L, Hermanová S.: Molekulové modelování – struktura a vlastnosti katalyzátorů na bázi karbenů. In *Sborník příspěvků studentské odborné konference Chemie a společnost*, p. 72-77, Fakulta chemická VUT v Brně, Brno, 2009.
7. Kulovaná, E. *Molecular modelling – structure and properties of carbene-based catalysts.* Brno: Vysoké učení technické v Brně, Fakulta chemická, 2010. 62 p. Vedoucí bakalářské práce RNDr. Lukáš Richtera, Ph.D.
8. Granovsky, A. A. *PC GAMESS/Firefly version 7.1.F* [computer program]. 2005 [ref. 2009-5-20]. Available from: http://classic.chem.msu.su/gran/gamess/index.html.

2-L16

HIGHER THERMAL EFFICIENCY FOR THE 2-ETHYL-HEXENAL HYDROGENATION PROCESS

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The actual industrial scale production of 2-ethyl-hexanol ($C8H18O$) through the 2-ethyl-hexenal ($C8H14O$) catalytic hydrogenation is one of the most important petrochemical process. 2-ethyl-hexanol is an oxo-alcohol, beeing used in the production of various compounds, such as: plasticizers (dioctylphthalate), acrylate monomers (2-ethylhexylacrylate) used to modify the properties of acrylic polyesters, solvent for wax, fats, vegetable oils, insecticides and in mining and as additive for diesel fuel (Nox reduction) and Lube oils.

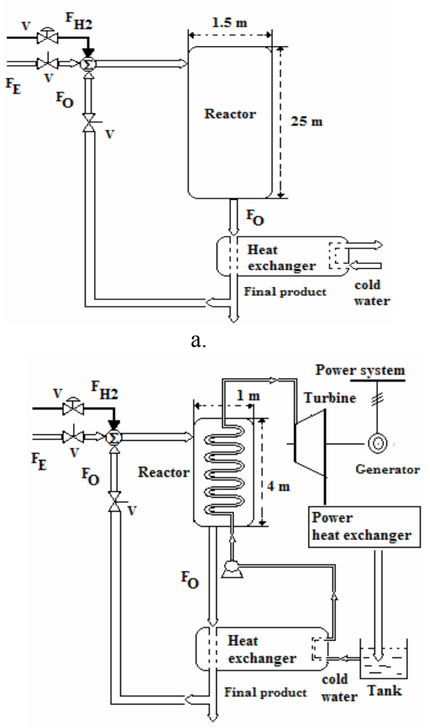
In a dedicated chemical reactor, the molecules of 2-ethyl-hexenal and hydrogen are both absorbed on the catalysts surface. Hydrogen is then dissociated to atomic form and the aldehyde is linked through the non participating electrons of the carbonyl group or of the double-bound carbon atoms. The final product (2-ethyl-hexanol) is desorbed from the catalysts surface and will migrate out from the system.

Because the hydrogenation reaction is highly exothermic, the temperature control is achieved through the dilution of 2- ethyl-hexenal with the final product itself, by recirculation of the 2-ethyl-hexanol. The mixture of the chemical compounds reactants diminishes the reaction rate and facilitates the process control. The 2-ethyl-hexenal input temperature lies in the temperatre range of 90°C to 110°C. Internal temperature of the reactants must be limited by 160°C in order to avoid side-reactions and increasing of the cooling process. The typical values of the dilution rate: 2-ethyl-hexanol flow over 2-ethyl-hexenal flow are: 6:1 – 8:1. The main drawback of the actual method consists of: high volumes of recirculated 2 ethyl-hexanol flow which needs huge dimension of the chemical reactor and relative high power consumption in the cooling process. The dissipated power is estimated at ~ 169 kW for a typical process[1].

The authors propose a new technological solution which leads to a new reactor structure given in figure 1.a. As adiacent advantage is the possibility to recover, at least partially, the thermal power of the exothermic reaction.

The main idea of the new solution is the rising of the thermal control efficiency with the cooling system placed inside the reactor. Due to the high value of the latent heat of vaporization the proposed system is based on water. The steam flow generated in the reactor, with respect to the safety condition, may be used to drive a low pressure, low power steam turbine with a proper electrical generator. The new structure consists of a pilot reactor with smaller dimensions. The flow ratio between the 2 ethyl-hexenal flow and 2 ethyl-hexanol flow is reduced to 1:1. In this case, due to the higher rate of reaction, the cooling system efficiency must be increased and the temperature reactor control needs more effective and modern strategies.

Considering a 90% steam transfer efficiency from the reactor to the turbine and a 85% turbine efficiency the available power will be around ~129 kW.



a.

b.

Figure 1. The actual structure a) and proposed structure b)

It must be emphasized that the control and protection systems will have a crucial role. If one of the systems fails, it may lead to runaway conditions (high temperatures) which will have a disastrous effect.

The given lecture is structured in 3 sections. At first an analysis of the classical method is presented. The second part consists of the thermal balance for the actual and new proposed solutions. The final part will present the efficiency impro-vement based on the modern control strategies.

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REFERENCES

1. S.C. Oltchim S.A, Romania. Internal report, (2010).
2. Mattos A. R. J. M., Probst S. H., Afonso J. C., Schmal M.: J. Braz. Chem. Soc. *15*, 760 (2004).
3. Both R., Festila C., Dulf E., Szelitzky T. O., Neaga A.: SACI 2011, in press

2-L17

SYNTHESIS AND APPLICATION OF SURFACE MODIFIED IRON OXIDE MAGNETIC NANOPARTICLES FOR REMOVAL OF POLLUTANTS

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The effluent water from dyeing, textile, pulp and paper industries contains various types of dyes which should be removed before discharging the effluent to the environment. Many of these dyes are toxic and even carcinogenic and this poses a serious hazard to aquatic living organisms.

In this study, the iron oxide magnetic nanoparticles were synthesized via chemical precipitation method with addition of ammonia to solutions of Fe^{2+} and Fe^{3+}. The structure and morphology of prepared nanoparticles were characterized by XRD and SEM instruments and the applicability of the synthesized nanoparticles for removing of cationic dyes from aqueous solutions was investigated. For this purpose, the surface of magnetic nanoparticles was modified with addition of an anionic surfactant. The effects of various parameters on the removal efficiency of dye pollutants such as type and amount of surfactant, pH of solution, salt effect and solution volume were studied and optimized. Finally, kinetic studies and dye adsorption equilibrium data were studied and calculated. The obtained results indicate that surface modified magnetic nanoparticles can be used as an efficient adsorbent material for adsorption of cationic dyes form aqueous solutions.

REFERENCES:

1. Qu S., Huang F., Yu S., Chen G., Kong J., J. Hazard. Mater. *160*, 643 (2008).
2. Faraji M., Yamini Y., Rezaee M., J. Iran. Chem. Soc. *7*, 1 (2010).

2-L18

FIVE MECHANISMS OF IRON CORROSION IN UNTREATED WATERS

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Carbon steels are the most used construction metallic material. It is given by versatility of their usage and low price. The great disadvantage of carbon steels is their low corrosion resistance in the most common corrosion environments such as diluted neutral water solutions acting in natural water cycle, in industry and water treatment. Majority of the corrosion protections manners are related to protection of carbon steel in neutral water electrolytes forming in atmosphere, soil or waters (sea, river, industrial, cooling, drinking, heating water). Corrosion rate of carbon steel in diluted neutral water solutions. Generally, iron does not resist to acidic environment, however its corrosion rate in alkaline environment is usually satisfactory (under 10 μm per year). In the neutral environment, iron corrosion is related to access of oxygen from atmosphere, composition of the electrolyte and temperature. An exchange of the electrolyte near the surface (viz. ratio of the electrolyte volume to the surface area of the metal) is a corrosion factor with significant importance but usually it remains unconsidered.

Corrosion of carbon steel in neutral water environment (3 g l^{-1} of chlorides) proceeds according to eight possibilities that are given by an oxygen access from air, by a ratio of a solution volume and surface area of the exposed metal and by an action of a differential aeration cell. The possibilities were verified experimentally by the study. Five of the corrosion mechanisms that lead to different corrosion products are characterized in this work.

It has been proven that cathodic passivation of iron by spontaneous alkalization of a small catholyte volume in a cell with differential aeration, which is a result of the prevailing oxygen reduction on the aerated electrode, causes the so called corrosion paradox. It was experimentally identified that a cathode corrodes faster than an anode in a cell with differential aeration in large electrolyte volumes, and the corrosion paradox does not occur. Asymmetric changes of pH in the cell with differential aeration with small volumes are caused by high solubility of ferrous hydroxide in the anolyte and by migration flow of hydroxide ions from the catholyte. A low corrosion rate of iron in a large volume of unaerated water may be a result of either hydrogen overpotential or the residual oxygen reduction. The corrosion rate of iron in a small volume of unaerated water may be low due to sponta-neous alkalization. However, it has not been proven by another experimental arrangement. Substantial impact of the subsequent formation of magnetite on the corrosion rate is rather unlikely.

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2-P1

SYNTHESIS AND SPECTRAL CHARACTERIZATION OF SOME NEW P-TERT-OCTYL CALIX[4]ARENE DERIVATIVES

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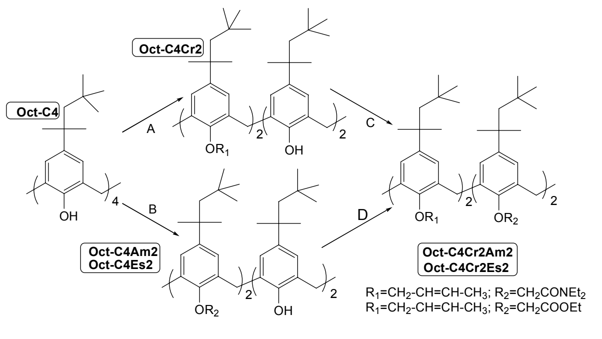
Calix[n]arene are cyclic molecules made of some phenolic units *meta* linked by methylene bridges. Owing to their cavity-shaped architecture, they are useful building blocks in the synthesis of receptors for cations, anions and neutral molecules[1]. Calix[n]arene derivatives can be synthesized by modification at the phenol OH groups (narrow rim) and in the *para*-position of the phenyl rings (wide rim). Calix[n]arene derivatives with ionophoric functional groups on the "narrow rim" exhibit excellent extraction/co-ordination abilities for metallic ions[2].

The purpose of this study is to prepare new O-ester and/or amide and/or alkenyl derivatives of *p*-tert-octyl-calix[4]arene for analytical purposes (Scheme 1).



Scheme 1. Synthesis of p-tert-octyl calix[4]arene derivatives

Elemental analysis, UV-Vis, FTIR, ^{1}H-NMR and ^{13}C-NMR investigations confirmed the formation of the desired calix[4]arene derivatives. The ability of these compounds to extract rare earths and/or precious metals ions from aqueous solutions was tested.

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REFERENCES

1. Atanassova M., Lachkova V., Vassilev N., Varbanov S., Dukov I.: Polyhedron *29*, 655 (2010).
2. Mellah B., Abidi R., Herchbach H., No K., Kim J. S., Arnaud F., Veronique H., J.: Inclusion Phenom. Macrocyclic Chem. *66*, 153 (2010).

2-P2

HUMIC ACIDS AS THE ADDITIVES IN POLYPROPYLENE

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Polyolefins, such as polypropylene (PP), show a high resistance against photo- and biodegradation, leading to the waste accumulation and environment pollution. One of the ways for turning them into environment compatible materials and for being assimilated in natural cycles is the blending with natural polymers, such as lignin or their derivatives[1]. Lignins are composed of polyphenolic compounds, which represent the major family of natural antioxidants[2] and are chemical analogues of synthetic stabilizers widely used in polymer industry as well.

Recently it has been shown that lignite humic acids (alkali extractable part of lignite) can be used as antioxidants in polymers probably because of the high content of polyphenols and unsaturated lipids. Previously we demonstrated that the addition of their ammonium salts into polyvinyl alcohol caused an increase in polymer oxidation stability. In contrast, when the degradation processes have already started, the presence of humic acids supported faster degradation of blends[3].

In this work we studied the thermal oxidation stability of humic acids/polypropylene blends using thermogravimetry. To assess the efficiency of antioxidant or pro-oxidant influence the isoconversional methods based on Arrhenius and non-Arrhenis temperature functions were used[4]. In addition, synthetic stabilizers such as Irganox and Irgafos were used to compare a stabilizing effect of HA in polypropylene.

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REFERENCES

1. Cazazu G., Pascu M. C., Profire L., Vasile C.: J. Environ. Prot. Ecol. *1,* 242 (2002).
2. Amorati R., Lucarini M., Mugnaini V., Pedulli G. F.: J. Org. Chem. *68*, 5198 (2003).
3. Kučerík J., Bakajová B., Pekař M.: Environ. Chem. Letters *6*, 241 (2007).
4. Šimon P.: J. Thermal Anal. Cal. *76*, 123 (2004).

2-P3

PRACTICAL ASPECTS OF BULK POLYMERIZATION OF STYRENE

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Bulk polymerization at high temperature with the aid of suitable initiators has significant industrial importance because it brings about increased the polymerization rate and narrowed polydispersity. Styrene is a reactive monomer that can be radically polymerized. The radical polymerization of styrene leads to a relation between polymerization rate and the molecular weight of the polymer. This is particularly true for monofuncitonal initiators. In the case of monofuncitonal initiators the molecular weight is inversly proportional to the polymerization rate. However, bi- and more functional initiators provide high polymerization rate without lowering the molecular weight of the polystyrene.

In this work, mono- and bifunctional organic peroxides were used to study of bulk radical polymerization of styrene. The free radical polymerizations were carried out at different temperature (80-134 °C) and time (1-24 hours). Relations between polymerization conditions, reaction conversion, molecular weight and polydispersity were observed. Monomer conversion was determined by gravimetry and infrared spectroscopy. Time dependence of the molecular weight and polydispersity of the result polystyrene (PS) were determined by gel permeation chromatography using PS solution in tetrahydrofurane at 23 °C.

Interesting results were obtained by comparison of polymerization rate of mono- and bi- functional initiators in initial stage of polymerization. In first 5 hours a lower polymerization rate was observed for monofunctional initiators in comparison to bifunctional initiators. Bifunctional initiators produced polystyrene with narrower polydispersity and higher polymerization rate.

This work was supported by the Ministry of Education of the Czech Republic under the project no. MSM 0021630501

2-P4

ADSORPTION OF TEXTILE ACID DYES IN THE PRESENCE OF A CATIONIC SURFACTANT ON MESOPOROUS MATERIALS AL-MCM-41

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Mesoporous Al-MCM-41 molecular sieves in the Si/Al ratios 20, 40 and 80 were synthesized under hydrothermal condition. The materials were characterized using several techniques. The mesoporous structure was confirmed by X-ray diffraction (XRD) technique. The surface area, pore size and pore volume were given by BET equation and BJH method using nitrogen sorption technique, transmission electron microscopy (TEM), Fourier transform-infrared (FT-IR) spectroscopy was used in order to obtain information on incorporated aluminum atoms in the mesoporous structure of Al-MCM-41, and differential scanning calorimetric DSC. The MCM-41 materials have effective adsorption characters to Textile Acid Dyes. The extent of adsorption was investigated as a function of solution pH, shaking speed, contact time, reaction temperature. These results demonstrate that these materials have potential applications in the treatment of yellow dye pollution from environment. Yellow dye adsorption was followed by UV.

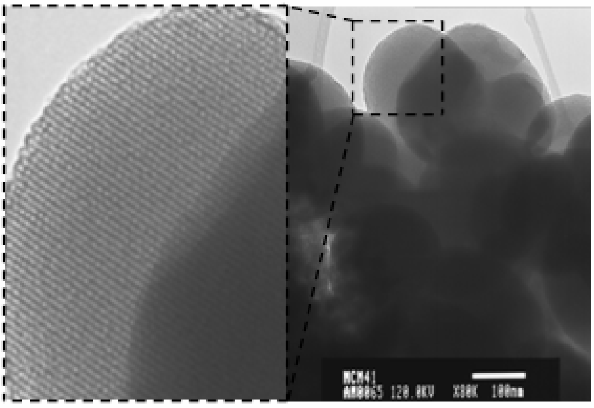

Figure 1. TEM image of Al-MCM-41 (Si/Al=80)

2-P5

GEOPOLYMERS – EARTH FRIENDLY BUILDING MATERIAL BASED ON ALKALI ACTIVATION OF INDUSTRIAL BY-PRODUCTS

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One of the possibilities how to produce more environmentally friendly concrete is to replace the OPC with alumino-silicate polymer, called geopolymer, synthesized by alkali activation of by-products materials such as fly ash, slag and another sources rich in silicon and aluminum. Geopolymers are formed by dissolution of solid aluminosilicate particles at high pH which quickly create a supersaturated aluminosilicate solution. Result is the formation of large network by condensation. Chemical structure can be described by $M_n\{\text{-(Si-O)}_z\text{-Al-O}\}_n \cdot wH_2O$ where "M" is a cation such as potassium, sodium or calcium; "n" is a degree of polymerization; and "z" is ratio of SiO_2 and Al_2O_3. The network is configured of SiO_4 and AlO_4 tetrahedrons united together by oxygen bridges. The fact that aluminum is four coordinated with respect to oxygen creates a negative charge imbalance and therefore the presence of cations such as K^+ and Na^+ is essential to maintain electric neutrality in the matrix. The C-S-H and C-A-H phases may also be originated in dependence on the composition of the starting materials and the conditions of the reaction[1-3].

This work was supported by the project "Centre for Materials Research at FCH BUT" No. CZ.1.05/2.1.00/01.0012 from ERDF.

REFERENCES

1. Davidovits J., in the book: *Geopolymer Chemistry and Applications*, chapt. 26, p. 592. Geopolymer Institute, Saint-Quentin 2008.
2. Shi C., Krivenko P. V., Roy D., in the book: *Alkali-Activated Cements and Concretes*, chapt. 13, p. 376. Taylor & Francis, London 2006.
3. Barbosa V.F.F., Mackenzie K.J.D., Thaumaturgo C.: Int. J. Inorg. Mater. *2*, 309 (2000).

2-P6

EFFECT OF pH ON PHOTOCATALYTIC ACTIVITY OF TITANIUM DIOXIDE SUSPENSIONS PREPARED VIA HYDROTHERMAL METHOD

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For preparation titanium dioxide suspensions was used as a precursor titanium oxo-chloride ($TiOCl_2$) and all samples were prepared by hydrothermal synthesis. The preparation was performed in two different pH. In acidic environment (pH 2) and in basic environment (pH 10). We investigated the influence of pH on final physical properties anf photocatalytic aktivity of prepared suspensions. Optical characterization of prepared symplex was accomplished using UV-VIS spectrometer with an integration sphere. The crystallinity and crystallite size was determined by X-ray diffraction (XRD). Consequently the particle size and shape were evaluated by transmission electron microscopy (TEM). The photocatalytic activity was studied on the basis of formic acid degradation.

Titanium dioxide (TiO_2) nanomaterials are used in a wide range of applications such as photocatalysis, separations, sensor devices, pigments and paints and dye-sensitised solar cells[1,2]. Hydrothermal treatment is one of the most widely used methods for increasing the crystallinity of TiO_2. The application of elevated temperatures and pressures in an aqueous solution facilitates the conversion of amorphous TiO_2 into crystalline TiO_2 and causes an increase in its crystallinity. Hydrothermal treatment can also be used to change the morphology, microstructure and phase composition of materials by varying the reaction parameters[3].

The pH value of the starting solution effects the crystallinity, stability and photocatalytic activity. Basic conditions suppor-ted the creation of pure anatase whereas acidic conditions brought about the formation of rutile and anatase mixture.

This work was supported by MŠMT ČR, projectOC 10050.

REFERENCES

1. Hagfeldt A., Gratzel M.: Chem. Rev. *95*, 49 (1995).
2. Chen X., Mao S. S.: Chem. Rev. *107*, 2891 (2007).
3. Byrappa K., Yoshimura M.: in the book *Handbook of Hydrothermal Technology*, chapt. 1. p. 1. William Andrew, New York 2001.

2-P7
THEORETICAL BACKGROUND OF GYPSUM DEHYDRATION TO ALPHA-GYPSUM IN CHLORIDE SOLUTIONS

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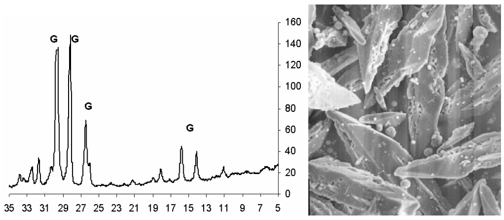
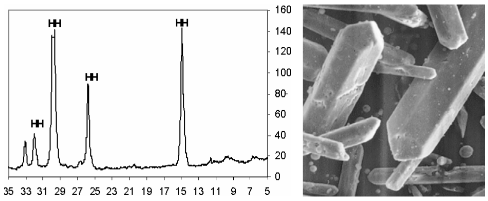
Brno University of Technology, solves in the long term the problem of alpha gypsum preparation by dehydrating the gypsum in the solution of chloride salts[1,2].

This study verified that alpha-gypsum can be trouble free prepared by gypsum dehydration in solution of a number chloride salts, among others $CaCl_2$, $MgCl_2$ or NaCl. In the same time it was found that owing to the different electro-chemical behavior of Potassium ions, the by heat conditioned reaction of gypsum with the KCl solution the dehydration doesn't take place, but a partial substitution of Potassium ions by Calcium ions takes place. The product of this reaction is the mineral görgeyit, $K_2SO_4.5CaSO_4.H_2O$.

This important problem was solved by reduction of the extremely electro-chemical high mobility of Potassium ions by the method of the mixed chloride solution with Potassium and Sodium ions, the mobility of which is in comparison with the preceding only one third. Samples of hemihydrate in the solution of mixed salts were prepared and tested in conclusion of the research.



Picture 1. RTG and REM after 300 min. dehydration in 100% KCl



Picture 2. RTG and REM after 300 min. dehydration in 75% KCl + 25% NaCl

This work was supported by grant No. FAST-S-11-15/11 and MSM0021630511.

REFERENCES

1. Dvořák, K.; Fridrichová, M.; Puttner, J.: Optimalizace výroby alfa-sádry ve směsném roztoku solí. In: *Maltoviny 2010*, p. 24 – 26, Vysoké učení technické v Brně, Brno, 2010.
2. Fridrichová M., Kalivoda K., Dvořák K.: Využití chloridových odprašků pro výrobu alfa-sádry. In: *Maltoviny 2008*. 1. Brno, 2008. p. 238 - 245.

P8
EFFECT OF THE RAW MATERIALS ON THE PROPERTIES OF TERBIUM DOPED YTTRIUM TANTALATE LUMINESCENT MATERIALS

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Terbium doped yttrium tantalate ($YTaO_4$:Tb) phosphor is a green emitting material that present a great interest from both scientific and application point of view. Usually, $YTaO_4$:Tb powders are prepared by ceramic method, from metal oxide mixtures and alkaline salts as flux.

The goal of our studies is to investigate the influence of the raw materials on the properties of $YTaO_4$:Tb phosphors prepared by solid-state reaction route. In this respect, synthesis mixtures containing appropriate amounts af Y_2O_3, Ta_2O_5, Tb-source and alkaline sulphate as flux were calcined in air, at 1200°C. Different activator concentration and terbium sources were used for phosphor synthesis namely: terbium (III) nitrate, terbium (III) oxide and terbium (III,IV) oxide. The luminescent powders were characterized by X-ray diffraction, FT-IR spectroscopy, scanning electron microscopy, and photoluminescence measurements (PL/PLE).

The morphological, structural and optical properties of $YTaO_4$:Tb phosphors are influenced by both the activator concentration and Tb-source (Fig.1).

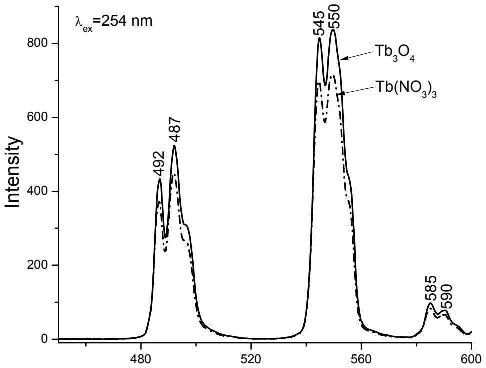

Fig.1. Emission spectra of YtaO4:Tb(7%) sample

This work was supported by the CNCSIS-UEFISCSU, project number PNII –IDEI 2488/2008.

REFERENCES

1. Popovici E. J., Nazarov M., Muresan L., Noh D. Y., Barbu-Tudoran L., Bica E., Indrea E.: J. Alloys Compd. *497*, 201 (2010).

2-P9
THE STOPPED-FLOW TECHNIQUE FOR 1-ALKENE POLYMERIZATION

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The stopped-flow polymerization technique enables to study early stages of α-olefin polymerization on Ziegler-Natta or metallocene catalysts. This technique allows performing the polymerization experiments shorter than average lifetime of the growing polymer chains (ca. 0,2 s). Thus the stopped-flow technique is considered as one of the most powerful tool for the investigation of the catalyst active sites performance and elucidation of the olefin polymerization mechanism based on the analysis of obtained polymer material.

In this work the stopped-flow technique is further utilized for the development and preparation of new polymer materials based on the preparation of special block copolymers as polypropen-block-poly(propen-co-ethen). For this purpose, the new experimental facility suitable for the preparation of relatively high amount of the copolymer materials was constructed. The facility is designed for the copolymer synthesis close to the conditions applied in industry (polymerization pressure up to 2,2 MPa, polymerization temperature up to 70°C), what facilitates the product preparation. Successful preparation of block copolymers will provide a new insight on the nature of active sites presented in the heterogeneous Ziegler-Natta catalysts and also allow investigating the role of block copolymers in heterogeneous materials consisting of crystalline matrix (polypropen) with inclusions of amorphous poly(ethen-co-propen) rubber (impact resistant sequential copolymers produced by commercial two-reactor technology).

2-P10
ANISOTROPIC FILM CONSTRUCTION USING PLASMA NANOTECHNOLOGY

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Plasma polymer films in the form of hydrogenated amorphous carbon-silicon (a-SiC:H) or carbon-silicon oxide (a-SiOC:H) alloy are often used as barrier or protective layers for polymer and metal substrates, cutting tools, electronic, and optoelectronic devices. Properties of the devices are influenced by interfacial phenomena. To eliminate or at least reduce the internal stresses and improve adhesion, mostly multilayered rather than a single layer film has to be used. Such multilayers may be used for passivation of organic devices[1], as a dielectric barrier in semiconductor devices[2], as tribological coatings in aeronautical applications[3], or as a functional coating in polymer composites with controlled interphase[4].

Bilayers consisting of a-SiC:H and a-SiOC:H alloys were deposited on silicon wafer using plasma-enhanced chemical vapor deposition (PECVD). The layered structures were subjected to ellipsometric measurements that enabled us to distinguish individual layers and determine the layer thickness and its optical constants[5]. In next study, single layer and multilayered a-SiC:H films, deposited from tetravinylsilane at different powers by PECVD, were investigated intensively by spectroscopic ellipsometry, nanoindentation, and atomic force microscopy (AFM) to compare optical and mechanical properties of the individual layer of decreased thickness (315 – 25 nm) with those of the corresponding single layer[6]. Multilayered a-SiC:H films consisting of ten layers were constructed from layers A (10 W) and B (0,1 W) using a layer-by-layer rotating system. Ellipsometric data were analyzed using the sample model in the 11-layer form Si-SiO_2/BABABABABA/OL (OL is a surface overlayer). The dispersion dependence of the dielectric function was fitted using the five-parametric Tauc-Lorentz formula and starting from optical constants determined for the single layer film. The experimental Ψ and Δ were compared with corresponding theoretical data. Such a structure was sectioned from the film surface at a small angle of 4 deg using ultramicrotomy. The uncovered multilayer was observed by AFM and characterized by nanoindentation. Ten individual layers could be differentiated from the bottom right corner to the upper left corner (Fig. 1) due to the surface morphology that corresponded to stiffness of the individual layer. Next progress was aimed at time-dependent deposition conditions enabling deposition of functionally gradient thin films. The gradient structures with linear or exponential distribution of the refractive index across the film were deposited and analyzed using spectroscopic ellipsometry, AFM, and nanoindentation.

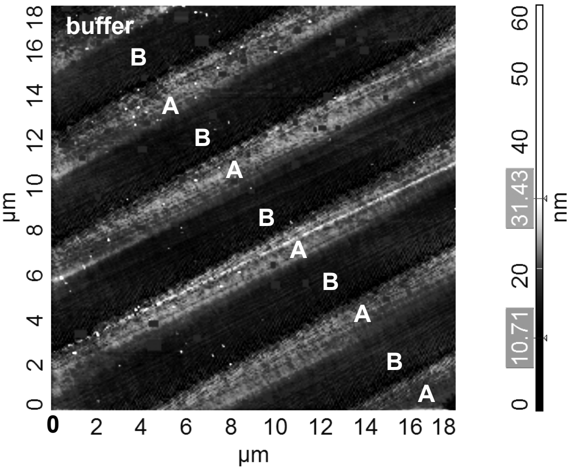

Figure 1. AFM surface morphology (scan area: 18 × 18 μm²) of sectioned ten-layered a-SiC:H film with buffer layer. Labels for layers A, B and the buffer layer are provided.

This work was supported by GACR, grant No. P106/11/0738.

REFERENCES

1. Lee J. H., Jeong C. H., Lim J. T., Zavaleyev V. A., Kyung S. J., Yeom G. Y.: Jpn. J. Appl. Phys. *8430*, 45 (2006).
2. Zhang Z., Wagner T., Sigle W., Schulz A.: J. Mater. Res. *608*, 21 (2006).
3. Joinet M., Pouliquen S., Thomas L., Teyssandier F., Aliaga D.: Surf. Coat. Technol. *2252*, 202 (2008).
4. Cech V.: IEEE Trans. Plasma Sci. *1148*, 34 (2006).
5. Studynka J., Cechalova B., Cech V.: Surf. Coat. Technol. *5505*, 202 (2008).
6. Cech V., Cechalova B., Trivedi R., Studynka J.: Thin Solid Films *6034*, 517 (2009).

2-P11

PHYSICAL CONDITIONS AFFECTING RHEOLOGICAL PROPERTIES OF THERMOSENSITIVE COPOLYMERS

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In this study rheological properties of triblock copolymers based on poly(ethylene glycol) and poly[(lactic acid)-*co*-(glycolic acid)] (PLGA-PEG-PLGA) were investigated. Since this copolymer is non-toxic, biocompatible, biodegradable and thermosensitive it might be used as injectable hydrogel for drug delivery or healing material for regenerative medicine. Injectable hydrogel is supposed to be a sol at room temperature and a gel at body temperature. Thus, it is necessary to test different physical conditions affecting the sol-gel transition prior the injection. Mainly, the influence of solvent, pH, storage temperature, sterilization and presece of ions on sol-gel transition of PLGA-PEG-PLGA copolymer were evaluated.

Rheological properties of series PLGA-PEG-PLGA copolymer concentrations (4-28 %) at miliQ water or phosphate buffer saline (PBS) at different conditions were studied by both test tube inverting method (TTIM) and rheometer. Since gel points (lower and upper) are detectable by both methods, TTIM show us the gel (no)transparency while viscosity and gel elasticity is measurable by rheometer.

Based on the results, the gel stiffness increased with the polymer concentration moving the gel point of copolymer to the lower temperature. As for physical conditions, the main effect on the sol-gel transition exhibited freezing the polymer solution prior the measurement by narrowing the gelation region (closing in the lower and upper gel points). However, the gel points were much more affected by physical conditions at polymers from unsublimated monomers (having some impurities and by-products) compare that from sublimated ones (very pure). The best results showed 26 wt% polymer obtained from sublimated monomers and dissolved in PBS having clear gel region from 32,6 to 38,6 °C while white gel is stable up to 46,3 °C. This material is promising as injectable hydrogel for regenerative medicine.

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2-P12

THE DISTRIBUTION OF P_2O_5 IN THE STRUCTURE OF THE CLINKER MINERALS

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This work dealt with the influence of P_2O_5 upon the phase composition of the clinker and its distribution in the structure of clinker minerals. The ash of bone meal was used as the source of P_2O_5. The samples with different content of the ash of bone meal were prepared. The free lime content in the samples increased with increasing ash of bone meal addition. The increase in f-CaO content in clinker is due to the fact that P_2O_5 enters C_2S and stabilizes it. The form of alite crystals was blocked due to high content of P_2O_5 in the raw meals. The distribution of P_2O_5 in the clinker minerals was captured with Electron Probe Micro Analyzer (EPMA). It was found out that P_2O_5 enters the structure of calcium silicates with three different ways. C_2S receives certain amount of P_2O_5 through P^{5+} for Si^{4+} substitution. The substitution of two atoms P^{5+} for two atoms Si^{4+} causes that Ca^{2+} atom must leave its position and so the vacancies is created ($Ca^{2+} + 2Si^{4+} \rightarrow V_0 + 2P^{5+}$). It means that during the synthesis of stabilized dicalcium silicate from pure dicalcium silicate and P_2O_5 certain amount of CaO has to be released. The contents of Al_2O_3, Fe_2O_3 and P_2O_5 in both calcium silicates (C_2S, C_3S) showed that P can enter the structure of calcium silicates through the substitution $Al^{3+} + P^{5+} \rightarrow 2Si^{4+}$ known as berlinite substitution and also the substitution $Fe^{3+} + P^{5+} \rightarrow 2Si^{4+}$. The results from chemical microanalysis showed that P doesn't enter the structure of C_3A and C_4AF. C_3A and C_4AF formed the gapping mass located between C_2S and C_3S grains.

2-P13

PHYSICAL-CHEMICAL PROPERTIES OF PRECURSORS FOR YAG:CE TYPE PHOSPHORS OBTAINED BY WET-CHEMICAL SYNTHESIS

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Under UV and blue excitation, cerium doped yttrium aluminium garnet $Y_3Al_5O_{12}$: Ce^{3+} (hereinafter YAG: Ce), exhibits efficient green-yellow luminescence that can be modulated toward orange red domain, by the partial or total substitution of Y^{3+} with Gd^{3+} ions, in $(Y,Gd)_3Al_5O_{12}$: Ce^{3+} (hereinafter YGAG:Ce) and $Gd_3Al_5O_{12}$: Ce^{3+} (hereinafter GAG:Ce) phosphors. Luminescence performances, particle morphology and crystalline phase purity of YAG:Ce type phosphors are decisive factors in determining their use in optoelectronics or as optical material for UV and IR optics.

Usually, the phosphor synthesis proceeds by the solid-state reaction route, at very high firing temperature (>1500°C), using several calcination-milling cycles. One alternative method for the synthesis of YAG: Ce type phosphor in mild thermal conditions is the wet-chemical synthesis route *via* the reagents simultaneous addition technique WCS-SimAdd[1].

The goal of this study is to determine the main physical-chemical characteristics of precursors obtained by WCS-SimAdd route, in order to correlate them with the specific properties of phosphors.In this respect, YAG:Ce, YGAG:Ce and GAG:Ce were prepared by calcination, from Y-Gd-Al-Ce precursors obtained using the WCS-SimAdd route, from metal (III) nitrate and urea as precipitating reagents, respectively.

Y-Gd-Al-Ce precursors were investigated using thermal analysis (TGA), infra-red absorption spectroscopy (FT-IR), FTIR-TGA measurements for gases evolved during the thermal process, ICP-optical emission spectroscopy, X-ray diffraction, scanning electronic microscopy, and photoluminescence (PL) measurements. Precursor composition, powder particle morphology and sizes, and crystalline state were determined in correlation with the precipitation conditions.

WCS-SimAdd proved to be a convenient method for the synthesis of homogeneous, amorphous nanopowders with general formula $xM(OH)_3.yM(HCO_3)_3.zM(NO_3)_3.vNH_3.wH_2O$, that can be used for low-temperature production of pure phase YAG:Ce, YGAG:Ce and YGAG:Ce powders with good PL properties.

This work was possible with the financial support of the CNCSIS –UEFISCSU, project number PNII – IDEI 2488/2008.

REFERENCES

1. Popovici E. J., Muresan L., Stefan M., Morar M., Grecu R., Indrea E., Barbu-Tudoran L.: J. Opt. Adv. Mat. - Symposia *2*, 136 (2010).

2-P14

LEACHABLE POLLUTANTS OF SECONDARY RAW MATERIALS

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Ever-increasing production of waste materials has become a serious economic and political worldwide problem. New approaches are required in almost all industrial procedures to reduce current trends of waste formation and to create new methods of ecological and economic recovery or disposal of waste. How to use these materials as a substitute for primary raw materials need to be learned, mainly because it is necessary to limit environmental damage by mining and by establishing of landfills. As far as the environment is concerned, the disposal of fly ash should not be regarded as a sustainable management strategy. Fly ash and the slag as secondary raw materials are considered as hazardous materials because of their high content of heavy metals such as lead, arsenic, barium etc. That is why both of them should be well treated to reduce their damage to the environment.

Environmental requirements for new building materials and also for secondary raw materials are very strict. The considerable aspect is to evaluate their leaching properties. These characteristics are the important part of determination about suitability of the secondary raw materials for incorporation into a final product or construction. Leaching properties consider not only efficiency of immobilization of pollutants in the created materials with the comparison of used secondary raw materials which include different types of fly ash and blast furnace slag, but also the behavior during the leaching and hydratation process. Many factors can influence this behavior including chemical speciation, particle size or minerals, leaching liquid etc.

This study deals with the use of different types of fly ash and other secondary raw materials as suitable materials for matrices. Leaching properties (concentration of selected elements) of these materials will be examined in the relation to time during the hydratation process.

This work was supported by the project "Centre for Materials Research at FCH BUT" No. CZ.1.05/2.1.00/01.0012 from ERDF.

REFERENCES

1. Odler I., in the book: *Special inorganic cements*, p. 240, E and FN Spon, London 2000.
2. Li G., Zhao X.: Cement. Concrete Comp. *25*, 293 (2003).
3. Izquierdo M., Querol X., Davidovits J., Antenucci D., Nugteren H., Fernandéz-Pereira C.: J. Inorg. Mater. *166*, 561 (2009).

2-P15
INCREASES IN WATER RESISTANCE OF MACRO-DEFECT-FREE COMPOSITES

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Macro-defect-free (MDF) cements are perspective materials providing unique properties relative to traditional cement pastes with great potential to incoming constructional utilization. But it is known, that MDF cements exhibit sensitivity to water, with swelling and reduction of strength. This work is aimed at monitoring of an organic polymer effect on the moisture resistance of MDF materials and phase changes. The effects of three different organotitanate cross-linking agents on the properties and moisture sensitivity were investigated. The organotitanate-modified MDF cement sam-ples exhibited improved moisture resistance as compared to the standard samples when exposed to 100 % relative humidity at 22°C. One way, how to eliminate the moisture sensitivity, is chemical modification the microstructure of the MDF cements, especially polymer matrix and the interphase. To increase the mechanical properties of MDF cements the fibre reinforcements were used. Results show that fibre reinforcements have positive effects on the growth of these properties.

This work was supported by the project "Centre for Materials Research at FCH BUT" No. CZ.1.05/2.1.00/01.0012 from ERDF.

REFERENCES

1. Birchall J. D., Howard A. J., Kendall K.: U.S Patent 4,410,366. 1983.
2. Lewis J. A., Boyer M. A.: Adv. Cement Base. Mater. *2*, 2 (1995).
3. Desai P. G., Young J. F., Wool R. P.: Materials Research Society Symposium Proceedings *245*, 179 (1992).

P16
APATITE-LAYER FORMATION ON LS_2-BASED GLASS-CERAMICS IN SBF AND DMEM SOLUTION

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In this study, the *in vitro* behaviour of lithium disilicate glass-ceramic in the Li_2O-SiO_2-CaO-P_2O_5-CaF_2 was investigated using simulated body fluid (SBF) and Dulbecco's Modified Eagle's Medium (DMEM). The formation of carbonated hydroxyapatite in body fluids is essential for both orthopaedic and, in particular, dental applications for caries inhibition. In this paper, we compare the results of bioactivity test for one type of glass-ceramics immersed in two medium, that are mainly composed of CaF_2 and $Ca_3(PO_4)_2$ linked in fluorapatite. The glass-ceramic samples were prepared by annealing, or thermal treating of parent glasses under optimised regime in a muffle furnace at 600°C for 6 hours. The bioactivity has been tested under static regime. The representative glass-ceramic samples were immersed in SBF and DMEM solution at 36,5°C for different period of time - 1, 3, 7 and 28 days. Scanning Electron Microscopy (SEM) and Electron Probe Micro Analysis (EPMA) were applied to characterise the glass-ceramic samples, as well as to detect the presence of a new phase onto the surface. The results suggest that the surface characterization and *in vitro* tests revealed a few variations in the reactivity with the different time of immersion in both fluids after their pristine form. The thickness of apatite-layer on the surface of samples is strongly influenced with time immersion in SBF and DMEM solution. For the formation of apatite-layer in earlier testing period (1, 3 and 7 days), was not observed striking difference between SBF and DMEM immersion. In the later testing period in SBF (28 days) the layer of hydroxyapatite (HA) was developed by periodical deposition of spherical apatite bullet that covers the whole surface. The use of the acellular culture solution DMEM resulted in a delay at the start of precipitation.

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2-P17
IMMOBILIZATION OF HEAVY METALS IN ALUMINOSILICATE MATRIXES

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Nowadays researchers all over the world try to find new methods and materials, which enable safety storage of the hazardous materials containing heavy metals. Good possibilities to fix and immobilize heavy metals show alkali activated aluminosilicate matrixes - geopolymers.

Structure of geopolymers can be described as a sialate network of connected SiO_4 and AlO_4tetrahedra linked together by sharing oxygen atoms. Negative charge of Al^{3+} is balanced by presence of positive ions Na^+ or K^+. Geopolymers have similar structure as zeolites.Contrary to zeolites geopolymers contain amorphous and semicrystalline structure.

Geopolymerisation can be classified as an inorganic polycondensation reaction. The reaction process is composed of mineral dissolution, aluminosilicate polycondensation and structural reorganization.

Geopolymers are synthetized by alkali activation of aluminosilicate source. Suitable sources are metakaolin, fly ash or blast furnaceslag.

Immobilization of heavy metals in geopolymer matrix is dependent on many various factors as a source of silica and alumina, type of alkali activator, pH, curing conditions, form of fixed element etc. Mechanism of inhibition of heavy metals in geopolymers hasn't been described precisely yet. Both chemical and physical interactions are expected to affect the immobilization. Chemical interactions mean, that metal cation can replace Na^+ or K^+ in the cavities and play the role of charge balancing ion or it can be chemicaly bonded within newly formed amorphous or crystalline aluminosilicate phases. Physical encapsulation is linked with size and quantity of pores and mechanical properties of matrix.

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REFERENCES

1. Komnitsas K., Zahakari D.: Miner. Eng. *20*, 1261 (1997).
2. Zhang J., Provis J. L., Feng D., van Deventer J. S. J.: J. Hazard. Mater. *157*, 587 (2008).
3. Phair J. W., vanDeventer J. S. J., Smith J. D.: Appl. Geochem. *19*, 423 (2004).

2-P18
BIOMEDICAL ELASTOMERIC POLYURETHANES BASED ON POLY(ETHYLENE GLYCOL)

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Biodegradable polyurethane (bio-PU) elastomers with potential use as scaffolds in a regenerative medicine were synthesized using poly(ethylene glycol) (PEG), poly(caprolactone) (PCL) and hexamethylene diisocyanate catalyzed with stannous octoate.

New methodology was developed to prepare functional samples with different molecular weight of PEG. Effect of bio-PU composition on the swelling and hydrolytic stability was investigated together with testing mechanical properties, monitoring of net formation and the degree of conversion of prepared samples. The morphology of prepared samples was analysed by SEM and optical microscopy. Chemical composition was tested by infrared spectroscopy and the thermal properties were measured by differential scanning calorimetry.

Synthesis of bio-PU was realized under the nitrogen atmosphere on high vacuum line in combination with nitrogen filled glove-bag. Samples were prepared on a mould (10x10 cm, 1mm thick) and cured for 48 hours at 65 °C. As for methodology development, it was found that it is necessary to degas all feedstocks before synthesis since non-homogeneous and bubbled samples have been obtained. Based on the physical conditions of samples preparations, flexible bio-PU films from transparent (clear) through cloudy (with visible phase separation) to no-transparent white were obtained. Phase separation was confirmed by optical microscopy showing typical spherulite structure of crystalline phase (PCL) in amorphous phase of PEG.

Mechanical properties testing showed that the higher molecular weight of PEG in the sample the tougher the material. Characterization of swelling exhibited higher water uptake for the samples with higher molecular weight of PEG, which correlated well with sample hydrolytic degradation. Bio-PU samples having higher molecular weight of PEG degraded in water faster in incubator at 37 °C.

In conclusion, the mechanical properties as well as the hydrolytic stability of resulted bio-PU elastomers might be effectively controlled by the both amount and the molecular weight of hydrophilic PEG in sample with a view to meet specific medical applications.

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2-P19
LUMINESCENT MATERIALS ACTIVATED WITH RARE EARTH IONS WITH GARNET STRUCTURE

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Garnets constitute one of the most important families of hosts for luminescent dopants, especially for rare earth ions (RE). Yttrium aluminate ($Y_3Al_5O_{12}$) is the best known garnet that can be doped with various RE such as: Ce^{3+}, Eu^{3+}, Tb^{3+}, Nd^{3+}, Ho^{3+}, Yb^{3+}, Pr^{3+}, Er^{3+} (cit[1]). Additionally, this kind of materials show very interesting applications in the field of optical materials, such as: scintillators for detection of high energy radiation; white LED lighting; classical and quantum information processing[2].

In this work, we present studies reffering to the preparation of rare earth doped yttrium aluminium garnet (YAG:RE) starting from suitable precursors by wet chemical method using different precipitating agent such as: urea, oxalic acid, ammonium carbonate etc., in order to establish the influence of some preparative conditions on the phosphor characteristics.

Morpho-structural and luminescence properties of YAG: RE phosphors were put in evidence using IR – spectroscopy (FT-IR), X-ray diffraction (XRD), scanning electron microscopy (SEM) and photoluminescent measurements.

Depending on the rare earth ions the garnet samples exhibit photoluminescent emission in the red, green and yellow-green spectral domain, with characteristic maxima at 609 nm (europium), 544 nm (terbium) and 530 nm (cerium).

The quality of precursors as well as the thermal synthesis regime, determines the photoluminescence properties and morpho-structural characteristics of YAG phosphor powders.

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REFERENCES

1. Speghini A., Piccinelli F., Bettinelli M.: Opt. Mat. *33*, 247 (2011).
2. William M. Yen, Shionoya S., Yamamoto H., in the book: *Practical Applications of Phosphors*, chapt. 2, p. 193, chapt. 3, p.254, chapt. 4, p308. CRC Press, New York 2007.

2-P20

PHYTO-TOXICITY OF SINGLE-WALLED CARBON NANOTUBES ON PLANT GROWTH

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Carbon nanotubes are a novel material with wide applications in areas such as electronics, cosmetics and medicine. Although many studies have carbon nanotubes as the focal point, their toxic potential to the enviornment is not yet determined. In this study three crop species (carrot, cucumber, corn) were subjected to the effects of pristine and functionalized carbon nanotubes.

Nanotubes were functionalized with carboxilic groups to enhance their water solubility and quantum-dots particles for imaging purposes. Root elongation was assesed in order to establish how different concentrations of carbon nanotubes affect the plants in this stage. Uptake and internalization of single-walled carbon nanotubes into the roots was evaluated using Raman spectroscopy, confocal microscopy and scanning electron microscopy. Different responses to carbon nanotube exposure were obtained in this study, depending on the type of carbon nanotubes employed, their concentration and plant specie.

Results obtained in this study are usefull in areas such as usage and safe disposal of carbon nanotubes.

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REFERENCES

1. Canas J. E., Long M., Nations S., Vadan R., Dai L., Luo M., Ambikapathi R., Lee H., Olszyk D.: Envioron. Toxicol. Chem. *27,* 1922 (2008).
2. Lin D., Xing B.: Enviorn. Pollut. *150*, 243 (2007).

2-P21

STUDY OF MECHANICAL AND BALLISTIC PROPERTIES OF TEXTILE MATERIALS USED IN BALLISTIC APPLICATIONS

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Different types of textile materials used in ballistic applications were tested. The aim of work was to measure material mechanical properties such as Young's modulus E [GPa], maximum load Fmax [N], tensile strength [MPa], elongatian [%] according to standard ČSN EN ISO 1421 and ballistic properties such as V50 balistic evaluation. The materials were compared by means of mechanical propertees and V50 values. The relation between mechanical and ballistic properties was discussed. Also flammability resistance according to standard ČSN 64 0757 was investigated.

Figure 1. Shooting range

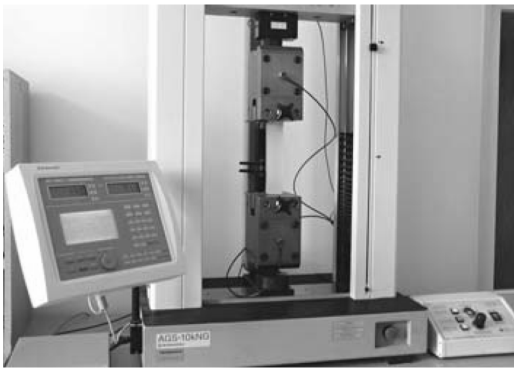

Figure 2. Testing of tensile strength

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2-P22

INFLUENCE OF THE ENVIRONMENT ON POLYMER ALUMINO-SILICATE MORTARS ON THE BASIS OF ALKALI-ACTIVATED FLY ASH

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Alumino-silicate polymer product of the (-O-Si-O-Al-Si-O-)n type is obtained by inorganic poly-condensation. Analogous materials practically occur in the nature only in the form of crystalline bodies, for instance zeolites, clay minerals. Alumino-silicate polymers represent materials at the border-line between hydrated inorganic binders, vitreous and ceramic materials.

Great attention has been paid recently to the properties of alumino-silicate polymers. The majority of papers dealing with the influence of the environment on the properties of alumino-silicate polymers are dedicated to the investigation into the effect of corrosive agents (salts, acids). It is evident from published data that alumino-silicate polymers exhibit excellent resistance to the action of corrosive environments which is substantially better in some cases than that of the materials based on Portland cement. However, a fact should be emphasized: there is practically no information about the influence of various types of possible exposures (aqueous, humid, hydro-thermal) on properties of alumino-silicate polymers. Such data about the properties resulting from various types of exposures namely belong to fundamental material properties in the same manner as in the case of Portland cement.

The main goal of the investigation was a systematic study of the long-term effect of external environment on mechanical properties of mortars containing a binder on the basis of the alumino-silicate polymer. The influence of the exposure of the above materials to the action of water, the humidity (the relative humidity amounting to 98 %) and to aqueous solutions with pH ranging from 12 to 14 was investigated. Furthermore, the materials were subjected to the process of "artificial aging" by a series of hydro-thermal cycles.

The strength characteristics were studied in intervals ranging from 7 to 360 days. The compressive strength of materials stored under dry conditions increases consistently in dependence on the exposure time. The values of the compressive strength of materials kept in the humid environment increase in the beginning but, later, a slight drop could be observed and, eventually, constant parameters are attained. The strength of materials exposed to water increases at first but, later, constant values are achieved. The time dependence of the bending strength of materials exposed to all three types of experimental conditions exhibits a constant or increasing trend. The exposure of alumino-silicate mortars to high pH environments results in a decrease in their strength because of the material destruction (probably caused by their dissolution).

2-P23

THERMODILATOMETRIC ANALYSIS OF KAOLIN

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Thermodilatometric analysis (TDA) is convenient method for investigation of sintering behaviour in ceramics. It can give insight into firing processes, phase transitions, densification and sintering properties, thermal shock, etc. It also provides understanding of reaction kinetics, additives and raw materials influence. These processes are conected with shrinkage of the sample which is measurable by dilatometer. TDA curve is then useful to estimate the firring behaviour and composition of ceramic green body[1-4].

The push-rod thermodilatometr (SETSYS – 2400 CS, Setaram Instrumentation) was used for TDA of kaolin (Sedlec Ia, Sedlecký kaolin a.s.). The 150 mg of sample was introduced into alumina crucible, pressed and heated up to 1000 °C. The temperature was increasing linearly with the heating rate of 0,25 $°C \cdot min^{-1}$. The results of the TDA ares shown in Figure 1.

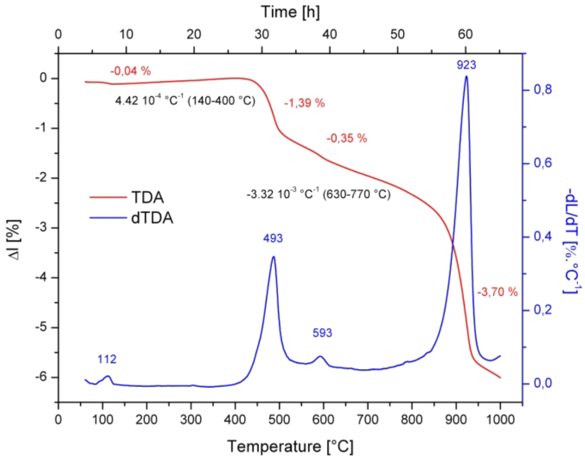

Figure 1. TDA curve of the ceramic kaolin Sedlec Ia.

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REFERENCES

1. Štubňa I., Trník I., Vozár L.: Ceram. Internat. *35* ,1181 (2009).
2. Paganelli M.: Am. Ceram. Soc. Bull. *81*, 25 (2002).
3. Šašek L.: *Laboratory Methods for Silicate Materials*, SNTL Praha – Alfa Bratislava, 1981.
4. Rath J., Pospíšil Z.: *Jemná keramika – metodika měření a zkoušek*, SNTL Praha – Alfa Bratislava, 1988.

2-P24

EFFECT OF ORGANOTITANATE CROSSLINKING AGENT ON GELATION OF POLYVINYL ALCOHOL

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Gelation is attributed to the copolymeric nature of the polymer, which is a result of incomplete hydrolysis of the basic polymer. Therefore, the stereoregularity also plays an important role: isotactic polyvinyl alcohol is dissolved easily in cold water, but syndiotactic polyvinyl alcohol does not dissolve even in boiling water. If the stereoregularity is not high, gels may be formed in aqueous solutions, where tiny crystallites act as multifunctional crosslinks. Possible three ways exists to form a cross-linked polyvinyl alcohol from solution. In water, where gelation is accompanied by formation of helical structures which are formed by syndiotactic sequences in the polyvinyl alcohol molecules. Secondaly aqueous solution, where PVAA gels are formed by cross-linking formation in terms of either hydrogen bonding followed by crystallization of PVAA chains or complexation with several kinds of inorganic ions such as cupric, borate, titanate, and vanadate ions and finally in non-aqueous solutions, where the process has to consist of organic solvents, e.g. ethylene glycol or dimethyl sulfoxide not necessarily mixed with water.

Polyvinyl alcohol is mixing with cement paste and Tyzors, organotitanate crosslinking agents, to crystallizate PVA chains and reached this way stabile MDF composite mixture with increased moisture resistivity.

The work contains the results of evaluation and selection of particular organotitanate cross-linking additives and optimizes the conditions of PVA cross-linking. The series of organotitanate compounds called Tyzors were examined as cross-linking additives with respect to the moisture sensitivity of the final macro-defect free composite.

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REFERENCES

1. Shibayama M., Adachi M., Ikkai F., Kurokawa H., Sakurai S., Nomura S.: Macromolecules. *26* ,623 (1993).
2. Peppas N. A., Merrill E. W.: J. Polymer Sci. *14*, 459, (1976).
3. Odler I., in the book: *Special inorganic cements*, p. 240, E and FN Spon, London 2000.

2-P25

OPTICAL AND ELECTRICAL PROPERTIES OF THIN LAYERS OF HUMIC SUBSTANCES

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Humic substances (HSs) are organic light-absorbing macromolecules found in nature[1]. HSs contain conjugated olefinic, aromatic, phenolic–quinone structures with different functional groups and chromophores that are capable of absorbing electromagnetic radiation[2]. HSs are used as an activator of soil, surfactants and also as material which is used for modification of surface in order to get higher conductivity[3].

The aim of this work is the study of optical and electrical properties of thin layers of HSs. Conductivity and optical properties of HSs were measured to provide an undersanding of the behavior of HSs as a semiconducting material usable for non-metallic electronic. UV-VIS and fluorescence spectroscopy were used for optical characterization; The HSs layers thickness have been investigated using confocal laser scanning microscopy (layers were prepared by spin coating), thickness of layers were in range 40 – 900 nm. To characterize the electrical properties current passing through thin layer were measured. Current-voltage characteristics were recorded for different conditions (dark and light). Thin layers of humic substances were also doped with phtalocyanine and fullerenes as modifiers of optical and electrical properties of humic substances.

Curent-voltage characteristic has typical expenential tendency which is characteristic for semiconductive materials. All the results taken into the consideration showed the same trends. Optoelectrical properties of HSs can be optimized by phtalocyanines. The results brought some relatively important information about behavior of humic substances in thin layers. The results partially support the investigated idea about semiconductive characteristic of humic substances and it is task for more measurements confirm and extend conclusions given in this work. Reported data can be considered only as a hint to push forward further effort to use the humic substances as semiconductive materials useful for non-metallic electronic.

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REFERENCES

1. Aguer, J.-P., Richard, C., Andreux, F.: J. of Photoch. Photobio. A *103,* 163 (1997).
2. Zhang, Z., Xue, W.: Spectrochim. Acta A *78*, 1018 (2011).
3. Stevenson, F. J.: *Humus Chemistry: Genesis, Composition, Reactions*, 2nd ed., Wiley, New York, p. 512, 1994.

2-P26

STUDY OF USABILITY OF SANDS FOR PREPARATION OF AEROCONCRETE IN HYDROTERMAL CONDITIONS

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A building industry is expanding branch all the time and it intervenes in the life of everyone. Aeroconcrete is very attractive and actual subject nowadays because economy is important point of view of production[1] . This material finds a widespread use thanks to its specific properties. The good manipulation and great thermal insulating properties are secured by its porous structure. Furthermore, proper material selection provides a saving of power of house for every customer demands. Due to aeroconcrete's sufficient compressive strength it is useful for both constructions and internal walls. Fundamental and very important for final material properties is raw material composition of aeroconcrete. A relationship between composition of filler (quartz sand) and resultant properties of aeroconcrete is an objective of the thesis.

This thesis deals with usability of sands for preparation of aeroconcrete materials in hydrotermal conditions. Materials for experiments were provided by Xella CZ, s.r.o., specifically 11 samples of sand from the nearest surroundings of factory in Hrusovany. The quality of aeroconcrete materials are influenced by composition of raw materials and a goal of this work was to analyze the samples and choose the best sand for use in a practice. For sample analysis XRD diffraction and ICP-OES[2,3] were used. The object of interest was relationship between composition and mechanical properties (e. g. compressive strength and grindability).

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REFERENCES

1. Svoboda L.: *Stavební hmoty*, Jaga group s.r.o., p. 471, Bratislava 2004.
2. Warren B. E.: *X- ray diffraction*. Dover Publications, Inc., Mineola, p. 381, New York 1990.
3. Hill. S. J.: *Inductively coupled plazma spektrometry and its applications*. 2nd edition. Blackwell Publishing Ltd., p. 427, Oxford 2006.

2-P27

MOLECULAR SIMULATION STUDIES OF ADSORPTION AND SELECTIVITY OF CO_2 ON ZEOLITIC IMIDAZOLATE FRAMEWORK-78

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In this work adsorption selectivity and atomic details of CO_2 and CH_4 in ZIF-78 were study using grand canonical monte carlo (GCMC) and molecular dynamic (MD) simulations, respectively. In the first step, suitable parameterizations were validated using the experiment adsorption isotherm. One lattice units of ZIF-78 of (1×1×1) and 8 unit (2×2×2) dimensional super cells were used in GCMC and MD simulations, respectively. Loadings of the guest molecule in MD simulation at 298 K are 10, 80, 150, 220 molecules for CO_2 and 5, 25, 45, 65 molecules for CH_4. The results show that adsorption site for CO_2 and CH_4 is O atoms of $–NO_2$ group and C atoms of nitroimidazole of ZIF-78, respectively. The result also found that the adsorption selectivity of CO_2 from CH_4 mixture at 1 atm is equal 8,15. This demonstrates that ZIF-78 can used to separate CO_2 from CH_4.

This work was supported by the National Research University Project of CHE, the Ratchadaphiseksomphot Endowment Fund (AM1078I) and the Thailand Research Fund (TRF:RTA 4980006) as well as the Deutsche Forschungsgemeinschaft (Schwerpunktprogramm SPP 1392). S.P. thanks the Asian Development Bank (ADB) scholarship for financial support. O.S. thanks the Ratchadaphiseksomphot Endowment Fund from Chulalongkorn University for Postdoctoral fellowship.

REFERENCE

1. Banerjee R., Furukawa H., Britt D., Knobler C., O'Keeffe M., Yaghi O. M.: J. Am. Chem. Soc. *131*, 3875 (2009).
2. Phan A., Doonan C. J., Uribe-Romo F. J., Knobler C. B., O'Keeffe M. and Yaghi O. M.: Acc. Chem. Res. *1*, 58 (2010).
3. Liu D., Zheng C., Yang Q., Zhong C.: J. Phys. Chem. C. *113*, 5004 (2009)
4. Acceleys, Inc., Materials Studio, 5.0V; Accelrys Inc.: San Diego,CA, 2009.
5. Hou X. J., Li H.: J. Phys. Chem. C *114*, 13501 (2010).
6. Casewit C. J., Colwell K. S., Rappe´ A. K.: J. Am. Chem. Soc. *114*, 10024 (1992).

2-P28

HYDRATION OF CARBOXYMETHYL CELLULOSE OF DIFFERENT MOLECULAR WEIGHT STUDIED BY SEVERAL DSC APPROACHES

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Various natural and synthetic polymers have been widely investigated for their specific interactions with water molecules[1]. It is well known that via these interactions water has a strong influence on physical and chemical properties of polymers. In addition polymer hydration properties such as water holding capacity, state of water, water self-diffusion coefficients etc. are important for their industrial and medicinal applications. Therefore, in order to improve their appplication potential, understanding of the influence of water on polymer physical structure under various conditions is of a great importance.

The traditional way of differentiation of water molecules in hydration shells of polymers using differential scanning calorimetry (DSC) is based on freezing/thawing experiments[1] in which the difference in physical properties between freezable water in form of ice and non-freezing water (does not form ice) is investigated. The aim of this study is to assess the hydration of carboxymethyl cellulose (CMC) with different molecular weights and different degrees of substitution by this conventional DSC approach and to compare obtained results with other alternative methods.

As pointed out recently, results obtained by DSC freezing/thawing experiments can provide different results when the hydration is determined from crystalization and melting peaks, mainly in case of water insoluble polymers[2]. It was also concluded that water cannot be monitored *in principle* by calorimetric analysis and term *thermally latent* water was introduced. In this work the hydration is studied using both processes and differences in freezing/thawing experiments are discussed with respect to presence of so-called freezing bond water which is understood as the interlayer between non-freezing and free-water. Further, the CMC derivatives are studied by method based on the water evaporation as recently suggested by Prusova et al.[3]

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REFERENCES

1. Hatakeyama H., Hatakeyama T.: Thermochim. Acta 308, 22 (1998).
2. Gemme-Ide M., Ohya A., Kitano H.: J. Phys. Chem. B. *114*, 4310 (2010).
3. Prusova A., Smejkalova D., Chytil M., Velebny V, Kucerik J.: Carbohyd. Polym. *82*, 498 (2010).

2-P29

ECOTOXICOLOGICAL EVALUATION OF CHOSEN FLY ASHES UTILIZABLE IN BUILDING MATERIALS

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Nowadays, waste material production grows worldwide as well as an effort of its further utilization. This effort leads to increasing efficiency of industrial production. It is possible to treat some waste materials again and we can use their properties which are a quality alternative for the common used natural materials whose mining is unbearable in light of economic and ecological. It is treated waste from metallurgy, chemical industry and power plants such as slag, ash and fly ash especially.

We were interested mainly in fly ash utilization in our work. It was necessary to do some experimental verification of ecological availability according to the Decree No. 294/2005 Coll. to break ash in building material. In agreement with that legislative a content of hazardous elements in dry matter and leach (As, Ba, Cd, Co, Cr, Cu, Hg, Mo, Mn, Ni, Pb, Sb, Se, V, Zn), an amount of radionuclide ^{226}Ra, ^{40}K, ^{228}Th and also ecotoxicity were controlled. Some organic pollutants were followed up in dry matter as well, such as extractable organic halogen (EOX), BTEX (benzene, toluene, ethylbenzene, and xylene), polycyclic aromatic hydrocarbon (PAH), hydrocarbons C_{10} – C_{40} and polychlorinated biphenyls (PCB). The ecotoxicological tests evaluated global effect and interaction of various factors of aqueous leach with waste or materials containing trash on various levels of ecosystem by using real background simulation.

Ecotoxicological tests were used for evaluation of ecotoxicity, represented by 4 trophic levels, with several bioindicators evaluated at different exposure time periods. The battery of classical tests includes the growth inhibition test of the freshwater alga *Raphidocellis subcapitata*, the immobilization test of the cladoceran *Daphnia magna* and freshwater fish *Poecilia reticulata.* Ecotoxicity was evaluated by test of inhibitive effects on the root growth of white mustards *Sinapis alba*.

At the end of testing the fly ashes satisfying specified limits were selected. Those were used for inorganic composite production which includes different content of fly ashes. The tests for ecological and technological (physico-chemical and physico-mechanical characteristics) suitability of this composite were made subsequently.

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2-P30
ANALYSIS OF CREEP BEHAVIOR OF HIGH DENSITY POLYETHYLENE

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High-density polyethylene (HDPE) pipes are used extensively for the transportation of natural gas, water, fuel etc. PE pipes have replaced clay, copper, asbestos-cement, iron and concrete pipes in various applications since the 1950s. HDPE pipes used for gas transport are under preassure for all lifetime period. In service, the time dependent phenomena notably creep take place during lifetime. Short- and long-term tensile creep tests of HDPE have been conducted at different stress levels and two different temperatures, 23°C and 80°C. All experiments were realized on two types of HDPE materials PIPELIFE 100 RC and HDPE XS10B. Specimens for creep experiments were moulded from granules. This technique of preparation excludes anisotropy of materials related to preferential orientation of macromolecular chains. Phenomenological approach was chosen for characterization of creep properties. This work is part of wide research: Fracture damage mechanism of multilayer polymer body. Obtained results serve like corroborative datas for numerical modeling of fracture damage mechanism of plastic pipes.

This work was supported by Grant Agency of Czech Republic ČR, grant No. 106/09/0279.

2-P31
THE STUDIUM OF THE EFFECT OF SELECTED MINERAL ADMIXTURES ON THE HYDRATION OF PORTLAND CEMENT BY ISOPERIBOLIC CALORIMETRY

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The possibilities of a multicell isoperibolic – semi-adiabatic calorimeter for the measurement of the hydration heat evolution and maximum developed temperature of cement mixture of different composition during setting and early hardening are presented. The measurements were aimed to determine the impact of selected components content on the course of portland cement hydration. As components used for the determination of the behavior in mixtures have been used: very finely ground granulated blast furnace slag (GBFS), silica fume (microsilica, SF), especially grinded quartz sand (SGQ) and calcinated bauxite (B). Commercial polycarboxylate type superplasticizer was also added.

2-P32
PYROLYSIS OF POLYMER COMPOUNDS

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Pyrolysis, as a way of biomass and waste processing, brings many benefits when the obtained products may provide a basis for energy gaining, but also raw material for a production of certain chemical compounds used in other sectors. Another advantage, of course, is the reduction of environmental pollution compared with conventional fuel combustion.

The process of pyrolysis occurs at three stages. At first stage organic material is drying and is released the moisture (temperature to 200 °C), at the second stage are decomposed mainly carbon-carbon, carbon-hydrogen bonds and carbon-oxygen bonds inside macromolecular organic materials and are forming volatile gaseous products, liquid simple hydrocarbon products and solid residue. At third stage (above 500°C) are hydrocarbons transformed into H_2, CO, CO_2 and CH_4[1].

The paper deals with the results of pyrolytic processing of various sample of organic polymer waste (lignocellulosic polymer and plastic polymer). The products of pyrolysis are char, pyrolysis oil and pyrolysis gas. The chemical composition of pyrolytic products and their utilization depend on chemical composition of material, pyrolysis temperature, heating rate, pressure, oxygen, pyrolysis technique[2].

We processed the samples of the wood sawdust, the straw, polypropylene and polyethyleneterephtalate (PET) in the laboratory pyrolysis equipment. The conditions of pyrolysis were 500 °C and non-oxidative atmosphere (nitrogen). We observed the weight decrease of the sample and the yield of products. The heighest decrease of weight was determined by the samples of plastic and the highest amount (about 70%) of pyrolytic gas too.

This work was supported by Slovak Grant Agency VEGA, grant No. 1/0471/10.

REFERENCES

1. Neves, D., Thunman H., Matos A., Tarelho L., Gomez-Barea A.: Prog. Energ. Combust. *37*, 611 (2011).
2. Bridgewater, A. V.: Chem. Eng. J. *91*, 97 (2003).

2-P33
THE CONTENT OF POLYCYCLIC AROMATIC HYDROCARBONS IN MOTOR OIL AND HYDRAULIC OIL

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Industrial waste oils are classed as hazardous waste with the amount of various classes of hazardous substances, mainly heavy metals, PCBs and PAHs. Polycyclic aromatic hydrocarbons are a group of over 100 different compounds with lipophilic character. They can be formed from natural and anthropogenic sources, when complex organic substances are exposed to high temperatures or pressures. Some PAHs have been identified as carcinogenic, mutagenic and teratogenic substances[2].

PAH compounds occured also in unused oils, due to the production process. Their content and composition during using of oils vary, that is dependent as well as way and time of using[1]. Therefore it is important compare their in samples of oil before and after using.

In this work, changes of PAHs content in motor oil and hydraulic oil during using, were analysed. Samples were treated with liquid-liquid extraction using solvents, that consits of hexane, methanol, water and N,N-dimethylformamide in various ratio. The concentrated extract was purified by SPE. The identification and quantification some PAHs was carried out by capillary column gas chromatography with mass spectrometer (GC/MS).

In used oil samples was determinated higher amount of PAHs and were identified also PAH compounds, that in unused oil samples were not occured. These compounds were mainly alkylderivate of PAHs.

This work was supported by Slovak Grant Agency VEGA, grant No. 1/0471/10.

REFERENCES

1. Domeno C., Nerín C.: J. Anal. Appl. Pyrolysis *67*, 237 (2003).
2. http://www.atsdr.cdc.gov [cit. 2011-03-04]

2-P34
DETERMINATION OF HYDROGEN CYANIDE FROM POLYURETHANE IN BUILDING INSULATION MATERIALS

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Polyurethanes are copolymers consisting of a chain of low molecular weight polyester or polyether molecules joint by covalent bond through characteristic urethane linkage, -NH-CO-O-[1]. Properties depend on degree of polymerization, structure with typical branched and crosslinked macromolecules and the length of chain between each urethane linkage[2].

Sandwich construction materials are often used nowadays in building industry. Their advantages are low weight, simple installation and good thermo and acoustic insulation properties. They are composed of two layers of metal alloy with a thermo insulation layer between them[3].

Disadvantages of polyurethane are high inflammability, high temperature of flames, high degree of smoke production and production of toxic fumes and compounds. These properties are considerable at polyurethane foam used as a acoustic and thermo insulation material. The use of fire retardants reducing inflammability can lead to a production of its own toxic fumes.

Combustion of polyurethane produces carbon oxides (CO, CO_2), water, nitrogen oxides (N_2O, NO, NO_2) and hydrogen cyanide[4]. Esperanza[5] identified also six hydrocarbons (methane, ethylene, etc.) and 31 volatile and semi volatile organic compounds (benzene, toluene, naphthalene, etc.).

This work brings new information on pollutants produced by combustion of building materials based on PUR. The subject of evaluation was the emission of hydrogen cyanide, a important PUR combustion product from toxicology point of view. Sample collection, laboratory sample analysis and comparison of results with the legislation limits were performed.

Sampling was conducted by standard sampling equipment supplied by co. EMIMAT. Samples were taken by stainless steel sampling probe connected to a set of absorbers filled with absorber solution by a silicone tubes. Subsequent to absorbers was a layer of silica gel to separate the gas moisture. Gas flow was determined by flow meter and supplied by membrane pump. Concentration of pollutants was analyzed by colorimetry, which is a analytic method recommended for HCN analysis by several authors[6]. Results were corrected for standard conditions (20°C, 101,3 kPa) and compared with highest acceptable exposition limit (NPEL). According to government regulation no. 300/2007 coll., appendix no. 1[7], highest acceptable exposition limit for HCN in work environment air is 2,1 mg m^{-3}. Measured values of HCN were in range from 10 to 160 mg in analysed combustion gases. Results indicate that acceptable limits according to legislation were exceeded several times.

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REFERENCES

1. Chian, K. S., Gan, L. H.: J. Appl. Polymer Sci. *68*, 510 (1998).
2. Smirnova N. N., Kandeev K. V., Markin A. V., Bykova T. A., Kulagina T. G., Fainleib A. M.: Thermochim. Acta *445*, 8 (2006).
3. Chow W. K.: Polym. Test. *23*, 974 (2004).
4. Walther D. C., Anthenien R. A., Fernandez-Pello A. C.: Fire Saf. J. *34*, 345 (2000).
5. Esperanza M. M., Font R., Garcıa A. N.: J. Hazard. Mater. B *77*, 119 (2000).
6. Le Tallec Y., Guillaume E.: *Hazards of combustion products*. The Royal Society, London, 2008.
7. Nariadenie vlády SR č. 300/2007 Z. z. o ochrane zamestnancov pred rizikami súvisiacimi s expozíciou chemickým faktorom pri práci.

2-P35

ELASTOMERIC POLYURETHANES FILLED BY BIODEGRADABLE POLYHYDROXYBUTYRATE

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Nowadays, with increasing PUs consumption, the chemistry of preparation is focused on the possibilities of recycling and/or degradation of the plastic materials in order to avoid environmental pollution by non-degradable polymer waste dump. One of the ways how to reduce the price along with increasing the degradability of these materials is addition of cheap biodegradable filler to the structure of PURs, which can partly or fully substituted one of the raw material, mostly polyol.

In the proposed work, new elastomeric bio-polyol based polyurethanes (PUs) with specific mechanical properties were prepared by one-shot process. Commercial non-degradable polyether polyol derived from petrochemical feed stocks was partly substituted by the biodegradable polyhydroxybutyrate (PHB), which belongs to group of polyhydroxyalcanoates. PHB is produced by microorganisms having physico-mechanical properties comparable to synthetic polypropylene.

Morphology of elastomeric PU composites was evaluated by scanning electron microscopy (SEM). Mechanical properties of prepared samples were obtained by both tensile measurements and prediction via Mooney-Rivlin equation.

Specimens of reference PU (no PHB) and PU filled with 1, 5 and 10 % of PHB were successfully synthesized on vacuum line apparatus. An analysis by SEM proves that prepared materials have character of particle reinforced composite material where PHB particles are regular with size of about 1 – 2 μm in diameter. Tensile measurement demonstrates that Young's modulus of each sample increase with increasing volume fraction of the filler. From measuring stress-strain data the first and the second term of Mooney-Rivlin equation were calculated. Getting constants were applied to recalculate stress-strain curves. It was found that Mooney-Rivlin equation corresponds well with stress-strain behavior of prepared specimens.

Proposed work shows that biodegradable filler, which in this case supplies polyol component, can improve mechanical properties of final product and possibly enhance the biodegradability of commercial Pus.

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2-P36

PROSPECTIVE NOVEL TRIAZOLE-BASED ALUMINIUM COMPLEXES AS CATALYSTS FOR POLYMERIZATION OF CYCLIC ESTERS

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Biodegradable aliphatic polyesters based on polylactones and polylactides are widely used in biomedical applications (Capronor) as well as in packaging (Mater-Bi, Novamont). Ring-opening polymerization (ROP) of lactones or lactides and functionally related monomers is major method to synthesize corresponding polyesters. Among known ROP catalysts, organoaluminum compounds (belonging to strong Lewis acids) represent highly selective catalysts being effective in the presence of alcohol as nucleophilic agent.

In this work, novel triazole-based aluminum compounds $\{O,O'\text{-[4,5-P(O)Ph}_2\text{tz]-Al}^i\text{Bu}_2\};O,O'\text{-[4,5-P(O)Ph}_2\text{tz]-Al}^i\text{Bu}_2\}$ and $\{\kappa_3\text{-N,N',S-[4,5-P(S)Ph}_2\text{tz)}_2(\mu\text{-tz)]-Al}^i\text{Bu}_2\}_2$ were investigated as possible catalysts for the ROP of ε-caprolactone and d,l-lactide. ROP reactions were performed in a chlorobenzene solution at 50 °C for 24 or 48 h in the presence of benzyl alcohol or butanol as initiators.

Based on the results, all Al complexes efficiently catalyzed ROP of ε-caprolactone (at 500 or 1000 excess to Al) in the presence of benzyl alcohol leading to high molecular weight polymers having molecular weight distribution (Mw/Mn) between 1,1 – 1,5. Catalytic efficiency expressed as the ratio of $Mn_{(Theor)}/Mn_{(NMR)}/Mn_{(GPC)}$ was found to be 1/1,1/1 under mentioned conditions.

Among examined complexes, only compound *O,O*'-[4,5-P(O)Ph_2tz]-Al^iBu_2} showed catalytic activity for the ROP of d,l –lactide in the presence of n-butanol initiator. However, with a view to Mw/Mn approximating to 2, the polymerization reaction was not well controlled exhibiting side reactions (e.g. transesterification, chain transfer, end-groups coupling).

The proposed work confirmed the newly used triazole-based aluminum compounds as suitable ROP catalysts of ε-caprolactone using benzyl alcohol initiator.

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2-P37
VISCOELASTIC PROPERTIES OF COLLAGEN I

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Proteins are materials with the characteristic hierarchical design, which involves formation of multi-functional and stimuli responsive materials. Collagen is Nature's most abundant structural protein and the basic building unit of human body. There are many types of collagen, but the most abundant is collagen I, which forms almost 90 % of collagen in human body. It is essential in forming bones, skin, tendons, cornea and ligaments.

Rheological properties of collagen are significant, because it is widely used as a biomaterial. It is well known that many biological soft tissues behave as viscoelastic materials with hysteresis curves being nearly independent of strain rate when loading frequency is varied over a large range[1].

Influence of structural parameters such as degree of crosslinking, concentration and pH on viscoelastic properties will be assessed in this work.

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REFERENCES

1. Zhang W., Chen H. Y., Kassab G. S.: Biomaterials *28*, 3579 (2007).

2-P38
MECHANICAL PROPERTIES OF CEMENT PASTE IN RELATION ON THE HYDRATION DEGREE

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This study presents a multi-scale approach to strength of cement paste, which is the binder for all cementitious materials, including mortar, concrete, high-performance concrete, sprayed concrete, jet grouted soil, etc. The strength of cementitious materials primarily depends on the mode of loading (tension/compression), on the composition of the material, and on the maturity, i.e. on the hydration degree.

Isothermal calorimetry is the experimental method for evaluating early-age hydration kinetics, typically up to ages ranging from 7 d – 14 d. A heat-flux differential calorimeter Toni Cal 7338 was used for the calorimetry experiments.

Compressive stress is the stress state caused by an applied load that acts to reduce the length of the material in the axis of the applied load. A simple case of compression is the uniaxial compression induced by the action of two opposite, pushing forces. To measure hardened cement paste samples, was used the universal testing machine: Walter & Bai LFM 150.

On the base of the results it can be concluded that the tests carried out at hydration degrees smaller or equal than approximately 50 % are not affected by lime leaching. These tests were used to back-analysis based on the micromechanics model[1] (the microscopic deviatoric hydrate strength of the hydration products). For cement CEM I 42.5 N the adjusted model prediction agrees very well with the measurements, i.e. the quadratic correlation coefficient amounts to very satisfactory (99,35 %). The correlation for CEM I 52.5 R, between the adjusted model prediction and the measurements is quantified by the quadratic correlation coefficient which amounts to 91,49 %.

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REFERENCES

1. Pichler B., Hellmich Ch.: Cement Concrete Res. *41*, 467 (2011).

2-P39
THIXOTROPY, AGING AND RHEOLOGICAL PROPERTIES OF CEMENT PASTES

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Rheological properties of cement paste are critical for the concrete industry, because they affect placement and workability. Moreover, these properties are responsible for the quality of concrete (mechanical properties, durability).

Fundamental and phenomenological rheological models have been proposed in the literature for characterizing the behaviour of fresh cementitious materials. Rheology, defined as „the study of deformation flow", provides a relationship between shear stress and rate of deformation[1].

Finding a rheological model describing flow curve for cementitious pastes has proved to be challenge. The most commonly used model is the Bingham model, which is given by: $\tau = \tau_0 + \mu_p\gamma$. In which τ is the shear stress [Pa], τ_0 the yield stress [Pa], μ_p the plastic viscosity [Pa.s] and γ the shear strain rate [s^{-1}] [2].

The thixotropy bebaviour is most common for cementitous materials[3]. According to Barnes[4], the accepted definition of thixotropy is „a gradual decrease of the viscosity under shear stress followed by gradual recovery of structure when the stress is removed".

The aim of this work has been to give a contribution to the fundamental understanding of the rheological behaviour of cementitious pastes. It was studied influence of water-to-cement ratio, superplasticizer dosage, tixotropy and aging of different cement pastes.

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REFERENCES

1. Chidiac S. E., Mahmoodzahed F.: Cement Concrete Comp. *31*, 535 (2009).
2. Vikan H., Justnes H., Winnefeld F., Figi R.: Cement Concrete Res. *37*, 1502 (2007).
3. Wallevik J. E.: Cement Concrete Res. *39*, 14 (2009).
4. Barnes H. A.: *An Introduction to Rheology*, Elsevier, Amsterdam 1998.

2-P40
WDS ANALYSES OF CONVERSION COATINGS ON MAGNESIUM ALLOYS

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Conversion coating belongs to best promising ways of surface treatment of magnesium alloys[1]. These alloys are very prospective due to their low specific weight and good mechanical properties. Their mean disadvantage is high reactivity leading to low corrosion resistance. New compositions used for the protective conversion coating fromation are described. Properties of the coatings are compared with values obtained at reference chromate coatings.

Up to now, almost no attention was given to use of wavelength dispersive x-ray spectroscopy (WDS) in connection with scanning electron microscopy. The elemental microanalysis was usually performed by EDS systems, since they are very easy to use, need no calibration, analyses are very fast and the spectral resolution and accuracy of results is satisfactory[2].

Since the chemistry of the coating formation is in focus of our research, WDS was used to analyse the coating layers compositions. Although the demanding calibration was needed, the two-order better spectral resolution enables to get more accurate quanifiaction, but mainly the shift of spectral lines can be observed caused by different chemistry environment[3].

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REFERENCES

1. Zhao M., Wu S., Luo J., Fukuda Y. and Nakae H.: Surf. Coat. Technol. *200*, 5407 (2006).
2. Hamdy A. S., Farahat M.: Surf. Coat. Technol. *204*, 2834 (2010).
3. Bastin G. F. and Heijligers H. J. M.: Scanning *12*, 225 (1990).

2-P41
DONOR-ACCEPTOR COMPLEXES OF TETRAHYDROFURAN WITH SULFUR TRIOXIDE AND SELENIUM TRIOXIDE

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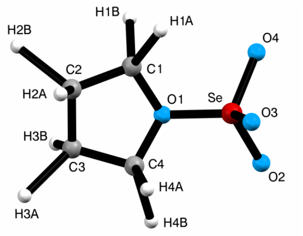
Sulfur and selenium trioxides react as Lewis acids with dialkyl and cyclic ethers to form donor-acceptor complexes having various degree of stability as primary products of this interaction. Quick rearrange of dialkyl ethers complexes to corresponding dialkyl sulfates or dialkyl selenates makes the isolation and subsequent structural characterization of these products more difficult. Donor-acceptor complexes with cyclic ethers as for exmaple 1,4-dioxanesupposed to be more stable towards rearrangement to sulfates or selenates but further reactionssuch as the ring-opening polymerization could take place.

Some representatives of these intermediates found their application also on industrial scale. Dimethyl sulfate can be manufactured and obtained in an excellent yield and purity on a continuous basis from dimethyl ether and liquid sulfur trioxide[1]. Complexes ofsulfur trioxidewith1,4-dioxane were used in the sulfonation of olefins[2] and aromatic compounds[2,3].

In our previous work, donor-acceptor complexes with the formula $Et_2O{\cdot}SeO_3$ and $(Me_2O)_2{\cdot}SeO_3$ were synthesized by the reaction of selenium trioxide with dialkyl ethers (R_2O, where R = Me, Et)[4]. The crystal and molecular structures of both complexes,which are stable only below their melting points, was determinedby X-ray structure analysis.Further, crystal and molecular structures of adducts with 1,4-dioxane: $C_4H_8O_2{\cdot}SO_3$ and $C_4H_8O_2{\cdot}2SO_3$ were determined by X-ray structure analysis and Raman spectroscopy[5].



Scheme 1. Simulation of $C_4H_8O{\cdot}SeO_3$ structure in HyperChem

In this work, the synthesis of donor-acceptor complexes with the formula $C_4H_8O{\cdot}SO_3$ and $C_4H_8O{\cdot}SeO_3$ was performed. Tetrahydrofuran (THF) was used in a high excess with respect to trioxide since it functions as solvent for the reaction as well.

The reaction of sulfur trioxide with THF was carried out at -30 °C to prevent the decomposition of organic component. Further,the solubility of sulfur trioxide was still sufficient to form a clear solution. Even at this low temperature the concomitant formation of gelous polymeric substance along with white crystalline solid was immediately observed. Unfortunately, all attempts to detect $C_4H_8O{\cdot}SO_3$ in reaction mixture failed.

The reaction of selenium trioxide with THF was carried out under same conditions. Long colourless needle crystals were isolated in a quantitative yield. However, regarding the extreme instability of these adduct, all attempts to isolatethe crystals of $C_4H_8O{\cdot}SeO_3$suitable for X-ray structure analysis failed.The identification of the product $C_4H_8O{\cdot}SeO_3$was performed by means of Raman spectroscopy.

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REFERENCES

1. Gilbert E. E.: Chem. Rev. *62*, 549 (1962).
2. Suter C. M., Evans P. B., Kiefer J. M.: J. Am. Chem. Soc. *60*, 538 (1938).
3. Hurd C. D., Kharasch N.: J. Am. Chem. Soc. *69*, 2113 (1947).
4. Richtera L., Taraba J., Toužín J.: Z. Anorg. Allg. Chem. *629*, 716 (2003).
5. Richtera L., Taraba J., Toužín J.: Collect. Czech. Chem. Commun. *71*, 155 (2006).

2-P42

PROCESSING OF MONOFILAMENTS FROM BIODEGRADABLE POLYSACCHARIDE MATERIALS

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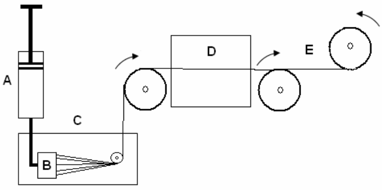
The interest of medical industry in the polysaccharidic materials is rising continuously, especially in those materials which are naturally occured in the human body. One of such materials is hyaluronic acid. The main reason for the increased attention about this material rises from it´s full metabolic resorbability[1]. This material property is advantageous for preparation of various fibrous and textile medical devices such as sutures or anti-adhesive textile layers.

The presented study describes application of the wet-spinning technology for the monofilamentous fibers formation[2,3]. The process is based on the coagulation of the fiber-forming polymer solution that is extruded through a spinnerette to a non-solvent baths. Various process parameters influencing the mechanical properties of the final fiber are studied.



Scheme 1. Fiber-spinning process, tested parameters: A (polymer solution properties), B (extruding spinnerette), C (coagulation bath), D (drying, coagulant evaporation), E (fiber elongation).

The resulting fibrous monofilaments were mechanically tested and the influences of various fillers implemented having branched structure, were evaluated. The study also shows some of the post-processing operations such as residual-solvent extraction. The final operation mentioned is the fiber-twisting into a form of threads that can be further fabricated into textile fabrics.

REFERENCES

1. Brown M. B., Jones S. A.: J. Eur. Acad. Dermatol. Venereol. *19*, 114 (2010).
2. Pakshver E. A., Kalabin A. L.: Fibre Chem. *37*, 5 (2005).
3. Moreland J. C., Sharp J. L., Brown P. J.: J. Eng. Fibers Fabr. *5*, 1 (2010).

2-P43

SYNTHESIS OF POLYVINYLALCOHOL-HYDROXYAPATITE COMPOSITE MEMBRANES

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Set of polyvinyl alcohol(PVA) and nanohydroxyapatite (HA) composite membranes were made with various HA weight percent - 0%, 10%, 20%, 30%, 40% and 50%. Nanohydroxyapatite was prepared by sol-gel procedure using diamonnium hydrogen phosphate and calcium nitrate tetrahydrate in water alkaline environment and then mixed with solution of polyvinyalcohol, which was prepared by dissolving it in water at 85°C. Different mixtures were casted in a mould and evaporated for 7 days at temperature 30 °C to obtain 1mm thin membranes. DSC, SEM and FTIR methods were used to characterise biocomposite membranes. Also, *in vitro* bioactivity in Simulated Blood Fluid (SBF) has been investigated as well as tensile properties. Then the effect of HA amount upon the membranes was discussed. It was observed formation of clusters within membranes with increasing amount of HA particles due to hydrogen bond and also the agglomeration and crystal growth of HA particles during drying of membranes. The tensile properties decreases with the increasing content of HA in membranes, as well as the onset temperatures and temperature peaks shown in DSC curves. The bioactivity was found increasing with the presence of HA in biocomposite materials.

2-P44

UTILIZATION OF FLUID BED FLY ASH IN THE PREPARATION OF RAW MATERIAL BASE FOR PORTLAND CEMENT BURNING

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The submitted research work is solved in the scope of the grant project ÚTHD FAST Brno. It records in principle the possibility of secondary raw material utilization in preparation of the raw material base for burning of Portland cement clinker. Concretely it concerns the fluid bed fly ash, produced by the power industry by modern low temperature combustion of mainly fossil fuels and of other alternative fuels too.

The optimization of the raw mix composition for the burning of Portland cement composed from two till four components was realized in the scope of the work. The effect of the selected regime on the burning process was preferentially evaluated on the base of the produced clinker phase composition, taking also into account its chemism. Not least the saving of carbon dioxide emissions were followed up and expressed as the decrease of carbon dioxide content in the given raw mix, in relation to the accordingly designed raw material mix.

This work was supported by GAČR, grant No. P104/10/0910.

2-P45

IMPROVEMENT OF CORROSION RESISTANCE OF EXTRUDED AZ61 MAGNESIUM ALLOY

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The use of magnesium alloys in structural applications is the most active area. A reduction in the weight of vehicles helps to minimize the fuel consumption and emissions. Disadvantage of magnesium alloys is low corrosion resistance. Therefore, the good corrosion protection is up to date topic for scientist around the world.

This paper deals with the investigation of corrosion resistance of extruded AZ61 magnesium alloy. Two different surface coatings were prepared. The first treatment was contained from the binary based Mastic and the polysiloxane top protective coating Hemphatane Topcoat. The second treatment was process based on phosphate-permanganate. This conversion coating was prepared like alternative to the chromating process which proved greatly results on magnesium alloys. Nevertheless, the use of chromates is limited by the environmental regulations in Europe.

The corrosion resitance of surface treatmensts were compared with that of uncoated metal. The corrosion test cabinet was used for assessment of corrosoin behavaiour. The course of the corrosion tests were in accordance with the standard ČSN EN ISO 9227. The experiments were carried out with increasing exposition. Every cycle was conducted in the test environment of 5% aqueous solution of NaCl with pH = 6,9 and temperature 35°C ± 2°C.

Final evaluation was based on measuring at analytical balances and metallographic evaluation.

This work was supported by project of specific research of Brno University of Technology FSI-J-10-38.

2-P46

INFLUENCE OF THE UREA ON CORROSION PROCESS OF ALUMINIUM ALLOYS

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Aluminium and its alloys are attractive in many technological applications due to its characteristics such as light weight, durability and good corrosion resistance. Frequent uses of these materials are in the automotive and aerospace industry. However, untreated aluminium alloys based on Al-Mg-Cu are susceptible to corrosion in aggressive atmosphere like NaCl aqueous solution.

The resistance of Al against corrosion in aqueous media can be attributed to a rapidly formed surface oxide film, which is composed maily of Al_2O_3, $Al(OH)_3$ and AlO(OH) phases. Nevertheless, the presence of aggressive ions like chlorides causes significant attack. The main corrosion process that is developed on the surface of Al alloys in a NaCl solution is the localized alkaline corrosion in the Al matrix surrounding Al(Mn, Fe, Cr) cathodic intermetallics.

This paper deals with corrosion behaviour of AlCu4Mg1 alloy in the aqueous solution inclusive 0,07 mol of urea + 0.03 mol of NaCl dissolved in the water. Two types of experimental samples were used. One of them was L-beam (AlCu4Mg1) with dimensions 14 x 14 mm with length 50 mm and thickness 2 mm. The second type was a AlCu4Mg1 sheet with 99,5% Al coating on the surface with dimensions 30 x 50 x 1 mm. Corrosion tests were performed as a modified OECD technique. The samples were placed into the small testing cells for provision no interaction of the samples. There were 30 ml of corrosion solution on the bottom of each cell and the samples were hung up above the surface. Testing mode was cyclical (8 hrs 35°C, 16 hrs 23°C – 1 cycle). The experiments were carried out with increasing exposition time (1, 5, 10, 20, 31 and 42 cycles).

The weight losses were evaluated and the statistic analysis was performed. First the Dixon test extreme deviations (maximal and minimal values) and than the corrosion rate [g m^{-2} d^{-1}] were calculated from the weight losses.

This research was supported by project of Ministry of Industry and Trade of the Czech Republic no. FR TI 1/274.

2-P47

RELATIONSHIPS BETWEEN REACTIVITY AND MOLECULAR ELECTROSTATIC POTENTIAL OF NITROAROMATIC ENERGETIC MATERIALS

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Energetic materials with $C_{(Ar)}$-NO_2 groups are generally characterized by thermochemical characteristics as homolytic bond dissociation energy *BDE* describing the C-NO_2 bond fission. This step is mostly the primary fission process under thermal, impact, shock, and electric spark initiation stimuli. To predict *BDE* energies the theoretical calculations at *ab initio* or DFT level are conducted but the reactions $ArC\text{-}NO_2 \rightarrow ArC\cdot + \cdot NO_2$ are substantially influenced by an incomplete treatment of electron correlation.

To overcome this substantial drawback the alternative method based on principle of well equilibrated isodesmic reactions was suggested recently[1]: The measure of bond strength is reaction energy of an isodesmic reaction $ArC\text{-}NO_2 + SArH \rightarrow ArC\text{-}H + SAr\text{-}NO_2$ where the pair SArH and SAr-NO_2 corresponds to standard nitroaromatic compounds. This reaction energy is expressed as the bond disproportionation energy *DISP*(C-NO_2) and should be free from electron correlation effects accompanying homolytic bond fission.

In analysis of reactivity of energetic materials the molecular surface electrostatic potential $V(\mathbf{r})$ is a promising tool[2].

$$V(\mathbf{r}) = \sum_A \frac{Z_A}{|\mathbf{R}_A - \mathbf{r}|} - \int \frac{\rho(\mathbf{r}')d\mathbf{r}'}{|\mathbf{r}' - \mathbf{r}|} \qquad (1)$$

In the case of polynitro aromatic compounds its positive value above the aromatic ring ($V_{S,max}$(ring)) can be used for QSPR studies.

The bond disproportionation energies *DISP*(C-NO_2) and bond dissociation energies *BDE*(C-NO_2) for 30 nitroaromatic energetic materials were calculated at DFT B3LYP/6-311+G(d,p) level with *ZPE* correction. The energies obtained were correlated with both electrostatic potential maxima $V_{S,max}$(ring) and total charges of the most reactive nitro group $Q(NO_2)$ in relevant structure. The results obtained show the closer correlation of *DISP* energies with $Q(NO_2)$ charges but without any structural patterns. The correlations with molecular surface electrostatic potential maxima $V_{S,max}$(ring) enable the more detailed insight into molecular structure of nitroaromatic energetic materials at primary fission process.

This work was supported by MŠMT ČR, grant No.FCH-S-11-2.

REFERENCES

1. Pexa M., Friedl Z.: Centr. Eur. J. Energet. Mater. *7*, 131 (2010).
2. Bulat F. A., Toro-Labbé A., Brinck T., Murray J. S., Politzer P.: J. Mol. Model. *16*, 1679 (2010).

3. Pexa M., Friedl Z.: *Proc. 14th Seminar New Trends in Research of Energetic Materials*, Part II, pp.893-899. UP Pardubice, April 2011.

2-P48
INDUSTRIAL POLLUTANTS PERMEATION RESISTANCE OF NANOSTRUCTURED FILMS

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Nanotech processes of thin barrier films preparation is currently used and developed in many industries. In the case of packaging materials for food, medicines and harmful substances, including construction materials for protection against toxic agents are the final outcomes of scientific work focused mainly on the preparation multilayer structures to obtain the barrier materials with very low permeation of O_2, CO_2, H_2O vapor, etc. Typically, polymer-based PET, BOPET, PP and PA foils are modified by combining the layers based on SiOx, metallic and organic layers, which are usually laminated with other polymer covering layer. This creates a great range of high quality products suitable for different types of packaging, whether in food, medicine, pharmacy, microelectronics and the like. From a military point of view, an effort is made to find cost effective ways of preparing new highly durable, solid, low-flammable and lightweight materials for personal protective equipment and containers or appropriate methods and procedures to increase the resistance of current construction materials. A key feature is their resistance to permeation by industrial pollutants (VOCs) and other highly toxic agents (CWA). For this purpose, polymer films based on PET are not very suitable, which, although they exhibit excellent resistance to permeation of most toxic substances, they are quite rigid and less resistant to periodic mechanical stress while wearing protective equipment.

With the aim to modify the in the Czech Army used copolymer PEVA foil (polyethylenevinylacetate) from which the single-use protective overcoat JP 95 is currently made up, barrier coatings based on melamine and SiO_x were applied to its surface with the aid of plasma technologies. The prepared nanocoatings were characterized in terms of surface morphology (SEM), chemical structure (FTIR) and oxygen permeability (OTR) at the University of Technology. The selected samples coated with a polymer matrix barrier nanocoatings were continuously evaluated for resistance to permeation of highly toxic chemicals (CWA) and industrial pollutants at the VTÚO Brno institute. In addition, microstructure of some selected samples was assessed before and after exposure to pollutants.

For testing the resistance of the samples, permeation by sulfur mustard (HD) was used, and as a replacement test substance cyclohexane first and then 1,6-dichlorhexane, the latter of which proved more advantageous. Presented are the resulting OTR values and also the permeation of toxic substances both of the initial uncoated and the barrier-layers-coated PEVA foil. The results show a difference in the mechanism of vapor permeation of CWA and organic solvents (VOCs) leading in both cases to swelling of the polymer matrix, in contrast to the permeation of oxygen at which the swelling does not occur.

2-P49
PRODUCTION TECHNOLOGY OF DENTAL COMPOSITE PREPREG

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Use of dental composites in dental prosthetics become increasingly important. With a wide range of properties of these materials can be used as fillers in a variety of front and posterior teeth (particle composites) or to preparation of post-orthodontic retention, leg braces and a reinforcement of platform structures (fiber-reinforced composites - FRC).

Fiber composites are used in dentistry as a material for the preparation of total dental prostheses (short-fiber composites) or as a reinforcement of platform applications, post-orthodontic plates, retention, etc. (long-fiber composites). Depending on the type of the resulting applications can be used unidirectional or multidirectional reinforcement. These fiber composites comes in to processors in the form of impregnated fiber bundle - prepreg.

The most common way to produce these materials is pultrusion. Pultrusion is a method for producing composite materials by pulling. As the reinforcement is frequently used form of glass fiber bundles oriented in one direction, braided fiber, fabric and nonwoven mats. Unsaturated Polyester Resin or vinylester resin is generally used as a matrix. In the case of dental fiber-matrix composites it is used matrix based on on bis-GMA. Pultrusion is a continuous process is basically composed of several basic stages of production, which over the years, much has not changed. They're unwinding fiber, their impregnation, molding, curing, extraction and shearing. In the case of dental fiber composites, which are produced in the form of uncured, impregnated strips called prepregs, curing process is omitted. In terms of production technology the material is therefore a very specific. Does not retain a linear form, bends and twists in the ratio between the length contraction alone impregnated tape, release paper and foil. Prepreg tape is very soft, which places increased demands on accuracy and proper component placement guidance device. A specific of uncured prepreg manufacturing is its cutting compared to conventional cutting of cured composites. When it is guided through the equippment, the end of cutted prepreg and cutting tool is contaminated by still sticky resin, which complicates any further processing.

This article describes the design of special system for dental prepreg production. Based on all our experiences cutting system and towing equipment that is capable to eliminated these problems were designed. The whole system has been continuously optimized and then tested during production.

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2-P50

ANALYSIS OF ORGANIC PHASE CHANGE MATERIALS AS LATENT HEAT STORAGE MEDIUM

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The thermal energy storage systems can operate with sensible heat storage or latent heat storage. The common way of thermal energy storage is sensible heat storage. The amount of stored heat depends on the mass of storage medium, specific heat and temperature difference during the storage process. The main advantage of sensible heat storage is the reversibility and low cost. The storage of heat can be detected from the change in temperature. As thermal storage medium can be used solids or liquids.

Phase change materials (PCMs) are latent heat storage materials. In latent heat storage the thermal energy is stored by means of a reversible change of a state or a phase change of the storage medium. Phase change materials are often used for more effective thermal energy storage. During the common process of phase change between solid and liquid state is stored large amounts of thermal energy. The latent heat storage method provides much higher storage density with a smaller temperature difference between storing and releasing heat. When a substance melts, vaporizes and sublimates, heat is absorbed. Thermal energy is released when the material solidifies. Organic phase change materials have poorer heat transfer properties and lower density. In general they are more expensive than inorganics. Paraffins have lower volumetric energy density without problems of phase separation and have good self-nucleating properties. These phase change materials are compatible with metal or plastic containers.

The most important properties of latent heat storage medium are capacity and temperature range of phase change. The correct determination of the properties is essential for practical use. Paper will be focused on the results from DSC analyses of different organic phase change materials.

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REFERENCES

1. Farid M., Khudhair A., Razack S., Al-Hallaj S.: Energy Convers. Manage. *45*, 1597 (2004).
2. Mehling, H.; Cabeza, L.: *Heat and cold storage with PCM: An up to date introduction into basics and applications*. Springer, Berlin 2008.
3. Sharma A., Tyagi V. V., Chen C. R., Buddhi D.: Renewable and Sustainable energy reviews *13*, 318 (2009).

2-P51

CORROSION DEGRADATION OF MAGNESIUM ALLOYS AS A NEW DEGRADABLE BIOMATERIALS

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Corrosion behavior of magnesium alloys is influenced by chemical composition, microstructure, grain size and heat treatment. Influence of enviroment on corrosion behavior must be also taken into consideration because it specifies the use of magnesium alloys. Magnesium ions are present in large quantities in the human body and are involved in many metabolic reactions and biological mechanisms. Biomaterials based on magnesium alloys compared to conventional metal biomaterials are more suitable for several reasons: density, tensile yield strength, young's modulus and the origin body elemets. It was found that the standard magnesium alloy AZ31 and AZ91 enhance and improve the process osteogenesis and newly formed bone have better properties compared to polymeric biomaterials poly-96L/4D-lactide. Option-specific degradation of magnesium alloys is causing great potential for their use as advanced biomaterials.

The rate of degradation of magnesium alloys in the solutions results from the aggressive behavior of chloride, sulfate, phosphate and carbonate ions, the chloride ions causes increased production of soluble magnesium chloride and simultaneously initiates the dissolution of magnesium. The magnesium alloys are highly susceptible to selective corrosion which results from heterogenous crystalline structure, such as precipitates $Mg_{17}Al_{12}$. On the surface it forms part of the traces of corrosion (pits), which may act as stress concentrators and significantly reduce the fatigue characteristics of the material.

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2-P52

CONVERSION COATINGS ON MAGNESIUM ALLOYS' SURFACE

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Magnesium alloys are one of the important structural materials. Problem is that corrosion rate of these materials is really high. Conversion coatings are one of the most effective protection against corrosion[1].

Surface of the magnesium alloys AZ31, AZ61 and AZ91 was treated by phosphate-permanganate and chromate conversion coatings. Chromating is one of the suitable way of conversion coating, but it cannot be used because of carcinogenic Cr(VI). In our case, chromate conversion coating was taken as standard[2].

Magnesium alloy AZ91 contains approximately 9 % of aluminium, which causes formation of β-phase ($Mg_{17}Al_{12}$). This phase is larger in AZ91 than in AZ31 and AZ61. Phase $Mg_{17}Al_{12}$ has the highest electrochemical potential, so it behaves like cathode in magnesium alloys. It seems that the higher contens of β-phase cause the bigger micro-cells and the elctrochemical corrosion is more significant.

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REFERENCES

1. Chong K. Z., Shih T. S.: Mater. Chem. Phys. *80*, 191 (2003).
2. Zhao M.: Mater. Chem. Phys. *99*, 54 (2006).

2-P53

CONSTRUCTION OF ELINGHAM´S DIAGRAM FOR THE PRESENTATION OF THERMODYNAMIC DATA ABOUT THERMAL STABILITY OF HYDRATES AND HYDROXIDES

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The Ellingham's type diagrams, where is plotted standard reaction enthalpy (standard reaction Gibbs energy) as the function of temperature is plotted, are used for presentation of thermodynamic and thermochemistry data. The main benefit of diagram is outline drawing of thermodynamic data in the graphic form even if the accuracy of data read-out is limited by the raster.

In literature were published Ellingham's diagrams for the thermal stability of oxides, sulphides, chlorides...[1-3]. In this work the Ellingham's type diagram for clay minerals and other hydrates (Figure 1) was constructed for the first time. The thermodynamic data used for the calculation are listed in works[4,5].

The lines in the diagram are representing the temperature dependence of standard reaction free enthalpy ($\Delta rG°$) that belongs to formation of hydrate from anhydrous form and water:

$$\Delta_r G° = -RT \ln K \quad (1)$$

Reaction equation is written for one mol of water so that:

$$\Delta_r G° = RT \ln P^*_{H_2O} \quad (2)$$

Where R, T and P^*_{H2O} is universal gas constant, temperature and relative pressure of water vapour, respectively. The relative pressure is defined as the dimensionless ratio of partial a standard pressure of water vapour in the system:

$$P^*_{H_2O} = \frac{p_{H_2O}}{P°} . \quad (3)$$

For example relative pressure is equal to 1 for the process that takes place under atmospheric pressure where $\Delta rG° = 0$.

The free energy of gas phase was calculated as following:

$$\Delta G = -(R \ln P^*_{H_2O})\, T . \quad (4)$$

It's obvious that $\Delta G > 0$ when p_{H2O} is lower than $P°$ and $\Delta G < 0$ in the other case.

For given value P^*_{H2O} the straight line starts in origin was obtained. The raster for P^*_{H2O} scale was constructed from these lines. The cross of the line with $\Delta_r G° = f(T)$ dependence belongs to the temperature of equilibrium between hydrate, anhydrous form and water. Hence the diagram can be used for prediction of thermal stability of hydrates under given value of relative pressure of water vapour.

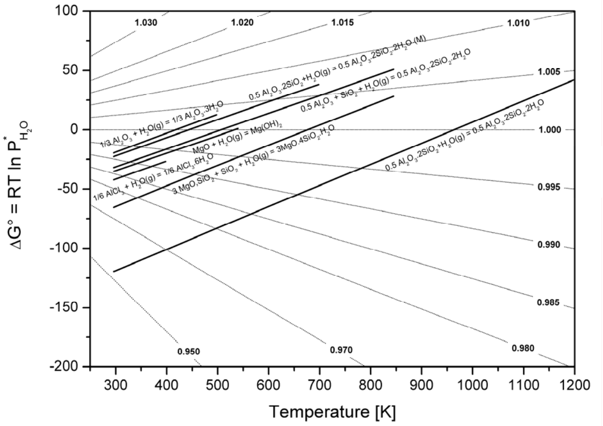

Figure 1. The Ellingham's diagram constructed for thermal stability of hydrates under different relative pressure of water vapour.

This paper arose of the research project supported by the project No. CZ.1.05/2.1.00/01.0012 "Centres for Materials Research at FCH BUT" supported by operational program Research and Development for Innovations.

REFERENCES

1. Šatava V.: *Fyzikální chemie silikátů I*, první vydání, vydalo: Ediční středisko VŠCHT, Praha (1991). ISBN: 80-7080-119-0.
2. Ellingham H. J.: J. Soc. Chem. Ind. *63*, 125 (1944).
3. Richardson F. D.: *Physical Chemistry of Melts in Metalurgy*, Acad. Press, London 1974.
4. Barin I.: *Thermochemical Data of Pure Substances, Part I*, VCH, 1993.

5. Barin I.: Thermochemical Data of Pure Substances, Part II, VCH, 1993. ISBN 3-527-28531-8.

2-P54
SYNTHESIS OF *β*-SPODUMENE GROG FOR LAS CERAMICS

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Ceramic materials based on the Li_2O – Al_2O_3 – SiO_2 (LAS) system have low thermal expansion coeficicent, excelent thermal and chemical durability (with except influence of H_2SO_4), high thermal schock resistance and low dielectric losses. The main crystaline phasese in LAS system are *β*-eucriptite ($Li_2O{\cdot}Al_2O_3{\cdot}2SiO_2$), *β*-spodumene ($Li_2O{\cdot}Al_2O_3{\cdot}2SiO_2$), virgilite ($Li_2O{\cdot}Al_2O_3{\cdot}6SiO_2$) and petalite ($Li_2O{\cdot}Al_2O_3{\cdot}8SiO_2$) and *β*-quartz solid solution with nominal composition $Li_2O{\cdot}Al_2O_3{\cdot}(2\text{-}10)SiO_2$. Also lithium silicate, disilicate and metasilicate are often formed in thermal treated working mixtures[1-3].

Lithic ceramics are prepared from the mixture of kaolin, quartz and lithium carbonate with molar ratio 1:1:4. Thermal tretment of material leads to of eucriptite (900 – 1200 °C) through rection of Li_2CO_3 with kaolinite. Spodumene phase was formed by rection of eucriptite with quartz under temperature at about 1300 °C. The product of calcination that consists from the solid solution of spodumene, slica and mullite in next grinded with clay, shaped and fired at ~1250 °C[1].

This article was didicated to synthesis of *β*-spodumene based grog (opening material) using kaolin, lithium carbonate and silica sol with particle size from 5 to 20 mn.

This paper arose of the research project supported by the project No. CZ.1.05/2.1.00/01.0012 "Centres for Materials Research at FCH BUT" supported by operational program Research and Development for Innovations

REFERENCES

1. Hanykýř V., Kutzendörfer J., *Technologie keramiky*, Druhé opravené vydání, vydala: Vega s.r.o. (2002). ISBN: 80-900860-6-3.
2. Ananthanarayanan A., Kothiyal G.P., Montagne L., Revel B.: J. Solid State Chem. *183*, 120 (2010).
3. Hlaváč J., *Základy technologie silikátů*, Druhé, upravené vydání, vydalo: SNTL – Nakladatelství technické literatury, Praha 1988.

2-P55
HYDROLYSIS OF (UN)SATURATED N-HETEROCYCLIC CARBENES

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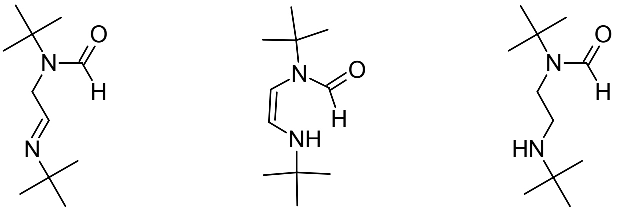
Thermally stable N-heterocyclic carbenes (NHCs) were synthesized as izolable crystalline solids by Arduengo *et al*. In 1991[1]. Generally, carbenes are sensitive towards moisture and thus readily hydrolysable. The computational as well as experimental study of the reactivity pattern including hydrolysis of some diaminocarbenes was reported[2,3]. 1,3-di-*terc*-butylimidazoline-2-ylidene and 1,3-di-*terc*-butylimidazole-2-ylidene react with an equimolar amount of water to give respective hydrolysis product; *t*Bu–NCHCH$_2$–N(CHO)*t*Bu and *t*Bu–NH–CH$_2$CH$_2$–N(CHO)*t*Bu, respectively in THF[2]. Hydrolysis of 1,3-bis(2,6-diisopropyl-phenyl)-imidazole-2-ylidene leads to ring opening and two isomeric products are formed[3].

In this work, the prediction of the structures of *N-tert*-butyl-*N*-[(2E)-2-(*tert*-butylimino)ethyl]formamide (***1***), its tautomer *N-tert*-butyl-*N*-[(Z)-2-(*tert*-butylamino)ethenyl]-formamide (***2***) and *N-tert*-butyl-*N*-[2-(*tert*-butylamino)ethyl]-formamide (***3***) (Scheme 1.) as possible NHCs hydrolysis products was performed. All structures were optimized at RHF and at DFT level with B3LYP, O3LYP and B3PW91 functional.



1 **2** **3**

Scheme 1. Studied compounds as possible NHCs hydrolysis product

Well corelation between experimental and theoretical data was found out for ***3*** by using B3PW91/6-31+G(d,p). From optimized structures ***1-3*** vibrational frequencies calculation at RHF and DFT level were performed. All predictions were computed by using PC GAMESS/Firefly[4].

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REFERENCES

1. Arduengo III A. J., Harlow R. L., Kline M.: J. Am. Chem. Soc. *113*, 361 (1991).
2. Denk M. K., Rodezno J. M., Gupta S., Lough A. J: J. Organomet. Chem. *617*, 242 (2001).

3. Holoczki O., Terleczky P., Szieberth D., Mourgas G., Gudat D., Nyulaszi: Am. Chem. Soc. *133*, 780 (2011).
4. http://classic.chem.msu.su/gran/gamess/index.html [cit. 2009-20-5].

2-P56

CHARACTERIZATION OF POLY[2-METHOXY-5-(3′,7′-DIMETHYLOCTYLOXY)-1,4-PHENYLENEVINYLENE] ELECTROPHORETIC SUSPENSIONS USED FOR THIN FILM DEPOSITION

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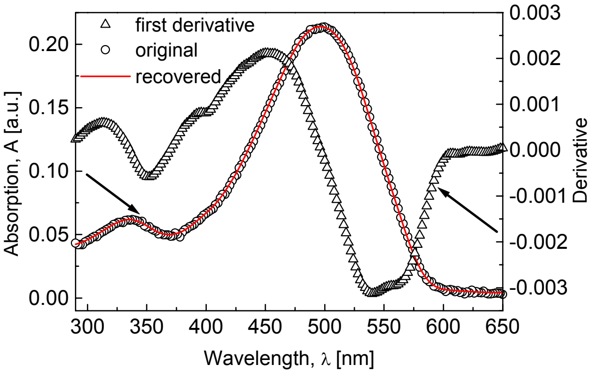
Electrophoretic deposition have been utilized recently for preparation of thin organic films for the microelectronics[1].

The presented work aims to optimize electrophoretic suspension used for thin organic film deposition.

Electrophoretic suspensions (0,0033 g l^{-1}) were prepared by precipitation of poly[2-methoxy-5-(3′,7′-dimethyl-octyloxy)-1,4-phenylenevinylene] toluene solution in acetonitrile.



Scheme 1. Typical absorption spectrum measured in the UV-VIS region, its first derivative calculated by spline procedure and the recovered from the derivative spectrum

The influence of the toluene/acetonitrile ratio on the suspension properties was studied by UV-VIS spectroscopy. For more detail study the first derivatives of the spectra were taken. It was found that bellow 30% of acetonitrile content, the precipitated material is too low to cause film deposition. Between 40% and 70% the deposition takes place from a solution/suspension mixture, while over 70% only suspension phase exists.

This work was supported by South Moravian Region and 7th Framework Programme for Research and Development (grant SIGA 885).

REFERENCES

1. Tada K., Onoda M.: Thin Solid Films *477*, 187 (2005).

3-KL

METALLOMIC STUDY OF THE FREE-LIVING MOUSE MUS SPRETUS FOR POLLUTION ASSESSMENT

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The conventional studies of environmental problems are usually based in the evalutaion of chemical parameters, mainly the presence of toxic metals, such as Cd, Hg or As, and organic contaminants such as pesticides, PAHs PCBs and others. In addition, living organisms have been proposed for a more unambiguous estimation of environmental concern by using a number of molecular biomarkers that reflect the effect of contaminants on cellular metabolism and global homeostasis, these include hundreds of cytochromes P450 (CYPs), dozens of glutathione-*S*-transferases (GSTs), heat-shock proteins and metallothioneins (MTs) that protect organisms from toxic metals[1]. Besides, antioxidant enzymes, such as superoxide dismutases (SODs) and glutathione peroxidases (GPXs), among others can also be considered.

However, the use of biomarkers requires a deep knowledge of their toxicity mechanisms and only provides a biased assessment about the contamination process, since the biological response is defined by a limited and pre-established number of metabolites excluding others whose relationship with pollution is unknown. Therefore, the use of omics can be a good alternative to alternative to biomarkers in environmental contamination assessment[1,2]. Metallomics is perhaps the most recent omic[2], in which metals or metalloids present in many cellular biomolecules are used as markers to track these molecules in complex biological matrices. These techniques make extensive use of high sensitivity elemental detectors as ICP-MS.

A number of bioindicators have been proposed in environmental omic studies, such as the mouse *Mus spretus*, the bivalve *Scrobicularia plana* and the crayfish *Procambarus clarkii*, in which high metabolic activity organs, such as liver and kidney for mammals and digestive gland for invertebrates, have been used. Recently, metallomics has been proposed to evaluate the biological response of free-living mouse *Mus spretus* in areas affected by different grade of contamination[3].

In the present study the up- and downregulation of metal-biomolecules under the action of contaminants have been studied in different áreas of Doñana National Park (southwest Spain) and surroundings, which are affected by differential contamination.

Free-living mice (*Mus spretus*) were captured in Doñana National Park (DNP), southwest Spain. Contaminated areas: "La Rocina Stream" (ROC), "El Partido" (PAR), "El Ajoli" (AJO) and "Matochal" (MAT) affected by inputs from agricultural and industrial activities were sampled for comparison with the non-contaminated point "Lucio del Palacio" (LDP) locatd at the center of the park. Adult animals were sacrified and dissected and cytosolic extract obtained.

The extracts were filtered through 0,2-mm pore size and analyzed by the coupling SEC-ICP-MS, using a Micromist nebulizer online connected to the chromatograph with a PEEK tubing (0,17 i.d. mm). The retention times corresponding to the peaks of standards used for SEC-ICP-MS column calibration are the following: ferritin 12 min, bovine serum albumin (BSA) 14 min, superoxide dismutase containing Cu and Zn (Cu,Zn-SOD) 16 min, metallothionein I containing Cd, Cu and Zn (Cd,Cu,Zn-MT1) 19 min, and reduced glutathione (GSH) 24 min.

The chromatograms of liver and brain extracts from contaminated and non-contaminated areas differ clearly, a Cu-peak with retention time at about 19,6 min was observed in liver extract with higher intensity in mice collected in ROC when compared with samples from the non-contaminated point LDP. This peal can be related with Cu-MT1 that exhibits very similar retention time. However, the peak Cu and Zn-peak at about 32 kDa that can be related with superoxide dismutases exhibits higher intensities in samples from non contaminated points. The As, Cd and Pb exhibit similar chromatograms in liver but the sensitivity of signals are very low. In brain the intensity of the peaks traced by the different elements are lower than in liver which can be attributed to the protective action of the blood brain barrier.

This work was supportedby CTM2009-12858-C02-01 from the Ministerio de Ciencia e Innovación, and the projects P08-FQM-03554 and P09-FQM-4659 from Consejería de Innovación Ciencia y Empresa (Junta de Andalucía). M.González-Fernández and M. Angel García Sevillano thanks the Ministerio de Ciencia e Innovación for a predoctoral scholarship. R. Jara-Biedma thanks to Consejería de Innovación Ciencia y Empresa (Junta de Andalucía) for a predoctoral scholarship .

REFERENCES

1. López-Barea J., Gómez-Ariza J. L. Proteomics *6*, S51 (2006).
2. González-Fernandez M., García-Barrera T., Jurado J., Prieto-Álamo M. J., Lopez-Barea J., Pueyo C., Gómez-Ariza J. L., Pure. Appl. Chem. *80*, 2609 (2008).
3. Gonzalez-Fernandez M., Garcia-Sevillano M. A., Jara-biedma R., García Barrera R., Vioque A., Lopez-Barea J., Pueyo C., Gomez-Ariza J. L.: J. Anal At. Spectrom. *26*, 141 (2011).

3-L1

REFLECTION OF UNDERLYING ROCKS IN CHEMISTRY OF UNDERGROUND AND SURFACE WATERS

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The following experimental site will be used to collect data for the proposed project. The site is involved in ERB (the Euromediterranean Network of Experimental and Representative Basins) described by Tesař[1]. All of them are also involved in GEOMON (Geochemical Monitoring Network), and have been coordinated by the Czech Geological Survey since 1994[2]. The long-term monitored experimental watershed, mentioned above and described below, was selected in order to represent the headwater regions of the Czech Republic.

Since 1983, the chemistry of underground as well as atmospheric waters has been regularly observed. The monitoring includes inflows and outflows of substances to and from the catchment in the form of rainfall, spruce and beech crown throughfall, stem flow (spruce and beech), deposited precipitation, soil water (suction lysimeters), and undergound water (drills).

The catchment in question is also remarkable due to the monitoring of the underground water chemistry and mineralogical and geological analyses carried out in order to clarify the character and intensity of weathering processes in relation to underground water chemistry. Complex investigation of the model catchment also includes the measurement of radon emanation in all monitored geofactors.

The dominant constituents in the LIZ watershed are as follows: migmatitized biotitic and sillimanite-biotitic paragneiss (sporadically with white mica and cordierite), often penetrated by dykes of pegmatite, aplite, and leucocratic granite, with thicknesses from decimeters to tens of meters[3]. The only insole rock present here is quartzite to quartzitic ore (sporadically with clinopyroxene). Paragneiss rocks are resistant to weathering and have lower buffering capacity. What is obvious is the dependence of sorption saturation and fine-grained soil content. The decisive factor in soil assessment from the viewpoint of mineral environment is soil pH, which can be classified, considering the type of water migration environment according to Perelman[4], as an acidic $pH = 4,0–6,5$ to strongly acidic $pH < 4$ water environment. The pH values, which correspond with the degree of buffering of cationic exchange to the degree of buffering of aluminium, are considerably affected by a high content of organic component.

Most soils which formed on the paragneiss substrate are oligotrophic to oligomesotrophic cambisols. Along the streams mainly organic gley soils and pseudogley cambisols developed. The higher elevations are marked by podsolization and by rankers in the places with abundant debris. With increasing depth there are decreases in C_{org}, cationic exchange capacity, and acidity, whereas pH values (H_2O) and pH (KCl) increase. The acidity of surface organic horizons is dominated by the presence of organic acids derived from the humification processes of surface horizons. The reserve of exchange cations (Ca, K, Mg) in the soils is low. Among the main buffering systems is the dissolution of Al secondary minerals. In soils with pH values below 5, the role of the exchange of H^+ for Ca, Mg, and K is only minor[5,6]. On the basis of the determined values of potential (exchange) pH, the soils can be characterized as strongly acidic.

The decrease in cations washed from the LIZ forest catchment into the surface waters indicates a decrease in acidic atmospheric deposition, which means there is less intensive rock weathering. The drop in nitrates and sulphates concentrations also signals that the chemical composition of surface waters is gradually improving in the area of interest.

This work was supported by the grant of the Academy of Science of the Czech Republic No. 6007665806 and research grant of Ministry of Agriculture of the Czech Republic NAZV QH 92298.

REFERENCES

1. Tesař M.: Acta Univ. Carol., Geol. *37*, 57 (1993).
2. Fottová D.: Water, Air, Soil Pollut. *150*, 73 (2003).
3. Oulehle F., McDowell W. H., Aitkenhead-Peterson J. A., Krám P., Hruška J., Navrátil T., Buzek F., Fottová D.: Ecosystems. *11*, 410 (2008).
4. Perelman A. I.: *Geochemistry of Landscapes.* Vysshaya Shkola, Moscow 1975.
5. Reif J., Rezek K., Charvátová S.: Mineralogie a geochemie zvětrávacích procesů za podmínek zvýšené kyselé atmosférické depozice. Úst. nerost. surovin *110*, 116 (1989).
6. Kinkor V.: *Research Report – Geochemical balance of the Jezeri catchment, Krusné hory Mts*. Czech Geological Survey, Prague 1988.

3-L2

WATER – PHYTOPLANKTON – FISH TRANSFER OF SOME HEAVY METALS IN THE DANUBE AQUATIC ECOSYSTEMS OF THE IRON GATES NATURAL PARK

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Located along the Danube River, in the South-Western part of Romania, the Iron Gates Natural Park hosts valuable wetland ecosystems and biodiversity resources (more than 1500 plant species and over 5000 animal species).

Due to a history of heavy metals contamination of the aquatic ecosystems in the region[1], the aim of this study was to quantify the level of some trace elements (Cd, Cu, Pb and Zn) in fish and aquatic plant species and emphasise a possible transfer of pollutants from water to living organisms.

In order to achieve the proposed aim, five herbivorous, zooplankton and predatory fish species (i.e. *Cyprinus carpio, Carassius gibelio, Sander lucioperca*, *Silurus glanis*, and *Aspius aspius*) and three plant species (i.e. *Elodea canadensis, Ceratophyllum* ssp. and *Salvinia natans*) have been chosen as indicators of metal contami-nation. For the fish species, several samples were collected – liver, muscle, gills, eggs – in order to emphasise a possible organotropism (the preferential accumulation of a pollutant in a certain organ) of the analysed elements[2, 3].

The results of the study will also be of a high importance for the local community, as fish is an important resource for the area, being the main component of the local inhabitants diet. Comparison of results obtained for lead and cadmium concentration in fish was performed with the maximum levels for contaminants in foodstuffs established by the European Commission[4]. As essential elements (copper and zinc) are not stipulated in the document, other regulations were used as threshold levels[5].

Two methods with similar limits of detection, selectivity, and sensitivity have been use for target analytes determination: Atomic Absorption Spectrometry (AAS) for water samples and Inductively Coupled Plasma – Optical Emission Spectrometry (ICP-OES) for biological samples, previously digested with concentrated acids (HNO_3 and $HClO_4$) in an open system.

Copper and zinc concentration values were usually situated below the threshold level for human consumption (20 $\mu g.g^{-1}$ and 50 $\mu g.g^{-1}$ respectively). Issues related to the fish consumption risk by humans were generated by the toxic elements, as several samples exceed the admitted limits for cadmium and zinc.

The concentration of metals in *Cyprinus carpio* were comparable with the results reported in a similar study performed in 2008 on individuals collected from Grote Nete River[6], Netherlands and with those of a captivity study in Belgium in 2009[7]. In the case of *Aspius aspius* and *Sander lucioperca*, the metal concentration levels were comparable to those registered in Věstonice Reservoir[8].

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REFERENCES

1. Matache, M., Pătroescu, C., Pătroescu-Klotz, I. V.: Rev. Chim. *53*, 623 (2002).
2. Kojadinovic, J., Potier, M., Le Corre, M., Cosson, R. P., Bustamante, P.: Environ. Pollut. *146*, 548 (2007).
3. Mieiro, C. L., Pacheco, M., Pereira, M. E., Duarte, A.C.: Arch. Environ. Contam. Toxicol., in press DOI: 10.1007/s00244-010-9591-5
4. European Commission Regulation No 221/2002: Official Journal of the European Communities *L37*, 4 (2002).
5. Ministry of Agriculture, Fisheries and Food, Aquatique Environment Monitoring Report No. 44. Technical Report. Directorate of Fisheries Research, Lowestoft, UK
6. Reynders H., Bervoets L., Gelders M., De Coen W. M., Blust R.: Sci. Total Environ. *391*, 82 (2008).
7. Bervoets L., VanCampenhout K., Reynders H., Knapen D., Covaci A., Blust R.: Ecotoxicol. Environ. Saf. *72*, 720 (2009)
8. Kenšová R., Čelechovská O., Doubravová J., Svobodová Z.: Acta Vet. Brno *79*, 335 (2010).

3-L3

INFLUENCE OF WATER EROSION PROCESSES ON SEDIMENT AND NUTRIET TRANSPORT FROM SMALL AGRICULTURAL CATCHMENT AREA

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Erosion processes in watersheds belong to serious ecological and economical problems because of negative consequences in terms of soil and water deterioration as well as on the environment as a whole. The soil particles washed down by water erosion are the biggest pollution factor due to the amount and volume. Sediments are the product of a selective process. In this process smaller and lighter particles are separated from eroded soil and taken away by water at the first. This means that the sediments contain a higher amount of organic, clay and silt particles than the the original soils. Washed down sediments consist mainly of particles smaller than 0,05 mm (40 - 90% of mixture).[1] Sediments detached by the erosion bind nutrients (particularly nitrogen and phosphorus), that can significantly affect the balance of the aquatic ecology, resulting in eutrophication of lakes and rivers.

More studies in Czech Republic have been focused on the assessment of soil erosion, based upon principles and parameters defined in the Universal Soil Loss Equation, but neither from them has dealt with nutrient transport assessment in consequence of water erosion. Researches deal with negative effects of water erosion on soil degradation and watercourses pollution in different range from small research area to big river basin area[2-4]. Advantage of small watercourses (to 10 km^2) is relative easiness of exact measurments of hydrologic and meteorological characteristics in the environment as well as accurate definability of natural and anthropic conditions. The most intensive water erosion in small agricultural watercourses is caused by snow melting or by local torrential rainfall[5]. This contribution deals with the nutrient transport from eroding upland fields by the small water basin.

The study of nutrient transport assessment in consequence of water erosion has been realized in vicinity of small catchment in cadastral area Hustopeče situated at the Starovicky stream in the Sout Moravia region. Experimental catchment Hustopeče was established on a thalweg of sloped arable land area of cca 29 ha severaly affected by erosion. It belongs to very warm and dry region. Soil types consist mainly from modal chernozem on loess.

The Thomson triangular spillway has been built up on the thalweg for experimental purposes. This spillway is equipped by flow and precipitation (S profile) measurement instruments. Water sampling is ensured by the automatic sampler integrated on bank upon the spillway. Water from increasing flow off wave part gradually fills up bottles sorted above them. Water samples are taken immediately after extreme precipitation-flow events, when one bottle of automatic sediment sampler is filled at least. Samples are analyzed for the total undissolved matter content in laboratory.

Excluding precipitation, runoff and water level is automatically continuously monitored using ultrasonic soundings and the results are continuously registered in 10 - minute step in data logger memory.

Catchment flow off has been registered in 7 episodes in 2008-2011. Snow melting was the most significant event for transport of undissolved matter and soil losses on the beginning of the year 2009. The relationship between the selected parameters were examined through correlation and regression methods.

This work was supported by Ministry of Agriculture, grant No. 0049002702 and by Ministry of Agriculture NAZV, grant No. QI91C200.

REFERENCES

1. Janeček M.: *Ochrana zemědělské půdy před erozí.* VÚMOP, Praha 2007.
2. Bečvář M.: Soil Water Res. *1*, 23 (2006).
3. Kvítek T., Doležal F.: Acta Hydrol. Slov. *4*, 255 (2003).
4. Owens P. N., Collins A. J.: *Soil erosion and sediment redistribution in river catchments.* CABI, Oxfordshire 2006.
5. Fulajtár E., Janský L.: *Water erosion of soil and erosion control.* Research institute of soil science and conservation, Bratislava, 2006.

3-L4

PERFLUORINATED COMPOUNDS LEVELS IN WILD FISH FROM THE CZECH REPUBLIC

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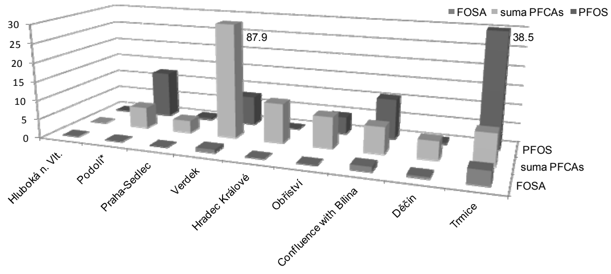
Perfluorinated compounds (PFCs) belong to one of the most important groups of "emerging" contaminants. Their presence in the environment has increased concerns about the potential health risk to organisms associated with the exposure to these contaminants especially in foodstuffs. Due to their unique properties PFCs are used in a variety of applications (surface treatment, fire-fighting foams, textile industry, paper wrappers, etc.). Any regulation over PFCs in foodstuffs has not been published yet. The European Commission has taken an action and in 2010 published Recommendation 2010/161/EU on the monitoring of perfluoroalkylated substances in food in response to occurrence of PFCs over the world[1]. The document recommends to examine three commonly reported compounds, which are perfluorooctane sulfonate (PFOS), perfluorooctanesulfonamide (FOSA) and perfluorooctanoic acid (PFOA), together with other perfluorinated compounds belonging to groups like perfluorocarboxylated acids (PFCAs), perfluorosulfonic acids (PFSAs), perfluorosulfonamides (FOSAs, FOSEs), perfluorotelomeric alcohols (FTOH) and polyfluoroalkyl phosphate surfactants (PAPS) in foodstuffs reflecting consumption habits.

Within this study, fish tissues such as muscle and liver were monitored for the occurrence of the 23 perfluorinated compounds. The common fish species traditionally living in the Czech rivers like bream (*Abramis Brama*), chub (*Leuciscus cephalus*), crucian (*Carassius Carassius*) and roach (*Rutilus rutilus*) were collected from June to August 2010. The sampling sites Bilina River (1), and the other on the Elbe River (5) as well as on the Vltava River (3) were distributed along their streams to cover both non-industrial and industrial areas. This kind of a large scale study was conducted in the Czech Republic for the first time. Finally, almost 100 pooled samples in three categories (*i*) 100–300g (*ii*) 300–900 and (*iii*) more than 900g were analysed.

The simple and fast analytical procedure for determination PFCs consists of methanol extraction followed by clean-up of a crude extract using activated charcoal coupled to the LC-MS/MS technique was employed[2].

The results found within this study confirmed our assumption that the lowest concentrations were found in the non-industrial locality such as Hluboka nad Vltavou on the Vltava River. From our previous monitoring study, it was identified as a relatively 'clean' area as shown in Figure 1.

In our preliminary studies dealing with the occurrence of PFCs, mainly linear isomer of PFOS (L-PFOS), PFOA and FOSA in the Czech aquatic ecosystem were monitored. Only L-PFOS was identified and sometimes also FOSA, but PFOA was not detected. In contrast to our previous finding in this survey also PFCAs were measured at relatively high concentrations mainly those with longer chains C9–C14 was determined in 100% of examined samples as well as PFOS, PFDS (98%) and FOSA. The usually studied PFOA was detected only in 40% of samples.



* - bream

Figure 1. PFCs concentration in chub muscle from different localities

Typically, PFOS was found at the highest level reached up 76 and 312 μg kg^{-1} of roach from the Bilina River in the muscle and liver, respectively. In comparison of branched (Br-PFOS) with the L-PFOS isomer, the latter one was dominant which correspond to the composition of technical mixture (79% of L-PFOS and 21% Br-PFOS). In spite of that, the composition of them a little varied depending on fish species.

The most abundant PFCAs were perfluorodecanoic (PFDA) and perfluorododecanoic (PFDoA) acids. In general, the concentration of ΣPFCAs did not exceed the level 5 and 14 μg/kg in muscle and liver, respectively excluding samples of chub' and bream' tissue collected in the locality Verdek (Elbe River). In this sampling-site the concentration ranged from 5 to 35 μg kg^{-1} of C10–C14 in muscle and from 13 to 100 μg kg^{-1} of C10–C14 in liver.

The potential sources of these chemicals could be the municipal waste water treatments (WWTPs), waste dumping sites or the industry located near the streams[3]. To our knowledge, there is no chemical industry producing PFCs in the Czech Republic. On the other hand, it can be assumed that the use of these compounds as ingredients in various technological processes might be responsible for documented emissions.

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REFERENCES

1. The European Commission recommendation (2010/161/EU) on the monitoring of perfluoroalkylated substances, Official Journal of the European Union, 2010.
2. Hrádková P., Pulkrabová J., Kalachová K., Hloušková V., Tomaniová M., Poustka J., Hajšlová J.: Arch. Environ. Contam. Toxicol., submitted.
3. Möller A., Ahrens L., Surm R., Westerveld J., van der Wielen F., Ebinghaus R., de Voogt P.: Environ. Pollut., *158*, 3243 (2010).

3-L5

BIOACCUMULATION OF SOME ORGANOCHLORINE PESTICIDES IN BIOLOGICAL SAMPLES FROM THE LOWER PRUT FLOODPLAIN NATURAL PARK

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The concentration of some organochlorine pesticides (DDT and metabolites – DDE, DDD, HCH isomers – alpha, beta, gamma, delta) has been studied in biological samples from the Lower Prut Floodplain Natural Park, a 8,000 ha protected area established in 2004 along the inferior sector of the Prut River, prior its junction with the Danbue River[1].

Romania is a country with a long history of producing and use of organochlorine pesticides, the removal of obsolete pesticides being finished only in 2005, from more than 100 deposits located all over the country[2].

The collected samples included mussels (soft tissue), fish (liver, gills, muscle) and bird eggs (whole eggs and chorio-alantoid membranes) and feathers.

Bird eggs and feathers were collected in order to use a non-invasive approach of the pesticides acumulation in the bird body, in order to limit the stress on the individual[3]. Eggs samples were collected directly from from the nests or its close vicinity immediatly after hatchling, while feathers were collected from the nest, captured individuals or nesting spots.

For fish, several body parts were analysed, for a more accurate assessment of the pollutant concentration in the fish body, but also for emphasising a possible organotropism of pollutants[4]. Mussel species identified in the field and collected for pollutants determination were *Sinanodonta woodiana* and *Unio pictorum*.

Chemical analyses were performed using Gas-Chromatography with Electron Capture Detector.

Similar to other studies reporting on organochlorine pesticides presence in Romanian wildlife[5-7], pollutants from the DDT group are the dominant organohalogenated compounds in the collected samples. Both DDE and DDD metabolites appear, so DDT degradation takes place following both aerobic and anaerobic path.

From the HCH group, gamma-HCH had shown the highest percentage, showing that lindane (with more than 95% of gamma isomer) was used in the field and not technical HCH (in which the gamma isomer exhibits not more than 12%). The lesser presence of this pesticide, although used more recently than DDT, might be a consequence of a higher water solubility, vapor pressure and biodegradability[8].

For the fish samples, gills were found as the most exposed organ, whilst the muscle samples contained the lowest pesticides concentration. For the bird samples, differences appear depending on the species feeding habits.

This study was supported by the Romanian Ministry of Education, Research, Youth and Sport through the PN II project no. 32111/2008 and the European Commission – Environment Directorate through the LIFE Nature project LIFE05/NAT/RO 000155.

REFERENCES

1. Ioja I. C., Savulescu I.: Comunicări de Geografie *25*, 541 (2007).
2. Stroebaek N., Dezsy S.: Environ. Eng. Manag. J. *5*, 757 (2006).
3. van der Oost R., Beyer J., Vermeulen N. P. E.: Environ. Toxicol. Pharmacol. *13*, 57 (2003).
4. Mahmoud N., Dellali M., El Bour M., Aissa P., Mahmoudi E.: Ecol. Indic. *10*, 696 (2010).
5. Aurigi S., Focardi S., Hulea D., Renzoni A.: Environ. Pollut. *109*, 61 (2000).
6. Covaci A., Gheorghe A., Hulea O., Schepens P.: Environ. Pollut. *140*, 136 (2006).
7. Matache M. L., Rozylowicz L., Hura C., Matache M.: Organohalogen Compd. *68*, 728 (2006).
8. Sarkar S. K., Bhattacharya B. D., Bhattacharya A., Chatterjee M., Alam A., Satpathy K. K., Jonathan M. P.: Environ. Int. *34*, 1062 (2008).

3-L6

ASSESSING HEAVY METAL CONTAMINATION IN THE VICINITY OF GOLD MINE, LOEI PROVINCE, THAILAND

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Fifty eight metal prospects including Au in the area of Loei-Chaenkain, northeastern part of Thailand, were investigated during the year 1963 to 1966[1]. Ore deposit was a Cu-Au bearing quartz vein. Average grade of the ore vein was 2-3 g Au ton^{-1}. Open pit gold mine operations in Loei Province began in 2006. Soon after, questions from local people about the possibility chemical leakage from gold mine processing arised. Due to possible deterioration of the environment near the mine, where there is a lot of agricultural land and rice fields, monitoring of metals concentration and concentration of other chemicals used in gold mining and processing in the vicinity of the mine is needed. The biggest problem is posed by heavy metals (Cd, Cu, Pb, As, Mn) and cyanides[2-4]. Therefore, objectives of this study were to evaluate and to assess concentration of heavy metals (As, Cd, Cu, Mn, Pb) and CN- in water and soil derived from mining activity, and to investigate the soil-water relationship in terms of availability of metals in the vicinity of gold mine.

Soil and water samples from seven locations were taken in October 2010. Three of them were taken from the site of gold mine (tailing storage pond, water slump and waste storage area), others were taken from the paddy field and crops area nearby. The concentration of Cd, Cu, Pb and Mn was determined using Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP-OES, Optima 7300 DV, PerkinElmer). Arsenic was determined by atomic absorption spectrophotometer with hydride generation technique (PerkinElmer, AAnalyst 800). Total CN in water was determined by reaction with chloramine-T at pH < 8 and pyridine-barbuturic acid. The pH value of water and soil was also measured during sampling period. Quality control of analysis was conducted using the ICP-multi-element standard solution IV CertiPUR® (Merck, Lot.-No.HC760060) as a spike sample.

Quality control (QC) samples were measured in terms of percent of recovery. Results indicated that the percent of recovery in soil and surface water samples spiked with 0,4 ppm of standard solution felt into 80-110 percent, which was in agreement with Horwitz's recommendation[5]

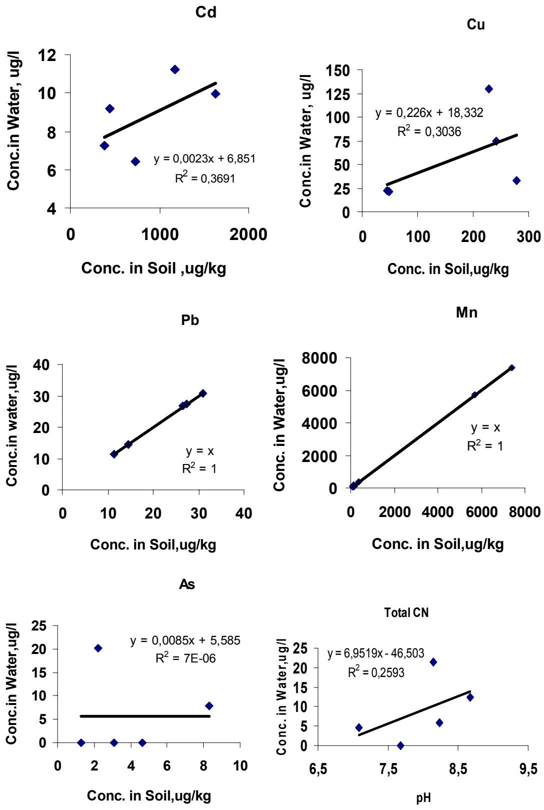

Fig. 1. Relationship of metal concentration in soil and in water

The highest concentrations of metals and CN in surface water from the vicinity mine area were 11,2; 129,3; 7385,7; 30,9; 20,2 and 21,36 µg l^{-1} for Cd, Cu, Mn, Pb, As, and total CN, respectively. In soil samples, the highest measured concentrations were 1627,3; 241,4; 7385,8; 30,9 and 8,31 µg kg^{-1} for Cd, Cu, Mn, Pb, and As, respectively. The pH of water samples varied in the range of 7,1-8,4. Copper and total CN concentrations in tailing storage pond were 2,379 and 11,942 µg l^{-1} respectively. The Cd, Cu and As concentrations in tailing storage pond soil were 6644; 454 and 538 μg kg^{-1}, respectively.

Relationship between concentrations of metal in soil and in water is shown in Figure 1. For total CN, relationship of concentration in water and pH of water was plotted. The obtained results revealed that the concentration of heavy metals in water had a positive correlation with those in soil. Correlation coefficients of Pb and Mn were among high statistically significant as R2 equals 1. However, Cd, Cu and As correlation coefficients were not statistically significant as well as total CN concentration and pH.

Results from this study will be used to further investigation regarding to possible application of alternative technologies of soil and water treatment and to sustainable environmental management.

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1. Jacobson B. S., Pierson C. T., Danusawas T., Japakasetr T., Inthuputi B., Siriratanamongkol C., Prapassornkul S., Pholphan N., in the book: *Geological Survey Professional Paper 618*, United States Government Printing Office, Washington, USA 1969.
2. Chon H., Ahn J. S., Jung M. C.: Environ. Geol. *38*, 101 (2005).
3. Eisler R. in the book: *Handbook of chemical risk assessment: health hazards, plants, and animals, Volume 1: Metals*. Lewis Publishers, Boca Raton, Florida 2000.
4. Kim J. Y., Kim K. W., Lee J. U., Lee J. S., Cook J.: Environ Geochem. Health. *24*, 215 (2002).
5. http://www.aoac.org/dietsupp6/Dietary-Supplement-web-site/HorwitzValid.pdf. [cit. 2011-11-01].

3-L7

EFFECT OF LAND CONSOLIDATION AND WATERSHED CONSERVATION MANAGEMENT ON WATER QUALITY IN HUBENOV WATER RESOURCE

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The aims of this study is to explore the relationship between the soil and water conservation through the process of Land Consolidation and changes in concentration ($N\text{-}NH_4$, $N\text{-}NO_3$ and total P) in surface waters of Maršovský stream as a tributary of Hubenov water resource dam. The Maršovský stream catchment (hydrological rank number 4-16-01-028) is located in Bohemian-Moravian highlands, close to the Jihlava city. The catchment area is 22,3 km^2. The goal was to find out how nutrient concentration has responded to changes in Land Use through the Land consolidation and watershed conservation management. The case study area Hubenov water resource dam was selected as a case study mainly for its natural conditions and high risk of soil erosion and sediment transport. Relief, geomorphology, the present state of the complex system of soil properties, the types of agricultural farming practices and land use, are all contributing to accelerated soil erosion with all its negative impacts on the environment. Apart from soil degradation and related decrease in soil fertility, water erosion causes further adverse effects. One of these effects is water pollution due to released and transported substances. Some soil particles are deposited at the slope base but most of them are transported with the water to the hydrographic network where they form the major part of sediment runoff (bed load). The sedimentary load causes silting in natural as well as artificial watercourses and reservoirs. Chemical substances (especially nutrients) linked to soil particles penetrate into surface water resources, representing a considerable threat to water resources use. The sources of these substances are commercial fertilizers and pesticides. A high content of nitrogen together with phosphorus from nonpoint pollution cause eutrophication and decrease water quality of water reservoirs, which impairs their use for water resource including some other functions. The determina-tion of the soil amount transported to the watercourse was carried out on the basis of the assumption of equal sedimentation. To some extent, this assumption means a simplified view. A significant part of the catchment area is suitable for the accumulation of water and serves as protection zone of a drinking water reservoir. This dam is a source of drinking water for the city Jihlava and other settlements. This makes it very important to find appropriate solutions for the prevention of the soil degradation in the selected case study area. The best decision was watershed conservation management through the process of Land Consolidation. In the case study area of Hubenov water resource dam the following long-term measures are applied by farmers: conversion of arable land to grassland (whole fields or waterways), creation of field banks and field roads when reshaping the field size and patterns, grass infiltration and buffer strips. The important from point of view of water quality were restrictions on the max. amount of N and P - fertilisation. The total amount applied should not exceed the equivalent of 170 kg N/ha. This rule is part of the prepared Action Plan. The same applies to phosphorus. This rule is applied regularly in the case study area. Protection measures such as restrictions on the maximum amount of manure, N and P fertilisation were applied in the case study area according to a special project based on detailed inputs of data using parametric methods. The soil loss is determined according to the method on the basis of DME (digital model of elevation) with the use of USLE 2D program and LS algorithm by McCool[1].

Monitoring from 1986-2010 for Hubenov Dam Water resource was established to continue collection water quality data ($N\text{-}NH_4$, $N\text{-}NO_3$ and total P). Monitoring provide a longer time frame overview by which to evaluate water quality changes in the water resources. The relationship between the selected parameters was examined through correlation and regression methods. Results from these monitoring studies indicate significand decreasing linear trend of $N\text{-}NH_4$, $N\text{-}NO_3$ and total P concentration since good soil and water conservation during the process of the Land consolidation (started 1993 and finished 1995).

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REFERENCES

1. http://geo.kuleuven.be/geography/modelling/erosion/usle2d/ [cit. 2010-09-03].
2. Lal R.: Land Degrad. Dev. *12*, 519 (2001).
3. Kovar J. L., Pierzynski G. M.: Southern Cooperative Series Bulletin *408*, 1 (2009).
4. Walling, D. E.: J. Hydrol. *65*, 209 (1983).

3-P1

Se (IV) AND $MeHg^+$ DETERMINATION BY HPLC-ICP-MS IN NATURAL WATERS AFTER HOLLOW FIBER PRECONCENTRATION

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Is known that selenium and mercury have several interactions and some seleno-compounds inhibit toxic effects of mercury compounds[1-4]. For this reason it is necesary to develop new methodologies that allows the simultaneous determination of these compounds in samples with environmental or health significance in which this species are presents at very low concentrations.

In the present study a new speciation method based on the use of hollow fiber for preconcentration and HPLC-ICP-MS for analysis has been developed for Se (IV) and $MeHg^+$ mixtures.

For this purpose Se (IV) was derivatized with o-phenylenediamine to form the corresponding piazoselenol to promote the preconcentration of this specie in the non-aqueous solvent present inside the hollow fiber[5]. The preconcentracion was carried out using a liquid-liquid extraction by U-shape hollow fiber configuration with a syringe connected to both ends. Finally, the separation of the species was carried out using two different mobile phases through a C18 column and detected with ICP-MS with hidrogen as collision gas for $^{40}Ar_2$ interference suppression.

The extraction efficiency of the compounds under study was satisfactory matching or improving those present in the literature for similar applications. The application of the procedure to the analysis of natural waters provides satisfactory results.

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REFERENCES
1. Suzuki K. T., Ogra Y.: Phosphorus Sulfur. *135*, 171 (2001).
2. Chen R. W., Whanger P. D., Fang S. C. Pharmacol. Res. Commun., *6*, 571 (1974).
3. Komsta-Szumska E., Chmielnicka J.: Arch. Toxicol. *38*, 217 (1977).
4. Cikrt M., Bencko V.: Toxicol. Lett., *48*, 159 (1989).
5. Saleh A., Yamini Y., Faraji M., Shariati S., Rezaee M.: J. Chromatogr. B. *877*, 1528 (2009).

3-P2

A NEW APPROACH FOR DETERMINATION OF POP'S IN WATER BY HF-LPME

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Nowadays, the use of plastics, flame retardants and several products to fight pests has led to expansion in the nature organohalogen compounds with high toxicity and persistence in the environment. Because of the importance of the presence of halogenated organic compounds in environmental samples and food, new analytical procedures for a sensitive, accurate and reproducible determination are mandatory.

The use of extraction techniques for organic pollutants based on HF-LPME for biological, environmental and food[1-3] samples has became the method of choice in a great number of works due to the significant advantages offered by this technique. We propose a new approach for hollow fiber membrane which is simple, inexpensive and accurate for the determination of organohalogenated contaminants in water. The new system has been applied successfully to the determination of 40 different POP's including pesticides, PCB's and PBDE's.

The proposed methodology has been optimised using a central composite design of the parameters that significantly affect the extraction efficiency. The combination of the response of all the analytes has been simultaneously optimised using a novelty chemometric appraisal. The optimisation allows the efficient extraction of all the analytes in about 37 minutes.

The detection limits obtained are in the range of $ng.l^{-1}$, recoveries around 100% and the enrichment factors are very high in comparison with other published studies. Important analytical features as quality and accuracy reach with the propossed analytical method a % RSD less than 17% for all analytes.

The approach is very simple and can be used for routine analysis of POPs in food and biological samples.

This work was supported by "Consejería de Innovación Ciencia y Empresa (Junta de Andalucía)" (projects P08-FQM-03554 and P09-FQM-04659) and the Spanish "Ministerio de Ciencia e Innovación" project CTM2009-12858-C02-01

REFERENCES
1. Sarafraz-Yazdi A., Amiri A.: Trends Anal. Chem. *29*, 1 (2010).
2. Ghambarian M., Yamini Y., Esrafili A., Yazdanfar N., Moradi M.: J. Chromatogr. *1217*, 5652 (2010).
3. Pedersen-Bjergaard S., Rasmussen K. E.: J. Chromatogr. A *1184*, 132 (2008).

3-P3
ISOPROSTANES AS MARKERS OF OXIDATIVE STRESS

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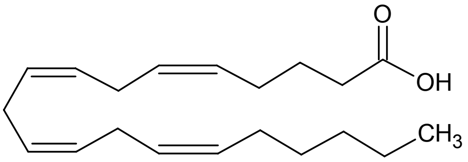
Oxidative stress is an imbalance between oxidants occurrence in organism and its antioxidant defense. Oxidants which include a reactive oxygen species (ROS), reactive nitrogen species (RNS) and free radicals are capable of inflicting injury on membrane lipids, proteins and nucleic acids mutation. They are involved in carcinogenesis, inflammation and degenerative diseases. Oxidative stress is also apparent in pathology associated with aging process [1,2].

Direct determination of oxidants is difficult, because these molecules are short-lived and highly reactive. Therefore the oxidative stress is assessed by determination of secondary products like malondialdehyde, 4-hydroxynonenal or isoprostanes. Isoprostanes are group of prostaglandin-like compounds that are formed via non-enzymatic radical oxidation of polyunsaturated fatty acids with at least four double bonds. One group that is formed following the auto-oxidation of arachidonic acid (Scheme 1) (F_2-iPs) consists of four classes of prostaglandin $F_2\alpha$ isomers ($PGF_2\alpha$)[3]. As a stable endproducts they are widely accepted as reliable markers of oxidative stress in biological samples such as urine, plasma, exhaled breath condensate or tissues (brain, liver, etc.)[4].

There are many types of instruments for monitoring the markers of oxidative stress. One of the most useful methods is determination of isoprostanes by HPLC with tandem mass spectrometric detection (HPLC-MS/MS).



Scheme 1. Arachidonic acid structure

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REFERENCES
1. Ahmed R. G.: Med. J. Islam. Acad. Sci. *15,* 129 (2005).
2. Rice-Evans C., Burdon R.: Prog. Lipid Res. *32,* 71 (1993).
3. John A. Lawson L. A., Li H., Rokach J., Adiyaman M., Hwang S. W, Khanapure S. P., Fitzgerald G. A.: J. Biol. Chem. *273*, 29295 (1998).
4. Gao L., Zackert W. E., Hasford J. J., Danekis M. E., Milne G. L., Remmert C.: J. Biol. Chem. *278,* 28479 (2003).

3-P4
OXIDATIVE STRESS ASSESSMENT OF SALMON EGGS

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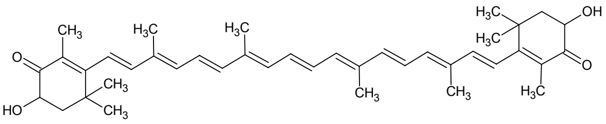
This study deals with the oxidative stress assessment of salmon eggs. M74 yolk-sac fry mortality syndrome in Baltic salmon is a result of combined environmental load of anthropogenic substances like organochlorine pollutants and their metabolites[1]. These compounds cause depletion of antioxidant defence which result in the presence of oxidative stress. The eggs of M74-offspring-producing females have lower astaxanthin levels and more oxidized fatty acids than eggs of females producing healthy offspring[2].

Freshly laid eggs, eyed embryos and non-viable eggs were used to test a more general hypothesis that egg viability can be affected by susceptibility to oxidative stress, either through the specific fatty acid concentration and/or the antioxidant capacity of the eggs.

The antioxidant defence is determined as the level of astaxanthin (Scheme 1) using the HPLC with UV detection at 474 nm. The GC/MS was used for the determination of fatty acid concentration in fish eggs samples after their methyl-esterification.

Higher content of PUFAs was observed in non-viable fish eggs than in eyed embryos. However, no significant difference of astaxanthin levels was found between those two samples. Arachidonic acid, PUFA from which prostaglandin $F_2\alpha$ isomers are formed via non-enzymatic radical mechanism, was found in all samples. The highest level of arachidonic acid was observed at freshly laid salmon eggs.



Scheme 1. Astaxanthin

This work was supported by MŠMT ČR, grant No. FCH-S-11-2; Education and Culture Lifelong Learning Programme ERASMUS; HIE Scottish Funding Council Promoting further and higher education; ERDF Investing in Your Future.

REFERENCES
1. Pickova J., Dutta P. C., Pettersson A., Frøyland L., Kiessling A.: Aquaculture *227*, 63 (2003).
2. Vuori K. A., Kanerva M., Ikonen E., Nikinmaa M.: Environ. Sci. Technol. *42*, 2668 (2008).

3-P5

SEASONAL DYNAMICS OF HEAVY METALS IN VEGETATION IN CONSTRUCTED WETLANDS FOR WASTEWATER TREATMENT

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Constructed wetlands are very complex systems, which are used for wastewater treatment, especially for domestic and municipal wastewater. Recently, increasing interest in the application of those systems for industrial and agricultural wastewater has been observed[1]. Constructed wetlands have been extensively monitored with focus on removal of organics, suspended solids, nutrients and microorganisms, whereas knowledge about the metals removal is still quite limited[2]. Only few studies on the accumulation of heavy metals in plants of constructed wetlands have been completed[3]. Information about the seasonal dynamics of heavy metals in the vegetation in constructed wetlands is entirely missing[2].

The aim of this work is to evaluate the seasonal dynamics of heavy metals in the biomass of *Phragmites australis* and *Phalaris arundinacea* in constructed wetlands. The plant biomass from the appropriate constructed wetlands will be harvested bimonthly and analyzed for heavy metals using the ICP-OES. In order to evaluate accumulation factors, also concentrations of monitored elements will be evaluated in the wastewater. The result should identify the period of maximum accumulation (standing stock) of different metals in the biomass and consequently the optimal time for vegetation harvesting in order to achieve maximum removal when heavy metals are the treatment target. This knowledge will contribute to more effective removal of heavy metals from wastewater.

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REFERENCES

1. Chen T. Y., Kao C. M., Yeh T. Y., Chien H. Y., Chao A. C.: Chemosphere *64*, 497 (2006).
2. Vymazal J., Kröpfelová L., Švehla J., Štíchová J.: Ecol. Eng. *36*, 939 (2010).
3. Lesage E., Rousseau D. P. L., Meers E., Tack F. M. G., De Pauw N.: Sci. Total Environ. *380*, 102 (2007).

3-P6

DEVELOPMENT AND OPTIMISATION OF SPE/CZE METHOD FOR THE ANALYSIS OF NON-STEROIDAL ANTI-INFLAMMATORY DRUGS FROM WATER

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Presented study builds on actual issue of the presence of pharmaceuticals in environmental compartments and on the necessity to apply new sufficient sensitive analytical methods for their determination. The presence and increasing concentrations of pharmaceuticals in the environment have been observed during last decades. The most heavily loaded environmental compartment is the aquatic one due to continuous discharge of these compounds into sewer system and their insufficient elimination during cleaning processes in wastewater treatment plants.[1] Pharmaceuticals are pollutants, which do not exhibit acute toxicity for water organisms, but their continuous release into water may cause chronic adverse effects on these organisms. Due to this potential threat it is necessary to control their presence in aquatic environment. Therefore, there is a need of reliable analytical methods for easy determination of these compounds. One of the most common extraction method for extraction of many compounds from water is solid phase extraction (SPE)[2,3].

This study is focused on the development of SPE-based method for extraction of selected acidic drug residues from water environment. Target drugs have been chosen from the group of non-steroidal anti-inflammatory drugs: salicylic acid, diclofenac, ibuprofen, ketoprofen, naproxen and acetaminophen. Optimisation of the SPE method covered effeciency of extraction, depending of pH and volume of loaded sample and volume of elution solvent. In addition, this study presents application of capillary zone electrophoresis as a very suitable analytical method for final analysis of these residues. Experiments in the range of this study cover experimental development of these methods for their possible use not only for determination of selected drug residues, but also for determination of other similar compounds from real samples of water.

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REFERENCES

1. Hernando M. D., Mezcua M., Fernández-Alba A. R, Barceló D.: Talanta *69*, 334 (2006).
2. Lin W. Ch., Chen H. Ch., Ding W. H.: J. Chromatogr. A *1065*, 279 (2005).
3. Fatta, D., Nikolaou, A., Achilleos, A., Meric, S.: Trend. Anal. Chem. *26*, 515 (2007).

3-P7

SOIL TO PLANT TRANSFER OF METALS IN BAIA MARE AREA, NW ROMANIA

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Topsoil samples (1-10 cm) and plants growing on them were collected from four areas in Baia Mare city and surroundings (three polluted areas and one reference area) and several metals (Cu, Zn, Pb, Cd, Ni, Cr, Co, Mn, Sn) were analyzed by inductively coupled plasma atomic absorption spectrometry. Baia Mare city is well known for its anthropogenic historical pollution of the soil due to the mining and metallurgical activities[1].

The pollution load index (PLI) was calculated as the nth root of the product of the n CF[2]: PLI = $(FC_1 \text{ x } FC_2 \text{ x } ….. \text{ x } FC_n)^{1/n}$, where n is the number of samples, and CF is the contamination factor expressed as the ratio obtained by dividing the concentration of each metal in the soil by the baseline or background value (background values are the content of metals in soil from a reference area).

The soil to plants transfer factors (TF) for the investigated metals were calculated and discussed in order to assess the "true" pollution level of the soil according to the possibility of metals entrance in the food chain. Soil to plant transfer of metals is the major way of human exposure to soil contamination[3]. TF values ranged between 0,1 for Pb and 16,466 for Cd. Average TF for each metal calculated for all the sampling locations in Baia Mare city decrease following the sequence: Cd > Ni > Sn > Cr ≅ Zn ≅ Mn > Co ≅ Cu > Pb. Similar results were obtained in several researches[4, 5].

Multivariate analysis, such as cluster analysis were conducted to classify metals of different sources. The statistical analysis of data was carried out using the software packages Statgraphics.

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REFERENCES

1. Oprea G., Michnea A., Mihali C., Senila M., Roman C., Jelea S., Butean C., Barz C.: Am. J. Environ. Sci. *6,* 33 (2010).
2. Rashed M.N.: J. Hazard. Mater. *178,* 739 (2010).
3. Cui Y. J., Zhu Y. G., Zhai R. H., Chen D. Y., Huang Y. Z., Qiu Y., Liang J. Z.: Environ. Int. *30*, 785 (2004).
4. Chojnacka K., Chojnacki A., Gorecka H., Gorecki H.: Sci. Total Environ. *337*, 175 (2005).
5. Intawongse M., Dean J. R.: Food Addit. Contam. *23*, 36 (2006).

3-P8

HEAVY METALS CONTAMINATION OF SELECTED BRNO CITY LOCALITIES

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This article is focused on the estimation of soil contamination by heavy metals in urban area of Brno. Samples were collected in five periods from October 2009 to January 2011, to represent different seasons and were made provision for their impact on the concentration of heavy metals in examined soil. At the time locations were chosen so that it can reach to compare also with resent collected results. These thirty-five locations represent different level of metal contamination, starting with busy traffic area, where the concentration of means was high continuing to areas located around habitation or parks, where present of heavy metals almost doesn't suppose. Given locations was placed in urban area of Brno to represent various distances from centre of the town up to his surrounding.

The soil samples were treated by method quartation and grain size 2 mm for analytical determination of heavy metals in soil. These samples were extracted by the help extraction surfactant nitric acid with concentration 2 mol dm^{-3}. This process proceeds in time duration 16 hours at laboratory temperature and continual shaking[1].

So prepared solution contained several form of heavy metals. In period from October 2009 to March 2010 total amounts of copper, cadmium, mercury and lead were determined. In other period from October 2010 to January 2011 the same elements were established but also with nickel and zinc. The all of samples were, measured pH, because it is one of the important factors which influence mobility of heavy metals and their bioavailability.

The concentration of copper and lead were measured by flame atomic absorption spectroscopy (F-AAS, SpectrAA 30), and concentrations of cadmium were determined by electrothermal atomic absorption spectroscopy (ET-AAS, AAS ZEEnit 60). The Advanced Mercury Analyzer (AMA 254) was used for the determination of mercury.

The results were compared not only with limits given by decree no. 13/1994 of the Ministry of the Environment, but also with data were provided by the Agency for Nature Conservation and Landscape Protection of the Czech Republic.

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REFERENCES

1. Zbíral J., Honsa I., Malý S.: in the book: *Analýza půd II : Jednotné pracovní postupy,* p. 84. ÚKZÚZ, Brno 2003

3-P9

THE ISSUE DETERMINATION OF ORGANOTIN COMPOUNDS IN THE ENVIRONMENT

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Tin has a large number of its organoderivatives in commercial use. The occurence of organotins in the environment has increased in recent decades because of the antropogenic activities. The bioavailability and toxicity of these organocompounds, in addition to their mobility and their environmental impact, is highly dependent on their chemical form[1-3].

Due to wide industrial applications, amounts of the organotins have entered various ecosystems. Among commercially used compounds are mono-, di-, tri- and tetraorganotin compounds, where the organic group is represented by methyl, buthyl, octyl, cyklohexyl or phenyl. Methyltin compounds can be produced in the environment by biomethylation of inorganic tin and may thus occur naturally. Monoorganotin compounds are in use as stabilizers of polyvinyl chloride (PVC) and for glass treatment. Diorganotin compounds are also used as PVC and polyurethane foams stabilizers and as catalysts for esterification. Triorganostannic compounds are applied as pesticides, fertilizers, wood preservatives and as an antifouling paint. Tetraorganotin compounds are used to prepare the substituted compounds, which are mentioned above. Using triorganotin compounds in anti-fouling paints has been restricted by the law (Organotins antifouling paints Control Act, 16 June 1988), which limited the types of ships on which these paints can be used[2,4,5].

Preferred analytical technique for the determination of organotin compounds is gas chromatography (GC) due to its high potential for a classification and detection flexibility. First of all, the organostannic compounds are extracted from samples using the suitable reagent, these adapted organostannic compounds are then transferred by derivatization into volatile form and only then they can be separated and detected using GC with a flame photometric detector (FPD), atomic absorption spectrometry (AAS) and mass spectrometry (MS) and also by many other types of separation and detection[2-4,6].

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REFERENCES

1. Centineo G., Gondález E. B., Sanz-Medel A.: J. Chromatogr. A. *1034*, 191 (2004).
2. Hoch M.: Applied Geochemistry *16*, 719 (2001).
3. Reuther R., Jaeger L., Allard B.: Anal. Chim. Acta *394*, 259 (1999).
4. Carvalho Oliveira R., Santelli R. E.: Talanta *82*, 9 (2010).
5. http://www.atsdr.cdc.gov/ToxProfiles/tp55-c5.pdf [cit. 2011-15-04].
6. Beceiro-González E., Guimaraes A., Alpendurada M. F.: J. Chromatogr. A *1216*, 5563 (2009).

3-P10

MONITORING OF DEAR CONTAMINATION BY ORGANOHALOGEN COMPOUNDS BASED XENOBIOTICS

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Various bio-indicators of animal or vegetable origin are used for the assessment of the environmental contamination. In thist study, the wild boar was chosen for the monitoring of organohalogen compounds based xenobiotics.

Limits of some contaminants have not been set for wild boar, and the samples of wild boar are assessed according to the limits set for the pork. Nevertheless, if the total number of wild boars caught in the Czech Republic in 2008 - 2009, the average weight caught pieces of 25 kg and carcass yield of 50 – 60 % are taken into consideration it could be concluded that the consumption of the wild game meat is relatively high.

The district health facility staff collected the samples of wild boars in the territory of Central Bohemia. The target substances were isolated from the matrix by petrolether extraction. The extract was further purified by column chromatography, final cleansing of the extract was made by acid hydrolysis. The determination of the selected analytes was realized using gas chromatography with electron capture detection. The results gave us basic information about the wild boar contamination by organohalogen pollutants.

PCB #28 and PBDE #153 were the most often detected congeners of PCBs and PBDEs; PCB #101, PCB #180, PBDE #100 and PBDE 99 were found with lower frequency. Other congeners were detected in one or two samples only. The highest PCB load was found in the samples from the locality of MS Chýňava, where six PCB and two PBDE congeners were detected (PCB #28, PCB #52, PCB #153, PCB #180, PBDE #47, PBDE #153). The highest levels were found in the sample originated from Brno, where the PCB #28 concentration was 2,25 ng g^{-1} and that of PCB #101 was 3,38 ng g^{-1}.

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3-P11

DECONTAMINATION OF SOIL POLLUTED BY PETROLEUM WASTES

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In the years 2008 – 2010 phytoremedial effects of petroleum contaminated soils on two different localities was studied. Two-phase decontamination based on utilisation of compost with rich microbial activity and subsequent growing of non-traditional crops (sweet clover and fodder mallow) with high dry matter production was used for decontamination of polluted soils. It was found that the use of compost and testing crops has greatly accelerated the degradation of non-polar extractable substances in the soil.

3-P12
SPRAY SCRUBBER SYSTEM FOR REMOVING CO_2 EMISSIONS FROM THE AIR

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This paper shows the common research activity of the team of Chemical Engineers from the Institute of Materials Chemistry and the Institute of Chemistry and Technology of Environmental protection, Faculty of Chemistry, Brno University of Technology, Czech Republic. The research activity is oriented to the absorption process and development of pilot-plant absorption scrubber prototype ZK 400.

The most important part of the absorption scrubber is spray tower, where contact between gas and liquid phase takes place. Scrubber is equipped with pH electrode, conductance electrode, pressure sensor. Function of gas scrubber was tested with absorption of carbon dioxide to the deionized water. Commercial sparklets were source of the carbon dioxide and the gas was of the food quality. For one meassurement were used app. 24 g (three spraklets) and 20 litres of destiled water as the absorbent. Used CO_2 had been circulated in gas loop for 20 minutes, for better distribution of the gas and after that, circulating gas were sent to the spray tower. The conductance of the solution was under the detection limits of the electrode, so the only constantly monitored physical property of the solution was pH.

The CO_2 gas electrode was used for analysis of carbon dioxide absorbed in the water samples and obtained data were evaluated with method of calibration curve. The 0,1 M stock solution of $NaHCO_3$ was used for preparation of calibration solutions. Citrate buffer solution was used as well. Citrate buffer was added to the calibration solutions and samples, for the acquirement of lower pH values of the solution.

Absorption was measured through different temperatures, cause the unfinished thermostatic device. The only way how to reach stabilization of temperature was to stabilize temperature in whole laboratory room.

The poster presents CO_2 concentrations of both the gas and luquid phases. The given hydrodynamics condition in the pilot-plant scrubber tower are presented too.

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3-P13
THE STUDY OF THE PRECONCENTRATION OF PLATINUM GROUP METALS ON MODIFIED SORBENTS AND THEIR DETERMINATION BY ICP-OES

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The analytical interest in microamounts of platinum group metals (PGMs) was awoken by massive introduction of automobile catalytic converters, containing especially Pt, Pd and Rh for elimination of toxic species of ignition motor exhaust gases. PGMs may have toxic properties, such as nefrotoxicity, genotoxicity, gastrointestinal and allergic irritation as well as anticancer activity or cytostatic influence of some platinum (II) complexes and their presence in the environment represents danger for human health. For this purposes, use in automobile industry and in anti-tumor medicine, increasing Pt and Pd concentrations have been found in various objects of environment. In spite of that, traces of PGM are still available in the environment, in comparison to common metals. For these facts the preconcentration and separation of PGMs is often necessary prior to their determination by ICP-OES. Octadecyl silica[1,2], octyl silica, phenyl silica, cyanoethyl silica, aminopropyl silica, silica, were tested for the separation and preconcentration of 1 - 20 μg in 50 - 1000 cm^3 sample volume of Pt(IV, II), Pd(II), Ir(IV), Rh(III), Os(VI), Ru(IV) in the form of ion associates of their chloroorbromo complexes with [1-(ethoxycarbonyl) pentadecyl]trimethylammonium bromide (Septonex®), benzyl-(dodecyl)dimethylammonium bromide (Sterinol®), trimethyl (tetradecyl)-ammonium bromide and hexadecyl (trimethyl) ammonium chloride[3] from 0,1 mol dm^{-3} HCl or 0,1 mol dm^{-3} HBr. A 100% recovery for Pd(II) and Pt(IV) was obtained with octadecylsilicas Separon™ SGX C18, Separon™ SGX RPS and Separon™ SGX Phenyl™ silica in the presence of 0,006 mol dm^{-3} Septonex® or 0,003 mol dm^{-3} Sterinol®. The retained PGMs were eluted with acetonitrile and determined by inductively coupled plasma atomic emission spectrometry (ICP-AES) after evaporation of organic solvent in the presence of 0,1 mol dm^{-3} HCl. The enrichment factor reached 100. A 1000:1 excess of Cl-, NO_3^-, SO_4^{2-}, Na^+, K^+, Ca^{2+}, Mg^{2+}, Fe^{3+} or Al^{3+} did not interfere. The behaviour of these sorbents was compared with that of a silica-based anion exchanger Separon™ SGX AX and macroporous sorbents Amberlite™ XAD2 and Amberlite™ XAD4 in the presence of 0,1 mol.dm^{-3} HCl and 0,006 mol dm^{-3} Septonex®. In this way, the recovery of 0,1 – 0,2 μg cm^{-3} Pd(II) and Pt(IV) was successfully tested with spiked matrices of soil extracts, power station dust samples and natural waters.

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REFERENCES

1. Otruba V., Strnadová M., Skalníková B.: Talanta *40*, 221 (1993).

2. Vlašánková R., Otruba V., Bendl J., Fišera M., Kanický V.: Talanta *48*, 839 (1999).
3. Komendová-Vlašánková R., Sommer L.: Collect. Czech. Chem. Commun. *67*, 454 (2002).

3-P14

PHOSPHORUS SORPTION - COMPARISON OF THE SORPTION CHARACTERISTICS OF FILTRATION MATERIALS

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This paper filling the knowledge obtained in research work which is aimed to determine the effect of phosphorus sorption through a filter material in the filtration process of mechanically purified sewage wastewater, respectively, water with a precisely defined quantity of total phosphorus.

Target of the investigations is a comparison of sorption characteristics and the possibility of using the current filter environments (river gravel, crushed gravel, sand, waterworks sand)[1] and non-traditional filtering materials (crushed blast furnace slag and cinders, sand saturated $KMnO_4$) in the process of sorption of phosphorus. Results of laboratory measurements used to calibrate the mathematical model, the sorption characteristics are calculated by HYDRUS 2D software.

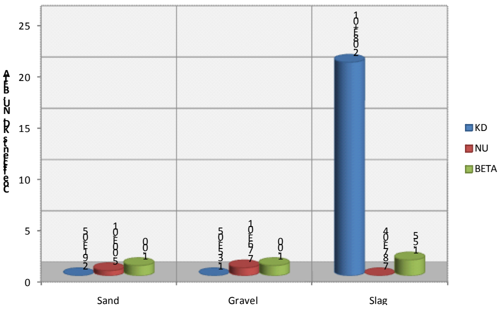

Figure 1. Sorption characteristics obtained from a mathematical model

Theoretical processing sorption properties problems of filter materials and findings from research on the sorption of phosphorus filter waste water from the environment shows that the use of appropriate filter materials containing an iron compounds, steady load and filter dont load the environment, it is possible to achieve a significant reduction of phosphorus in the effluent cleaned waste water. The results of the research can be fully used in combination with a suitable mathematical tool for designing the optimal soil (earth) filters, to assess the effect of cleaning of soil in filtration wastewater, assessing accidental releases, etc.

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REFERENCES

1. Sakadevan, K., Bavor, H. J., in the book: *Phosphate adsorption characteristics of soil, slags and zeolite to be used as substrates in constructed wetlands systems*, p. 393-399. Elsevier Science Ltd., No.2, Great Britain 1998.

3-P15

THE USE OF COMPREHENSIVE TWO-DIMENSIONAL GAS CHROMATOGRAPHY WITH MASS SPECTROMETRIC DETECTION FOR THE ASSESSMENT OF DRUG-RESIDUE CONTAMINATION OF WASTEWATER AND SURFACE WATER

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This study is focused on application of analytical method for analysis of selected drug residues from wastewaters and surface waters. Target compounds were acidic drugs mainly from the group of non-steroidal anti-inflammatory drugs (salicylic acid, acetylsalicylic acid, clofibric acid, ibuprofen, acetaminophen, kofein, naproxen, mefenamic acid, ketoprofen, diclofenac). Pharmaceuticals have become very controlled environmental contaminants in recent years, due to their increasing concentration in environmental components. It is mainly caused by their high produce and everyday consumption. It is proved, that these substances are not completely eliminated in organism and about of 80 % are eliminated in unchanged form and discharged into sewer system with feces[1]. So they enter wastewater treatment plant (WWTP), where they are only partly removed during the cleaning processes. Therefore they can be found in effluent of WWTP and so they are discharged into surface water[2,3], where they can cause adverse effects on non-target water organisms[4]. Due to this case, there is a need to apply new and sufficient sensitive analytical methods, which can detect presence of contaminants even in very low concentrations.

Analytical method used in this study includes solid phase extraction (SPE), derivatization by MSTFA (*N*-methyl-*N*-(trimethylsilyl)trifluoroacetamide) and final analysis by orthogonal comprehensive two-dimensional gas chromatography with mass spectrometric detection Time-of-Flight (GCxGC-TOF MS). This instrumental analytical method offers much higher separation efficiency and sensitivity compared with liquid chromatography or conventional gas chromatography. Therefore it is very suitable for trace and ultra-trace analysis of organic compounds such as pharmaceutical residues. In addition, mass spectrometric detection with electron ionization enables reliable identification of separated compounds on the base of library searches.

Detection limits of presented method varied from 0,18 to 5 ng l^{-1}, depending on the specific compound. The method was successfully applied for the determination of presence and concentration of selected drug residues in Svratka River and in wastewater from WWTP in Brno – Modřice. Concentrations of drug residues ranged from units to tens of ng l^{-1} for river water and from units to tens of µg l^{-1} for wastewaters.

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REFERENCES

1. Togola A., Budzinsky, H.: Anal. Bioanal. Chem. *388*, 627 (2007).
2. Baranowska I., Kowalski B.: Water Air Soil Pollut. *211,* 417 (2010).
3. Hernando M. D., Mezcua M., Fernández-Alba A. R., Barceló D.: Talanta *69,* 334 (2006).
4. Mompelat S., Bot B. L., Thomas O.: Environ. Int. *35*, 803 (2009).

3-P16
CHROMIUM REMOVAL FROM WATER BY SYNTHETIC GOETHITE

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Synthetic goethite α-FeOOH was studied as potential sorbent for the removal of chromium from model aqueous solutions. The pure goethite was prepared from an Fe^{II} system, involving oxidative hydrolysis of Fe^{II} solutions. As various Fe oxides may be produced by this method, careful control of factors such as the rate of oxidation, pH and the nature of the anion present is necessary to ensure formation of pure goethite. The material was characterized by chemical analysis as well as X-ray diffraction, electron microscopy, specific surface measurements. Specific surface area of sorbent was 54 m^2 g^{-1} and magnetic susceptibility was 435×10^{-6} u. SI. Chromium removal from aqueous solutions was investigated using batch adsorption-equilibrium experiments. Initial Cr concentration was 50 $mg.l^{-1}$ and the sorbent concentration was 2 g l^{-1}. The experiments were carried out at ambient temperature (23 ± 1°C). The effect of pH, ionic strength and initial chromium concentration were investigated. Experi-mental data were modeled using Freundlich equation and adsorption parameters (K, b, R^2) were determined. The sorp-tion capacity of goethite has been about 25 mg of Cr per g of sorbent.

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3-P17
METHOD OF SEQUENTIAL EXTRACTION OF HEAVY METALS FROM SOLID ENERGETIC WASTE PRODUCTS

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The physical and chemical properties of industrial waste product, in general, are quite variable, as they are influenced by coal source, particle size, and type of coal burning process and degrese of weathering. Water extracts from fly ash are commonly alkaline and contain excessive amounts of dissolved solids. Leachability of heavy metals presents a hazard for natural waters. Since many trace elements existing in fly ash can leach out and groundwater resources, the study of them has been regarded in recent years as important in connection with protection of the environment[1-3].

In the study a sequential extraction method was applied for Cd, Cd, Cu and Hg in accordance with the scheme proposed by the Tessier. Five steps of extraction with different extraction agents were carried out at temperatures 25°C and 40°C. The samples were collected from four heating plants and with two different types of combustion apparatus.

The concentration of metals were determined by flame atomic absorption spectroscopy (F-AAS, SpectrAA 30), by electrothermal atomic absorption spectroscopy (ET-AAS, AAS ZEEnit 60). The Advanced Mercury Analyzer (AMA 254) was used for the determination of mercury.

The determined concentration of metals from sequential extraction at various tempreraturе were identified to different fractions, it means metals in exchangeable fraction, exchangeable fraction and associated with carbonated phases, reducible fraction or fraction associated with Fe and Mn oxides or hydroxides, oxidisable fraction or bound to organic matter and residual fraction and results also confirmed influence of temperature of extraction.

This work was supported by the institution research plan No. FCH-S-11-3 from the Ministry of Education, Youth and Sports of Czech Republic.

REFERENCES

1. Passos E. A., Alves J. C., Santos I. S., Alves J. P. H., Garciav C. A. B., Costa A. C. S.: Microchem J. *96*, 50 (2010).
2. Fuentes A., Iloréns M., Sáez J., Soler A., Aguilar I., Ortuňo J. F., Meseguer V. F.: Chemosphere *54*, 1039 (2004).
3. Shiowata J., Tantidanai N., Nookabkae W. S., Nacapricha D.: J. Environ. Qual. *30*, 1195 (2001).

3-P18

SPATIAL DISTRIBUTION OF METALS IN TOP SOILS AND CONTAMINATION ASSESSMENT METHODS

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In urban and industrial areas, chemical pollution sources are numerous. Diffuse metal contamination of soils is caused mainly by atmospheric fallout from various sources, the most important being industrial and traffic emissions. For human and ecological risk assessment, a growing body of evidence has shown the necessity of determining the spatial distribution of pollutants.

This environmental study is focused on the distribution of heavy metals in the top soils of selected localities. The samples were from various localities of Moravia and Slovakia. The criterium for samplings was different industrial, traffic and agricultural situation and supposed different metal contamination of top soils.

The concentration of metals were determined by flame atomic absorption spectroscopy (F-AAS, SpectrAA 30), by electrothermal atomic absorption spectroscopy (ET-AAS, ZEEnit 60). The Advanced Mercury Analyzer (AMA 254) was used for the determination of mercury.

To assess the level of contamination indexes such as the pollution index (PI), integrated pollution index (IPI), contamination factor (C_f) and the degree of contamination (C_{deg}) were calculated to assessing the contamination level of heavy metals in top soils.

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REFERENCES

1. Ajmone-Marsan F., Biasioli M., Water Air Soil Pollut. *213*, 121 (2010).
2. Maas S., Scheifler R., Benslama M. M., Crini N., Lucot E., Brahmia Z., Benyacoub S., Giraudoux P.: Environ. Pollut. *158*, 2294 (2010).

3-P19

MODIFIED DIGESTION METHOD OF TOTAL PHOSPHORUS DETERMINATION IN RIVER SEDIMENTS

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A digestion method of total phosphorus determination in river sediments has been modified, verified and compared with three generally used methods. Real sediment samples were used for the phosphorus analyses. They were collected in the upper part of Bílá Opava River and in small watercourses in the Brno suburban area, Czech Republic.

Principle of the digestion method is based on Hach method for solids[1], the parameters of which have been changed to suite the purpose of decomposition of river sediments. Organic matter is decomposed by sulphuric acid and hydrogen peroxide at the temperature of 440 °C.

The compared methods include heating and effects of strong acids or alkalis. In the first of them, the sediment is mineralized by sulphuric acid. The sample solution is then neutralized by ammonium hydroxide[2] (2,4-dinitrophenol indicator). The second method involves sediment calcination and subsequent hydrochloric acid extraction[3]. The third method is based on sodium carbonate fusion[4] followed by reaction with sulphuric acid; the sample is neutralized by sodium hydroxide (p-nitrophenol indicator).

The released phosphates have been determined spectrophotometrically by the phosphomolybdenum blue method in the last analytical step of each method.

Mineralization by sulphuric acid has given lower results than both fusion and digestion methods. Calcination method has given zero values. Method of sodium carbonate fusion, which is supposed to be very efficient, and modified digestion method have given comparable results. Thus, the modified digestion method, which is simple and fast, has proved to be suitable for total phosphorus determination in river sediments.

This work was supported by Brno University of Technology - Faculty of Civil Engineering - Specific Research project No. 1161.

REFERENCES

1. Hach Company in the book: *Digesdahl Digestion Apparatus Instrument Manual,* Colorado, USA 1999.
2. Bíliková A.: *Hydrochémia 92*, p. 311-328, Bratislava 1992.
3. Pardo P., Rauret G., López-Sánchez J. F.: J. Environ. Monit. *5*, 312 (2003).
4. Kovar J. L., Pierzynski G. M.: Southern Cooperative Series Bulletin *408*, (2009).

3-P20
EFFECT OF TEMPERATURE ON TREATMENT OF HUMIC WATER BY DIFFERENT COAGULANTS

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Coagulation is used in drinking water treatment to destabilize and subsequently to remove colloidal particles and dissolved organic substances. The most widely used coagulants are aluminium and iron salts, however the use of natural organic coagulants, such as chitosan, may be an interesting alternative.[1]

Chitosan is a linear cationic polymer of high molecular weight obtained by deacetylation of chitin. Most of amine groups of chitosan in acidic solution are protonated and they can destabilize particles with negative surface charge that are present in water (e. g. humic substances).[2]

Many chemical and physical factors, such as coagulant dosage, pH, natural organic matter (NOM), turbidity or temperature, affect coagulation processes in water treatment practice. It is commonly found that metal coagulants perform less effectively at low temperatures.[3-5]

The paper presents results of coagulation experiments studying the influence of temperature upon aggregation in treatment of humic water by different coagulants. While coagulation with aluminium or iron sulpate was negatively influenced by low temperature, results with chitosan and prepolymerised aluminium coagulants Flokor were hardly affe-cted by low temperature. Separation efficiency by chitosan is comparable with metal coagulants. Small influence of low water temperature is a positive feature for water treatment practice.

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REFERENCES

1. Rizzo L. Di Gennaro A., Gallo M., Belgiorno V.: Sep. Purif. Technol. *62*, 79 (2008).
2. Zang X., Bai R.: J. Colloid Interface Sci. *264*, 30 (2003).
3. Dolejs P.: Environ. Prot. Eng. *9*, 55 (1983).
4. Dolejs P. in the book: *Chemistry for Protection of the Environment*, p. 169-178. Elsevier, Amsterdam 1984.
5. Duan J., Gregory J.: Adv. Colloid Interf. Sci. *100-102*, 475 (2003).

3-P21
COMPARISON OF BIOLOGICAL RESPONSE OF THE CLAM *SCROBICULARIA PLANA* FROM DIFFERENT ENVIRONMENTAL AREAS BY SEC-UV-ICP-MS

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The study of environmental issues is generally performed by the use of bioindicators that reflect the biological response to the presence of contaminants. Mamals[1] and invertebrates[2] have frequently proposed for this aim. Conventional biomarkers have been used for this purpose but they require a deep knowledge about the toxicological mechanisms involved in the release of substance. For this reason the omics are now the new tool to evaluate the global biological response to contamination.

Metallomics is the most recent omic focused in the study of metal-binding biomolecules using instrumental couplings, generally based on SEC-ICP-MS and organic mass spectrometry. In the present study we apply this approach to compare the biological response of *Scrobicularia plana* collected from polluted and non-polluted areas from the southwest of Spain and Portugal.

Digestive gland of the clam was extracted to obtain the cytosolic fraction by cryogenic homogenization followed by treatment with 20 mM ammonium acetate solution at pH 7,2; 1 mM GSH to avoid the formation of disulfide groups and 1 mM PMSF (phenyl-methanesulfonylfluoride) for proteases inhibition[3].

Chromatographic profiles of the extracts denote the presence of low molecular mass Cu and Zn molecules about 300 Da that can be related to the Cu- and Zn-glutathiones and As metabolites such as arsenobetaine and arsenocholine. The response of Cu-biomolecules are higher in Cadiz area against Portugal zone while Zn-biomolecules present a opposite behaviour. In several samples have been observed the presence of a remarkable Cu-peak of about 67 kDa.

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REFERENCES

1. Bonilla-Valverde D., Ruíz-Laguna J., Muñoz A., Vallesteros J., Lorenzo F., Gómez-Ariza J. L., López-Barea J.: Toxicology *197*, 123 (2004).
2. Pérez E., Blasco J., Montserrat S.: Mar. Environ. Res. *58*, 275 (2004).
3. González-Fernández M., García-Sevillano M. A., Jara-Biedma R., García-Barrera T., Vioque A., López-Barea J., Pueyo C.and Gómez-Ariza J. L.: J. Anal. At. Spectrom. *26*, 141 (2011).

3-P22

SPECIATION OF ARSENIC METABOLITES IN THE FREE-LIVING MOUSE MUS SPRETUS FROM DOÑANA NATIONAL PARK USED AS BIOINDICATOR FOR ENVIRONMENTAL POLLUTION MONITORING

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The use of free-living organisms in environmental pollution assessment is a useful approach because biological responses can be more unequivocal information than direct analysis of contaminant to assess pollution of a given ecosystem. *Mus spretus* from Doñana National Park (SW Spain) has been used for this purpose[1]. This free-living organism has not been yet genetically sequenced although exhibits a clear genetic homology with the typical laboratory sequenced mouse *Mus musculus*. Doñana Natural Park is one of the most important European biological reserves but it is subject to ecological pressures, due to the input from adjacent agriculture, minig and industrial activities. Arsenic is commonly found in several chemical forms whose toxicity, environmental mobility and accumulation in living organism usually depend on the form in which the element is present. Thus, inorganic forms of arsenic are more toxic, while methylated forms are considered only moderately toxic and non-toxic as AsB, AsC and other arsenosugars.

Several organs from *Mus spretus* have been studied but the highest concentration of arsenic has been found in the kidneys. The metallo-metabolites profiles in kidney from *Mus spretus* have been probed using SEC with ICP-ORS-MS detection. The results show the presence of LMW molecules linked to arsenic in these tissues with differents intensities in the differents areas, which can be related to environmental stress caused by contamination. These fractions has been collected and has been analyzed using AEC-ICP-ORS-MS. Characterization of these fractions by organic mass spectrometry such as HPLC-ESI-MS have been performed in relation to environmental issues.

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REFERENCES

1. González-Fernández M., García-Sevillano M. A., Jara-Biedma R., García-Barrera T., Vioque A., López-Barea J., Pueyo C., Gómez-Ariza J. L.: J. Anal. At. Spectrom. *26*, 141 (2011).

3-P23

NOVEL APPROACHES TO ANALYSIS OF 3-CHLOROPROPANE-1,2-DIOL DIESTERS IN PALM OIL EMPLOYING LIQUID CHROMATOGRAPHY–MASS SPECTROMETRY AND DIRECT ANALYSIS IN REAL TIME–MASS SPECTROMETRY

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The 3-chloropropane-1,2-diol (3-MCPD) is a well known contaminant in various food such as acid hydrolyzed vegetable protein (HVB), soy sauce, various food ingredients and bakery products[1]. 3-MCPD esters were recently detected in a wide range of foodstuffs, especially in refined vegetable oils and products made of refined vegetable oils. The highest levels of these emerging processing contaminants were found in hydrogenated fats, palm oil and solid frying fats, at concentration levels largely exceeding those of free 3-MCPD[2]. Esters of 3-MCPD with higher fatty acids are formed at high temperatures during the refining process of edible oils and fats, mainly during the deodorization step. It is still unknown whether 3-MCPD esters have the same toxic effect as free 3-MCPD. One reason is that the bioavaibility of 3-MCPD esters have not been completely elucidated. The 3-MCPD esters have been put by the European Food Safety Authority (EFSA) on the priority list for the health risk assessment process. Until recently, the limiting factor in obtaining more comprehensive data on MCPD esters was their difficult determination.

In the present study, development and validation of two alternative MS-based high-throughput approaches to analysis of individual 3-MCPD diesters are described. The new method is capable to replace the established, time consuming procedure based on hydrolytic step followed derivatization of released 3-MCPD for gas chromatographic (GC) analysis by simple straight forward procedure[3]. The target analytes isolated from edible oil samples by fast silica gel column chromatography were determined with the use of ultrahigh pressure LC coupled to a high-resolution Exactive^{TM} Orbitrap mass spectrometer. Additionally, the potential of a novel ambient pressure desorption ionization technique, direct analysis in real time (DART), for both qualitative and quantitative analysis of these processing contaminants was explored.

REFERENCES

1. Hamlet C. G., Sadd P. A., Crews C., Velisek J., Baxter D. E.: Food Addit. Contam. *19*, 619 (2002).
2. Svejkovska B., Novotny O., Divinova V., Reblova Z., Dolezal M., Velisek J.: Czech J. Food Sci. *22*, 190 (2004).
3. Seefelder W., Varga N., Studer A., Williamson G., Scanlan F. P., Stadler R. H.: Food Addit. Contam. *25*, 391 (2008).

3-P24

ANALYSIS OF TAR GENERATED FROM BURNING BIOMASS

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In the last 15 years, the intensive progress was achieved in area of biomass gasification. This fact is based on general social interest to obtain energy from renewable sources. The technologies of gasification are more effective than classical combustion, they have lower emissions and primarily, it could be realized smaller local units with lower power of congeneration. Until recently, the research of waste gasification was only insignificant matter. Mostly, it was used only as a substitute of natural gas in the lime and cement kilns where the fill of kiln is used for the combustion products treatment.

By thermal gasification, combustible components are formed but also many other undesirable compounds mainly polycyclic aromatic hydrocarsons, sulphur componuds and chlorine compounds are formed. This components cause the problems during gas application in congeneration units. But they could cause also potential risks during possible breakdown because many compounds have carcinogenic and toxic effects.

In this work, the methods were optimalized for the analysis of tar which is formed during gasification. In obtained samples it was determined volatile organic componds (BTEX) using gas chromatography with flame ionization detector (FID). 1 ml of tar sample was clean up and was transfered into isooctan. Than the polycyclic aromatic hydrocarbons (PAHs) were analysed. It was used gas chromatography with mass detection (GC/TOF-MS). The last analysing group were n-alkanes C10 – C32 (FID). The highest concentrations were set for low molecular PAHs – naphthalene, anthracene, acenaphthylene and acenaphthene. Further, it was identified more than 30 compounds.

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REFERENCES

1. Harvey R. G. in the book: *Polycyclic aromatic hydrocarbons: Chemistry and carcinogenity*, p. 405. Cambridge University Press, Cambridge 1991.
2. Callen, M., Hall, S., Mastral, A. M., Garcia, T., Ross, A., Bartle K. D.: Fuel Process. Technol. *1*, 34 (2000).
3. Ravindra K., Sokhi R., Van Grieken R.: Atm. Env. *13*, 42 (2008).

3-P25

SYNTHETIC AROMATIC SUBSTANCES IN WASTEWATERS

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Synthetic fragrances are artificially synthetized organic compounds which are used as fragrant substances in assorted personal care products, perfumes, cosmetics, soaps, detergents, cleansing articles, etc. so-called musk compounds create significant group of synthetic fragrances which are distinguished by specific fragrance reminiscent of musk. Recently great attention is devoted to musk compounds because they were found in different environmental compartments, especially in aquatic ecosystem. Due to their persistence and their ability of bioaccumulation they can negatively influence the water biota.

The aim of this study was the determination of selected linear musk compounds produced and used by the Czech company Aroma Praha, Ltd. (arocet, aroflorone, linalool 925, lilial, isoamyl salicylate) in real samples of waste water in the influent and effluent from the municipal waste water treatment plant Brno-Modřice. At first optimization of selected method was performed and then this method was used for the determination of target analytes. Head-Space Solid Phase Microextraction (HS-SPME) was used as the method of isolation and preconcentration, gas chromatography with mass spectrometric detection was used for final analysis. The results were used for the evaluation of the linear musk compounds removal efficiency during the cleaning process.

The only compound which was found in all analysed samples was lilial; its concentrations varied between 0,017 – 0,065 ng ml^{-1}. The presence of Arocet wasn't proved in any of analysed samples, aroflorone and isoamylacetate levels were bellow LOQ. Linalool 925 was found only in 3 samples. The removal efficiency during the water cleaning process for all target compounds was very good – above 99% with one exception which was lilial with removal efficiency between 79 – 96 %.

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3-P26

PROBLEMS WITH THE SPECTROPHOTOMETRIC AND ELECTROTHERMAL AAS DETERMINATION OF TRIVALENT CHROMIUM IN WATERS

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The aim of this study was the spectrophotometric determination of trivalent chromium with chromazurol S (CAS), eriochromcyanine R (ECR), 4-(2-thiazolylazo) resorcinol (TAR) and its comparison with the determination of total chromium as Cr(III) by atomic absorption spectrometry with electrothermal atomization.

The reactions of Cr(III) with spectrophotometric agents are catalytically hindered by the previous existence of inert cationt $[Cr(H_2O)_6]^{3+}$ and all reactions are initialized by longer increased temperature only.

Most suitable agent is chromazurol S. This determination is provided at pH 3,5 in presence of hydrazin dihydrochloride as reducing agent after heating for 40 minutes at 95 °C. The stechiometry of formed complex (λ_{max} = 575 nm) under these conditions was Cr(III):CAS = 1:2.

Conditions of the spectrophotometric determination Cr(III) with eriochromcyanine R, are similar, at pH 4,2 – 4,8. But absorbance of the agent and Cr(III)complex (λ_{max} = 550 nm) are rather unstable in time.

The determination with 4-(2-thiazolylazo)resorcinol is provided at pH 5 in presence of 5% dimethylformamide at 545 nm. Since nitrate interferences, in this case chloride medium must allways be used. Under these conditions the Cr(III)-$(TAR)_2$ complex is rather formed.

The Fe^{2+}, Fe^{3+}, Al^{3+}, Na^{+}, $H_2PO_4^{-}$, and HCO_3^{-} ions interfere all spectrophotometric determinations of Cr(III). Sensitivities and various kinds of detection limits were evaluated. The most sensitive reaction with the highest value of molar absorption coefficient and the lowest detection limits of studied spectrophotometric methods, provides the determination with chrom-azurol S.

The ET-AAS determination of total chromium as Cr(III) has been provided at 357,9 nm in a graphite cuvette with platform and Zeeman correction of background in the presence of the same reductant without modifier with charring and atomization temperature 900 °C and 2300 °C. The Fe^{2+}, Fe^{3+}, Cl^{-} and Na^{+} ions could also interfere the determination.

Concentration of Cr(III) was succesfully determined in various kinds of real waters and both the ET-AAS and spectrophotometric determination with chromazurol S were compared.

3-P27

MONITORING OF PRESENCE OF SELECTED HEAVY METALS IN WELL WATERS

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The toxic metals in drinking water present one of the main health risks for human. The goal of this research was to monitor the presence of selected metals (Pb, Cu, Zn, Mn, Fe) in well waters in the region of Rimavská Sobota. During the six periods of 2009 (20-24 May, 5-10 June, 3-7 August, 7-10 September, 4-7 November and 8-12 December) water samples from 10 wells in various villages (Uzovská Panica, Figa, Radnovce, Chrámec, Husiná, Horné Zahorany, Bátka, Hačava - Skálie, Hňúsťa and Tisovec) were taken and analyzed.

Iron was determined photometrically, Pb, Zn and Cu were determined by atomic absorption spectrometry. Most of the samples from the wells of the selected areas of Rimavská Sobota region met the requirements for the quality of drinking water. The Pb concentration in the collected samples was mainly < 3,0 $\mu g\ l^{-1}$). Copper concentration did not exceed 100 $\mu g\ l^{-1}$ in most of collected samples. The exceptions were found in the well water collected in September and November from the well in Bátka. Above-limit values of iron were detected in water from wells in Uzovská Panica, Figa, Chrámec and Tisovec. Slightly elevated concentrations of manganese were detected in water from wells in Uzovská Panica and Tisovec. We did not observe regular seasonal incidence of the elements in the well waters.

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3-P28

RESPONSE OF SOYBEAN ROOTS TO IONS OF CADMIUM AND ARSENIC

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Heavy metals are serious contaminants of the environment with negative impact on living organisms including plants. Plants are considered as one of enter points of heavy metals into the food chain. Heavy metals contaminated plants thus endanger animals and humans.

A set of soybean (*Glycine max L.*) genotypes, that are grown in Slovakia (cvs. Cordoba, Essor, Merlin a Kent), Ukraine (cvs. Chernyatka, Ustya, Kyivska 98, Vorskla) and

Hungary (cvs. Evans a Boróka), were screened for sensitivity and tolerance to ions of cadmium and arsenic (both at concentrations of 5 mg l^{-1}).

Based on measurements of root growth two genotypes with contrasting sensitivity to heavy metal exposure were selected: the most sensitive Kyivska 98 and the relatively tolerant Chernyatka. In both genotypes a significantly enhanced rate of lipid peroxidation was observed, indicating membrane damage as the consequence of applied stress. Protein extracts from the roots of tested plants were separated on polyacrylamide gels. The subsequent staining for chitinase activities revealed genotype specific chitinase accumulation as a component of plant defense against the heavy metal stress. The involvement of detected chitinase isoforms in metal tolerance and sensitivity is evident and requires further investigations in future.

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3-P29

RESPONSE OF SOYBEAN ROOTS TO CADMIUM, NITROGEN STRESS AND THEIR COMBINATIONS

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In environment polluted with heavy metals plants represent an entry point to the food chain and by this way they are endangering the consuming organisms by metal intoxication. The metal uptake by plant is affected by nutrient availability. In this work we show that relatively low doses (1,2 mM) of nitrogen in the growth media slightly increased, while high doses (24 mM) of nitrogen significantly suppressed the uptake of cadmium (applied as 50 μM Cd^{2+}) by roots. Despite of that, both of the high nitrogen concentration and exposure to Cd resulted in stunted root growth with most detrimental effect after the combination of these stressors. To clarify the occurring defense strategy in roots, total proteins were separated on polyacrylamide gels and subsequently stained for activities of chitinase enzymes, generally involved in plant stress against pathogens. We found that activities of certain isoforms that typically increased during Cd exposure, again dropped when high, but not low doses of nitrogen were also applied. Activity of certain chitinase isoforms indicates to a complex plant defense strategy under the exposure to a combination of metal and nitrogen stress.

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3-P30

EVALUATION OF BIOAVAILABILITY OF TOXIC METALS IN AQUATIC SYSTEM BY USING PASSIVE SAMPLERS CHEMCATCHER

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The issue of monitoring of aquatic ecosystem contamination by toxic metals is actual till now. Although there are efforts to reduce the technical use of toxic metals and to lower the production of waste with their contents, however, these substances are present in all environmental compartments, and in some areas they are found in very high concentrations till now. Toxic metals represent a potential danger to aquatic organisms. They can adsorb on solid particles in aquatic environment, depending on temperature, pH value and content of humic substances. Due this process only part of the present toxic metal is available for the organisms [1, 2].

The methods of spot sampling and biomonitoring are used to assess the contamination of aquatic system by toxic metals. The later method is based on the collection of appropriate plants and animals living in the area of interest and offers information about bioavailability of target analytes. The use of passive samplers is a suitable alternative to biomonitoring. Passive sampler uptakes only substances truly dissolved in water which usually corresponds to their bioavailable fraction. Another advantage of passive samplers is the fact that they are able to reflect the fluctuations of concentrations of monitored metals during exposition period [2,3].

Chemcatcher sampler is the type of the passive sampler suitable for determination of the metals bioavailability in water. The sampler is composed of the body made from Teflon and the receiving phase covered by the diffusion membrane. According to the monitored analytes the suitable combination of the receiving phase and the diffusion membrane is chosen. For toxic metals chelating Empore disc as a receiving phase and cellulose acetate or polyethersulphone diffusion membrane are generally chosen [2-4].

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REFERENCES

1. Vardanyan L. G., Ingole B. S.: Environ. Int. *32*, 208 (2006).
2. Lobpreis T., Vrana B., Dercová K.: Chem. Listy *103,* 548 (2009).
3. Vrana B., Mills G. A., Allan I. J., Dominiak E., Svensson K., Knutsson J, Morrison G., Greenwood R.: Trends Anal. Chem. *24*, 848 (2005).
4. Agilar-Martínez R., Goméz-Goméz M. M., Greenwood R., Mills G. A., Vrana B., Palacios-Covillo M. A.: Talanta *77,* 1483 (2009).

3-P31

DETERMINATION OF POLYBROMINATED DIPHENYLETHERS AND PERFLUORINATED COMPOUNDS IN SOIL AND MATRICE FROM FIREPLACES

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Polybrominated diphenyl ethers belong among the persistent compounds, which have been classified as priority organic pollutants. In environmental compartments are observed in the past decade. Polybrominated diphenyl ethers are detected in abiotic and biotic matrices. For these studies were chosen the matrices from seats of fire, taken in various localities of the Czech Republic and Slovakia. Their analysis should demonstrate whether they in these specific matrices remain. There were examined following polybrominated diphenyl ether congeners: BDE- 28, 47, 99, 100, 153, 154, 183. For their isolation from the matrix were used there different extraction techniques, namely ultrasonic extraction, microwave extraction and pressurized solvent extraction. For the determination was chosen method of GC/ECD. In these studies are also described basic chemical, physical and environmental properities of diphenyl ethers in environmental compartments.

The other theoretical part of these studies consists of current matters regarding the presence of perfluorinated organic compounds in living environmental elements. The attention has been focused on perfluorooctanoic acid (PFOA), (PFOS) and (FOSA). Physical-chemical characteristics of these organic pollutants and their toxicological and environmental aspects have been described. The experimental part has explored effects of extraction techniques (sonication, pressurized solvent extraction, solid-phase extraction) used for PFAS separation from seats of fire soil sample. Identification of the analytes has been achieved by liquid chromatography/mass spectrometry.

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3-P32

EVALUATION OF CONTENT OF PCB AND PBDE IN PLANT BIOINDICATORS

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Contamination in the environment is usually assessed on the basic of plant and animal bioindicators. Plant bioindicators include needles of coniferous plants and moss. Needles of coniferous plants and moss belong to a group of "detectors" i.e. naturally occurring species that react not only to changes in the environment.

Moss can be found almost everywhere: from the coast of Arctic seas through tropical zones to Antarctica. Conifers are wooden trees or shrubs that usually live many years. Moss and needles are recommended as bioindicators to monitor the levels of persistent organic pollutants (POPs). POPs released in the environment, can be transported by air or water into remote regions far from the original source of contamination. In addition, POPs show high stability in all components of the environment. They enter the air from various industrial sources such as power stations, heating plants, incineration plants and also from local domestic fireplaces, etc. Air-borne POPs may occur as vapour or may bind to the surface of solid dust particles.

Our study particularly focused on PCBs and PBDEs which belong to a group of pollutants that spread easily into the environment by means of remote transport. The levels of PCBs and PBDEs in plant bioindicators (needles of coniferous plants, moss) were determined using gas chromatography combined with an electron capture detector. The main aim of this study was to find out whether the selected bioindicators are suitable to assess the level of environmental contamination with PCBs and PBDEs. The highest levels of PCBs were found in the Norway spruce (Picea abies). PCB-28 congener was the most frequently detected congener, but congeners PCB 153, 138 and 180 being also detected. Polybrominated diphenyl ethers were not detected using the method chosen. The selected type of a bioindicator was not therefore found suitable to determine the level of environmental contamination with PBDE.

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3-P33
DUMP WATER QUALITY AND WETLAND VEGETATION STRUCTURE

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Fossil fuels - coal, oil and gas are even today among the most important source of energy used by human society. Anthropogenic activities associated with their mining adversely affect the natural ecosystems on our planet. Efforts to recover these damaged ecosystems to their original form must be in accordance with the principles of their natural functioning.

The area is located in Karlovy Vary region, west of the Sokolov Down in the Sokolov brown Coal Basin. Surface mining includes the complete removal of vegetation cover and the surface mines significantly change the water cycle on the landscape (transfer a large number of streams, drainage of large areas and last but not least, a significant decline in groundwater levels). The total area of Sokolov Brown Coal Basin is approximately 219 km^2 with the landscape area by directly affected by extraction being nearly 89 km^2.

An excavated overburden creates mining dumps. Water runoff from the mine dumps has a specific composition, the vast majority of the water is acidic. Dump waters are a very serious problem, because due to their chemical composition they can greatly affect the quality of surface water, into which they discharge[1,2]. The principle of acid drainage water is very well known. The water quality is affected by oxidation of pyrite in the presence of oxygen, giving rise to dissolved iron, sulfate and acidity. Seepage from mine dumps can be a source of acidic water for decades[3].

Conventional methods of treatment of acid mine water and seepage from waste dumps are mostly represented by continuous addition of expensive chemicals such as lime or sodium hydroxide to produce a bulky sludge with low solids about 5%. Natural or artificial wetlands have been used for treatment of acidic mine water and seepage from the dumps since the 1980s. The results fom many systems have revealed that wetland systems are a suitable alternative to the currently used technologies based mainly on the chemical cleaning[4-6]. The advantage of wetland systems is their low cost and low operating costs without using the electricity[7].

Information about the importance of plants in wetlands for the treatment of mine and dump water are very scarce, however the results so far have suggest that the uptake of iron, manganese and aluminum by plants forms only a very small proportion of these metals, which enter the wetland[8-9]. Sheoran[10] reported that the effectiveness of different wetland vegetation may vary, and it is likely that the choice of plants can affect the cleaning efficiency. This study[10] also reported that a selection of plants for the wetlands used for treatment of mine water and dump has not been given due to the limited informaion avaible.

Ziemkiewicz et al.[11] reported that 82 of the 83 studied artificial wetlands for the treatment of acid mine water in the eastern United States showed a positive effect. At the same time, however, point to the specifics of individual systems that do not allow unambiguous determination of the parameters involved in cleaning, and note that further research in this area is needed.

The research results can be used to determine the appropriate composition of wetland vegetation for optimal function of wetlands for water purification dump. They will be designed wetlands with such artificial vegetation composition to promote cleaning function and contribute to the restoration and rehabilitation of the country.

1. Flanagan N. E., Mitsch W. J., Beach K.: Ecol. Eng. *3*, 135 (1994).
2. Nyquist J., Greger M.: Ecol. Eng. *35*, 630 (2009).
3. Sheoran A. S.: Mine Water Environ. *25*, 23 (2006).
4. Ye Z. H., Whiting S. N., Qian J. H., Lytle C. M., Lin Z. Q., Terry N.: J. Environ. Qual. *30*, 1710 (2001).
5. Mays P. A., Edwards G. S.: Ecol. Eng. *16*, 487 (2001).
6. Johnson, D. B., Hallberg K. B.: Sci. Total Environ. *338*, 3 (2005).
7. Woulds C., Ngwenya B. T.: Appl. Geochem. *30*, 1710 (2004).
8. Mitsch W. J., Wise K. M.: Water Res. *32*, 1888 (1998).
9. Karathanasis A. D., Johnson C. M.: Mine Water Environ. *22*, 22 (2003).
10. Sheoran A. S.: Mine Water Environ. *25*, 23 (2006).
11. Ziemkiewicz P. F., Skousen J. G., Simmons J.: Mine Water Environ. *22*, 118 (2003).

3-P34
SIMULTANEOUS DETERMINATION OF METAL TRACES BY ICP-MS IN ENVIRONMENTAL WATERS USING SPE PRECONCENTRATION ON DIFFERENT POLYMERIC SORBENTS

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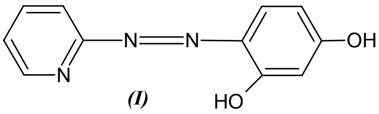
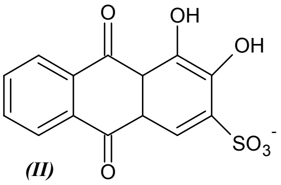
Direct determination of extremely low concentrations of trace elements by modern spectroscopic methods is often difficult. Limitations are associated not only with insufficient sensitivity of analytical equipments, but especially with matrix interference. For this reason, preconcentration and separation of trace elements from matrix are required. This study has been conducted for the determination of nine trace elements (Be, V, Co, Ni, Y, Cd, Pb, Th and U) by inductively coupled plasma mass spectrometry (ICP-MS) in natural waters and water samples after mining and processing of uranium.

An ICP-MS spectrometer Agilent 7500ce, with a plasma generator of 27.12 MHz, power output 1500 W and concentric nebulizer MicroMist™ with a cooled Scott chamber was used. The nonspectral interferences were eliminated by using a solution of internal standards ^{6}Li for ^{9}Be, ^{45}Sc (^{51}V), ^{72}Ge (^{59}Co, ^{60}Ni, ^{89}Y), ^{103}Rh (^{111}Cd), ^{209}Bi (^{206}Pb, ^{207}Pb, ^{208}Pb, ^{232}Th). The spectral interferences were eliminated by using a Helium collision cell during measurement. Monitored interfering elements were K^+, Na^+, Fe^{3+}, Ca^{2+}, Mg^{2+}, Al^{3+}, HCO_3^-.

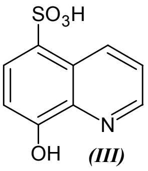
For the preconcentration non-polar sorbents based on polymeric sorbents (copolymers of styren-divinylbenzen, especially XAD-16 and XAD-1180) were used. Elements were retained as their chelates with different organic reagents - 4-(2-pyridylazo) resorcinol (PAR) ***(I)***, Alizarine S ***(II)***, 8-Hydroxyquinoline-5-sulphonic acid (8HQS) ***(III)*** and Ammonium pyrrolidinedithiocarbamate (APDC) ***(IV)***.



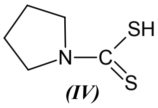
(I)



(II)



(III)



(IV)

The sorption process was studied under various conditions. The influence of pH is very important, because of the formation of metal chelates and their interaction with the sorbent. Sorbents were also conditioned with different surfactants: Benzyldimethyltetradecylammonium chloride (Zephyramine), 1-ethoxycarbonyl-pentadecyltrimethyl-ammonium bromide (Septonex®), Brij 35 or Sodium Dodecylsulphate (SDS).

Various elution mixtures were also tested, the best results were obtained for the mixture of acetone and ethanol (1:1) in the presence of 1 mol l^{-1} HCl. The excess of acid and organic solvents were evaporated in Teflon® dishes prior to the determination by ICP-MS.

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3-P35

SUITABILITY OF EXTRACTION METHODS FOR DETERMINATION OF DRUG RESIDUES IN THE RIVER SEDIMENT

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In this study, three different methods for extraction of drug residues from river sediments were tested. Sulphonamide antibiotics, which belong to a group of pharmaceuticals widely used in human and veterinary medicine[1,2], were selected as target compounds. They are discharged to the sewer systems together with the urine and faeces and enter to the wastewater treatment plant, where they are not completely removed. Their further negative impact on the ecosystem cannot therefore be excluded[3,4].

Sulphonamide antibiotics were isolated from sample of river sediment by three different methods: ultrasonic extraction (USE), pressurized solvent extraction (PSE) and microwave-assisted extraction techniques (MAE). These three methods were compared and the most suitable method was found. Samples were concentrated and cleaned using solid phase extraction (SPE). For determination these antibiotics high performance liquid chromatography with diode array detector (HPLC–DAD) was used.

The highest recoveries for all analytes were achieved by using PSE method with methanol as an extraction solvent. The most suitable conditions for PSE extraction are following: pressure 60 bar; temperature 40 °C; 2 cycle time; 7 min static time; 20 s purge time and 2 min nitrogen drying, when the drug recoveries were in a range of 65 – 85 % depending on the drug. The recoveries by using MAE method and methanol as an extraction solvent were in a range 40 – 65 %. When we used acetonitrile as an extraction solvent the recoveries dropped under 50 %. The lowest recoveries were achieved by using USE method, when the recoveries of all studied drugs with using methanol or acetonitril as an extraction solvent were under 50 %.

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REFERENCES

1. García-Galán M. J., Díaz-Cruz M. S., Barcelo D.: Trends Anal. Chem. *27*, 1009 (2008).
2. Ben W., Zhimin Q., Craig A., Zhanga H., Chenc L.: J. Chromatogr. A *1202*, 173 (2008).
3. Watkinson A. J., Murbyd E. J., Kolpine D. W., Costanzof S. D.: Sci. Total Environ. *36*, 2711 (2009).
4. Kümmerer K.: Chemistry *75*, 417 (2009).

3-P36

APPLICATION OF VOLATILE COMPOUNDS DISTRIBUTION FOR THE ASSESSMENT OF THE CZECH BEER AUTHENTICITY

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The Czech beer occupies privileged position at the world market due to its unique and unmistakable taste, which is result both of the quality of raw materials used for brewing and also the know-how of Czech brewers. The final product – finished beer – is from chemical point of view very complicated mixture of various compounds differing in their nature, chemical properties and taste effects, present in very different concentrations.

The main goal of this study was the evaluation of the possibility of Czech beer authenticity evaluation on the base of distribution of volatile compounds. For this purpose it is necessary in the first step to tune up suitable analytical procedure, as simple as possible, in the second step to analyse representative set of beer of Czech origin as well as imported brands and finally to evaluate the obtained dataset in order to find similarities in the beers of Czech production and their significant differences from the foreign products.

Solid Phase Microextraction (SPME) was the method of choice as simple, effective and fast method of isolation of volatile compounds from beer. Headspace sampling set-up was used; the removal of dissolved CO_2 which lowers the partial pressures of volatile compounds in the headspace was achieved by alkalization of the liquid phase - beer - by potassium hydroxide. Several fibers with various collecting phases were tested; temperature and time of sorption were optimized as well as the salting-out effect. Gas chromatography/mass spectrometry (GC/MS) and orthogonal comprehensive gas chromatography with fast TOF-based mass spectrometry (GCxGC-TOF MS) were used as final analytical methods. Using this analytical procedure set of beer of various origin has been analysed; obtained data are still under evaluation.

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3-P37
MOBILE RAMAN SPECTROMETER IN SERVICE OF FIRE BRIGADE UNITS

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This contribution describes the first pieces of experience gained during the evaluation of newly purchased mobile Raman spectrometer Ahura First Defender for the support of Fire Brigades Units in our country. The main goal of this instrument is the fast *on-place* identification of unknown compounds. This new instrument was subjected to thorough evaluation with the aim to evaluate the possibilities, to specify the advantages and manly to find weaknesses and limitations of this instrument before its introduction to regular service.

The instrument First Defender (mass 1,85 kg) is based on the principle of Raman spectrometry. The excitation laser diode power is up to 300 mW at a wavelength of 785 ± 0,5 nm (red light), working range is 250 – 2875 cm^{-1}. Instrument is able to scan spectra either in original packing (in case it is transparent for visible light – glass, PET bottles, plastic bags) or in 4 ml glass vials placed in measuring holder of the instrument. Spectra of spilled liquids could be obtained directly by measuring from the distance of several centimeters from the surface; the same method is applicable for solid substances. The instrument automatically controls the time of scanning in order to achieve necessary quality of obtained spectrum. For the identification of unknown compound the instrument is equipped with the IR spectral library containing several thousands of entries (3200 or more[1]). The experimental spectrum is compared with the library and the list of hits is presented on the instruments display together with appropriate spectra.

The possibilities of this instrument were checked up on the set of unknown compounds which were gathered by fire rescue brigade unit in the cellar of a basic school (69 compounds of various origin – inorganic as well as organic) and by other specially prepared binary and ternary mixtures of liquids and solids. The obtained results proved that the identification of individual compounds is usually succesful, but in the case of mixtures the results are much worse.

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REFERENCES

1. RMI, s.r.o.: Operating Manual for Raman spectrometer First Defender. RMI, Lázně Bohdaneč, 2007.

3-P38
ECOTOXICOLOGICAL EVALUATION OF SEDIMENTS

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Sediments of lotic and lentic water could be a source of inorganic and organic material in which significant conversion of cyclic organic compounds, and basic elements exists. On the other hand they can be a reservoir of a number of contaminants, which can enter the aquatic ecosystem. Sediments can hereby affect not only the aquatic ecosystem, but also the terrestrial system in the case that they will be used as fertilizer or as a material for the recultivation. The last possibility is to depone the sediment as waste material. In Europe hazardous wastes are classified by 14 criteria including ecotoxicity (H14). Appropriate ecotoxicity tests are used in accordance with valid Czech legislation to predict the ecotoxicity and the environmental impact of various matrices.

The aim of our study was the ecotoxicological testing of samples originating from differently contaminated water ecosystem in South Moravia. In bioassays following testing organisms were used: water crustaceans *Daphnia magna* and *Artemia Salina*, representative aquatic plants *Lemna minor* and seeds of terrestrial plant *Sinapis alba*. The values of 24hLC50 and 48hEC50 obtained for *Artemia salina* and *Daphnia magna* and 168hIC50 and 72hIC50 values gained for *Lemna minor* and *Sinapis alba* are the basic data for the ecotoxicological assessment of the sediments and for their classification following the Czech legislation.

Results of our study indicated that ecotoxicity of all tested sediments for all testing organisms were negligible. Values of calculated LC(EC, IC)50 were ≤ 10 ml l^{-1} which is limit value for declaration of ecotoxicity properties. It is necessary to point out the fact that ecotoxicity assessment folowing Czech legislation is based on the results obtained from ecotoxicity evaluation of the water leaches of waste prepared by distilled water. Therefore, it is important to deal with the principle of ecotoxicological testing of solid matrices and in the future as much as possible to classify the ecotoxicity on the basis of results obtained by the contact bioassays. In our opinion current set of tests is relevant only for water ecosystem and does not support sufficient prediction for eventual ecotoxicologic impacts on terrestrial ecosystem.

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3-P39

ECOTOXICITY OF SELECTED PESTICIDES

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Most of the chemicals produced and used in human activities entered to the ecosystem and end up in the aquatic ecosystem. Similarly many of chemical substances which are part of effective agrochemicals used in agriculture either to protect crop production or as fertilizer end up in the aquatic ecosystem, especially if they are soluble in water. Chemical analyses are able only to identify and quantify these xenobiotics but the prediction of the effects of these substances on ecosystem is possible via ecotoxicology which covers the assessment, monitoring and diagnosis of effects on populations, communities and ecosystems[1, 2].

The aim of our study was to evaluate ecotoxicity of selected substances used in agriculture and horticulture via aguatic organism *Daphnia magna*, which is the longest-used organism in ecotoxicology. It is representative of the zooplankton, sufficiently sensitive to toxic substances, easily grown in laboratory conditions, it is capable of rapid reproduction and its biology, toxicology and specifics are already well studied[3]. Via *Daphnia magna* acute and chronic toxicity of commercially sold herbicides was determined: Finalsan (active substance nonanoic acid), the product of Neudorff GmbH KG Company and Dominator (active substance glyphosate, isopropylamine salt – IPA salt Gly) by Dow AgroSciences Ltd. Next tested substances were potassium nitrate and sodium nitrate – common agricultural fertilizers. Methodology of following standard methods were used: ČSN ISO 10706 Water quality - Determination of long term toxicity of substances to *Daphnia magna* Straus (*Cladocera*, *Crustacea*) and ČSN EN ISO 6341 Water quality, Determination of the inhibition of the mobility of *Daphnia magna* Straus (*Cladocera*, *Crustacea*)-Acute toxicity test. The values of EC50 or EC40 were calculated and the above mentioned substances according to the toxicity to aquatic organisms were classified (EU Directive 93/67/EEC Commission of the European Communities, 1996). Toxicity of tested substances decreased in the following order: IPA salt-Gly, nonanoic acid, KNO_3, $NaNO_3$.

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REFERENCES
1. Newman C. M., Clements H. W.: *Ecotoxicology a Comprehensive Treatment*. CRC Press, Taylor & Francis Group, LLC, 2008.
2. Håkan B.: Crop Protect. *20*, 897 (2001).
3. Enserink L., de la Haye M., Maas H.: Aquat. Toxicol. *25*, 111 (1993).

3-P40

MATRICES FROM FIRE PLACES - THEIR POSSIBLE ECOTOXICITY

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In the case of fires frequently wide range of inhomogeneous material is burnt. A lot of combustion products arising during this event may adversely affect the environment. Some of the burning products entered the atmosphere (gaseous components - carbon dioxide, carbon monoxide, sulfur dioxide and other), some of them are either adsorbed on the solid particules or dissolved in water based estinguishers (oganic compounds like PAHs, PCBs, TCDDs and other) and end up in soil or water ecosystem, where they can negatively influence aquatic and soil organisms[3]. It is possible to identify and quantify most of them by chemical analysis, but their impact on the ecosystem is not predictable on this basis. An effective tool to predict the impact of combustion products on the ecosystem are ecotoxicity tests[2].

In our study, the water leachates of samples from fire places were prepared. Testing was carried out in accordance with current legislation on waste management when ecotoxicity of solid waste is determined on the basis of ecotoxicological values obtained by testing water leachates. Two alternative tests of ecotoxicity on aquatic organisms were used: Thamnotoxkit F^{TM} with organism *Thamnocephalus platyurus* and Daphtoxkit F^{TM} with *Daphnia magna.* Another testing aquatic organism was *Artemia salina*. Ecotoxicity was also tested using two standard phytotoxicity tests - white mustard (*Sinapis alba*) root growth inhibition tests and lesser duckweed (*Lemna minor*) growth inhibition test. On the basis of obtained ecotoxicological values LC (EC, IC)50 the impact of matrices from fire places on the ecosystem was evaulated. We can concluded that matrices do not exhibited significant ecotoxicity to the tested organisms with the exception of samples whose aqueous extracts showed foaming. We suppose with accordance to the literature that tensides used as aditives to the extinquishing agents are responsible for their ecotoxicity[3].

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REFERENCES

1. Eom I. C., Rast C., Veber A. M., Vasseur P.: Ecotox. Environ. Safe. *67*, 190 (2007).
2. Hoffmann D. J., Rattner B. A., Burton G. A., Cairns J. *Handbook of Ecotoxicology*. 2nd edition, Lewis Publisher 2003.
3. Cserháti T., Forgács E., Oros G.: Environ. Int. *28*, 337 (2002).

3-P41

ELIMINATION OF RESIDUES OF ESTROGENS DURING DRINKING WATER TREATMENT

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Presence of some compounds with very low concentrations in water ecosystems and in drinking water can have adverse effect on environment and human health (including drinking water sources). At the present time, attention is given especially to pharmaceuticals and hormones. We focused on the problem of occurrence of two estrogenic hormones – estradiol and ethinylestradiol in drinking water sources and on possibilities of their elimination during drinking water treatment.

Selected compounds occur in water environment in quantities on the level of even tens ng per liter[1]. They can cause feminization of fish males[2] and dysfunction of human (both male and female) reproductive system[3]. The above mentioned substances are carcinogens to humans[4].

An analytical method consisting of SPE preseparation, vacuum distillation and HPLC/MS/MS determination was chosen as a suitable tool for quantification of low concentrations of both tested compounds in water under conditions of technological tests. Efficiency of coagulation using ferric sulphate as a coagulant under optimized conditions and efficiency of pulverised activated carbon adsorption were tested in laboratory scale for elimination of these compounds from two types (natural river water and model water with increased level of humic matter) of artificially contaminated water. Efficiency of ozonation in elimination of tested compounds from artificially contaminated demineralised water was also tested. Conditions of technological tests were chosen with respect to real conditions in technical practice.

Relatively low efficiency of coagulation was found during the treatment of artificially contaminated natural (river Svratka Brno, Czech Republic,) and model water under conditions optimized for elimination of non-specific organic pollution. The efficiency was approximately at the level of reduced values of characteristic indicators of non-specific organic matter content. Obtained low efficiency is consistent with results of other authors[5, 6].

On the other hand, the lowest operationally useable dosages of activated carbon or ozone lead to almost complete elimination of selected compounds from artificially contaminated demineralised water. The above phenomenon is caused by hydrophobic character of these compounds[7] and by the presence of phenolic structure in molecule[8]. The data for determination of parameters of numeric models of adsorption isotherms were obtained in adsorption experiments. These models allow finding proper conditions for adsorption of contaminants from contaminated water.

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REFERENCES

1. Ying G. G., Kookana R. S., Ru Y. J.: Environ. Int. *6*, 545 (2002).
2. Kidd K. A., Blanchfield P. J., Mills K. H., Palace V. P., Evans R. E., Lazorchak J. M., Flick R. W.: Environ. Sci. *21*, 8897 (2007).
3. Pohanish R. P.: *Sitting's Handbook of Toxic and Hazardous Chemicals and Carcinogens: 5th Edition*. William Andrew Publishing, Norwich 2008.
4. http://monographs.iarc.fr/ENG/Classification/index.php, [cit. 2011-30-04].
5. Bodzek M., Dudziak M.: Desalination *1–3*, 24 (2006).
6. Chen Ch. Y., Wen T. Y., Wang G. S., Cheng H. W., Lin Y. H., Lien G. W.: Sci. Total. Environ. *3*, 352 (2007).
7. Westerhoff P., Yoon Y., Snyder S., Wert E.: Environ. Sci. Technol. *17*, 6649 (2005).
8. Huber M. M., Canonica S., Park G. Y., von Gunten U.: Environ. Sci. Technol. *5*, 1016 (2003).

3-P42

THE CONTENT OF COOPER AND CHROMIUM IN FISH TISSUE FROM THE WATER RESERVOIRS IN GOLIANOVO AND KOLÍŇANY

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Metals in trace amounts normally found in living organisms, which are involved in important biochemical processes. On the other hand, metals have the ability to bioaccumulate in the body and increased quantities behave toxic. Fish, as living organisms in the aquatic ecosystem at the end of the food chain by becoming the latest recipients. Compared with warm-blooded vertebrates, the fish is characterized by continuous weight, and physical growth. This may affect the actual elements of metal bioaccumulation in fish.

Serious problem is the long-term contamination of surface water. Contamination of the aquatic environment caused by the disappearance of sensitive species, slowing their growth, deterioration of reproductive ability and condition, eventually reducing their resistance. Another serious aspect is the contamination of fish with foreign substances, causing deterioration, or impairment of the quality of meat and fish.

In the present work we focused on the determination of the risk elements studied in carp muscle (Cyprinus carpio L.) in water reservoirs in Nitra region (Golianovo and Kolíňany). The contents of copper and chromium in fish muscle was determined using microwave digestion of biological material. Ending analytical was AAS (Varian AA 240 FS). The obtained results oft he content of hazardous elements (Cu: 0,28 ± 0,11 mg kg^{-1} - Kolíňany and 0,40 ± 0,21 mg kg^{-1} - Golianovo) and (Cr: 0,15 ± 0,06 mg kg^{-1} - Kolíňany and 0,09 ± 0,06 mg kg^{-1} - Golianovo) were compared with the maximum accetable limits (MAL) of heavy metals, which are listed in the Codex Alimentarius SR. The contents of heavy metals in any case not exceed MAL.

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3-P43

THE CONTENT OF HEAVY METALS IN SEDIMENTS FROM THE THE WATER RESERVOIRS

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Lake sediment is a material dredged from the bottom of water reservoirs or ponds. It is the result of natural processes, they are actually wash away the agricultural and forest soils, streams and river tributaries. According to the flow velocity, settling velocity and water depth to identify the composition of bottom sediments as gravel and sand, clay or clay to clay material, in most cases with a high content of organic matter. According to the grain composition is mainly used in construction to a variety of construction or modifications to the fertilization of agricultural soils.

The work deals with the migration of selected heavy metals in small basins, which are bound in bottom sediments. Bottom sediments may act as significant sources of pollution, since they can accumulate pollutants such as heavy metals and organicsubstances.

In this work we focused on identification of risk elements in a small pond sedimetoch Nitra (Kolíňany and Golianovo) used for breeding fish for food purposes. Majority representation is abyčajný carp (*Cyprinus carpio L.*), which as is typical of benthic fish exposed to an increased impact of possible contamination risk elements of the bottom sediments. The content of the heavy metals (Cu and Cr) in sedimetoch we detect the mineralization of pre-wet samples. Analytical ending the AAS (Varian AA 240 FS). The results obtained content of hazardous elements (Cu – 4,0 ± 0,42 mg kg^{-1} - Kolíňany and 16,3 ± 3,57 mg kg^{-1} - Golianovo) and (Cr – 0,60 ± 0,53 mg kg^{-1} - Kolíňany and 36,6 ± 9,16 mg kg^{-1} - Golianovo) were compared with the limit values set by relevant legislation (Act no. 188/2003 Coll. Z.). All of us detected elements do not exceed the levels observed in either case the limit values.

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3-P44

CONTENTS OF CADMIUM AND NICKEL IN SOILS AND PLANT MATERIAL AFTER BIOSLUDGE APPLICATION IN VPP KOLÍŇANY

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The application of decomposed substrate after continual biogas production is one of the possible ways how to use alternative energy sources with following monitoring of its complex influence on the hygienic soil state with the emphasis on heavy metal input. The substances from bilge and drain sediments from water panels, also biosludge gained by continual co-fermentation of animal excrements belong among these compounds. The biosludge application is connected with possible risk of cadmium and nickel, also other risky elements input into the soil. The Cd and Ni fractions in soils were by the method selective sequential extraction (SSE) determined. By Cd fractionation it was found out that by the application of biosolid into the soil the deal of individual fractions was changed only minimally. The composition of 1-4 fractions presented by the plants as available Cd forms is in individual variants 11-13%. This knowledge is important, because the total Cd content in soil is higher at 48% as limitv value and real risk of its intake by plants is high. The total Ni content in soil is higher than limit value at 9%. By the method SSE it was found out that Ni in soil is occuring mostly in residual fraction with percentual structure 61-79%, fractions and available forms (1-4 fractions) form only 8-11% deal of total Ni content in soil. This fact is important and it suggests the low Ni plant uptake inspite of its high content in soil.

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3-P45
CONTENT OF RISK ELEMENTS IN SOIL AND WHEAT AROUND THE CHEMICAL FACTORY OF EAST SLOVAKIA

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Strednozemplínska locality - Strážske ranks to the regions that are monitored as one of nine most contaminated sites in Slovakia. Contamination in this region has anthrophogenic character that has been caused by industrial enterprises in Vranov nad Topľou, Strážske and Humenné. Concentrations of Cd were exceeded at all sampling points. ranged from 1,10 to 1,90 mg kg^{-1}, what present an increase of the limit value by 57,1 to 171 %. The content of Ni was increased in seven sampling points while its content ranged from 40,4 to 70,4 mg kg^{-1}. This present exceeding of limit values up to 40,8 %. The limit value was also exceeded for Pb, Hg and As. In the vicinity of chemical enterprise of Humenné the Co concentration ranged from 19,6 to 29,6 mg kg^{-1} at all sampling points and exceed the limit value from 30,7 to 97,3 %. Pseudototal content of selected elements showed local exceeding of all selected heavy metals in horizon B. In soil extract from horizon B of *aqua regia* the limit values of Cd, Pb, Zn, Ni, Cr and Co were exceeded. The Cd content in monitored horizon ranged from 1,3 to 2,0 mg kg^{-1} and exceed the limits at all sampling points. The highest value 2,0 mg kg^{-1} present an increase of 185 % compared to the limit value. The lowest concentration exceeded the limit value by 85 %. The Pb concentration ranged from 39,6 to 114,8 mg kg^{-1}. Lead belongs among high toxic metals and the background value in soils in Slovakia varies from 7,95 to 10,79 mg kg^{-1}. The limit value of Pb defined by legislative (70 mg kg^{-1}) was exceeded at two sampling points. The highest content represent an increase of 64 %, the second highest content presented an increase of 62.8 % in comparison with the limit value. Content of Ni ranged from 32,8 to 76,4 mg kg^{-1}. The highes value exceed the limit by 52,8 %. The content of Zn defined by legislative was exceeded at two sampling points. The highest value of Zn 164,8 mg kg^{-1} exceeded the limit by 9,8 %. Average content of Cu and Cr in soil from Slovakia ranged from 5,34 to 6,34 mg kg^{-1}, and from 1,78 to 1,87 mg kg^{-1}, respectively. At all of sampling points higher valeues of Cu and Cr were determined. The highest value of Cu exceed the limit value by 12,6 %. The highest assessed value of Cr 86,6 mg.kg^{-1} represented an increase of 24 % in comparison with the limit value. Evaluation of soil hygiene revealed increased content of Cd, Pb, Ni and Co, partially Zn, Cu and Cr, what reflects their contents in plant production, but increased moiety of mobile form besides Cd in soil was not manifested in increased content in production parts of grown crops. In grain of wheat (*Triticum aestivum,* L.) the highest acceptable amount (0,1 mg kg^{-1}) of Cd was exceeded in seven sampling points of chosen site. Increased content of Cd was in range from 0,12 to 0,18 mg kg^{-1}, what represents an increase of 20 - 80 % when compared to highest acceptable amount (HAA). The highest values of Cd and Ni in grain of oat (*Avena sativa,* L.) were found at sampling point 8. The highest value of Cd in grain exceed over 0,1-times the HAA.

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3-P46
THE TOLERANCE OF SELECTED GRASS SPECIES TO PETROLEUM STRESS

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The presence of petroleum hydrocarbons in soils is a negative factor for plant growth and development[1]. Their harmful effects include inhibition of seed germination, reduction of photosynthetic pigments, decreased nutrient assimilation and shortening of roots and aerial organs[2]. This toxicity may be exerted through the hydrophobicity of petroleum-polluted soils which induce drought stress[1], and also by the high biochemical oxygen demand (BOD) characteristic of these soils and their poor agronomical properties, both causing anoxic stress[3]. Phytoremediation has emerged as a viable biotechnology for remediation of petroleum polluted soils, f.e. with species like bermudagrass[4] or gabiroba[5].

We studied the influence of petroleum originated from Korňa petroleum spring (Northern Slovakia) to germination and growth parameters of 3 grass species - perennial ryegrass (*Lolium perenne* L.) cv. Kelt, red fescue (*Festuca rubra* L.) cv. Barborka and tall fescue (*Festuca arudinacea* SCHREB.) cv. Tulsa. The experiment was established with three treatment groups: 100% of petroleum sample, 50% of petroleum sample and control treatment (distilled water). Seeds germinated in Petri dishes under growth chamber constant conditions (T 20 °C, RH 60%, photoperiod 12 h light/12 h dark period, PPFD 400 μmol m^{-2} s^{-1}). We determined *Lolium perenne* resistance to petroleum stress, because of statistical analyse did not confirmed significant differences for all observed parameters (the length of germinated leaf and root, the number of roots, the fresh weight of germinated leaf and roots, the total dry weight) between control and petroleum treated plants. *Festuca rubra* and *Festuca arundinacea* belong to partially tolerant species to petroleum stress, because of statistical analyse confirmed significant differences only within some observed parameters between control and petroleum treated plants.

The results of our study can be practically applied for greening or recultivation of petroleum contaminated localities or petrol gas areas.

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REFERENCES
1. Feng Li. Y., Sawatsky N.: Plant Soil *192*, 219 (1997).
2. Adam G., Duncan H.: Environ. Pollut. *120*, 363 (2002).
3. Rentz J. A., Chapman B., Alvarez P. J. J., Schnoor J. L.: Int. J. Phytorem. *5*, 57 (2003).
4. Peña-Castro J. M., Barrera-Figueroa B. E, Fernández-Linares L., Ruiz-Medrano R., Xoconostle-Cázares B.: Plant Sci. *170*, 4 (2006).
5. Gogosz A. M., Bona C., Santos G. O., Botosso, P. C.: Braz. J. Biol. *70*, 4 (2010).

3-P47

CHLOROPHYLL FLUORESCENCE RESPONSE OF CORN SEEDLINGS TO LEAD STRESS

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All environmental factors, such as excessive irradiance, water or heavy metal stress either directly or indirectly affect the photosynthetic function of leaves, the pigment composition and the ultra-structure of the photosynthetic apparatus. Early stress detection in plants, before visual damage symptoms are noticeable, is required in order to reactivate the plant's vitality by suitable countermeasures. Today the non-destructive fluorescence methods are the most promising techniques for early stress detection in plants[1].

Corn (*Zea mays* L.) cv. Theresa seeds were germinated for 7 days in Petri dishes containing different concentrations of Pb^{2+} (50, 300, 500, 1000 and 1500 mg l^{-1} Pb solution). Lead was provided as lead nitrate $Pb(NO_3)_2$. Seeds germinated in Petri dishes under growth chamber constant conditions (T 20 °C, RH 60%, photoperiod 12 h light/12 h dark period, PPFD 400 $\mu mol.m^{-2}.s^{-1}$). Chlorophyll fluorescence measurements were realised by fluorescence spectrophotometry method (GFPCam kinetic fluorescence camera, PSI, Czech republic)[2]. We determined minimal fluorescence (F_0), maximal fluorescence (Fm) and maximum quantum yield efficiency (Fv/Fm) by fluorescence method. Statistical analysis confirmed significant influence of lead on minimal fluorescence (F_0) of corn seedlings between F_0 of control plants and all lead treatments. Treated plants had higher F_0 values than control plants. Although without insignificant differences between Fm of control and treated plants (with exception of 1500 mg Pb treatment with significant difference) lead application induced Fm increasing. The maximum quantum yield efficiency (Fv/Fm) decreased with increasing lead concentration, significant differences between Fv/Fm of control and treated plants were confirmed by statistical analyse. Average values of Fv/Fm lead treated plants were lower from 15,6 % to 20,4 % with increasing lead concen-tration comparing control plants.

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REFERENCES
1. Lichtenthaler H. K. , Langsdorf G., Lenk S., Buschmann C. Photosynthetica *43*, 355 (2005).
2. Trtílek M., Kramer D. M., Koblízek M., Nedbal L.: J. Lumin. *72*, 74 (1997).

3-P48

CHLOROPHYLL FLUORESCENCE RESPONSE OF PISUM SATIVUM TO LEAD STRESS

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Lead (Pb) contamination in soil is a widespread phenomenon and originates from automobiles, metal smelting plants, mines, lead-contaminated sewage sludge, industrial wastes, etc. The Pb exposure to plants causes effects such as the disturbance in mitosis, induction of leaf chlorosis, depression of photosynthetic rate[1], inhibition in root and shoot growth[2].

We used the method of growth analysis and fluorescence spectrophotometry method (GFPCam kinetic fluorescence camera, PSI, Czech republic)[3] for determination of lead effect on growth processes and chlorophyll fluorescence of pea (Pisum sativum). Its transfer from soil was accelerated with phytoextraction induced by using the chelating agent EDTA (ethylenediaminetetraacetic acid). Application of lead caused non-significant reduction of plant height, fresh weight and dry weight of aboveground biomass. A stronger effect of lead was presented on the root system, we have found a reduction of root weight from 5,3 to 14% comparing with untreated plants. In fact, we have presented higher lead content in roots by the chemical analysis by AAS (atomic absorption spectrometry), than in aboveground biomass. The most pronounced effect of lead was found on the fresh weight of root biomass after the addition of EDTA, with increasing concentration of the chelating agents the fresh weight decreased from 36% to 56%. Chelating agent strongly supported the income of observed metal. We determined lead content in the aboveground part of plants from 17 to 43 times higher and from 0,5 to 2,5 times higher lead content in roots than in lead treated plants. The maximum quantum yield of chlorophyll fluorescence (Fv/Fm) gradually decreased with increasing concentration of lead and EDTA. Application of heavy metal and EDTA caused a statistically significant reduction in Fv/Fm from 9% to 20% and consequent reduction in photochemical efficiency of PSII photosystem. Inhibition of photosynthesis and biochemical processes of photosynthesis means the change in physiological status of plants, and therefore measurement of chlorophyll fluorescence can be used as an indicator of the impact of environmental stress on the functionality of photosynthesis apparatus. Results of this work can be used in the field of phytoremediation of heavy metals contaminated areas.

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REFERENCES
1. Parys E., Romanowska E., Siedlecka M., Poskuta J. W.: Acta Physiologiae Plantarum *20*, 3 (1998).

2. Hadi F., Bano A., Fuller M.P.: Chemosphere *80*, 4 (2010).
3. Trtílek M., Kramer D.M., Koblízek M., Nedbal L.: J. Lumin. *72*, 74 (1997).

3-P49
EFFECT OF CADMIUM AND ARSENIC IONS ON THE CONTENT OF PHOTOSYNTHETIC PIGMENTS IN LEAVES OF GLYCINE MAX (L.) MERILL.

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Heavy metals belong to significant pollutants of the environment since they accumulate in organisms and are not degradable. The goal of our research was to determine the content of photosynthetic pigments in the different developmental stages of leaves of studied soybean varieties (*Glycine max* cvs. Bólyi 44 and Cordoba) exposed to cadmium (50 mg kg^{-1} Cd^{2+}) and arsenic (5 mg kg^{-1} As^{3+}). After 10 days of growing the plants in contaminated soil, leaves of cv. Cordoba responded sensitively to Cd^{2+}. In plants of cv. Cordoba we observed statistically significant decrease of *chlorophyll a* content in lower (by 30,86%) and upper leaves (by 19,09%). Consequently, significant decrease of *chlorofyll a+chlorofyll b* in lower (by 14,73%) and upper leaves (by 14,21%), as well as decrease of *chlorofyll a/chlorofyll b* in lower (by 15,93%) and upper leaves (by 11,35%) were detected. We did not record any significant change in content of *chlorophyll b* and *carotenoids* as a result of the Cd^{2+} effect on this variety. In contrast, no such changes occured in cv. Bólyi 44. The applied dose of As^{3+} did not induce significant changes in the content of photosynthetic pigments in either of studied soybean varieties. The applied dose of Cd but not As negatively affected the photosynthesis in dependency on genotype.

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3-P50
CHLOROPHYLL FLUORESCENCE IN LEAVES OF SOYBEAN AS INDICATOR OF CADMIUM STRESS

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Photosynthesis is sensitive to cadmium, because it directly disturbs the chloroplast function by inhibiting the activities of enzymes of chlorophyll biosynthesis and CO_2 fixation. The intensity of chlorophyll fluorescence can be used as an indicator of environmental stress effects on photosynthesis apparatus effectiveness. The goal of our research was to determine the parameters of chlorophyll fluorescence (F_0 – minimum fluorescence, *QYmax* – the maximum quantum yield of Photosystem II photochemistry) in the different developmental stages of leaves of studied soybean varieties (*Glycine max* (L.) Merill. cvs. Bólyi 44 and Cordoba) exposed to cadmium (50 mg kg^{-1} of soil). We examined differences in tolerance index value of two genotypes based on fresh weight of shoots (95,73% for Cordoba, 97,62% for Bólyi 44). In plants of cv. Cordoba we observed statistically significant decrease of F_0 only in upper leaves 1 (by 9,85%). Differences in *QYmax* were observed in lower leaves of cv. Cordoba (decrease by 11,94%) and in upper leaves 1 and upper leaves 2 of cv. Bólyi 44 (decrease by 15,52% and 18,18% respectivelly). We examined genotypic variability in chlorophyll fluorescence and differences in developmental stages of leaves in consequence of the applied Cd^{2+}.

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3-P51
CONTAMINATION LEVEL OF UPPER FLOW RIVER NITRA ALLUVIAL AREA AFFECTED BY LOCAL INDUSTRIAL ACTIVITY

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Present state-of-art of environment is widely affected by various impacts of man which significantly eleminate negative affecting of its influence on the environment. In the past this fact was not so implemented and thus there was uncontrolled escape of contaminants of organic, but also inorganic origin into various components of environment. The most sensitive component is water ecosystem and its close plates (base sediments, banks sediments and biosphere near the flows).

River Nitra ranks among the most affected water ecosystems in Slovakia that was in 1965 the recipient of sludge, that was by accident in Zemianske Kostoľany uncontrolled spilled into the river with aftermath of long–term contamination of all sub-components in ecosystem, mainly by heavy metals (Hg, As, Pb, etc.).

In our work there was shown that the level of banks sediments contamination in River Nitra exceed severalfold the limit values, mainly for Hg. Total Hg concentration in bank sediments samples that were dried before analysis to constant weight was assessed using an AMA 254 (AAS).

Middle value of total content of Hg was 1,064 ± 0,612 $mg.kg^{-1}$. The highest concentration was recorded at sampling site „Bošany“ (1,893 mg kg^{-1} Hg) what presents almost 4-fold exceeding of limit value (0,5 mg kg^{-1}).

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3-P52

CHIRAL ANALYSIS OF DRUG RESIDUALS IN WASTE WATERS

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Many of the pharmacologically active substances present in the environment are chiral. However, there is only little data concerning their fate in the environment on the enantiomeric level. Chiral pharmaceuticals are known to exhibit stereoselective behaviour when situated in biological system such as human body. Therefore, stereoselective behaviour of chiral drugs is very likely to happen in the environment. Since the drug enantiomers could differ in toxicity, it is of great importance to develop chiral analytical methods capable of trace analysis in the environmental matrices.

Enantioselective HPLC-DAD method using Chiralpak AD as a chiral column was developed for the analysis of two non-steroidal anti-inflammatory drugs ibuprofen and ketoprofen in wastewater samples. The samples were collected from the municipal wastewater treatment plant in Brno-Modřice and then extracted by SPE using Oasis HLB cartridges. Before the chiral analysis was applied, the sum of enantiomers was quantified by HPLC-ESI-MS. After this, the samples were analyzed by chiral HPLC using diode array detector.

It was found out that the diode array detector is not sensitive and selective enough for the chiral analysis of wastewater samples. Drugs are generally present in the wastewater in very small amounts and when they are divided into individual enantiomers, their concentrations are too low for UV detection and detection by mass spectrometry is needed.

Since the developed chiral method is using normal phase liquid chromatography it is not compatible with electrospray ionization which was used when the sum of enantiomers was determined.

It was concluded that if the developed chiral HPLC method should be applied for the environmental purposes it must be used in combination with mass spectroscopy and atmospheric pressure chemical ionization.

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3-P53

DETERMINATION OF SYNTHETIC MUSK COMPOUNDS IN WASTE WATER

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Synthetic musk compounds or synthetic fragrances are organic substances commonly used as fragrant constituents of perfumes, detergents, cosmetics and personal care products. These compounds are discharged after use via domestic wastewater and sewage treatment plants to the aquatic environment. The result is accumulation in the environment and occurrence in a food chain especially of aquatic and marine ecosystems. Synthetic musk compounds are also found in surface water, fish tissues and also in body fluid (blood, urine, milk) and tissues of human body. Synthetic musks have raised considerable attention in environment chemistry because of their persistence, bioaccumulation potential and toxicity.

This study is focused on 5 nitromusk compounds [musk ambrette, musk xylene, musk moskene, musk tibetene, musk ketone] and 4 polycyclic musks [phantolide, traseolide, galaxolide, tonalide]. These compounds were studied in influent and effluent waters from waste water treatment plant in Brno-Modřice (February 2010). The optimization method solid phase microextraction (SPME) was used for isolation of analytes from samples. The head final parameters are: SPME fiber PDMS/DVB (65 μm), direct immersing, temperature 80 °C, exposure time: 30 min. The identification and quantification of analytes was carried out by gas chromato-graphy-mass spectrometry (GC 6890N, capillary column: DB-5MS; MSD 5973N, EI 70 eV, Q-SIM) [Agilent, USA]. The head final GC parameters are: injector in the splitless mode at 250 °C, constant flow of carrier gas He (0,8 mL min^{-1}), oven temperature program: 50 °C (hold 3 min), rate 15 °C/min to 110 °C, rate 5 °C/min to 165 °C (hold 8 min), rate 15 °C/min to 285 °C (hold 2 min) [total time: 36 min][1].

Galaxolide and tonalide were detected at highest concentrations – 2765 ng l^{-1} for galaxolide and 422,8 ng l^{-1} for tonalide. The most extended nitromusks were found at concentrations 89,85 ng l^{-1} (for musk ketone) and 40,39 ng l^{-1} (for musk xylene). Musk moskene and musk tibeten were not detected in any sample analysed. The overall removal efficiency of synthetic musks from the waste water in the WWTP ranged from 37,43 % to 99,99 % (e.g. for galaxolide 55,48 % and for musk ketone 39,10 %).

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REFERENCES

1. García-Jares C., Llompart M., Polo M., Salgado C., Macías S., Cela R.: J. Chrom. A *963*, 277 (2002).

3-P54

DETECTION OF POLYCHLORINATED BIPHENYLS IN AIR AT JAMES ROSS ISLAND, ANTARCTICA

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Herewith we present results of analyses of polychlorinated biphenyls (PCBs) in the air from the northern part of James Ross Island (JRI). The presence of these compounds in these far-away areas is induced by long-distance transport.

Passive sampling using polyurethane foams (PUFs) as collecting medium was used for this purpose. The sampling periods were one month (Jan-Feb) in 2009, 2011 and one year in (Jan-Jan or Feb-Feb) 2009/2010, 2010/2011 at two different sites. Contamination in Crame Col is influenced by prevalent south winds, whereas below the Berry Hill by dominant westerly winds due to a different topographic position.

Exposed PUFs were extracted by dichloromethane using Soxhlet extractors and obtained extracts were cleaned-up by acidic treatment. Comprehensive orthogonal two-dimensional gas chromatography with TOF-based mass spectrometric detection (GCxGC-TOF MS) was employed as the final analytical method.

Total of 32 PCBs containing from 3 to 7 chlorines were identified. For the identification and quantification of indicator congeners PCB #28, #52, #101, #138, #153 and #180 the external standard method was used. Classification of other polychlorinated biphenyls was based on their mass spectra and retention behaviour.

Supposing sampling rate of passive air sampling devices about 10 m^3 per day, the average atmospheric concentrations of the sum of all of 32 PCBs detected congeners varied between 1,5 and 9,8 pg m^{-3} for one month austral summer sampling and for sum of indicator congeners between 0,8 and 4,5 pg m^{-3} for one-year sampling.

The contamination evaluated during one-year exposition was higher in Crame Col than below the Berry Hill; one-month austral summer exposition of PUFs showed on the other hands higher contamination below the Berry Hill. Meteorological and geomorphological conditions principally affect timing and distribution of PCB contaminants in the air above the JRI.

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3-P55

DETERMINATION OF SURFACTANTS BY CAPILLARY ZONE ELECTROPHORESIS

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Capillary zone electrophoresis is a technique with a wide range of applications. The most commonly, this method is used in medicine in the separation of proteins, lipids, enzymes, nucleic acids, etc. This method reached great importance also in the pharmaceutical industry, where it is used to analyze drugs and their metabolites and the important role capillary zone electrophoresis play also in determination of environmental pollutants[1,2].

Surfactants are substances with anti-corrosion, emulsifying and disinfecting properties. They represent an essential ingredient for hair products, cosmetics, detergents and cleaning products. Increasing consumption of these products increases the amount of surfactants in the environment. Even trace amounts of surfactants can disrupt the biological balance, and reduce the surface tension of water. In this way, these agents reduce aeration of the water, which encourages such undesirable growth of algae. Some surfactants are highly toxic and poorly biologically degradable substances. From these reasons it is very important their monitoring in the environmental compartments. Surfactants can be divided into four groups: anionic, cationic, nonionic and ampholytic[3,4]. So far, anionic surfactants are the most used in the Czech Republic. That is why the work is focused on their determination by capillary electrophoresis.

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REFERENCES

1. Blažej A., Hodul P., Markušovská E., Novák L., Paulovič M., Vyskočil I.: in the book *Surfactants*, Alfa, Bratislava (1997).
2. Heinig K., Vogt C., Werner G.: J. Chromatogr. A *745*, 281 (1996).
3. Myers, D.: *Surfactant Science and Technology*. 3rd edition, Hoboken: Wiley-Interscience, 2006.
4. Fainerman, V. B., Möbius, D., Miller, R.: *Surfactants: Chemistry, Interfacial Properties, Applications*. 1st edition, Elsevier Science, Amsterdam 2001.

3P-56

SIM – GC/MS DETERMINATION OF BTEX IN AMBIENT AIR

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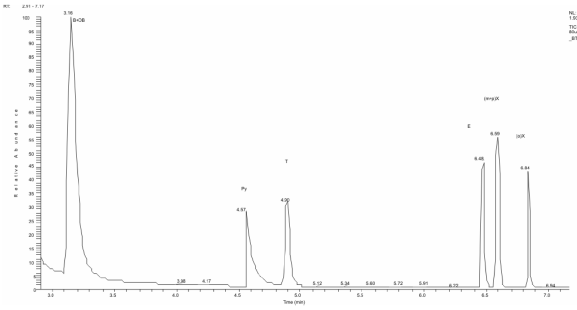
The assessment of air pollution and its public health impacts in an evolving world has become increasingly important, the air quality, both indoors and outdoors, being closely related to morbidity and mortality from respiratory and cardiovascular diseases.

This research has the aim to develop a quantitative method for determination of benzene (B), toluene (T), ethylbenzene (E), and isomeric (*o-*, *m-*, *p-*) xylenes (X) levels in ambient air by gas chromatography - mass spectrometry (GC/MS). BTEX are toxic compounds with high impact on human health. The method was applied for BTEX levels determination in the indoor/outdoor air. As internal standard were used pyridine or deuterated benzene.



Scheme 1. BTEX separation by SIM-GC/MS

The adopted method proved to be simple and fast, with good linearity, precision and accuracy, its validation demonstrating the capacity to reliably identify and measure the yields of BTEX compounds.

REFERENCES

1. Culea M., Cozar O., Melian C., Ristoiu D.: Indoor Built Environ. *14*, 241 (2005).
2. Defino R. J., Gong H., Zin W. S., Hu Y., Pellizzari E. D.: J. Expo. Anal. Environ. Epidemiol. *13*, 348 (2003).
3. Weisel C. P., Park S., Pyo H., Mohan K., Witz G.: J. Expo. Anal. Environ. Epidemiol. *13,* 393 (2003).
4. Nicoara S., Culea M., Nica A, Culea E., Cozar O.: Indoor Built Environ. *14*, 405 (2005).
5. Jamieson S., Dimitroulopoulou S., Brown V, Simion H., Colvile R.: Indoor Built Environ. *14*, 259 (2005).
7. Solomon S. J., Schade G.W, Kuttippurath J., Ladstatter – Weissnmayer M., Burrows J. P: Indoor Built Environ. *17*, 260 (2008).

3P-57

TRACE OF HEAVY METALS IN FISH TISSUES, SEDIMENT AND WATER FROM DANUBE RIVER IN ROMANIA

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The presence of heavy metals in aquatic ecosystems is the results of two main sources of contamination: natural processes or natural occurring deposits and antrophogenic activities[1-4].

In this study, some heavy metals (manganese, cadminum, nickel, chromium, zinc, iron, lead and copper) were determined in water, sediment and some tissues of *Gobius cephalarges* from Danube River, which is an important source of food and drink for populations and for agriculture in Romania.

Metals concentrations were measured using graphit furnance atomic absorption spectrophotometer 650 Zeenit, from Analytik Jena. Fish tissues and sediments were extracted using wet digestion method. The quality control of digestion was tested using Certified Reference Material that lead to a recovery of 95-110 %.

The obtained results for water were compared with national water quality guidelines. The analysis of heavy metals in sediments indicated no exceeded of recommended levels. Fe was maximally accumulated, followed by Ni, Cu, Pb, Mn, Zn and Cd.

In the *Gobius cephalarges* samples, the heavy metal concentrations were found to decrease as Zn > Fe > Cu > Ni > Mn > Cd > Pb. Heavy metal concentrations in fish samples were found to be above than maximum tolerable values provided by national law, which lead that the fish is safe to be eaten.

REFERENCES

1. Iordache A., Sandru C., Geana I., Bara L., Ionete R., Culea M.: Bulletin UASMV Animal Science and Biotechnologies *67*, 1 (2010).
2. Ekeanuyanwu C. R., Ogbuinyi C. A., Etienajirhevwe O. F.: Ethiopian Journal of Environmental Studies and Management *3*, 3 (2010).
3. Ozturk M., Ozozen G., Minareci O., Minareci E.: Iran. J. Environ Health. Sci. Eng. *6*, 73 (2009).
4. Ahmad A. K, Shuhaimi-Othman M.: J. Biomed. Sci. *10*, 93 (2010).

3P-58

INVESTIGATION AND MONITORING OF AMBIENT POLLUTANTS OF ATMOSPHERE FROM IN SITU COLLECTED SAMPLES IN AN INDUSTRIAL AREA

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The air pollution monitoring is one of the most pressing environmental problems today. A direct air pollutant is one that is emitted directly from a given source as products of combustion. They are classified as either directly released or formed by subsequent chemical reactions, the atmosphere being a proper medium for pollutants to spread in the environment. The paper describes the air pollutants monitored in the environmental system of an industrial area, in the southern-east of Romania, near Calarasi.

Air pollution measurement strategy may differ from area to area, depending on a number of factors such as geographical location, temperature, wind and humidity, the pollution being differently dispersed.

Portable analyzers, part from a complex system of mobile laboratory, were used for in situ analysis. These automatic analyzers monitored a wide range of pollutants besides meteorological parameters like wind speed, wind direction, temperature and humidity. The most air significant pollutants to asses include carbon monoxide, nitrogen monoxide, nitrogen dioxide, nitrogen oxides, sulfur dioxide on ambient atmosphere, and sulfur dioxide on exhaust gases from furnace, particulate matter, ozone, methane and total hydrocarbons. In addition to these conventional parameters, we also monitories special parameters like NH_3, H_2S. The carbon monoxide, sulfur dioxide, nitrogen dioxide, ozone and particulate matter in the outdoor atmosphere concentrations were measured and recorded according with the Romanian legislation no. 592/25.05.2002, and no 462/01.07.1993.

REFERENCES

1. Michulec M., Wardencki W., Partyka M., Namiesnik J.: Crit. Rev. Anal. Chem. *35*, 117 (2005).
2. Manoj A., Rajput S. P. S.: Int. J. Environ. Sci. *1,* 1069 (2011).
3. Kansal A., Khare M., Sharma C. S.: Atm. Poll. Res. *2,* 99 (2011).
4. Kankal S. B., Gaikwad R. W.: Adv. Appl. Sci. Res. *2*, 63 (2011).

3P-59

EFFECT OF POLYCICLIC AROMATIC HYDROCARBONS ON THE ENVIRONMENT OF ERBIL CITY

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Decades of research have provided the scientific foundation for understanding the role of the environment in disease. For many pollutants, scientists know with some certainty that exposure to these pollutants, at sufficiently high concentrations; can cause a variety of health effects. For other pollutants, where scientific evidence is less conclusive, scientists can only establish an "association" between exposure and health problems. Some effects on health may be short-term and reversible, such as irritated eyes from smog. Other effects, such as emphysema, heart disease, and cancer are chronic or even fatal. Some effects may appear shortly after exposure. Others, such as cancer, may require a long lead time before the disease appears. In many cases, pollution likely is just one of several factors—including diet, exercise, alcohol consumption, and genetic make-up—that influence whether an exposed person will ever become sick. Further complicating the picture is the fact that several segments of the population may be at higher risk for damage or disease from environmental pollutants. Potentially sensitive groups include children; older Americans; people with existing health problems such as diabetes, respiratory disease, or heart disease; and persons with compromised immune systems, including those who have HIV/AIDS or are undergoing cancer chemotherapy. Many studies in people have demonstrated an association between environmental exposure and certain diseases or other health problems. Examples include radon and lung cancer; and cancer arsenic in several organs; lead and nervous system disorders; disease-causing bacteria such as E. coli O157: H7 (e.g., in contaminated meat and water) and gastrointestinal illness and death; and particulate matter and aggravation of heart and respiratory diseases. In this work the effect of polycyclic aromatic hydrocarbons pollutants on the environment of Erbil City was studied.

3P-60

SPATIAL AND TEMPORAL DISTRIBUTION OF PHOSPHORUS IN COASTAL MARINE SEDIMENT FROM THE NORTHWESTERN ARABIAN GULF

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This study reports the spatial and temporal variability of total phosphorus (TP) and its forms in sediments from Kuwait Bay, a semi-enclosed water body in the northwestern corner of the Arabian Gulf. The mean (and range) concentration of TP in surficial sediments is 19,4 (12,2 to 24) μmol-P g^{-1} dry weight. Of this, inorganic phosphorus (IP) comprised the largest reservoir of phosphorus, contributing about 83,5 ± 6,8% with organic phosphorus (OP) contributing 16,5 ± 7,3% of the measured TP. The highest concentrations were measured in the deeper portions of the Bay which is characterized by fine grained sediments. The vertical profile in the sediments was characterized by a gradual increase in concentration from depth to a subsurface maximum (at approximately 4-5 cm) followed by an exponential decrease to the sediment-water interface. The sedimentary profile of TP correlated remarkably well with changes in the population of Kuwait over the last three decades, suggesting that wastewater inputs into the Bay from treatment plants is an important source of phosphorus in the Bay. The decrease in concentration in sediment deposited over the last five years maybe as a direct consequence of the commissioning of an improved wastewater treatment plant with higher phosphorus removal efficiency. This study shows how better wastewater management practices can lower the levels of phosphorus inputs to coastal marine waters and reduce the likelihood of eutrophication particularly in semi-enclosed water bodies.

3P-61

CLUSTER ANALYSIS APPLIED TO ENVIRONMENTAL SEDIMENT DATA IN SULAIBIKHAT BAY, KUWAIT

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Statistical technique was used in this work to assess the controls on the importace of the different geochemical fractions as hosts for trace elements and to identify possible pollution sources. Surface sediments samples were collected from 35 locations in Sulaibikhat Bay, Kuwait. Concentrations of As, Cd, Co, Cr, Cu, Hg, Ni, Pb and Zn were determined. Grain sizes, TOC, carbonate and mineralogical data were determined too. The abundance of elements, their geographical distribution and their relationships with each other and with sediment and other environmental parameters are described. Cluster analysis was applied to the data matrix to identify the possible pollution sources. The cluster analysis identifies two cluster groups, suggesting that the sediment geochemistry in Sulaibikhat Bay is strongly influenced by competitive sedimentation between coarse, aragonite-rich sediments on the one hand and fine-grained, TOC and trace elements rich material on the other hand. The cluster analysis results show that the compositions of sediment sampled near the Ghazali outfall differ markedly from those of sediments from Sulaibikhat Bay. The statistical techniques applied in this study proved to be useful tools for interpreting the environmental data in Sulaibikhat Bay.

4-IL

MASS SPECTROMETRY AND SDAP FOR IDENTIFICATION OF PR-PROTEINS/TYPE I ALLERGENS FROM PLANTS

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Using of plants with improved resistance against pathogenic microorganisms or substances which are not malicious for living organisms but are able to support the natural resistance of plants, an increase of productivity and quality of agriculture was expected in a near future. From this reasons, plants with higher content of PR-proteins (pathogenesis-related pro-teins) or substances with the ability to induce their increased production gained the attention of researchers and growers. Some of PR-proteins were successfully expressed to the gene-tically modified plants.

Rapid increase of type I allergy on agricultural crops coerced the researchers to focus on the health risks bound with the cultivation of such plants. Some of PR-proteins were directly convicted as a reason of so-called latex allergy, syndrome of oral allergy or cross-reactivity allergy of fruits, vegetable and pollen. The problem is so mandatory that the WHO and FAO permanently deal with. Already in 2001 (January 22nd – 25th) FAO/WHO Allergenicity Rules based on the cross-reactivity of structurally similar proteins were formulated. Simplified, they are based on identity of six consecutively bound amino acids in a searched protein and in allergen or more than 35% identity of amino acids between them (at least 80 amino acids consecutively bound are required). The following conditions lead to a high probability, that the searched protein is an allergen, too. Specialized databases containing all known structures of protein allergens were established. One of them, free Structural Database of Allergenic Proteins (SDAP) is refreshed each third month[1]. Except of a database of allergens it offers the direct alignment of a new structure with known ones.

With the increasing information about sequences of genes and proteins in databases, it becomes more and more easy to provide complex analysis of protein series using methods known from proteomics, the two-dimensional electrophoresis and mass spectrometry. In the last time, the term allergenomics is used for the fast and complex analysis of potential protein allergens. The same strategy as in proteomics is applied. Allergenomics allows us to detect and find putative allergens (proteins interacting specifically with IgE antibodies in the patient body) in a very short time as well as to analyze qualitative and quantitative changes in antigens in dependence on the environment leading to the allergic reaction.

An exemplary allergenomics starts with the protein extraction after allergic reaction. The extracted proteins are separated by two-dimensional electrophoresis and then transferred to a membrane and led to interact with the patient serum. Subsequently, the antigens specifically interacting with IgE antibodies are detected. The next step is the “in-gel digestion” with some specific proteases, mostly trypsin. The generated peptide fragments are extracted from the gel and analyzed by mass spectrometry to obtain “peptide mass map” (typical “finger-print” for defined structure). From some intense peptide ions are then obtained MS/MS spectra (tandem mass spectra) which interpretation represents directly the amino acid sequence of analyzed peptide. The comparison of obtained data with databases leads mostly to the direct identification of the allergen. The actual allergenicity of identified candidate have to be confirmed with other techniques as for instance leather or histamine test because of disproportionate rate between IgE-binding activity of the protein and the ratio of induced symptomps.

In the case, that the sequence is not registered in any database the identification requires so-called *de-novo* sequencing. The interpretation of obtained tandem mass spectra offers partial amino acid sequences of analyzed peptides what can lead to identification of unknown protein on the basis of its similarity with known ones in the database. Fortunately, just PR-proteins have unusually high homology caused by relati-vely fixed protective mechanisms of higher plants during evolution. In the same conditions, the most plants produce or accumulate structurally and functionally similar PR-proteins regardless of their morphological differences. Some enzymes, as they necessary for biosynthesis of low-molecular antibiotics or some reserved proteins in seeds and fruits belong to PR-proteins. As a consequence, PR-proteins were classified to 17 families regardless of their variety[3], where the sequence similarities, serological and immunological relationship and similarities of enzymes were the basis for this classification.

This presentation will be focused on the PR-proteins of family 10. For interest, their action pattern in the plant protection is fully unknown. Their similarity within the family of *Apiaceae* and their cross-reactivity with type I allergens represented with the well-known pollen allergen of *Betula verrucosa* (birch) caused by the similarity of their tertiary structures will be discussed, too.

This contribution is the result of the project implementation: Centre of excellence for glycomics, ITMS 26240120031, supported by the Research & Development Operational Programme funded by the ERDF (50%). The financial support from the Institutional Research Plan No. MO0FVZ0000501 from the Faculty of Military Health Sciences, University of Defence, Czech Republic is greatly appreciated, too.

REFERENCES

1. http://www.fermi.utmb.edu/SDAP [cit. 2011-05-04].
2. http://dmd.nihs.go.jp [cit. 2011-02-05]
3. http://www.bio.uu.nl [cit. 2011-02-05].

4-L1

FOOD SAFETY FROM PERSPECTIVE OF MICROBIOLOGIST

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Food safety from perspective of microbiology is getting to be more important nowadays as incidence of foodborne diseases caused by new emerging pathogens is increasing. Microbiological criteria for foodstuffs and raw materials of plant and animal origin are controlled in the EU according to Regulation EC 2073/2005, but this regulation recommends testing only for major pathogens such as *Salmonella sp.*, *Enterobacter sp.*, *E. coli*, *Listeria monocytogenes* and coagulase positive staphylococci. No legislative criteria are set up to monitor the presence of other foodborne bacteria and viruses. People are usually infected with ingestion of contaminated food or water used for drinking or for washing.

Mycobacteria could be considered as possible thread for human health in some cases. *Mycobacterium avium* subsp. *paratuberculosis* (MAP) is an agent of the economically important disease in ruminants called paratuberculosis (Johne's disease). It is estimated that up to 50% of cattle herds in Europe are infected[1]. The disease is also increasingly being detected in wild ruminants. MAP is excreted into the environment via feces and milk of infected animals. Such contaminated environment could be the source of infection for other animals[2]. MAP also features a long-term ability to persist in the environment[3]. MAP is considered to play possible role in the etiology of Crohn's disease in humans due to the similarity of clinical symptoms with Johne's disease and the clinical trials[4].

Group of alimentary food-borne viruses includes not only human but also animal viruses that can infect humans. Diarrhea, gastroenteritis or in worse cases severe liver inflammation (hepatitis) could be observed. Foodborne viral infections are mostly caused by genera Norovirus (NoV) and Hepavirus (HAV)[5]. Hepatitis E virus (HEV) is new emerging pathogen, which is the leading causative agent of enterally transmitted hepatitis non-A non-B worldwide. Outbreaks of hepatitis E occur most commonly in developing countries with poor sanitation conditions and are usually associated with fecal contamination of drinking water or water for industrial purposes[6].

The aim of this presentation will be to provide an insight into the field of microbiology with the respect to food safety. New emerging pathogens and risk factors connected with their spread will be discussed. Additionally, available methodologies applicable for the control of food quality will be summarized and some cases will be presented.

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REFERENCES

1. Nielsen S. S., Toft N.: Prev. Vet. Med. *88*, 1 (2009).
2. Grant I. R.: J. Appl. Microbiol. *98*, 1282 (2005).
3. Whittington R. J., Marshall D. J., Nicholls P. J., Marsh A. B., Reddacliff L. A.: Appl. Environ. Microbiol. *70*, 2989 (2004).
4. Hermon-Taylor J., Bull T.: J. Med. Microbiol. *51*, 3 (2002).
5. Koopmans M., Duizer E.: Int. J. Food Microbiol. *90*, 23 (2004).
6. Vasickova P., Psikal I., Kralik P., Widen F., Hubalek Z., Pavlik I.: Vet. Med. Czech *52*, 365 (2007).

4-L2

CHARACTERIZATION OF MAGNETIC AND NON-MAGNETIC SOLID POLYMER MICROSPHERES

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Magnetically driven separation techniques using micro- and nanosized magnetic solid carriers have become common place in molecular diagnostics of microorganisms in recent years. Magnetic separation is one of modern methods to speed up and facilitate the previously used separation and purification procedures.

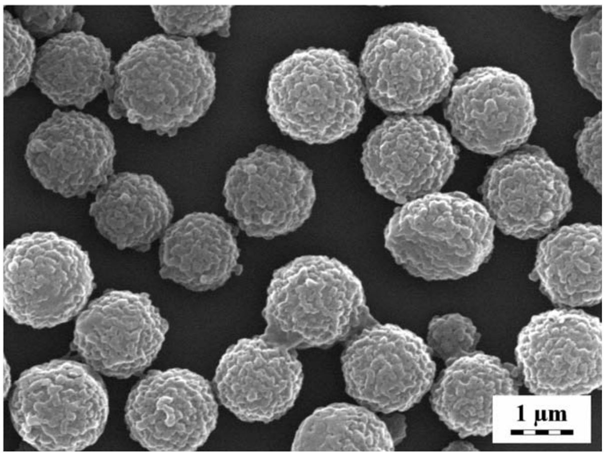

Figure 1. A scanning electron micrograph of magnetic nonporous P(HEMA-co-EDMA) microspheres[1].

Rapid and sophisticated DNAisolation strategies are based on the utilisation of solid phase systems selectively or non-selectively adsorbing genomic DNA. The sensitivity of PCR may be reduced when this method is applied to complex biological samples (including foods) due to the presence of extracellular PCR inhibitors in tested samples. The problem can be solved by means of various isolation and purification methods, such as solid phase reversible imobilization (SPRI) using carriers. In previously published papers[2,3] magnetic non-porous P(HEMA-co-GMA) poly(2-hydroxyethyl methacrylate -co-glycidyl methacrylate) microspheres, P(HEMA-co-EDMA) poly(2-hydroxyethyl methacrylate-co-etylene dimethacrylate) and (PGMA) poly(glycidylmethacrylate) ones were success-fully used in the presence of high concentrations of PEG 6 000 and NaCl for the isolation of DNA from various samples containing polymerase chain reaction (PCR) inhibitors. Prerequsite for application of magnetic particles in DNA analysis is their noninterference in PCR.

The aim of this work was to study the potential inhibition effect of different types of commercial and non-commercial magnetic and non-magnetic carriers on PCR course. Total of 25 types of nano-/micro-particles such as magnetic non-porous P(HEMA-co-GMA), P(HEMA-co-EDMA), PGMA particles covered by carboxyl groups, and thermosensitive non-magnetic poly(N-isopropylacrylamide) microspheres – P(NIPAAm)[4] were tested. Real-time PCR (qPCR) was used for an estimation of the PCR interference by comparing the amplification efficiencies of purified DNA containing solid nanoparticles with the DNA standards free of any nanoparticles. It was shown that most of carriers does not influence the course of PCR. In second part, reversible DNA adsorption on surface of magnetic particles was tested in the presence of an aqueous phase system containing 16% PEG 6000 and 2 M NaCl from complex samples, namely dairy products. The quality and quantity of DNA isolated magnetic nano- and microspheres were verified by PCR and real time PCR using primers specific for domain *Bacteria*[5] and genus *Lactobacillus*[6]. It was found out that the amount of isolated DNA correlated with the content of -COOH groups on the microspheres surface. Higher quantity of DNA was isolated using microspheres with higher content of carboxyl groups. From the results it can be conclu-ded that tested nano- and microspheres are suitable for the PCR and qPCR ready DNA isolations from different types of food samples.

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REFERENCES

1. Rittich B., Španová A., Horák D.: Food Res. Int. *42*, 493 (2009).
2. Rittich B., Španová A., Šálek P., Němcová P., Trachtová Š., Horák D.: J. Magn. Magn. Mater. *321*, 1667 (2009).
3. Kahánková J., Španová A., Pantůček R., Horák D., Doškař J., Rittich B.: J. Chromatogr. B *877*, 599 (2009).
4. Macková H., Králová D., Horák, D.: J. Polymer. Sci. Part A: Polymer. Chem. *45*, 5884 (2007).
5. Haarman, M., Knol J.: Appl. Environ. Microbiol. *72*, 2359 (2006).
6. Dubernet S., Desmasures N., Guéguen M.: FEMS Microbiol. Lett. *214*, 271 (2002).

4-L3

NEW INTEGRATED STRATEGY FOR QUICK ANALYSIS OF PERSISTENT ORGANOHALOGEN CONTAMINANTS AND POLYCYCLIC AROMATIC HYDROCARBONS IN FOOD

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Presented study focuses on emerging ubiquitous environmental contaminants with emphasis on polychlorinated biphenyls (PCBs), brominated flame retardants (BFRs) and polycyclic aromatic hydrocarbons (PAHs) whose occurrence was found out in food and feed and that might be transferred also into human food chain. On this account, the request for the occurrence data has been raised by the European Food Safety Authority (EFSA) with the aim to advice the European Commission on the risks that may pose these chemicals to human health[1-4].

With regards to similarities in physico-chemical properties of these chemicals (good thermal stability, hydrophobicity, etc.) the “classic” analytical methods for determination of PCBs, BFRs (e.g., polybrominated diphenyl ethers - PBDEs), and PAHs in various types of complex food matrices are often based on similar multistep sample preparation procedures which include non-selective isolation of lipids portion from sample followed by various clean-up steps which are not only laborious and time consuming but also require high volumes of organic solvents[5-7].

PCBs, PAHs, and BFRs suggested for this study are semivolatile compounds amenable to gas chromatography (GC). Nowadays, this separation technique hyphenated to mass spectrometry (MS) represents the most preferred option for their identification/quantification[5,6]. With regard to high boi-ling points of target analytes, temperature resistant low bleed high resolution capillary columns coated with non-polar to medium polar stationary phases are employed. In addition to this approach, reversed phase high performance liquid chroma-tography (HPLC) with fluorescence detection (FLD) comm-only employed in case of PAHs, although not all the PAHs are fluorescent (e.g., cyclopenta[cd]pyrene and benzo[j]fluor-anthene)[7].

In this study, a new rapid, flexible, high throughput and robust method for the simultaneous determination of above mentioned compounds including among the others also dioxin-like PCBs, 32 PAHs together with their methylated analogues and emerging non-PBDE BFRs (e.g. decabromodiphenyle ethane, hexabromobenzene, bis(2,4,6–tribromphenoxy)ethane, etc.) in fish by gas chromatography coupled to tandem mass spectrometry with triple quadrupole mass analyzer (GC-MS/MS) was developed and validated. Homogenized sample was after addition of water shaked with ethyl acetate and the transfer of analytes into organic layer was further supported by addition of inorganic salts. Crude extract was in the next step clean-up using silica handmade columns.

Compared to “conventional” approaches, up to 6 samples can be processed in less than one hour compared to 8 hrs needed for Soxhlet extraction and another 1 hr for GPC.

Moreover, the volume of an extraction solvent and consumption of other chemicals could be significantly reduced.

During the optimization of final instrumental analysis different injection techniques (pulsed splitless and programmable temperature vaporization), chromatographic columns, oven temperature programs and other related parameters were tested with the aim to reached the best chromatographic separation of all target analytes and the lowest limits of quantification (LOQ) as possible. Further, the back flush mode which enables to lower the chemical noise raising from the potential „carry over“ effect, decrease the ion source contamination and prolong the column lifetime, was tested. Together with all above mentioned facts triple quadrupole mass analyzer com-pared to routinely used single quadrupole or time-of-flight mass analyzer allows obtaining lower LOQ that are necessary when relevant data concerning food contamination for exposure studies should be collected.

The key performance characteristics of the entire sample preparation procedure together with the instrumental analysis met the criteria applied in the European Union in food contaminants control (SANCO document No. 10684/2009 originally designed for pesticides residues analysis but commonly applied also for other organic food contaminants)[8]. None of recoveries was out of 70–120% range, the repeatability of measurements was less than 20% for all analytes, even at the lowest spiking level (1 μg kg^{-1}).

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REFERENCES

1. Scientific report of EFSA: Results of monitoring of dioxin levels in food and feed, EFSA Journal *8*, 1385 (2010).
2. Opinion of the scientific panel on contaminants in the food chain on request from the commission related to the presence of non dioxin-like polychlorinated biphenyls (PCB) in feed and food (Question N°EFSA-Q-2003-114), EFSA Journal *284*, 1 (2005).
3. Scientific opinion of the panel on contaminants in the food chain: Polycyclic aromatic hydrocarbons in food (Question N°EFSA-Q-2007-136), EFSA Journal *724*, 1 (2008).
4. http://www.efsa.europa.eu/en/data/call/datex091215.htm [cit. 2010-09-08].
5. van Leeuwen S. P. J., de Boer J.: J. Chromatogr. A. *1186*, 161 (2008).
6. Fidalgo-Used N.; Blanco-Gonzáles E., Sanz-Medel A.: Anal. Chim. Acta. *590*, 1 (2007).
7. Plaza-Bolanõs P., Frenich A. G., Vidal J. L. M.: J. Chromatogr. A. *1217*, 6303 (2010).
8. Document No. SANCO/10684/2009.

4-L4

ARSENIC SPECIATION IN RICE: SURVEY OF THE CZECH MARKET

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Humans are exposed to arsenic through drinking water and such food as seafood and rice. The toxicity of arsenic is highly dependent on its chemical form and oxidation state, therefore the speciation analysis of arsenic plays an important role in determination of its actual toxicity to living organisms. Arsenic provides a very good example of the importance of speciation in relation to toxicological aspects, because total arsenic content does not provide complete information for correct risk analysis. Nowadays, only some techniques and their combination are used for arsenic speciation analysis. A fundamental tool for speciation analysis has been combination of chromatographic separation techniques with element specific detector (ICP-MS).

In general, inorganic arsenic compounds are more toxic than the organic arsenic ones and the trivalent species are considered to be more toxic than pentavalent species. As known, inorganic arsenic is a confirmed human carcinogen causing skin and bladder cancer. The major arsenic species in drinking water are arsenate As^V and arsenite As^{III}, both of which have high acute toxicity. On the other hand, most arsenic found in seafood occurs in relatively nontoxic organoarsenic forms such as arsenobetaine or arsenosugars. Inorganic arsenic is metabolized by methylation in the liver and is largely excreted via urine. Biomethylation seems to be a crucial detoxification mechanism because the major methylated arsenic metabolites monomethylarsenic acid (MA) and dimethyl-arsenic acid (DMA) are excreted and are less toxic than inorga-nic arsenic species.

Rice has attracted a lot of attention because it is a staple food in many countries and the major arsenic species in rice are inorganic arsenic (As^V and As^{III}) and organic one dimethyl arsinate (DMA). As known, rice is grown under flooded conditions and it is able to accumulate arsenic from the soil and water.

This study was aimed at determination of arsenic species in rice retailed at Czech food market to find eventual health risk associated with the consumption of this food. Ten rice samples were analyzed for their arsenic content by ICP-MS after microwave-assisted acid digestion. As found, arsenic content varied from 0,04 to 0,22 mg kg^{-1}. Speciation analysis was performed by HPLC-ICP-MS using anion exchange separation conditions. The key step of arsenic speciation analysis is the extraction procedure to reach sufficient extraction yields. In this work, the microwave-assisted extraction using 0,02 mol l^{-1} trifluoroacetic acid (TFA) was used for the extraction of arsenic species from milled rice powder. The sum of the arsenic species extracted and determined by HPLC-ICP-MS ranged from 86 to 100 % of the measured total arsenic content. Inorganic arsenic and DMA were the predominant arsenicals found in rice, inorganic arsenic ranged between 54–78 % of the total arsenic content in rice.

The accuracy of the arsenic speciation procedure was followed by analysis of the certified reference material NIST CRM 1568a Rice Flour (National Institute of Standards and Technology, NIST, USA) and it was found, that measured data were in good agreement with certified values, what enables to use this method for evaluation of arsenic content species in the rice.

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4-L5

ORGANIC AND CONVENTIONAL WINES CHARACTERISATION BY MODERN SPECTROSCOPIC TECHNIQUES

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In the last decade organic viticulture in Europe has noticed great progress. The main reason is the increased consumers demand for organic wines, usually connected with arguments of some experts that organic wines contain much more health beneficial substances and should be therefore healthier than the conventional ones. There are only few previous studies supporting these claims. However, there exist some comparative studies of organic and conventional wines, indicating that main differences between the organic and conventional grapes and wines are to be found especially in the content of biologically active compounds, such as polyphenols (i.e. trans-resveratrol, total phenolic compounds), organic acids, biogenic amines, minerals and essential elements (especially Ca, Fe, Mg and Cu)[1-4]. Therefore, the differences in antioxidant activities of these wines could be expected.

In addition, with respect to organic viticulture sector development, the legitimate demands on quality control and authentication of organic grapes and wines have arisen. Up to the present, there are still no validated methods applicable for the authenticity characterisation of organic wines and their unambiguous differentiation from wines produced by conventional methods. Thus, there is an urgent need to find objective markers allowing clear distinction both types of wines.

The objective of the present study was to evaluate antioxidant/radical-scavenging activities and some other relevant characteristics of different Slovak organic and conventional wines originating from the same wine-making region. To achieve these goals, methods of UV-VIS and EPR spectroscopy were applied. Antioxidant activity of wine samples was tested by EPR spectroscopy using 2,2-diphenyl-1-pickrylhydrazyl ($^{\bullet}$DPPH) free radical, 2,2′-azino-bis-(3-ethylbenzthiazoline-6-sulfonic acid) cation radical (ABTS$^{\bullet+}$) and 4-hydroxy-2,2,6,6-tetramethylpiperidin-N-oxyl free radical. The ability of samples to terminate $^{\bullet}$OH radicals was tested directly in the experimental system, radicals were generated via thermal decomposition of radical initiator ($K_2S_2O_8$). The UV-VIS was involved to monitor the content of polyphenols (TPC) in the samples, formation of oxidation products in the presence of thiobarbituric acid (TBARS value), together with their $^{\bullet}$DPPH and ABTS$^{\bullet+}$ quenching and ferric ions reducing abilities. Besides them, CIE Lab colour coordi-nates of all samples were assessed under standardized condi-tions.

All samples showed significant antioxidant and radical-scavenging properties, generally, the higher for red wines, without respect on the way of production. As expected, the concentration of polyphenols in red wines was several times higher than in white ones, being thus one of key factors responsible for the observed differences between the red and white wines[5,6]. As regards the capability of conventional wines to reduce ferric ions, it was just slightly higher compared to organic wines. EPR experiments proved that the ability of conventional wines to scavenge TEMPOL free radicals was significantly higher compared to organic wines. On the contrary, the higher capability to terminate $^{\bullet}$OH radicals was observed for organic wines compared to conventionally produced wines.

First results indicate that the unequivocal differentiation of organic wines from conventional wines represents still a complex problem. All the experimental data will be after the finishing of experiments that are still in progress processed by multivariate statistics enabling comparison and correlation of objective analytical data obtained by various methods. Moreover, it could enable also the characterisation and differentiation of wine products according to their origin and way of production.

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REFERENCES

1. Vrček I., Bojić M., Žuntar I., Mendaš G., Medić-Šarić M.: Food Chem. *124*, 1 (2011).
2. Mulero J., Pardo F., Zafrilla P.: J. Food Compos. Anal. *23*, 6 (2010).
3. Provenzano M. R.., El Bilali H., Simeone V., Baser N., Mondelli D., Cesari G.: Food Chem. *122*, 4 (2010).
4. Yildrim H. K., Üren A., Yücel U.: Food Tech. Biotechnol. *45*, 1 (2007).
5 Polovka M., Šťavíková L., Hohnová B., Karásek P., Roth M.: J. Chromatogr. A, *1217*, 51 (2010).
6 Katalinić V., Možina S. S., Skroza D., Generalić I., Abramović H., Miloš M., Ljubenkov I., Piskernik S., Pezo I., Terpinc P., Bodan M.: Food Chem. *119*, 2 (2010).

4-L6

OPTIMISATION OF GC/MS MULTIRESIDUE METHOD FOR DETERMINATION OF PESTICIDES IN FRUIT AND VEGETABLE

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Pesticides are chemical substances used to protect food crops against various pests. Under certain conditions their residues may enter food chain and pose a health risk for consumers. To avoid or minimize such negative effects the MRLs (maximum residue limits) for various commodities are set for a wide range of active ingredients of pesticide preparations by Regulation (EC) No 396/2005[1].

Multidetection methods capable to determine a large number of pesticides simultaneously – in a single run – are used for pesticide residues monitoring and/or MRL control. The determination of GC-amenable pesticides in food samples is traditionally performed by gas chromatography (GC) coupled to mass spectrometry (MS).

The aim of this study was to optimise and validate the multiresidue method based on "QuEChERS" (Quick, Easy, Cheap, Effective, Rugged, and Safe) approach based on (i) acetonitrile – water partition (transfer of analytes into organic phase is supported by inorganic salts) and (ii) dispersive SPE clean-up of crude extracts. For identification and quantitation of target pesticides, GC-HRTOF-MS system was used. Within our experiments, the amount and the type of sorbents in dispersive SPE were optimised for two common types of fruit and vegetable matrices such as apples and lettuce.

For the purpose of validation, homogenized samples were spiked at two concentration levels – 0,1 and 0,01 mg kg^{-1}. The obtained recoveries were, in most cases, in the range 70 % - 120 % and RSDs were below 20 %. The limits of quantitation (LOQ) for most pesticides were below 0,01 mg kg^{-1}. Matrix effects were eliminated by the use of matrix-matched standards for each matrix. The obtained performance characteristics will be compared with those of obtained by "classic" method employing ethylacetate for residues extraction and gel permeation chromatography for extract purification.

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REFERENCES

1. http://ec.europa.eu/sanco_pesticides/public/index.cfm [cit. 2011-20-4].
2. Method validation and quality control procedures for pesticide residues analysis in food and feed. Document SANCO/10684/2009.
3. Lehotay S. J., Maštovská K., Lightfield A. R: J. AOAC Int. *88*, 615 (2005).

4-L7

QUANTIFICATION OF FATTY ACIDS IN BUFFALO MILK AND CHEESE USING GC-FID

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The romanian buffalo is exploited mainly for its milk production and less for its meat production capability. Due to its unique physical and chemical characteristics, buffalo milk, is considered to have some advanteges to cow milk in the manufacturing process of dairy products. Lipids are a major component of buffalo milk with great implications in nutrition and economical aspects. The nutritional value of buffalo milk is derived form its complex chemical composition and its high rate of asimilitation. The objective of this study is to identify and quantify the fatty acids present in the buffalo milk of the romanian buffalo breed and also for the cheese obtained by employing a traditional manufacturing process. For the identification and quantification of fatty acids a Gas Chromatograph coupled with a Flame Ionization Detector was used. Major fatty acids found in both milk and cheese were: palmitic acid (16:0), oleic (18:1) and stearic (18:0). The ratio between UFA/SFA was 0,3; lower than the threshold of 1,5 which is considered not to raise negative health effects.

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REFERENCES

1. Varricchio M. L., Francia A. Di, Masucci F., Romano R., Proto V.: Ital. J. Anim. Sci. *6* (Suppl. 1), 509 (2007).
2. Kuldip C. S., Veer K. S. and Sudarshan S.: Dummerstorf *43*, 123 (2000).
3. Bergamo P., Fedele E., Iannibelli L., Marzillo G.: Food Chem. *82*, 625 (2003).
4. Jensen R. G., in the book: *Fatty Acids in Food and their Health Implication*, Ed. Ching Kuang Chow, Marcel Dekker Inc, New York (USA), p109-123, (2000).
5. Jensen, R. G.: J. Dairy Sci. *85*, 295 (2002).

4-L8

MULTIRESIDUE ANALYSIS OF MORE THAN 300 PESTICIDES IN FOOD OF PLANT ORIGIN USING LC-MS/MS: EVALUATION OF QUECHERS PROCEDURES

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Analysis of pesticides poses complicated tasks since more than 800 pesticides representing various chemical groups exists worldwide. Therefore, multiresidue methods have became an effective tool for control of legislation limits nevertheless none of them is appropriate for all existing or registered analytes. However QuEChERS[1] has been showed as a great extraction procedure appropriate for LC and GC amenable pesticides. Nowadays a lot of modifications of the original version exist, and QuEChERS has become an official method: citrate-buffering version was set by the European Committee for Stan-dardization (CEN) as a Standard Method EN 15662[2] and the acetate-buffering version became AOAC Official Method 2007.01[3].

In this study, three versions of QuEChERS sample prepa-ration method have been compared: original, citrate-buffering and acetate-buffering, for 325 LC-amenable analytes (pesti-cides and their metabolites involved in their residue defini-tions).

Samples were processed according to procedures described in the literature[1-3] and different commodity groups were chosen according to the Document No. SANCO/10684/2009: (i) High water content: apples and lettuce, (ii) High starch and/or protein content and low water and fat content: wheat, (iii) High acid content and high water content: oranges. Each matrice was spiked at 0,1 mg kg^{-1} (0,3 mg kg^{-1} in wheat) in six replicates, relevant matrix-matched standards were prepared from blank matrices and analysed by ultraperformance liquid chromatography coupled to triple-quadrupole tandem mass spectrometry.

Results of our validation study for each version were excellent; nevertheless few differences among the recoveries in each version appeared. Figure 1 demonstrates that all versions are appropriate for almost all studied pesticides and their metabolites. Differences were evident especially for acidic and basic analytes and also for pesticides sensitive to pH value.

The acetate-buffered version achieved higher recoveries for basic pesticides, presented in our experiments only by pymetrozine. However recoveries of acidic pesticides that are more often applied to protect crops (for example 4-CPA, quinmerac, imazapyr) were lower in most commodities using acetate-buffered version (see Figure 2).

Recoveries of pesticides known as base sensitive (tolylfluanid, dichlofluanid, thiodicarb) were lower than 20% using both buffered versions. The reason is addition of water to dry samples that is recommended by Document No. SANCO/10684/2009. Nevertheless some base sensitive pesticides degraded during swelling of wheat with non-modified water foregoing buffered versions. In case of original version we used acidified water that we know from previous experiment is necessary for good recovery for acidic pesticides and finally also for prevention of degradation of other base-sensitive pesticides. Thus we demonstrated that pH adjustment is sometimes appropriate also using buffered versions.

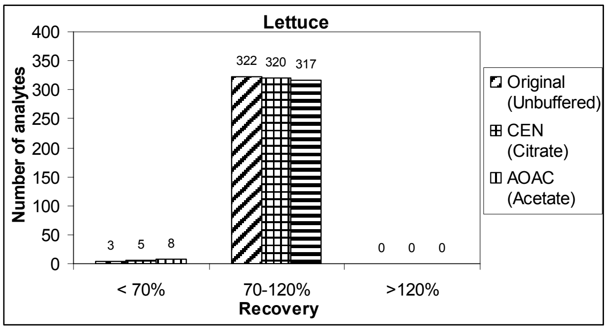

Figure 1. Distribution of recoveries obtained during the method validation for 325 tested pesticides in lettuce (six replicates spiked at 0,1 mg kg^{-1}).

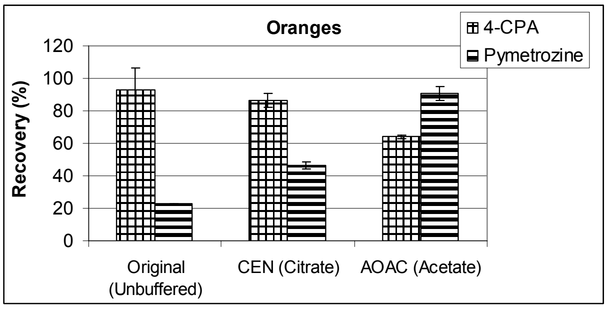

Figure 2. Distribution of recoveries of acidic and basic pesticide in oranges (six replicates spiked at 0,1 mg kg^{-1}).

The QuEChERS sample preparation method was demonstrated as a flexible and rugged approach that gave for most pesticides excellent results using its different modifications. To conlude, the acetate-buffered version of QuEChERS is not very appropriate for the analysis of acidic pesticides; on the other hand it is the best choice for the analysis of basic pesticides – in our study represented only by pymetrozine. However, acidic pesticides are more commonly applied for the crop protection and therefore an original or citrate-buffered version with pH adjustment that is in some cases necessary (e.g. for the extra-ction of cereals, in this study represented by wheat) is pre-ffered.

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REFERENCES

1. Anastassiades M., Lehotay S., Stajnbaher D., Schenck F.: J. AOAC *2*, 86 (2003).
2. EN 15662:2008: *Foods of plant origin. Determination of pesticide residues using GC-MS and/or LC-MS/MS following acetonitrile extraction/partitioning and clean-up by dispersive SPE. QuEChERS-method* (2008).
3. Lehotay S. J.: J. AOAC Int. *90*, 485 (2007).

4-L9

ANALYSIS OF FLAVONOID GLYCOSIDES AFTER HYDROLYSIS WITH HPLC-DAD-ELSD METHOD FOCUSED ON IDENTIFICACTION OF SACCHARIDES

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The glycoside analysis presents an indivisible part of a nature compounds analysis. The identification of both basic parts (aglycone and carbohydrate), type of their chemical bond and the position of glycosidic bond in polyhydrohylic aglycone are essential for the determination of the structure. HPLC-MS or GC-MS are the typical means of glycoside identification. But the complete identification of a glycoside is often impossible though usage of mass spectrometry (MS) because of carbohydrate isomers. Than it can be used instrumentally difficult connection HPLC-NMR-MS, 2D NMR or HPLC-PAD techniques. UV-VIS detector cannot be used for the detection of carbohydrates because of absence of chromophore. Alternative RI detector can be used only in isocratic conditions and it is low sensitive. Evaporative Light Scattering HPLC Detector (ELSD) is detector suitable for carbohydrate analysis with many advantages (high sensitivity, stable baseline, possibility of gradient elution)[1-4].

This work is engaged in spreading possibilities of nature compounds identification especially glycosides. The method of separation and identification carbohydrates with the HPLC-ELSD enable differ saccharide isomers that is not possible with one of the most used methods HPLC-MS. Acid and enzymatic hydrolyses were studied in this work. The method of detection saccharide units after glycoside hydrolysis was developed on a model system of flavonoid glycosides formed from two different aglycones with different bonded glycones (Tab.I).

All the model compounds were hydrolysed to basic parts which were detected. The enzymatic hydrolysis is very specific method which depends not only on the type of saccharide but also on its position. Full enzymatic hydrolysis was obtained in hesperidin and neohesperidin, but not in 3-O-glycosides. The connection HPLC-DAD-ELSD represents system able to iden-tify original glycoside and also aglycone and sugar parts after hydrolysis. The column LiChrospher 100 DIOL was used for separation of compounds. The new created experimental conditions enable to hydrolyse, separate and identificate all the glycoside and its parts. The acid hydrolysis is with respect to financial and time requirements cheaper and faster method but enzymatic hydrolysis gives also information about sequence of bounded sacharides and partially about position of glycosidic bound. On the other hand specifity of enzyme is limiting in hydrolysis of unknown glycoside.

The method was used for identification of unknown compound isolated from *Polygonum lapathifolium* (Polygonaceae). The acid hydrolysis demonstrates presence of galactose bounded to kaempferol. The glycoside was not enzymatically hydrolysed so we can suspect that galactose is bounded in position 3 of the flavonoid. These results were also confirmed by GC-MS analysis.

Table I, Standards of glycosides

Quercetin	R = H
	R = glc (Quercetin 3-β-D-glucoside)
	R = gal (Quercetin 3-β-D-galactoside)
	R = rha (Quercitrin)
	R = glc-rha (rutinose) (Rutin)
Hesperetin	R = H
	R = glc-rha (rutinose) (Hesperidin)
	R = glc-rha (v pol.2) (Neohesperidin)

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REFERENCES

1. Brito-Arias M., in the book: *Synthesis and Characterization of Glycosides*, chapt. 7, p. 304. Springer, New York 2007.
2. Vystrčil A.: *Rostlinné glykosidy*, ČSAV, Praha 1955.
3. Rotrekl V.: Chem. Listy *92*, 883 (1998).
4. http://www.hplc.cz/Teorie/ELSD.htm [cit. 2010-24-11].
5. Day A. J., DuPont M. S., Ridley S., Rhodes M., Morgan M. R. A., Williwamson G.: FEBS Lett. *436*, 71 (1998).

4-L10

NON-ISOTHERMAL KINETICS OF BENZO[A]PYRENE PHOTOOXIDATION IN NON-POLAR FOOD MATRIX.

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Polycyclic aromatic hydrocarbons are permanent contaminants found in various foods as a result of technological procedures carried out at elevated temperatures such as grilling, drying, frying and mainly smoking[1,2]. The toxicity of these compounds can be increased by solar radiation which induces formation of reactive oxidised products[3,4]. As known, PAH contain two or more conjugated benzene rings in molecules that facilitate the absorption of ultraviolet A radiation (320–400 nm), ultraviolet B radiation (290–320 nm), and in some instances, visible light (400–700 nm). This leads to photoactivation of PAH and increase their toxicity via the photosensitized production of singlet oxygen, and photomodification of original molecules, that results in formation of oxy-PAH compounds[5,6]. Many of products generated through the photooxidation exhibit greater toxicity than the parent PAH and have the potential to generate toxic compounds that could negatively impact living systems and human health. The most representative compound of PAH group is benzo[a]-pyrene (BaP) due to its carcinogenic effects to living organisms[7].

BaP was dissolved in glyceryl trioctanoate, heated in a glass reaction vessel within temperature range 297,95 – 361,85 K with heating rate of 1 K/min and simultaneously exposed to UV light at wavelength λ = 365 nm at radiation power 20 mW cm^{-2} in order to study the decrease of BaP concentration due to its photooxidation. The concentration of BaP was determined by the HPLC-FD periodically in 5 min intervals.

As observed, the BaP concentration decreased from 62,2 μg l^{-1} to 19,8 μg l^{-1}. From the experimental results, the parameters characterizing the non-isothermal kinetics of BaP photooxidation have been obtained. The parameters were tested at two various isothermal conditions (290,16 and 323,26 K) to verify a suitability of derived parameters. Comparing calcu-lated and measured data of half-lives of BaP decrease at mentioned temperatures it was found that calculated half-lives are in a good accordance with those experimentally obtained values, when relative standard deviations at 290,16 K was 17,0% and 5,4 % at 323,26 K, respectively. The kinetic parameters enable the calculation of the rate constant for any temperature in isothermal regime and make possible to model, in general, the kinetics of such processes without a deeper insight into their mechanisms.

This contribution is the result of the project implementation "Establishment of a „HiTech“ centre for research on formation, elimination and risk analysis of contaminants in food" and "Centre of Excellence for Contaminants and Microorganisms in Food", supported by the Research & Development Operational Programme funded by the ERDF and supported by Science and Technology Assistance Agency of Slovak Republic under the contract No. APVV-0602-07.

REFERENCES

1. Tamakawa K., in the book *Handbook of Food Analysis,* chapt. 43, p.1449. New York, Marcel Dekker, 2004.
2. Šimko P.: J. Chrom. B. *770,* 3 (2002).
3. McConkey B. J., Duxbury C. L., Dixon D. G. Greenberg B. M.: Environ. Toxicol. Chem. *16*, 892 (1997).
4. Newsted J. L., Giesy J. P.: Environ. Toxicol. Chem. *6*, 445 (1987).
5. Arfsten D. P., Schaeffer D. J. Mulveny D. C.: Ecotoxicol. Environ. Safe. *33*, 1 (1996).
6. Brack W. R. Altenburger E., Kuster B., Meissner K., Wenzel D., Schürmann G.: Environ. Toxicol. Chem. *22*, 2228 (2003).
7. Šimko P.: Mol. Nutr. Food Res. *43,* 637 (2005).

4-L11

DETERMINATION OF BIOLOGICALLY ACTIVE COMPOUNDS IN FLAXSEED (LINUM USITATISSMUM L.)

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Flaxseed has been widely used in human nutrition as a functional food for thousands of years, while flax oil (more commonly known as linseed oil), because of its strong flavor and limited stability, has a predominant usage in industrial applica-tions[1,2]. The presented study has been conducted within the Project "Flaxseed for human health" which aims to breed a flaxseed with higher levels of the oleic acid at the expense of the unstable α-linolenic acid and, thereby, make conditions for a broad use of flaxseed oil in food and medicine. In order to assess health benefits / risks associated with flaxseed consumption, also biologically active compounds present in this crop, namely phytoestrogen secoisolariciresinol (SECO), carotenoid lutein and cyanogenic glycosides were determined in several cultivars grown under various conditions. For this purpose, high performance liquid chromatography coupled with diode array detector (for determination of SECO and lutein) or with tandem mass spectrometry detector (for determination of cyanogenic glycosides) was evaluated and validated. Total levels of SECO ranged between 1,4 – 8,7 g kg^{-1}, of lutein 0,5 – 8,2 mg kg^{-1} and of cyanogenic glycosides 1,0 – 7,5 g kg^{-1}. Results were used to assess the impact of the variety, cultivation, and growing conditions on the levels of these compounds.

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REFERENCES:

1. Thompson, L. U., Cunnane, S. C., Eds. *Flaxseed in human nutrition*, 2nd ed.; AOCS Publishing, 2003.
2. Krajčová A., Schulzová V., Hajšlová J., Bjelková M.: Czech J. Food Sci. *27*, S252 (2009).
3. Krech M. J., Fieldes M. A.: Can. J. Bot. *81*, 1029 (2003).

4-L12
AMINO ACIDS DETERMINATION IN DRY FERMENTED SALAMI

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Curing is a conservation technique widely used since antiquity to prolong the life of food products. Rapid curing processes used in the manufacture of dry cured sausages, allowing the meat industry to reduce the costs associated with a long curing process, results in the loss of the typical organoleptic characteristics in the final product. Meat fermentation is a low energy, biological acidulation, preservation method which results in unique and distinctive meat properties such as flavor and palatability, color, microbiological safety, tenderness. The study of starter cultures and the influence of salt concentration, ripening time and the different curing ingredients are important in the quality of fermented sausages[1]. Free amino acids content is directly related to the meat flavor and taste[2]. Meat consumers and processors are interested in the development of efficient and economical technology for proce-ssing meat into a product that is palatable and reasonable in cost[3]. A gas chromatography mass spectrometric technique was used for the quantitative characterization of amino acids in different steps of salami processing. Several batches containing 0,3% GDL (glucono delta-lactone) and 0,05% sodium ascor-bate, and several batches to which was added only 0,1 % sodium ascorbate were manufactured.

The aim of the work was to determine the content of free amino acids at different time intervals over 45 days of storage. The extraction procedure was followed by a suitable derivatization method, to separate and to identify the amino acids extracted from salami samples. Quantitation was obtained by using ^{15}N-methionine (^{15}N-Met) as internal standard by selecting the specific ions m/z 171 and 172 from the mass spectrum of Met and respectively of labeled methionine.

Amino acids were purified on Dowex 50W-W8 exchange resin, eluted with NH_4OH 4M and derivatized. The N-trifluoro-acetyl n-butyl ester derivatives were obtained by following a two step derivatization method, esterification at the C terminal edge and trifluoroacetylation at the N terminal one[4]. The measurements were performed by gas chromatography mass spectrometry (GC-MS) using a Trace GC equipped with a Rtx-5MS capillary column (30 m x 0,25 mm, 0,25 µm film thickness) and coupled to a Trace DSQ Thermo-Finnigan quadrupole mass spectrometer. The oven temperature program for separation of amino acids trifluoroacetyl butyl esters was: 50°C for 1 min, 6°C/min to 100°C, 4°C/min to 200°C, 20°C/min to 300°C, for 3 min. Helium 5.5 was used as carrier gas, with a flow rate of 1 ml.min^{-1}.

Differences in amino acid levels at different days of storage were determined in the dry fermented salami. The amino acids increased significantly with the storage time, especially in GDL variety.

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REFERENCES

1. Jin S. K., Kim I. S., Kim D. H., Choi Y. J., Hur S. J.: Poultry Sci. *86*, 2676 (2007).
2. Koga K., Fukunaga T., Kawagoe, S.: Mem. Fac. Agr. Kagoshima Univ. *23*, 121 (1987).
3. Hermanussen M., Gonder U., Jakobs C., Stegemann D., Hoffmann G.: Eur. J. Clin. Nutr. *64,* 88 (2010).
4. Culea M., Hachey D.: Rapid Commun. Mass Spectrom. *9*, 655 (1995).

4-L13
PRODUCTION, ISOLATION AND APPLICATION OF POLYHYDROXYALKANOATES – BIODERADABLE ALTERNATIVE TO PETROCHEMICAL PLASTICS

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Polyhydroxyalkanoates (PHA) are a group of polyester materials which are accumulated in the form of intracellular granules by a wide variety of bacterial strains. Due to their mechanical properties similar to those of plastics of petrochemical origin and their biodegradability, PHA are considered being an environmentally-friendly alternative to common plastics such as polyethylene or polypropylene. Moreover, thanks to their biocompatibility, PHA find many potential applications in medicine[1].

In terms of chemical structure, PHA are polyesters of hydroxyalkanoic acids. Up to date, more than 100 monomers have been found in the naturally synthesized polyester polymers[2]. Mechanical properties of PHA strongly depend on monomer unit composition. Homopolymer of 3-hydroxybutyrate, poly(3-hydroxybutyrate) (PHB), which is the most ubiquitous PHA in nature, possesses highly crystalline structure that makes it relatively stiff and brittle. Also the fact that the melting point of PHB (173-180°C) is close to the temperature of decomposition (approx. 200°C) has impractical consequences. Nevertheless, mechanical properties of PHA can be signi-ficantly improved by incorporation of other monomer units into PHA structure[3].

This work is focused on production, isolation as well as on application of polyhydroxyalkanoates as delivery systems for selected bioactive substances.

In our work we studied biotechnological production of PHA from various waste substrates such as cheese whey or waste frying oils. This strategy should result in reduction of production cost of PHA. Further, we tested involvement of PHA into stress response of bacteria and we observed that introduction of stress condition caused by ethanol or hydrogen peroxide significantly enhanced PHA yields. Thus, simple application of cheap substance significamtly improved PHA production process. We decided to look into metabolic metabolic details and we observed that stress response of bacterial strain to either ethanol or hydrogen peroxide involves activation of PHA biosynthetic pathway. Moreover, molecular weights of PHA accumulated under stress conditions were significantly higher. Hence, application of cheap substance facilitated significant improvement of both PHA yields and mechanical properties. Furthermore, introduction of propanol enhanced both biomass and PHA yields when produced on waste oils and also resulted in incorporation of 3-hydroxyvalerate as a monomer into polymer structure.

Besides cost of carbon source, one of the most important factors contributing to production price of PHA is the cost of down-stream process[4]. Therefore, a lot of afford have been made to improve such a process. We have developed environmentally-friendly isolation protocol based on controlled application of enzymes and surfactants into the cultivation media after the end of cultivation. During the isolation process, non-PHA biomass is digested and solubilized while PHA stays untouched. The purity of PHA materials isolated in this way reaches up to 95%.

Microparticles made from polymers are considered being very promising delivery systems for various pharmaceuticals. They can be injected into blood stream, administrated subcutaneously or intramuscularly and adapted for oral administration or inhalation. As compared to chemically produced polymers such as polyglyconate, polylactate etc., which are mostly well known as biologically degradable drug carriers with good retarding characteristics, PHA have advantages that they are easily processed, fully biologically degradable and compatible with animal and human tissues[5]. We have been working on development of PHA based delivery systems for native proteins. Various phase ratios, PHA and enzyme concentrations as well as type of emulsifier were tested in order to optimize process of PHA microspheres production. We have reached relatively high efficiency of encapsulation process (about 70 %). Microparticles were able to release native protein when incubated in physiological buffer and the release followed Higuchi equation for diffusion of drugs from spherical matrices.

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REFERENCES

1. Kessler B., Wilholt B.: in the book: *Encyclopedia of bioprocess technology - fermentation, biocatalyst and bioseparation*. John Willey, New York 1999.
2. Steinbuchel A, Valentin H. E.: FEMS Microbiol. Lett. *125*, 219 (1995).
3. Sudech K., Abe H., Doi Y.: Prog. Polym. Sci. *25*, 1503 (2000).
4. Jacquel N., Lo C. W., Wei Y. H., Wu H. S., Wang S. S.: Biochem. Eng. J. *39*, 15 (2008).
5. Ueda H., Tabata Y.: Adv. Drug Deliver. Rev. *55*, 501 (2003).

4-L14

ANALYTICAL APPROACHES TO ANALYSIS OF MYCOTOXINS IN FEEDSTUFFS; OCCURRENCE IN REAL LIFE SAMPLES

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Occurrence of mycotoxins in silages, forages, and other complex feeding mixtures has an adverse effect on animal health. Supposing the transfer of parent toxins and/or their metabolites into edible tissues, eggs or milk, also the consumers' health might be indirectly influenced[1,2].

In the case of silages and forages, their mycotoxicological quality can be significantly worsened under the incorrect agricultural practice conditions; poor anaerobic conditions during silage making can lead to instability of low pH and follow-up growth of fungi, including the toxinogenic species. Although several studies concerned with mycotoxins in feed have been already published[3-5], the overall knowledge is fairly less extensive in comparison with cereal grains (wheat, barley, maize), whose analysis is not as complicated as in the case of fermentated feed and complex feeding mixtures.

The aim of presented study was to develop and validate a simple and sensitive analytical method for determination of a wide range of mycotoxins such as trichothecens, zearalenones, aflatoxins, alternaria toxins, ergot alkaloids, enniatins, beauvericin, fumonisins, citrinin, ochratoxins, sterigmato-cystin, verrucarol, gliotoxin, roquefortin C, penitrem A, penicilic acid, and mycophenolic acid in forages and silages, and employ this method for identification and quantificaton of these mycotoxins in real life feed samples.

At the beginning of method development, four most common extraction procedures were tested: (*i*) acetonitrile:water (50:50, v/v) extraction, (*ii*) QuEChERS based extraction (formerly widely known from pesticides residues analysis area, but nowadays, commonly applied for analysis of mycotoxins[6]), (*iii*) extraction with pure acetonitrile, (*iv*) extraction with pure acetonitrile followed by water dilution (in this particular case, water addition causes precipitation of polar matrix co-extracts which improves purification of sample extract).

As seen in Figure 1, the lowest amount of matrix co-extracts demonstrated as total ion current (TIC) response was obtained for acetonitrile extraction. In this particular case, the matrix effects for particular analytes did not exceed ±18%, and also recoveries gained from analysis of spikes ranged from 82 to 116%. However, as we uncovered later, when analysing real silage samples, pure acetonitrile was not able to properly extract naturally occurring mycotoxins. Based on this fact, the QuEChERS based method was chosen as the best alternative.

To ensure the acidic pH of extract necessary to proper extraction of acidic analytes typically occurring in silages (e.g.

fumonisins, mycophenolic acid, penicillic acid) even in case of highly fungi contaminated sample (pH of „hot spots“ is often >7), addition of formic acid into the extraction water was optimized as 1%. Matrix effect ranged between -56 and 37%, recoveries of most of analytes lied between 76 and 122%.

For the purpose of analytes separation and detection, ultra high performance liquid chromatography coupled with ultra high resolution Orbitrap mass spectrometry (UHPLC–orbitrapMS), and ultra high performance liquid chromatography coupled with tandem Qtrap mass spectrometry (UPLC–QtrapMS/MS) were used. Detection potential of both of the methods will be discussed.

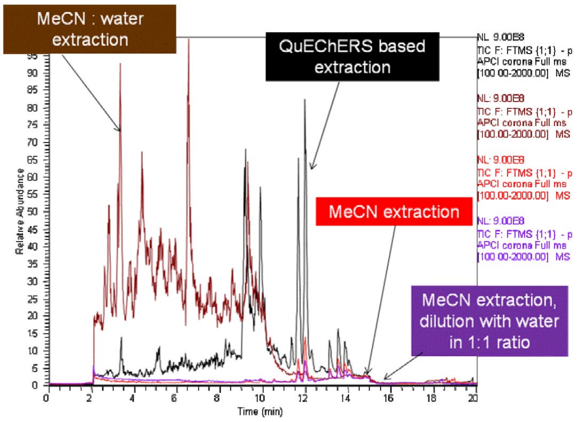

Figure 1. TIC of four proposed methods by UHPLC–orbitrapMS.

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REFERENCES

1. Duca R. C., Bravin F., Delaforge M., Vladescu L., Badea I. A., Criste R. D.: J. Agric. Food Chem. *57*, 10497 (2009).
2. Beltran EIbanez M., Sancho J. V., Cortes M. A., Yusa V., Hernandez F.: Food Chem. *126*, 737 (2011).
3. Schollenberger M., Müller H. M., Rüfle M., Suchy S., Plank S., Drochner W.: Mycopathologia *161*, 43 (2006).
4. Rasmussen R. R., Storm I. M. L. D., Rasmusen P. H., Smedsgaard J., Nielsen K. F.: Anal. Bioanal. Chem. *397*, 765 (2010).
5. Mol H. G. J., Plaza-Bolanos P., Zomer P., de Rijk T. C., Stolker A. A. M., Mulder P. P. J.: Anal. Chem. *80*, 9450 (2008).
6. Zachariasova M., Lacina O., Malachova A., Kostelanska M., Poustka J., Godula M., Hajslova J.: Anal. Chim. Acta *662*, 51 (2010).

4-L15

DEVELOPMENT AND VALIDATION OF A RAPID METHOD FOR THE ANALYSIS OF PESTICIDE RESIDUES IN TEA USING GC–MS

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Tea (*Camellia sinensis*) represents a popular beverage worldwide. However, it might be also considered as a potential source of human exposure to pesticides. During recent years, contamination of tea by pesticide residues has been reported by Rapid Alert System for Food and Feed. The insecticides such as fenvalerate, cypermethrin, fenpropathrin, buprofezin and triazophos were found most frequently in this commodity[1].

The current approaches for the analysis of pesticide residues in foods are commonly based on gas and/or liquid chromatography coupled with mass spectrometry (GC–MS, LC–MS, respectivelly) procedures enabling selective, sensitive and robust determination of a broad range of target compounds. Although LC–MS methods combined with QuEChERS sample preparation strategy have been developed for tea[2], GC–MS analysis has always been complicated due to the high complexity of tea matrix. In the initial part of our study, we observed presence of a high amount of coextracts (*e.g.* caffeine) when preparing the sample extracts using QuEChERS method for subsequent GC–MS analysis. On this account, we focused on streamlining the sample preparation in order to eliminate the most of the matrix interferences and improve the performance characteristics of this method.

The implemented method involves QuEChERS-like sample preparation procedure using (i) acetonitrile as an extraction solvent, (ii) dispersive solid-phase extraction (dSPE) clean-up, and (iii) liquid-liquid extraction (LLE) of target analytes into hexane. GC–MS was then used for identification / quantification purpose. Within the method development, the type and amount of sorbent for dSPE as well as LLE parameters were optimized, taking into account the need of matrix co-extracts reduction. Two performance characteristic achievable by two alternative mass analyzers – high resolution time of flight and tandem ´triple qadrupol´ were critically assessed.

For approx. 100 pesticides tested at a spiking level 0,1 mg kg^{-1} the recoveries were in the range 70–120%, and repeatabilities (expressed as RSD) were below 20%, what means criteria of SANCO/10684/2009 document were met. The applicability of the new method was demonstrated by analysis of 45 commercial tea samples.

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REFERENCES

1. Huang Z., Li Y., Chen B., Yao S.: J. Chromatogr. B *853*, 154 (2007).
2. Chen G., Cao P., Liu R.: Food Chem. *125*, 1406 (2011).

4-L16

PHYTOSTEROL CONTENT OF SOME ROMANIAN WILD AND CULTIVATED BERRIES POMACE

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Large quantities of both liquid and solid wastes are produced annually by the food processing industry and their disposal into soil or landfills poses significant environmental and ecological problems because of its relatively low pH.

Wild and cultivated berries are widely used as raw materials for juice maker industry in Europe. A major coproduct during juice production is the pressing residue called pomace, which consist of the pulp/peel fraction and the seeds of the berries. The pomace still contains a significant amount of the fruit functional components, such as polyphenols, proteins, polysaccharides, fatty acids and phytosterols. Available studies of berry pomace concern the properties of phenolic compounds, antioxidant activities and fibre, but few reports relate to lipid compositions.

The objective of this study was to evaluate the phytosterol composition, using gas chromatography (GC) with flame ionization detection (FID), of five different Transylvanian (Romania) berries pomace: wild and cultivated blueberries *(Vaccinium myrtillus)*, wild lingonberries (*Vaccinium vitis-idaea*) and raspberry (*Rubus idaeus*) and cultivated chokeberry (*Aronia melanocarpa*). Samples were collected in our labora-tory after the fruits had been pressed.

The fat content was 5% in the blueberries and lingonberry pomaces (fresh weight, f.w.), 6% in chokeberry and 10 % in raspberry pomaces. The highest amount of sterol (168,21 mg/100g fat) was determined in lingonberry pomaces and the lowest concentration (101,68 mg/100g fat) was detected in cultivated blueberry pomaces. The major sterol in the studied pomaces was β-sitosterol, which comprised ca. 55 % (lingon-berry) –74% (chokeberry) of the total phytosterol content. The next major components were stigmastanol with isofucosterol. Cholesterol was detected in relative high concentration only in cultivated blueberries pomaces (5,2 mg/100g fat).

The total sterol concentrations in studied berry pomaces were comparable with the total sterol amount from the olive oil (150-160 mg/100g oil).

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REFERENCES

1. Yang B., Ahotupa M., Määttä P., Kallio H.: Food Res. Int., doi:10.1016/j.foodres.2011.02.025 (2011).
2. Zlatanov M. D.: J. Sci. Food Agric. *79*, 1620 (1999).
3. Helbig D., Böhm V., Wagner A., Schubert R., Jahreis G.: Food Chem. *111*, 1043 (2008).
4. Phillips K. M., Ruggio D. M., Toivo J. I., Swank M. A., Simpkins A. H.: J. Food Compos. Anal. *15*, 123 (2002).

4-L17

DETERMINATION OF POLYCYCLIC AROMATIC HYDROCARBONS (PAHS) IN SEAFOOD USING GC-MS: A COLLABORATIVE STUDY

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Following the Mexico Gulf oil spill (April 2010), AOAC INTERNATIONAL launched the call for submitting rapid analytical methods suitable for quantification of polycyclic aromatic hydrocarbons (PAHs) in the raw edible portions of fin fish and seafood. The purpose was to carry out evaluation through the AOAC Official Methods SM program and, supposing the collaborative study is successful, replace existing ´conventional´, time and labour demanding methods, by the ´new´ one. Having a rapid method is essential for quick determination of contaminants in food, especially after environmental disasters.

Within the CONffIDENCE project (Contaminants in food and feed: Inexpensive detection for control of exposure) efficient, cheap, rapid and simple multiresidue analytical method for simultaneous determination of PAHs, polychlorinated biphe-nyls (PCBs) and polybrominated diphenyl ethers (PBDEs) in fish and seafood samples was developed. This method based on rapid ethyl acetate extraction (partition supported by inorganic salts addition), followed by clean-up on silica SPE mini-column need for sample preparation (including extraction, clean-up and concentration prior to the gas chromatography-mass spectrometry (GC-MS)) about 30 min and multiple samples can be processed at the same time. Identification and quantitation is performed using GC-MS in original procedure with time-of flight analyser. The method was validated for 32 PAHs incluting both EU and EPA PAHs and also their methylated analogues, 18 PCBs (dioxin-like) and 7 major PBDEs. The recovery and repeatability (expressed as relative standard deviation RSD,%) of the developed method, were determined for six replicates of the two different matrices (fish and shrimps) spiked with target analytes at two n levels of 1 and 5 $\mu g\ kg^{-1}$. For fish, the PAH recoveries ranged from 73% to 97% with RSDs of 2-13% and for shrimp from 73 to 109% with RSDs of 2-15%. The method trueness was verified by analysis of a NIST Standard Reference Material 2977 (mussel tissue) sample, with results being within the certified values. Limits of quantitation (LOQs) for BaP was 0,05 $\mu g\ kg^{-1}$ and 0,05 to 0,25 $\mu g\ kg^{-1}$ for the other studied PAHs. Because of the different types of MS techniques used by the collaborators GC-MS and GC-MS/MS for determination of target analytes were also tested.

This efficient, cheap, rapid and simple method had been submitted to the AOAC and of 30 reviewed submissions PAH Working Group on Quantitative Methods recommended the ICT Prague method as the most promising candidate.

The purpose of the collaborative study is to evaluate the method's intra-laboratory and inter-laboratory performance. The results of this study will be submitted to the AOAC INTERNATIONAL for adoption as an Official Method for the determination of PAHs in seafood. In the study all together 19 analytes (14 US EPA PAHs and 5 methylated homologues) were included which were tested on three seafood matrixes (mussels, shrimp, and oysters).

Fourteen laboratories participate in this study. The Collaborative study consist of two parts: i) Laboratory qualification and ii) Analysis of Test Materials. In the laboratory qualification phase, the collaborators were conducted seven steps to check their GC-MS and solvent evaporation conditions, selection of appropriate silica SPE column (with low background), reagent blank contamination and familiarize themselves with the method. Also some problems/difficulties during the qualication part were established. As a first step GC separation of critical pairs (benzo[a]pyrene and benzo[e] pyrene, anthracene/phenanthrene and benzo[b]fluoran-thene, benzo[j]fluoranthene and benzo [k]fluoranthene) was tested. Most problematic was the third step - evaporation test because of the presence of highly volatile naphthalene. The absolute recoveries of all analytes have to be above 70%. The problem with volatile compounds was resolved using keeper in both evaporation steps and optimalized evaporation. The fourth step was determination of the elution profiles of PAHs and fat on in-house prepared or commercially obtained silika SPE columns. The next problematic step for some participant laboratories was procedure blank test because of high background contamination of naphthalene in the laboratory. The sixth step was low level spike test on shrimp matrix where participants prepared and analyzed spiked samples with concentration of BaP 1 $\mu g\ kg^{-1}$. The recoveries of this test were in range 75-120% and no problems were detected. But in the last step where practice samples (two spiked samples of shrimps and one sample of SRM1974b-mussel tissue) were analyzed, problems with recoveries were detected. No problem with recoveries was detected when analyzed mussel tissue, but low recoveries (40-60) were obtained when analyzed samples of shrimps spiked few weeks ago and stored in a freezer. After this finding, few experiments for resolution of this problem were prepared. This problem will be discussed in the lecture. Nowadays the first part of the collaborative study is completed and the second part will started in next few days.

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4-L18

TYRAMINE CONTENT AND ENTEROCOCCI ISOLATED FROM THE SLOVAK EWE´S CHEESES AS A POTENTIAL SOURCE OF BIOGENIC AMINES

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Many varieties of foods of animal or plant origin are sources of biogenic amines. Tyramine is natural component of banana, cheese, and avocado, and bacterial action on the amino acids found for istance in meats and fish in which can be produced putrescine and cadaverin. Of course, also, histamine and ß-phenylethylamine have been implicated as ethiological agents in several outbreaks of food poisoning[1].

Tyramine content can be high in oriental preserved foods such as soy sauce, soybean paste, and various condiments. The tyramine presence in cheeses can be associated with the presence of desirable or undesirable bacteria like genus *Entero-coccus*[2,3]. The aim of this study was to (i) identify enterococci isolated from the ewe´s milk lump cheeses from the market vet in Košice, particularly the *Enterococcus faecalis* and *E. faecium;* (ii) also evaluate them for the presence of *tyrdc* gene (tyrosine decarboxylase gene) responsible for the produ-ction of tyramine, (iii) from the identified enterococci to detect the tyramine production in medium according to MAIJALA (1993)[4-6], and (iv) to detect the tyramine content in ewe´s milk cheeses used in the experiment by ion exchange chromatography.

In this study, the presence of *Enterococci* spp. grown on Slanetz-Bartley agar plates from cheeses made from ewe's milk was investigated[7]. The Gram positive catalase negative bacilli identified in this study belonged to the *Enterococcus* species. A multiplex PCR reaction was used for confirmation of isolated bacteria. Out of 34 isolates, 17 strains (50,00%) were identified as *Enterococcus* spp., 17 isolates due to lack of identification by PCR method. In further investigation, the confirmed isolated strains by PCR method (88,24% *E. faecalis* and 5,88% *E. faecium,* and 5,88% *Enterococcus* spp.) were studied for the presence of *tyrdc* gene responsible for tyramine production. In all isolated strains of *Enterococcus* spp. (100%) were found to carry the *tyrdc* gene responsible for tyramine production. The same results were achieved with the tyramine production by the isolated strains (mentioned above) in medium according to Maijala after incubation at 15°C and 6°C for 1, 2, 3, and 6 days.

The detected content of tyramine in the ewe´milk lump cheeses included in the experiment reached the mean value of 24,27 $mg\ kg^{-1}$ (3,70 – 68,20 $mg\ kg^{-1}$).

The ability of enterococci to decarboxylase amino acid tyrosine onto tyramine, and the presence of *tyrdc* gene for the tyramine production appear to be a marker of food safety that enhances the ability of isolates to cause food-induced traits in humans being.

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REFERENCES

1. Dičáková Z., Dudriková E.: Chem. Listy *102*, 617 (2008).
2. Franz C. M. A. P., Stiles M., Schleifer K. H., Holzapfel W. H.: Internat. J. Food Microbiol. *88* 105 (2003).
3. Foulquié Moreno M. R., Sarantinopoulos P., Tsakalidou E., De Vuyst L.: Internat. J. Food Microbiol. *106*, 1 (2006).
4. Maijala R.: Lett. Appl. Microbiol. *17*, 40 (1993).
5. Trivedi K., Karpíšková R.: Acta Scientiarum Polonorum Medicina Veterinaria *7*, 29 (2008).
6. Trivedi K., Borkovcová I., Karpíšková R.: Czech J. Food Sci. *27*, 357 (2009).
7. Jurkovič D., Križková L., Dušinský R., Belicová A., Sojka M., Krajčovič J., Ebringer J.: Lett. Appl. Microbiol. *42*, 553 (2006).

4-L19

LEAD, CADMIUM, AND ARSENIC IN EWE´S MILK AND BLOOD SERUM OF EWES AND THEIR LAMBS AS CHEMICAL HAZARD ON FARMS IN EAST SLOVAKIA

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The estimation of the risk associated with dietary intakes of heavy metals residues by the consumer is an integral part of regulatory processes. Slovakia is a country with very well sheep farming programme and ewe´s milk is very popular for the cheese making.

The aim of this study was to evaluate the concentrations of lead, cadmium, and arsenic in ewe´s milk and blood serum of ewes and their lambs, and to compare the possible exposure of these heavy metals to the tolerable daily intake (TDI)[1,2].

Milk and blood samples were collected from ewes and their lambs in five farms in East part of Slovakia. Four of these farms are situated in an area with high pollution index and one in the area with low pollution index served as control[3,4].

Concentrations of heavy metals present in milk (density 1,035 g cm^{-3}) and blood serum were studied by using graphite furnace atomic absorption spectrometry (AAS).

Data of this experimental work showed that heavy metals such as lead, cadmium and arsenic can be still present in raw ewe´s milk for further processing used for human consumption.

The compounds detected at lowest concentration were cadmium in milk, ewes and lambs serum (0,018 μmol l^{-1} ≅ 0,2 μg kg^{-1}, 0,122 μmol l^{-1} and 0,118 μmol l^{-1}) and arsenic in milk (0,006 μmol l^{-1} ≅ 0,45 μg kg^{-1}).

Cadmium concentrations did not vary greatly between the samples evaluated, suggesting that this compound passed through the blood into the lambs and only limited concentrations (mean value 0,002 μmol l^{-1}) were observed in milk (from 0,001 μmol l^{-1} to 0,002 μmol l^{-1} ≅ 0,12 to 0,22 μg kg^{-1}). Overall cadmium concentrations were comparable to values reported in literature and were below the maximum limit of 10 μg kg^{-1} (Codex Alimentarius of the Slovac Republic, 2006).

The highest concentrations of lead were detected in lambs (0,152 μmol l^{-1}), following milk and ewe´s serum, respectively (0,014 μmol l^{-1} ≅ 2,63 μg kg^{-1} *versus* 0,128 μmol l^{-1}).

Experimental results also showed different patterns of heavy metal contents in milk and serum of ewe and lamb among the samples received from different part of the East Slovakia. The area of ewe´s farms were located in the part of Slovakia, in which in the 80-ies the higher content of heavy metals in the environment was detected.

EU regulations have not yet fixed limits for arsenic in milk or dairy products. Our results showed that the lowest concentration of arsenic was observed in milk (0,017 μmol l^{-1} ≅ 1,23 μg kg^{-1}), and the highest content was observed in serum of ewes and lambs (0,16 μmol l^{-1} *versus* 0,127 μmol l^{-1}, respectively).

Differences in the detected heavy metals concentrations in evaluated samples were in correlation with the industrial areas in which farms were located. Concentration of heavy metals in samples evaluated by AAS documented the presence of heavy metals not only in the blood serum of experimental ewes and their lambs but also in the milk from the area with low pollution index (e.g. cadmium 0,1 μmol l^{-1} in ewe´s serum, 0,002 μmol l^{-1} in milk ≅ 0,22 μg kg^{-1}, and 0,09 μmol l^{-1} in lamb´s serum, respectively). This effect can be caused by the wind orientation, and its speed.

Despite the exposure of Slovakian consumers to lead, cadmium, and arsenic was not superior to the TDI. The mean daily intake for all heavy metals investigated in ewe´s milk was lower than stated mean daily intakes in some EU countries, and it was less than 1% of the estimated mean daily intake (8,76×10^{-8} mg day^{-1} for cadmium, 1,15×10^{-6} mg day^{-1} for lead, and 1,97×10^{-7} mg day^{-1} for arsenic, respectively)[5].

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REFERENCES

1. Anastasio A., Caggiano R., Macchiato M., Paolo C., Paino S., Cortesi M. L.: Acta Vet. Scand. *47*, 69 (2006).
2. Caggiano R., Sabia S., D´Emilio M., Macchiato M., Anastasio A., Ragosta M., Paino S.: Environ. Res. *99*, 48 (2005).
3. Herian K.: *Proceedings of Hygiena Alimentorum XXXI*, p.78, Štrbské Pleso, 2010.
4. Matlovič R.: Urbánne a krajinné štúdie *3*, 383 (2000).
5. Nasreddine L., Parent-Massin D.: Toxicol. Lett. *127*, 29 (2002).

4-L20

ANALYSIS OF NON-TRADITIONAL FRUITS (*HIPPOPHAE RHAMNOIDES* AND *CORNUS MAS*)

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Sea buckthorn (*Hippophae rhamnoides* L.) is a kind of hardy bush with orange oval berries, naturally distributed in various regions of Asia and Europe. Due to the multiple positive effects, including lowering plasma cholesterol level, inhibiting platelet aggregation, promoting the cure of injuries on skin and mucosa, regulating immune function, and anticancer effects, many medicinal preparations of sea buckthorn have been used in clinical practice and traditional medicine in many countries. Recently, the nutritional value of the berries has also been recognized in the western world because of their special chemical composition. Sea buckthorn berries contains many nutritious substances: flavonoids, lipoid substances, triterpenes, phenolic acids, sterols, sugars, vitamins, etc. It's suitable for production of juices, sirups, preserves, jams, candies, tea mixes and other. It takes a place in cosmetics too.

Cornelian cherry (*Cornus mas*) is a shrubby species of cornels native to southern Europe and southwest Asia with characteristic yellow blossoms and dark red oval stone-fruits. Fruits from several Corpus spp. have been used for improved liver and kidney functions. It is also reported to have anti-bacterial, anti-microbial and anti-allergic properties. From a nutritional point of view is a source of different type of sugars, anthocyanins, vitamins and other biologically active substances. In the past, the cornelian cherry was plentifully utilized for production of preserves, jams or spirits.

Nowadays, these species of small berry fruit, also called superfruit, are not utilized a lot in food industry, unless they have a big potential to be a good source of biologically active substances which are important for humans health and can be suitable as raw material for various kinds of functional food. Both species are unassuming for soil and climate requirements and readilly grows in our latitudes.

This work is focused on characterization of 7 varietes of sea buckthorn and 9 varietes of cornelian cherry grown in the experimental orchard of Brno Mendel University. Dry matter, titrable acids, nutritionally important and toxic elements, content of vitamin C, total polyphenols and total antioxidant activity were determined.

Vitamin C was estimated as L-ascorbic acid by modified standard method[1]. After extraction with metaphosphoric acid solution the content of vitamin C was determined by HPLC with UV detection at 254 nm. For the separation the Gemini C18 column and mobile phase consisting of phosphate buffer with 10 % of methanol were used.

Dry matter of fruits was determined by drying at 70 °C to constant weight. Titrable acids were estimated by titration with sodium hydroxide and expressed as grams of malic acid in fresh fruit weight.

The total polyphenolics content using Folin-Ciocalteau's reagent were spectrophotometricaly determined using modified method from ref. 7 and expressed in mg of gallic acid in fresh fruit weight.

The concentration of selected elements was determided using ICP-MS Thermo X-series Quad after mineralization of fruit samples (nitric acid and hydrogen peroxide) in microwave oven Milestone 1200. For sample introduction CETAC autosampler ASX 520, peristaltic pump (50rpm) and traditional Meinhard nebulizer with a conical spray chamber cooled at 5°C were used. The experimental conditions of ICP-MS were following: cool gas flow 13 l min^{-1}, auxillary gas flow 0,7 l min^{-1}, nebuliser gas flow 0,9 l min^{-1}, forvard power 1300 W, reflected power < 5W. Oxide ions (CeO+/Ce+) and doubly charged ions Ba++/Ba were less than 3%. Calibration of the instrument was done using dilluted standards of metals of interest.

Antioxidant activity of the samples were investigated by Electron paramagnetic resonance spectroscopy, using stable radicals 1,1-diphenyl-2-picrylhydrazyl (DPPH)[2]

Among cultivars of each fruit were found discernible variances in all studied parameters.

Content of vitamin C ranges between 715,6–3100 mg per kg of fresh sea buckthorn berries and 198,5–433 mg per kg of fresh cornelian cherry fruits.

Values of total polyphenols expressed in gallic acid equivalents were varying from 1,038 to 3,009 mg kg^{-1} in sea buckthorn berries and from 1195 to 6479 mg kg^{-1} in cornelian cherry fruits.

In investigated fruits sixteen elements were analysed. The results shows that sea buckthorn and cornelian cherry are rich sources of boron, zinc and manganese. Little differences in elemental composition between cultivars were discovered. None of the samples did not exceed concentration limits for toxic elements (As, Cd, Pb) according to czech legislation.

Antioxidation properties of the samples in presence of $^{\bullet}$DPPH was quantified by comparsion of $^{\bullet}$DPPH relative concentration in time 14 min after mixing with the sample[2]. Cornelian cherry fruits have higher antioxidant activity than sea buckthorn berries. Among each cultivars of cornelian cherry significant differences (up to 40 %) in radical scaveging activity were found. Sea buckthorn shows slightly lower antioxidant activity but it has much smaller differences between each cultivar (approx. 10 %).

REFERENCES

1. ČSN EN 14130*: Potraviny - Stanovení vitamínu C metodou HPLC,* Český normalizační institut, Praha, 2004.
2. Stasko A., Polovka M., Brezova V., Biskupic S., Malik F.: Food Chem. *96*, 185 (2006).

4-P1

GAS CHROMATOGRAPHIC-MAS SPECTROMETRIC DETERMINATION OF LOW-MOLECULAR PRODUCTS FORMED DURING EXPOSITION OF POLYCAPROLACTONE TO *ASPERGILLUS*-LIPASE

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The degradation of poly(ε-caprolactone) (PCL) by the action of enzymes – lipases was recognized already in 1977 by Tokiwa *et al.*[1] Lipases function as depolymerases cleaving the long polymer chains up to the formation of lower-molecular products such as oligomers and monomers[2]. These oligomeric products become water-soluble and hence diffuse readily to the aqueous medium during surface erosion. Monitoring of these released products gives qualitative as well as quanitative evaluation of degradation process. Oligomeric products were detected by gel-permeation chromatography (GPC)[3], the monomeric ones were analysed by a solid-phase micro-extraction method in combination with gas chromatographic-mas spectrometric determination (GC-MS)[4].

This work reports on the determination of possible degradation products released from PCL films aged in the presence of lipase-type enzyme by means of the gas chromatographic-mass spectrometric analysis. The analysis was performed on HP-6890 gas chromatograph equipped with the mass spectrometric detector HP-5673. HP-5MS column (30 m x 0,25 mm x 0,5 μm, film of bonded 5 % phenyl, 95 % dimethylpolysiloxane, Hewlett-Packard, Waldbronn, Germany) was used. PCL substrate in the film form (M_n = 19 300 g/mol) was added to a phosphate buffer system with pH = 7 containing 0,05 mg ml^{-1} *Aspergillus sp.* lipase with the initial enzymatic activity of 0,3 mmol min^{-1} ml^{-1}. The degradation experiments were carried out for 42 days at 37 °C. For analyses purpose, the aliquots of buffer solution, used in degradation tests, were withdrawn in certain intervals and extracted with dichloromethane. Subsequently, the presence of ε-caprolactone was confirmed in the extracts. The decrease in the amount of ε-caprolactone (18,1 – 0,65 μg g^{-1}) was observed during the degradation period. Moreover, the presence of compound (Figure. 1) having the structure close to the trimer of ε-caprolactone was detected.

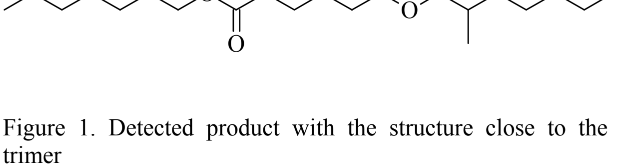

Figure 1. Detected product with the structure close to the trimer

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REFERENCES

1. Tokiwa Y., Suzuki T.: Nature *270*, 76 (1977).
2. Mueller R.-J.: Process Biochem. *41*, 2124 (2006).
3. Shalaby S. et al., in the book: *Polymers as Biomaterials*, p. 181. Plenum Press, New York 1985.
4. Hakkarainen M.: J. Chrom. A *1010*, 9 (2003).

4-P2

OPTIMIZATION OF LIPASE PRODUCTION BY *BACILLUS SUBTILIS*

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Extracellular lipases were proved as effective and selective biocatalysts in a variety of organic chemistry synthesis and transformations such as the synthesis of chiral pharmaceuticals[1]. The lipases are triacylglycerol acylhydrolases EC 3.1.1.3 and hence are able to catalyze the reactions on ester bonds including the degradation of aliphatic polyester chains[2]. Since these enzymes combine the outstanding performance as the catalysts with both high enantio- and stereoselectivity and the possibility to be produced in high yields from renewable sources i.e. bacteria and fungi they are in the centre of research interest[3].

Among bacterial lipases, those from *Bacillus subtilis,* bacterial strain widely distributed in soil, display interesting properties from the view of biotechnological application.[4] Further, the bacterium produces several extracellular hydrolytic enzymes such as a phospholipase, a lipase LipA, and esterase LipB and hence is capable to utilize complex nutrient sources[5]. The production of extracellular lipase by the microorganism is induced by the presence a lipid substrate alone or together with sugars as a carbon source in the cultivation media[6].

The aim of this work was to study the effect of pH and temperature on the activity of lipase produced by the submerged culture of *Bacillus subtilis* CCM 1999. A series of cultivation media differing in the carbon source and the nutrient content *i.e.* brain heart infusion broth, nutrient broth, and nutrient broth supplemented with 2 % glucose was used. The activity of lipase was measured spectrophotometrically using *p*-nitrophenyl laurate in ethanol as the substrate. The highest lipase activity 0,136 μmol min^{-1} ml^{-1} was obtained in nutrient broth with glucose as the carbon source after three days of cultivation. The lipase was most active at pH of 8. The optimum temperature was observed at 37 °C. The thermal inactivation of the lipase under investigation was observed at 80 °C. It retained about 60 % relative activity at 60-70 °C.

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REFERENCES

1. Hasan F., Shan A. A., Hameed A.: Enzyme Microb. Technol. *39*, 235 (2006).
2. Mochizuki M., Hirano M., Kanmuri Y., Kudo K., Tokiwa Y.: J. Applied Polymer. Sci. *55*, 289 (1995).
3. Mateos Dias J. C., Rodríguez J. A., Roussos S., Cordova J., Abousalham A., Carriere F., Baratti J.: Enzyme Microb. Technol. *39*, 1042 (2006).
4. Sanches M., Prim N., Randez-Gil F., Pastor F. I. J.,Diaz P.: Biotechnol. Bioeng. *78*, 339 (2002).
5. Eggert T., Pouderoyen G., Pencreac'h G., Douchet U., Verger R., Dijkstra B. W., Jaeger K. E., Colloids Surf. B Biointerfaces *26*, 37 (2002).
6. Sharma R., Christib Y., Banerjee U. C., Biotechnol. Adv. *19*, 627 (2001).

4-P3

STUDY OF CHANGES IN ANTIOXIDANT LEVELS IN SEASONAL BERRY FRUITS DURING LONG-TERM STORAGE BY FREEZING

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The phenolic composition of fruits and, hence, their antioxidant properties may be modified by environmental and post-harvest factors, including storage and processing.

This work was focused on the study of changes in the content of selected low molecular weight antioxidants in five local seasonal berry fruits available in the Czech Republic (strawberries, raspberries, blueberries and red/black currants). The fruit was freezing for 6 months due to seasonality of the fruit. Berries were stored as whole fruits, whole osmotically processed fruits by sucrose, raw pulp and sweetened pulp. At first, starting values of selected antioxidant parameters were measured in fresh fruits (ascorbate, total antioxidant activity, total phenolics, catechins and individual flavonoids). After freezing the same parameters were measured regularly in two month periods.

Total antioxidant capacity was measured by Randox kit. Individual flavonoids were analyzed using RP-HPLC/UV-VIS method (Biospher PSI 200 C18) and on-line LC/MS using the extrenal standards to phenolic quantification. Ascorbic acid was measured by RP-HPLC/UV-VIS (Hypersil APS-2, NH_2). Sensory analysis (taste, texture, flavour) was proved for the whole fruits and also for whole sweetened fruits.

During long-term freezing total antioxidant activity (TAS) changed individually in different kinds of berries and was related to the sample processing before freezing. The highest TAS values were found in raspberries and strawberries. In contrary, the lowest value was found in red currant.

Flavonoids constituted 90% of total polyphenols in strawberries, 5% in raspberries, about 10% in currants and 30% in blueberries. Overall, the highest content of flavonoids was observed in strawberries. Higher levels of flavonoids showed blueberries, other samples of black currant showed relative comparable content of flavonoids.

As the most suitable method of fruit processing, with regard to antioxidant content, storage of the whole fruits or pulp can be recommended. Content of ascorbate decreased in all forms and species of the processed fruits during all the time of storage (about 80% in the whole berries). Oppositely, the content of other studied antioxidants exhibited fluctuations or increasing trend. Sensory analysis exhibited that the best taste and texture was evaluated in both currants independently on sugar addition, while the worst evaluation was found in strawberries and raspberries.

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4-P4

ANALYSIS OF POLAR AND NONOPLAR LIPIDS IN THE SURFACE LAYERS OF LONG-STORED APPLES IN THE ATMOSPHERE WITH DIFFERENT OXYGEN CONTENT

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Lipids are important structural and metabolic constituents of plant cells. They are essential components of membranes. Disturbances in membrane lipid composition of fruit often have severe consequences on the ability of the cell to adapt to high/low temperatures and other stress conditions, which may lead to various storage disorders.

This work was focused on analysis of polar and nonpolar lipids in the surface layers of different varieties of apples stored for 160 days in normal atmosphere and modified atmosphere with low oxygen content. This study was supplemented by analysis of microbial contamination and by microscopy analysis of surface layers for assessing the impact of storage process on quality of fruits.

We used the combination of two chromatographic separation techniques (TLC and GC) for the determination of the individual lipids and fatty acids (FA) composition in the surface layers of apples (GC 6890N Apparatus Agilent Technologies, column DB23). To monitoring the microbial contamination of stored fruit contact tests with selective media (Merck - Contact Envirocheck ®) were used. Stored apples were acquired from the cooperating Department of Post-Harvest Technology of Horticultural Products, Mendel University in Lednice.

To the major fatty acids in apple surface layers belong palmitic, stearic, linoleic and oleic acids. During long-term storage, the minor changes of fatty acids content were observed in flesh of apples. These effects are caused by FA deposit in the deeper layers of fruits and less contact with the storage atmosphere. The total degree of saturation increased with the storage period and with decreased oxygen concentration. The amount of contaminating microorganisms on the surface of apples increased gradually to their aging. The highest microbial containation was found in the aged fruit stored in normal atmosphere, probably due to increased invasiveness of fungi that are capable invades through pinched wax layer into the already weakened apple fruit.

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4-P5

COMPARISON OF SACCHAROMYCES STRAINS ISOLATED FROM TWO TYPES OF WINE

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Alcoholic ferementation is a process involving a great number of genera and species of yeast and bacteria. The conversion of the grape must into wine is almost exclusively performed by more alcohol-tolerant species of *Saccharomyces* genus, especially by *Saccharomyces cerevsiae*. Due to great genetic as well as physiological similarity of the individual species of the genus *Saccharomyces*, identification at species level is very complicated[1,2].

In this study we analysed *Saccharomyces* genus at species level. For this purpose we used PCR-fingerprinting method. Futher, we also identified this group by PCR-RFLP method by using ITS1-ITS4 primers and *Hae*III restriction endonuclease[3].

Saccharomyces genus (length of ITS-PCR amplified product – 880 bp) was identified by PCR-fingerprinting. We used single repetitive primers (GAG)$_5$, (GAC)$_5$ and M13 phage. All identified yeast strains were isolated from grapes and must during spontaneous fermentation process. As a region of our interest, we have chosen South Moravian region (2009, 2010) and 2 different variety of wine – Sauvignon (white wine) and Rulandske modre (red wine). Both varieties were produced in integrated and ecological agriculture mode[4,5].

Together 59 yeasts strains of *Saccharomyces* genus were identified; 32 from Rulandske modre (18 from ecological, 14 from integrated) and 27 from Sauvignon variety (12 from ecological, 15 from integrated).

We also identified collection yeasts strains (from CCY Bratislava) and then we compared PCR profiles of this collection strains with isolated yeasts. Finally, electrophoreograms were evaluated by BioNumerics software and as a result, we are able to compare genetics similarity of isolated yeasts. In conclusion, we were able to identified yeasts of *Saccharomyces* genus at species level.

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REFERENCES:

1. Dos Santos S. K. B., Basilio A. C. M., Brasileiro B. T. R. V:. World J. Microbiol. Biotechnol. *23*, 1613 (2003).
2. Le Jeune C., Erny C., Demuyter C.: Food Microbiol. *23*, 709 (2006).
3. Steve-Zarzoso B., Belloch C., Uruburu F., Querol A.: Int. J. System. Bacteriol. *49*, 329 (1999).
4. Zane L., Bargelloni L., Patarnello T.: Molecular Ecology *11*, 1 (2002).
5. Nikolou E., Andrighetto C., Lombardi A.: Food Control *18*, 1458 (2007).

4-P6

ANTIOXIDANT AND ANTIMICROBIAL PROPERTIES OF MONOFLORAL BEE POLLEN USABLE IN HUMAN NUTRITION

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Bee pollen is regarded as valuable food. Several new studies are dealing with the properties of bee pollen[1,2]. The aim of the study was investigate antioxidant properties and an antimicrobial activity of frozen bee pollen samples from three plant species: poppy (*Papaver somniferum* L.), rape (*Brassica napus subsp. napus* L.), sunflower (*Helianthus annuus* L.).

Samples of bee-collected pollen were obtained from beekeepers, which respected qualitative criteria for gathering and storing as proposed by Bogdanov[3]. The samples were collected from west Slovakia. The fresh bee pollen was stored at -18 °C, 20 % moisture, six months until analyzed. Antiradical activity was determined by modified DPPH method according to Brand-Williams et al.[4] and expressed as the % of DPPH inhibition. Reduction power of pollen compounds was evaluated spectrophotometrically and expressed as quantity of ascorbic acid necessary to achieve the same effect. Antioxidant activity was further evaluated by DNA-based biosensor using a voltametric procedure based on the protective effect of antioxidants against the oxidative DNA damage. Total polyphenols content was quantified according to the Folin-Ciocalteau method using tannin as reference standard. The antimicrobial effect of the bee pollen samples were tested using the agar well diffusion method. For extraction methanol in 99,9 % and 70 % concentrations and ethanol in 96 % and 70 % concentrations were used. Five different strains of bacteria *Listeria monocytogenes* CCM 4699; *Pseudomonas aeruginosa* CCM 1960; *Staphylococcus aureus* CCM 3953; *Salmonella enterica* CCM 4420 and *Escherichia coli* CCM 3988 were tested after 24 and 48 hours.

Average value of antiradical activity was 70,05 ± 17,17 %. Reduction power of frozen bee pollen compounds was 3575,56 ± 749,04 μg ml^{-1} and the antioxidant activity was 1,09 ± 0,68. In the case of polyphenols the average content reached 964,22 ± 319,31 mg kg^{-1}. The antiradical activity of bee pollen samples occurred in the range from 47,68 to 87,51 %, reduction power of compounds from 2775 to 4501 μg ml^{-1}

and antioxidant activity from 0,32 to 1,95. Total polyphenols were presented in amount 682-1389 mg kg^{-1}. The antioxidant properties significantly differ in particular plant species. The highest values of all antioxidant parameters were detected in *B. napus* pollen. All examined antioxidant properties decreased in the order: *B. napus* > *P. somniferum* > *H. annuus*. More research is needed to identify the antioxidant properties and to evaluate disease preventing effects of bee pollen.

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REFERENCES

1. Kňazovická V., Kačániová M., Dovičičová M., Melich M., Kadási-Horáková M., Barboráková Z., Mareček J.: Potravinárstvo *5*, 27 (2011).
2. Kolesarova A., Capcarova M., Bakova Z., Galik B., Juracek M., Simko M., Sirotkin A. V.: J. Environ. Sci. Health B *46*, 207 (2011).
3. Bogdanov S.: Apiacta *38*, 334 (2004).
4. Brand-Williams A., Cuvelier M. E., Berse, C.: Lebensm. Wiss. Technol. *28*, 25 (1995).

4-P7

POLYUNSATURATED FATTY ACIDS IN FLAX (*LINUM USITATISSIMUM* L.) GENOTYPES WITH HIGH AND LOW CONTENT OF ALFA-LINOLENIC ACID

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At the present flax seeds are known as functional food[1]. Whole linseed contains 41 % of fat of which 70 % belong to polyunsaturated fatty acids (PUFA) and more than half of fatty acids (FA) belong to alfa-linolenic acid (ALA - C18, n-3 FA) – 50 – 62 % of flax oil or 22 % of whole seed[2-5]. The second important essential FA is linoleic acid (LA - C18, n-6 FA). It represents 17 % of all FA[6]. The aim of our work was to characterize linseed (*Linum usitatissimum* L.) genotypes divided to groups with high and low content of (ALA).

There were analyzed 33 genotypes of linseed, thereof 21 breeding lines and 12 varieties. Proportional representation of FA was realized by the norm ČSN EN ISO 5508. The oil content was analyzed according to internal methodology of Agritec Ltd., which was based on the norm ČSN EN ISO 659.

From all analyzed 33 linseed genotypes, the 69,7 % represent high alfa-linolenic genotypes and 30,3 % are genotypes with low ALA content. The average content of total fats was 40,33 %, content of ALA was 38,20 % and content of LA 33,63 % in analyzed seed samples (n= 489). Distributional analysis confirms normal distribution of experimental groups ($p < 0,05$). In the group with high ALA was average content of ALA in the range from 31,9 to 65,2 %; and in the group with low ALA was in the range from 1,1 to 6,6 %. High content of ALA worsen the qualitative parameters of flax oil, but on other hand the high content of ALA brings many health benefits. The breeding is then oriented to low content of ALA and increasing of LA content. In the group with low ALA was detected content of LA in the range from 63,2 to 75,0 % and in the group with high ALA was in the range from 11,1 to 42,6 %. In the groups of genotypes with brown and yellow seeds was found out the very high variability of analyzed fatty acids (62,79 – 78,38 %). There were not confirmed negative correlation between content of ALA and LA in the group with low ALA, but in the group with high ALA was this negative correlation confirmed ($p < 0.0001$; $r^2 = -0,67$ to $-0,85$).

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REFERENCES

1. Thompson L.U., Cunnane, S.C., in the book: *Flaxseed in human nutrition*. Champaign, IL: AOCS Publishing, 2003.
2. Bhatty R.S., in the book: *Flaxseed in human nutrition*. p. 22 Champaign, IL: AOCS Press, 1995.
3. Bloedon L. T., Szapary P. O.: Nutr. Rev. *62*, 18 (2004).
4. Hall C., Tulbek M. C., Xu Y.: Adv. Food Nutr. Res. *51*, 1 (2006).
5. Muir A. D.: J. AOAC Int. *89*, 1147 (2006).
6. Velíšek J., in the book: *Chemie potravin 1*. p. 344. Ossis, Tábor 2002.

4-P8

CONTENT OF POLYUNSATURATED FATTY ACIDS IN LINSEED GENOTYPES (*LINUM USITATISSIMUM* L.) AND COVERING OF RECOMMENDED NUTRITIONAL AMOUNTS BY CONSUMPTION OF LINSEED

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Primary function of fatty acids (FA) is creation of prostaglandins, which regulate body functions, like heart frequency, blood pressure, blood coagulation, fertility, conception. Prostaglandins also play important role in immunity, they regulate inflammation and stimulate body to infection fight[1,2]. The FAO and WHO recommend proportion of omega-6:omega-3 FA between 5:1 and 10:1. They propose more food consumption with high content of essential FA[3].

The aim of this work was to characterize selected genotypes of linseed, from the view of content of total fat, polyunsaturated fatty acids (ALA – alfa-linolenic, LA – linoleic acid). Also there were calculated covering of recommended nutritional amounts by consumption of linseed according to determined values in selected groups of population (adolescent boys and girls (15 – 18 years old), working women (35 – 54 years old), men (35 – 59 years old) and nursing women.

Total of 33 genotypes of linseed were analyzed. Proportional representation of FA was realized by the norm ČSN EN ISO 5508. The oil content was analyzed according to internal methodology of Agritec Ltd., which was based on the norm ČSN EN ISO 659.

From the analyzed genotypes (n=33) 21 breeding lines and 12 varieties of linseed were available. The number of analyzed seed samples was 489. The highest content of oil reached variety ASTRAL (43,9 %) with high content of ALA and the lowest had genotype AGT 892/05 (36,22 %) with low ALA. In the group with high ALA the maximum content of ALA achieved variety ASTRAL (64,20 %) and lowest the variety FLANDERS (39,70 %). In the group with low ALA the lowest content of ALA reached genotype AGT 981/05 (1,1 %) and the highest the genotypes AGT 892/05 and AGT 987/02 (6,4 %). The variety AMON has the lowest content of ALA (1,7 %) and the highest content of ALA achieved variety LOLA (6,4 %). The highest content of LA had variety LOLA (75,0 %) and the lowest content reached the genotype AGT 892/05 (63,20 %) in the group with low ALA. The consumption of 20 g (i.e. circa 2 tablespoons) of linseed covers energy requirement for 2,96 up to 4,11 % (it is less then tenth of recommended nutritional amount). The values of LA covered recommended intake for 11,90 to 17,55 % (it is less then fifth of recommended nutritional amount). The values of ALA covered recommended intake for 116,78 to 181,67 % (it is more than 100 % of recommended nutritional amount).

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REFERENCES

1. Rudin D., Felix C. in the book: *Omega-3 oils: a practical guide*. p. 224. Avery Trade, USA 1996.
2. Stoll A. in the book: *The omega- connection: The groundbreaking antidepression diet and brain program.* p. 304. Free Press, New York 2002.
3. Institute of Medicine: Dietary Reference Intakes for Energy, Carbohydrate, Fiber, Fat, Fatty Acids, Cholesterol, Protein, and Amino Acids. National Academies Press, Washington, DC, 2002.

4-P9

MONITORING OF YEASTS POPULATION IN ECOLOGICAL AND INTEGRATED PRODUCED MORAVIAN WINES

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In enology, yeasts play an important part in the characteristics of the final product. Many factors such as chemical and microbial composition of grape juice can influence wine quality. Yeasts are predominant in the biochemical interaction with components of must, more information about yeasts composition of grape must is important to produce high quality wine[1]. Rapid identification of yeasts population is necessary for monitoring of fermentation process and for obtaining good quality wine[1,2].

In this study, we identified yeasts isolated from red variety Rulandske modre from South Moravia. Yeasts were isolated from must during different stages of spontaneous fermentation process. We chose two types of vineyard agriculture – ecological and integrated for both varieties. Together, we isolated 60 strains from ecological variety and 64 from integrated. In order to identify species we used PCR-RFLP method (Polymerase Chain Reaction and Restriction Fragment Length Polymorphism). We amplified 5.8S-ITS segment of rDNA by using ITS1 and ITS4 primers. Then amplified DNA was chopped by five restriction endonucleases – *Hae*III, *Hinf*I, *Taq*I, *Alu*I and *Mse*I[3,4].

All obtained electrophoreograms were processed by BioNumerics software. We obtained 8 different groups of isolated yeasts from integrated vineyard and 10 groups for ecological vineyard. Finally, based on our results we processed dendrograms which show representation of yeasts during wine production and compare their genetics similarity.

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REFERENCES

1. Di Mario E., Ercolini D., Coppola S.: Int. J. Food Microbiol. *117*, 201 (2007).
2. Heras-Vazquez F. J., Mingorance-Cazorla L., Clemente-Jimenez J. M., Rodriguez-Vico F.: FEMS Yeast Res. *3*, 3 (2003).
3. Sences-Ergul S., Agoston R., Belak A., Deak T.: J. Food Microbiol. *108*, 120 (2006).
4. Steve-Sarzoso B., Bellech C., Uruburu F., Querol A.: Int. J. Syst. Bacteriol. *49*, 239 (1999).

4-P10
ISOLATION OF DNA FROM PROBIOTIC PRODUCTS USING SOLID CARRIERS

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Lactic acid bacteria are historically defined as a group of microaerophilic, Gram-positive organisms that ferment hexose sugars to produce primarily lactic acid[1]. Bacteria of the genus *Lactobacillus* are important group of the lactic acid bacteria. In humans, *Lactobacillus* species are symbiotic and are found in normal gastrointestinal microflora[2]. They play an important role in the food industry because they significantly contribute to the flavour, texture, and the nutritional value of food products.

Molecular methods such as PCRs are essential for correct identification *Lactobacillus* cells at the genus or species levels. Polymerase chain reaction (PCR) can be used for identification of probiotics in food products. The aim of this work was to isolate PCR-ready DNA. Bacterial DNA was isolated from lysed cells of *Lactobacillus* in probiotic products. Reversible DNA adsorption on the surface of solid particles. Carboxyl coated nonporous poly(2-hydroxyethyl methacrylate-*co*-glycidyl methacrylate) (P(HEMA-*co*-GMA)) magnetic particles and silicagel coated manganase Perovskite ($La_{0,75}Sr_{0,25}MnO_3$) nanoparticles were used for this purpose. Classical procedure of phenol extraction and DNA precipitation with ethanol was used as control. DNA was adsorbed on the surface of the microspheres in the presence of 16 % poly(ethylenglycol) (PEG 6000) and 2 M sodium chloride (NaCl) concentrations[3]. The adsorbed DNA was released from the surface of particles by low ionic strength TE buffer (pH= 8,0). The quality of isolated DNA was checked by spectrofotometric measurement and PCR amplification.

All the DNA samples isolated using magnetic particles and phenol extraction procedures were PCR-ready. The DNA isolated from lysed cells of probiotic products was quantified in real-time qPCR.

REFERENCES

1. Carr F. J., Chill D., Maida N.: Crit. Rev. Microbiol. *28*, 281 (2002).
2. Zhang Z., Ye Z.,Yiu L., Peng S.: BMC Evol. Biol. *11*, 1 (2011).
3. Rittich B., Španová A., Šálek P., Němcová P., Trachtová Š., Horák D.: J. Magn. Magn. Mat. *321*, 1667 (2009).

P11
FUNCTIONALIZED SINGLE-WALLED CARBON NANOTUBES FOR LIPASE IMMOBILIZATION

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Single walled carbon nanotubes (SWCNTs) have attracted wide interest for their biochemical applications, in particular the immobilization of enzymes, delivery of bio-active molecules, visualization of biological molecules, bio-therapy etc.

In this work, a lipase from *Candida rugosa*- an almost universal lipolytic biocatalyst - was covalently anchored onto SWCNTs chemically modified with 1,6-hexandiamine via diimide-activated amidation. The whole process of functionalization is characterized by FTIR, TEM and Raman spectroscopy. The SWCNTs-lipase biomaterials showed increased solubility in some common used organic solvents, such as DMF, THF and chloroform.

The activity of immobilized lipase was compared with that of lyophilized powdered enzyme in the hydrolysis reaction of carotenoid esters.

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4-P12
CONCANAVALIN A LECTIN-BASED AFFINITY CHROMATOGRAPHY FOR ENRICHMENT AND DETERMIANTION OF GLYCOPROTEINS IN BARLEY MALT

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Of all known protein post-translational modifications (PTM), glycosylation is the most common and complex one, which is predicted to occur on over 50% of all proteins and shows a great of diversities. Glycoproteins play important roles in many biological processes, such as embryonic development, cell division processes and protein regulations and interactions[1]. For identification and detailed analysis of glycoproteins is required to separate them from complex samples. Lectin affinity chromatography has been widely used for purification and enrichment of glycoproteins or glycopeptides. Lectins are proteins capable to recognize and bind specific carbohydrates. There are many lectins available with very specific ligands whereas one of the most well characterized and widely used is concanavalin A (ConA) derived from *Canavalia ensiformis* (Jack been) seeds. ConA binds specifically to α-mannosyl and α-glucosyl residues[2,3].

In this study, glycoproteins of barley malt used in brewing were investigated. Plant glycoproteome analysis is important for agriculture (because of their biological role in plants) and in biotechnological industry, in our case in malting and beer production. Glycosylated proteins were first separated from non-glycosylated proteins using high performance liquid chromatography (HPLC). Capture of glycoproteins was performed on a new monolithic ConA affinity column (Dionex). Then required proteins were separated on SDS-PAGE for fast screening of protein representation or on different HPLC columns, respectively. Separated proteins were digested and identified using MALDI/TOF-TOF mass spectrometer. The aim of this study is to optimise separation and enrichment of individual glycoproteins with the intention of *N*-linked glycoproteins for following study of attached glycans after degycosylation. Used techniques can be used for investigation of glycoproteins in other samples as well.

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REFERENCES

1. Hao P, Ren Y., Xie Y.: PLoS One *5*, 11 (2010).
2. Twyman R. M., in the book: *Principles of proteomics*, chapt. 8.3, p.182, BIOS Scientific Publishers 2004.
3. Dionex: Product manual ProSwift ConA-1S, 065346-02, 2010.

4-P13

THE USE OF ENZYMES CONTAINED IN THE WASHING POWDERS TO THE PREPARATION OF THE CRUDE LYZATES OF *LACTOBACILLUS* CELLS FOR MAGNETIC DNA ISOLATION

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The preparation of crude lysates of bacterial cells is relatively expensive procedure because of enzymes used during the procedure. This problem can be solved using the washing powders for the cell lysis.

The aim of this work was the isolation of DNA from the *Lactobacillus* at quality for the PCRs and to test whether the DNA isolated by the magnetic microsperes (P(HEMA-co-GMA) is appropriate to run the PCR. We tried to find out if the composition of the washing powders is suitable for disruption of the cell wall instead of lysosyme. The lysis of the *Lactobacillus paracasei subsp. paracasei* CCDM 212/106 cells was performed with lysis buffer containing five types of washing powders. Two exposure times (1 and 3 h) were tested. At first, 1 ml of the cell suspension was centrifugated at 15 000 g and washed. After second centrifugation (10 000 g) the pellet was resuspended in 500 µl of lysis buffer containig washing powder solution or lysozyme solution. Lysis buffer containing lysozyme (3 mg ml^{-1}) was used as a control with the addition of SDS and proteinase K during the crude cell lysates preparation. We optimalised the cell lysis of the *Lactobacillus* cells. At the begining we tested washing powder solutions (powder 1) of different concentrations (1, 2, 3 and 4 %) and we prepared the crude cell lysates. DNA isolation was performed using magnetic microspheres[1]. DNA was adsorbed on the magnetic microsperes surface and than it was eluted to the TE buffer. The quality of eluted DNA and the presence of target DNA were verified by PCR using LBLMA 1 and R16 primers specific to the *Lactobacillus* genus[2]. The next aim of our work was to prove the applicability of different washing powders. We tested five types of washing powders and all of them were suitable for cell lysis and subsequent DNA isolation using magnetic particles.

Developed method is very usefull for the laboratory practice or for regular research. It is very simple, cheap and friendly to use method. Enzymes and detergents presented in the powders are very good agents for the cell wall lysis.

REFERENCES

1. Rittich B., Španová A., Šálek P., Němcová P., Trachtová Š., Horák D.: J. Magn. Magn. Mater. *321*, 1667 (2009).
2. Dubernet S., Desmasures N., Guéguen M.: FEMS Microbiol. Lett. *214*, 271 (2002).

4-P14

OPTIMALIZATION AND VALIDATION OF SPME-GC-FID METHOD FOR DETERMINATION OF SUSPECTED ALLERGENS IN SELECTED COSMETICS AND FOOD SAMPLES

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Products containing scent are a part of daily life. The majority of cosmetics, toiletries, laundry products etc. contain fragrance. However, the Scientific Committee on Cosmetic Products and Non-Food Products (SCCNFP) has selected 26 fragrance compounds as likely to cause allergies[1]. The European Cosmetic Directive requires that any cosmetic product containing any of 26 fragrances suspected allergens above certain trigger levels must impart these ingredients on the label. Labeling is required when the level of the individual ingredient exceeds 10 mg kg^{-1} in a product intended to leave on the skin or 100 mg kg^{-1} in a product to be rinsed off of the skin. Even though some of these fragrance compounds are present in food, there is no similar directive related to these allergens in food.

A method by solid-phase microextraction (SPME) and gas chromatography with flame ionization detection (GC-FID)[2] was applied for fragrance allergens identification, extraction and measurement. The procedure was based on headspace sampling (HS-SPME) using carboxen/polydimethylsiloxane fiber. The optimal extraction conditions of SPME-GC method were investigated including equilibrium time, extraction time, extraction temperature, desorption time and injector temperature to increase extraction efficiency. In addition to that SPME-GC-FID method was validated and following performance parameters were determined: repeatability, linearity, limits of detection (LOD), limits of quantification (LOQ). The future work will be focused on an application of validated method to the determination of allergens in real food samples (e.g. strewn tea and tea in bag) and cosmetics (e.g. cosmetics for children, natural cosmetics).

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REFERENCES

1. SCCNFP/0690/03, Notes of Guidance for Testing of Cosmetic Ingredients and Their Safety Evaluation, 6th revision, SCCP 10th Plenary Meeting of 19 December 2006.
2. Kataoka H., Lord H. L., Pawliszyn J.: J. Chrom. A *35*, 880 (2000).

4-P15
USE OF DESCRIPTIVE SENSORY METHODS FOR EVALUATION OF VARIOUS TYPES OF FOODS

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Descriptive sensory analysis (DA) is highly sophisticated technique that is used to obtain complete sensory description of a food product. It involves discrimination of both the qualitative and quantitative traits of a sample; the qualitative aspects of a product include aroma, appearance, flavour, texture, aftertaste and sound properties[1].

The training phase of DA techniques usually begins with the development of a common language which comprehensively describes the product attributes[2,3]. Generally, a new panel will develop the sensory language themselves, however, input from an experienced panel leader can assist the learning process. The final descriptive language should be precisely defined and contain enough terms to include all attributes likely to be encountered, but should not be so large as to be cumbersome in use[1]. Once terms are selected, the panel is trained to use them to illustrate/define the product attributes and their intensity. This is generally achieved by exposing the panel to the range of products in the category under test. Many authors also recommend the use of reference standards[4]. All DA methods require a panel with some degree of training.

DA may be used as a research guideline tool, or as a method of quality assurance or quality control. There are several different methods of DA, including the Flavour Profile Method, Texture Profile Method, Quantitative Descriptive Analysis, the Spectrum method, Quantitative Flavour Profiling, Free-choice Profiling and generic descriptive analysis. The specific methods reflect various sensory approaches[1].

The aim of this work was to apply several DA methods for evaluation of various types of foods. The special sensory lexicons of terms and forms for evaluation of chocolate, pastry, fruits, meat products, cheeses, yoghurts and other food products were developed. The chosen foods available in Czech market were then evaluated and compared. Students of our food department were used as assessors. Recent surveys suggest that the use of DA methods has increased rapidly[5,6] and will continue to do. Acquired knowledge allows product optimisation and is highly desirable within the food industry[1].

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REFERENCES

1. Murray J. M., Delahunty C. M., Baxter I. A.: Food Res. Int. *34*, 461 (2001).
2. Barcenas P., Perez Elortondo F. J., Salmeron J., Albisu M.: J. Sens. Stud. *14*, 161 (1999).
3. Johnsen P. B., Civille G. V., Vercellotti J.R., Sanders T.H., Dus C. A.: J. Sens. Stud. *3*, 9 (1998).
4. Murray J. M., Delahunty C.M.: J. Sens. Stud. *15*, 179 (2000).
5. Cristovam E., Paterson A., Piggott, J. R.: Eur. Food Res. Technol. *211*, 65 (2000).
6. Delahunty C.M., McCord A., O'Neill E., Morrissey P. A.: Food Qual. Prefer. *8*, 381 (1997).

4-P16
TRACKING CHANGES IN THE GENOME OF CAROTENOGENIC YEASTS ANALYZED BY TWO DIFFERENT GEL ELECTROPHORESIS TECHNIQUES – PFGE AND DGGE

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Survival of all living organisms critically depends on their ability to sense and quickly adapt to changes in the environment. One aspect of this adaptation is the reorganisation of genomic expression to the program required for growth. The changes in genome induced by stress conditions have been studied in six strains of carotenogenic yeasts and some comparative yeast strains. Moreover, influence of random mutagenesis induced by ethyl methylsulfonate was tested as well. In this work two molecular gel electrophoretic techniques were used for yeast DNA analysis: pulsed-field gel electrophoresis (PFGE) and denaturing gradient gel electrophoresis (DGGE)[1-3].

Chromosomal DNA was separated in agarose plugs using PFGE with contour - clamped homogenous electric field. Plugs were subjected to enzymatic lysis, and incubated with proteinase K in NDS buffer. The agarose gel was than run at 6 V/cm using pulse times of 150 - 500 seconds for 85 hours at 10 °C in 0,5x TBE buffer with Yeast chromosome PFG marker as a standard. In case of PFGE, more than ten bands of yeasts chromosomes were observed.

For DGGE technique first yeasts DNA isolation was done using the Microbial ultra clean isolation kit followed by nested PCR (primers NL1 with NL2 and NL4 with NS2). The small (200-700 bp) genomic restriction fragments were run on a low to denaturation gradient acrylamide gel; at 60°C, 120V for 4 hours and about 4 similar bands in all red yeast strains were observed.

DGGE can be used for group identification of red yeasts and other yeast and fungal species in mixed natural samples and in special environments, while PFGE karyotypes exhibited characteristic patterns and enabled relatively detailed yeast identification.

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REFERENCES
1. Libkind D., Gadanho M., van Broock M., Sampaio J. P.: J. Basic. Microbiol. *48*, 93 (2008).
2. Walker G. M., in the book: *Yeast Physiology and Biotechnology*, chapt. 6, p. 362. John Willey, West Sussex 1998.
3. S. Hohmann S., Mager W. H., in the book: *Yeast Stress Response*, chapt. 6, p. 389. Springer-Verlag, Berlin 2003.

4-P17

DETERMINATION OF IRON (II), IRON (III) AND TOTAL IRON IN SOME β-THALASSEMIA PATIENTS USING DIFFERENT ANALYTICAL TECHNIQUES

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Thalassemia is an inherited autosomal recessive blood disease. The disease is particularly prevalent among Mediterranean people. Kurdistan people are also suffered from this disease. The most common heterogeneous group of genetic disorders, which the production of normal hemoglobin (Hb) is partly or completely suppressed of defective synthesis of one or more globin chain[1]. There are many well-known analytical methods for determination of iron (II) and iron (III)[2]. Among these methods: gravimetric, titrimetric, potentiometric, conductometric, batch and flow injection spectrophotometric methods. In the present study, two batch spectrophotometric, atomic absorption spectrometric and biolabo kit methods have been used for determination of iron (II), iron (III) and total iron. The present methods have the advantages of high sensitivity, low cost reagent, low operation cost, simplicity, speed and their applications for determination of iron (II) and iron (III) in some serum samples of normal human and β-thallasemia patients in Erbil city.

For the first time especially in Erbil city attempts were made to use zero, first and second derivative spectra to identify the serum samples of some β-thallasemia patients from the normal human serum samples due to the appearance and resolution of peaks in both cases.

REFERENCES
1. Mansi K. M., Aburjai T. A.: Int. J. Hematol. Oncol. *18*, 94 (2008).
2. Hosseinimehr S. J., Bagheri M., Gholipoor M., Mokarami H.: J. Biol. Sci. *7*, 451 (2007).

4-P18

ALTERNATIVE PROCEDURE FOR PREPARITON OF *LACTOBACILLUS* CELL LYSATES

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The *Lactobacillus* species play an important role in the food industry.They are also very important for human health due to their positive influence of the gastrointestinal tract functions. Identification of the *Lactobacillus* species is based on the DNA amplification methods. Molecular methods are essential for correct identification of *Lactobacillus* at the genus or species levels. Polymerase chain reaction (PCR) is common amplification method used in biological laboratories. PCR-ready DNA must be available to amplification. Enzymes and detergents are used for the crude cell lysates preparation. Enzymes disrupt the cell wall and cell proteins and detergents destroy the plasmatic membrane of the cells. DNA is relased into medium and it is available for purification procedure. Lysozyme, sodium dodecyl sulphate (SDS) and proteinase K are the standard components in the bacterial crude lysates preparations. They are relativelly expensive and sophisticated to storage. The aim of this work was find out if enzymes and detergents presented in the washing powders are capable of cell lysis at adequate quality. Bacterial cells of *Lactobacillus* species were used for cell lysis experiments. They were grown in MRS medium to the absorbace A_{600nm} 3,0. Four types of lysis solutions for the crude lysates preparation were tested: lysis buffer (10 mM, Tris-HCl, pH 7,8 , 5 mM EDTA, pH 8,3 mg/ml lysozyme), SDS (0,5 %) and proteinase K (0,2 μg μl^{-1}) - control, 4% solution of washing powder, 4% solution of wasching powder and SDS, 4% solution of washing powder, SDS and proteinase K. Differnt times of treatment were also tested. DNA was isolated from cell lysates using the magnetic microsperes (P(HEMA-co-GMA) covered by carboxyl groups In the presence of 16 % PEG 6000 and 2 M NaCl[1.] Isolated DNA was eluted into TE buffer and tested in PCR with primers specific to genus *Lactobacillus*[2]. The PCR products were detected by agarose electrophoresis. It was proved that the washing powders contain enzymes and detergents which can be use to disrupt cell wall and plasmatic membrane.

REFERENCES
1. Ritrich B., Španová A., Šálek P., Němcová P., Trachytová Š., Horák D.: J. Magn. Magn. Mater. *321*, 1667 (2009).
2. Dubernet S., Desmasures N., Guéguen M.: FEMS Microbiol. Lett. *214*, 271 (2002).

4-P19

ANALYSIS OF PHYSICO CHEMICAL PROPERTIES OF COLLAGEN ISOLATED FROM DIFFERENT ANIMAL TISSUES

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Traditional sources of collagen such as pork and bovine have limitations because of religious issues in first and potential BSE risk in second case. Possible use of carp collagen also presents health hazard as it is important allergen source. Chicken skin can be valuable alternative. Collagen isolated from chicken skin was analyzed and compared to collagen from bovine tendon, and collagen from carp, poultry and livestock animals skins.

Collagen was isolated by modified extraction procedure. TNBSA method was used for free amino group analysis. Stability and molecular properties of collagen was analyzed by PAGE-SDS. Thermal and structural stability of collagen isolates was tested using ultrasonic spectroscopy (HRUS 102 Ultrasonic Scientific). Viscosity measurements were performed by rotational rheometer (Model AR-G2 TA Instruments). Elemental trace analysis was done by ICP-MS. Amino acid composition was analyzed in hydrolyzed samples using AAA.

After collagen was extracted, chicken skin proved to be vast source of collagen type I consisting of typical fractions α1 and α2 chains in ratio 2:1. Predicted yield was 25% and water content was around 67%. The proportional lysine content was 7,07%, proline 5,88% and glycine 10,07%. Denaturation temperature was determined at approx. 50 °C for chicken, 60 °C for hen and 78 °C for mature hen collagen. Heating induces the denaturation of native proteins. These denatured proteins tend to aggregate and if aggregation proceeds long enough, a gel is formed. Pre-aggregation occurs at lower temperatures and is indicated by signal decrease in temperature interval from 20 °C to 27 °C, which is proof of disaggregation and liquefaction of samples. From 40°C upwards second phase occurs as signal constantly increases while liquefaction process finishes and aggregation continues. When compared with electrophoresis of heat pretreated samples, obtained results confirmed that bovine collagen denaturation starts at 40 °C and finalizes at 70 °C. According to results in chicken samples actual denaturation is not visible till 50 °C, and ends at almost 80 °C which explains chicken collagen thermal stability. These data were confirmed by viscosimetry too.

Obtained results suggests that waste chicken skin has potential to be an excellent alternative source of raw collagen, which could find it's application not only in food industry but, after further research and processing, as a component of various collagen based biomaterials in biomedicine.

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4-P20

FAST METABOLOMIC APPROACH FOR ALZHEIMER DISEASE DIAGNOSIS BASED ON HIGH RESOLUTION MASS SPECTROMETRY

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Alzheimer's disease (AD) is the most common neurodegenerative disorder, described by broad cognitive decline (involving memory, reason and orientation). However, its ethiology is not completely known due to the complexity of this disease. Currently, the criteria for diagnosis of AD require the presence of significative cognitive deficit when the brain damage is already widespread and it is usually too late to receive effective drug treatment. Thus, the discovery of biomarkers that allow early diagnosis of this disease is of great importance[1]. Have been studied several biological markers in cerebrospinal fluid (CSF), in close proximity to the pathology. However, the ideal sample is the blood serum which is available through non-invasive techniques. In the discovery of biomarkers can be remarked the importance of the omics, techniques of massive information. The most incipient omic science is metabolomics, wich study the enteriry of low molecular weight compounds involved in cellular processes. This approach is of great interest, since metabolomic analysis involve factors both genetic and external (food, exercise), giving us a real view of the pathology.

In this field, the application of high resolution mass spectrometry techniques (hybrid systems such as QqQ-TOF, triple quadrupole – time of flight) let us to obtain wide metabolomic profiles. Moreover, the high resolution allows the analysis by direct infusion of the sample, without chromatographic separation, giving us a very fast methodology. In this way, blood serum samples of Alzheimer patients and healthy controls were analyzed by direct infusion ESI-QqQ-TOF-MS, obtaining characteristic metabolic profiles, that allows establish differences between the diseased and control groups. More important signals were studied and identified as potential biomarkers of Alzheimer disease: glycerophospholipids[2], ceramides[3], aminoacids.

REFERENCES

1. Schneider P., Hampel H., Buerger K.: CNS Neurosci. Ther. *15*, 358 (2009).
2. Mielke M., Lyketsos C.: Int. Rev. Psychiatry *18*, 173 (2006).
3. Mielke M., Lyketsos C.: Neuronil. Med *12*, 331 (2010).

4-P21

USE OF THE MICROALGAE CHLORELLA SOROKINIANA FOR THE BIOTECHNOLOGICAL PRODUCTION OF IODINE AND SELENIUM FUNTIONAL FOOD

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The need for food supplemented with essential elements has becoming an important issue to assure the health of people, and microalgae, such as *Chlorella sorokiniana*, exhibit a high potential in the fast production of biomass with high level of bioactive molecules that can be use for this purpose. *Chlorella* has been studied by Burianova et al[1] to be used as iodine supplement, but selenium is another essential very related to iodine in some important diseases such as thyroid disorders, that need to be also incioporated to these foods.

An important question is the chemical forms (metabolites) in which the heteroelements are transformed in the biological systems, which determine their potenrtial health effects (metabolomics). Therefore, ICP-MS coupled to multidimensional chromatographic techniques and assisted by tandem mass spectrometry (metallomics) represents a powerful tool for this purpose.

Chlorella has been is exposed to Se in the form of selenate which is rapidly absorbed in few minutes. After 24-48 hours, about 40% of the total Se intake was as organic-Se species inside the cell, maily in the form of selenomethionine[2]. The study of cellular response to Se exposure showed that Chlorella[3] tolerates sodium selenite in the culture up to a concentration of 100 mg l^{-1}. Exposure to iodine showed that both dose and chemical forms of this element are critical factors in the cellular response. Experiments with potassium iodide at different concentrations showed that Chlorella cells can tolerate up to a concentration of 3000 mg l^{-1}.

The characterization of iodine species in the microalgae was performed using the coupling of multidimensional chromatography with inductively coupled plasma-mass spectrometry (ICP-MS) deteccion. Further studies have to consider the purification of the ICM-MS fractions for their identification by tandem MS.

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REFERENCES

1. Burianová J., Machát E., Niedobová, Doucha. J, Kanický V.: Chem. Listy *273*, 99, (2005).
2. Fan T. W. M., Lane A. N., Martens D., Higashi R. M.: Analyst. *875*, 123, (1998).
3. Pepan D., Cohen E.: Plant Growth Regul. *225*, 45, (2005).

4-P22

CAROTENOIDS-ENRICHED BIOMASS PRODUCTION BY RED YEASTS CULTIVATED ON VARIOUS WASTE SUBSTRATES HYDROLYZED BY MICROBIAL HYDROLASES

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All types of cells are characterized by the ability to respond to changes in their environment. Yeast species *Rhodotorula*, *Cystofilobasidium* and others might be commercially used in food and feed industry to natural pigment synthesis, but the limiting factor is the high cost of production. This can be achieved in the use of waste materials and industrial by-products as a source of nutrients[1-3].

In presented comparative study production properties of four red yeast strains (*Cystofilobasidium capitatum, Rhodotorula sp.*) were tested. Production of carotenoids and biomass using various waste substrates and industrial by-products as nutrient sources was studied. The substrates were also subjected to enzymatic hydrolysis with commercial microbial enzymes, and complex enzyme mixtures isolated from selected fungi in our laboratory. Pigments were analyzed by HPLC/PDA/MS/ESI, enzyme activities were measured spectrophotometrically. Changes of carbohydrate composition in processed waste substrates were analyzed by HPLC/RI.

All tested *Rhodotorula* strains exhibited similar biomass and pigments production properties than *Cystofilobasidium capitatum*. Use of some of tested hydrolyzed substrates (pasta, apple fiber, wheat bran, rice) led to an induction of production of targeted metabolites and biomass, while some of them exhibited inhibitory effect. The best production properties showed strain *R. mucilaginosa* grown on hydrolyzed pasta.

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REFERENCES:

1. Rosa C. A., Péter G., in the book: *Biodiversity and Ecophysiology of Yeasts (The Yeasts Handbook)*, chapt. 9, p. 179. Springer, Berlin 2006.
2. Frengova G. I., Beshkova D. M.: J. Ind. Microbiol. Biotechnol. *36*, 163 (2009).
3. Bhosale P., Gadre R. V.: J. Ind. Microbiol. Biotechnol. *26*, 327 (2001).

4-P23

FERMENTATIVE HYDROGEN PRODUCTION BY BACTERIAL STRAIN *CLOSTRIDIUM TYROBUTYRICUM* S5.

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Presence of clostridium in milk is a technological problem for cheese making as they cause late-blowing defect. This damage is characterised by gas formation. The ability to produce gas by some species of genus *Clostridium* could possibly find an industrial use. Research has been focused on *Clostridium* strains deposited at MILCOM – Dairy Research Institute (Tábor, Czech Republic). It was found out that good candidate for hydrogen production is *Clostridium tyrobutyricum* S5 strain from this collection. From this reasone its properties were evaluated and the cultivation of *Clostridium tyrobutyricum* S5 under different conditions was studied.

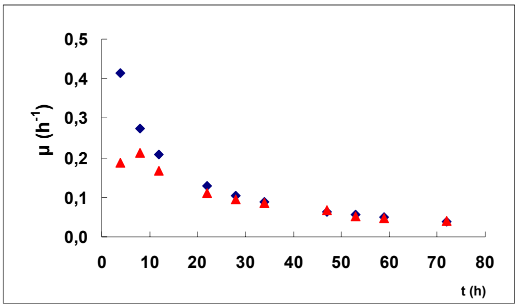

Figure 1. Effect of temperature 37 °C (♦) and laboratory temperature (20-23 °C) (▲) on the maximal specific growth rate (μ) of *Clostridium tyrobutyricum* S5 in RCM with optimal pH value 6,5 and lactose as substrate

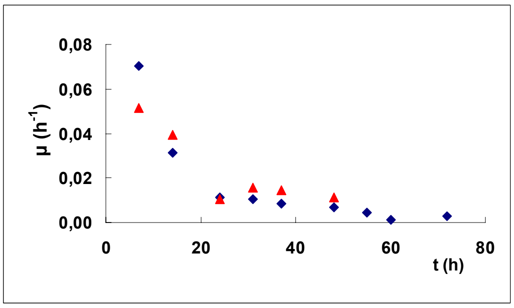

Figure 2. Effect of temperature 37 °C (♦) and laboratory temperature (20-23 °C) (▲) on the maximal specific growth rate (μ) of *Clostridium tyrobutyricum* S5 in RCM with cheese whey as substrate

The cultivation was carried out in liquid Reinforced Clostridial Medium (RCM) with lactose (5 %) and cheese whey with lactose content 5 % instead of glucose as substrate under anaerobe conditions. Growth of bacterial cells was observed at laboratory temperature (from 20 to 23 °C) and at 37 °C, pH values range from 4,0 to 8,0 with 0,5 unit. Optimal pH was evaluated at 6,5. Optimal pH and different temperatures (20-23 °C/37 °C) were thereafter used in last experiment (see Fig. 1 and Fig. 2.). The content of lactose and volatile fatty acids was also determined in supernatants.

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4-P24

IMPACT OF ELEVATED CO$_2$ CONCENTRATION ON KEY ENZYME OF PHOTOSYNTHESIS – RUBISCO

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We studied seasonal changes of Rubisco content and activity in seven-year-old seedlings of *Fagus sylvatica* L. exposed for three growing seasons to ambient (A = 385 μmol mol^{-1}) and elevated (E = 700 μmol mol^{-1}) CO$_2$ concentrations. The highest content and activity of Rubisco enzyme was measured immediately after full leaf expansion and then gradually decreased during the growing season. Rubisco content in E was reduced relative to A after full leaf expansion (down-regulation), whereas Rubisco activity in E was stimu-lated relative to A at the same time. This effect is very pronounced at specific activity of Rubisco. Because no signi-ficant differences were found in Rubisco activation state between A and E, we assume that stimulation of Rubisco activity in E was not a consequence of higher carbamylation but was caused by release of inhibitors from active Rubisco sites under the influence of elevated CO$_2$.

This work is part of the research supported by grant 522/06/0930 (GA ČR).

4-P25

CHARACTERIZATION OF POLYGALACTURONASES PRODUCED BY *GEOTRICHUM CANDIDUM*

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The *Geotrichum candidum* CCY 16-1-29 (teleomorph *Galactomyces geotrichum*) was able to grow and produce high activities of polygalacturonase on pectin or grape pomace as a sole carbon source. The highest activities of extracellular enzymes were found in the third and in the seventh day of cultivation. After extraction and precipitation, polygalacturonases produced in these cultivation periods were characterized.

The production of multiple forms of polygalacturonase was observed in both cultivation periods. Two major forms, polygalacturonase with random action pattern (endoPGase, EC 3.2.1.15) and oligogalacturonate hydrolase (exoPGase, exopolygalacturonase preferring oligogalacturonides as substrates) were detected as well as numerous minor forms (IEF-PAGE with print technique detection). EndoPGase was identified by mass spectrometry, too.

The major forms have similar isoelectric points (below pH 6,0) and pH optima (4,6 and 4,8). pH Optimum 4,6 was associated with exoPGase and the second one with endoPGase. Both enzymes were stable after freeze-drying and storage at 4 °C. EndoPGase had molecular weight determined by gel fitration about 29 kDa (36 kDa by SDS-PAGE), optimum of temperature about 45 °C and was stable only to 35 °C. ExoPGase had molecular weight about 50 kDa, optimum of temperature about 60 °C and was stable to the same temperature. Optimal substrate for exoPGase was pentamer, for endoPGase pectate. K_m for optimal substrate of exoPGase reached the value of 11,4 . 10^{-5} mol l^{-1} and of endoPGase 6,6. 10^{-5} mol l^{-1}.

This work was partially supported by grants No. 2/0011/09 and 2/0005/10 from the Grant Agency for Science VEGA (Slovakia), by financial support from Ministry of Education, Youth and Sports of Czech Republic under project MSM 021630501 and by ITMS 26220120054 supported by the Research & Development Operational Programme funded by the ERDF (25%). The financial support of the Institutional Research Plan AVOZ40310501 of the Instute of Analytical Chemistry, v.v.i. ASCR is greatfully acknowledged, too

4-P26

FATTY ACIDS DETERMINATION IN BIOLOGICAL SAMPLES BY GC-MS

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A gas chromatography-mass spectrometric (GC/MS) technique was used for qualitative and quantitative analysis of fatty acids in meat and fish. The aim of the paper was to develop an analytical method for the determination of fatty acids in biological samples. The method involves extraction procedure, derivatization and gas chromatography/mass spectrometric analysis (GC/MS). The extraction of the fatty acids was performed by mixing plasma and chloroform: methanol (2:1) during 30 seconds, at room temperature[1]. Fatty acids from plasma and meat were derivatized as methyl ester derivatives[1,2]. Finally, the extracted analytes were detected by GC/MS in the electron impact (EI) mode. A Trace DSQ ThermoFinnigan quadrupole mass spectrometer coupled with a Trace GC was used. The identification of fatty acids was obtained by comparison of fatty acids methyl esters (FAME) mass spectra with the mass spectra of FAME standards of NIST library[2].The method was validated by using fatty acid standard samples. Figure 1 presents the important fatty acids identified in fish plasma.

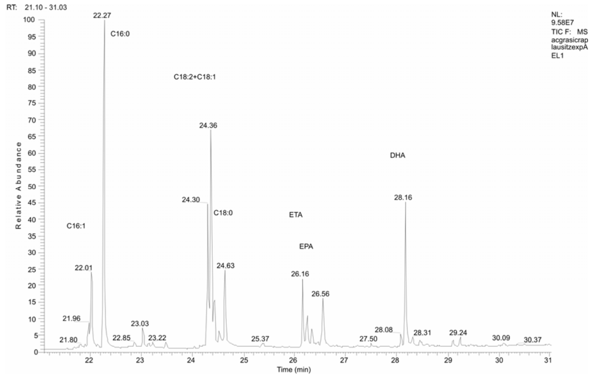

Figure 1. Fatty acids separation chromatogram in fish plasma

Eicosapentaenoic acid (EPA) and docosapentaenoic acid (DHA) are the major fatty acids found in fish. These fatty acids are produced by unicellular algae and phytoplankton which are consumed and then accumulate in fish[3].

The method developed showed good precision in the analysis of fatty acids from different biological media. The high values of (n-3) highly unsaturated fatty acids (HUFA) in fish plasma prove the high nutritious value of fish.

REFERENCES

1. Freije A.: J. Oleo Sci., *58*, 379 (2009).
2. Shapira N., Weill P., Sharon O., Loewenbach R., Berzak O.: J. Agric. Food Chem., *57*, 2249 (2009).
3. Kołodziejczyk M.: Rocz. Panstw. Zakl. Hig. *58*, 287 (2007).

4-P27

MD SIMULATION ON MUTANT NEURAMINIDASE OF INFLUENZA B VIRUS AND ITS INHIBITOR

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Not only influenza A virus is associated with annual outbreak of seasonal flu, influenza B virus is also a cause of influenza epidemic. There are only 2 available inhibitors of influenza B virus, zanamivir (ZNV) and oseltamivir (OTV), these drugs block the neuraminidase (NA) and thus inhibit the release of new viruses[1]. Although, OTV is more suitable than ZNV but unfortunately influenza B virus strains had become resistant to OTV. The single mutations on NA of influenza B virus were detected, E117G[2] and R150K[3]. Therefore, this work provides the information of OTV resistant toward NA of influenza B virus by using molecular dynamics simulations. The results show that both of wild-type and mutant systems reach the equilibrium in 6 to 10 ns, thus, the last 4 ns of simulations were performed to analyze drug-target interactions. The resistance of OTV in mutant systems due to the loss of hydrogen bonding between functional group of OTV and NA active site which satisfy the binding free energy.

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REFERENCES

1. Ferraris O., Lina B.: J. Clin. Virol. *41*, 13 (2008).
2. Jackson D., Barclay W., Zurcher T.: J. Antimicrob. Chemoth. *55*, 162 (2005).
3. Mishin V. P., Hayden F. G., Gubareva L. V.: Antimicrob. Agents. Ch. *49*, 4515 (2005).

4-P28

DETERMINATION OF PHENOLIC GLYCOSIDES AND AGLYCONS IN SELECTED KINDS OF RICE

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Cereals are very important material for mankind. They are applied to main part of human nutrition, they are used as feed for farm animals and small amount is used to technical applications (starch, ethanol).

A grain is the most important part of cereal. Cereal grains are milling on flour. A lot of various bakery products are made from this flour. Rice (*Oryzae sativa*) is the most common cereal for direct consumption. It is formed from complex saccharides, further it contents high amount of fibre, contrary low amount of fat, and it doesn´t content cholesterol, sodium or gluten.

Rice also contains many types of phenolic compounds, which are attributed to the significant antioxidant properties. Phenolic compound can be free or bound as the aglykon part to glycosides. The aim of this work was to analyse content and composition of phenolic compounds in selected kinds of rice.

Individual mono- and disaccharides were determined by RP-HPLC with refractometric detection, acetonitrile:water mixture was used as mobile phase. Individual phenolics were analyzed by HPLC/UV-VIS and HPLC/PDA. Separation was done in isocratic mode using methanol:water as mobile phase for catechin elution and acetonitril:methanol:phosphoric acid for flavonoid separation. Quantitative analysis was performed using external calibration.

Antioxidant activity of most rice samples was about 20 mg/100 g except Natural Red (78 mg/100 g). Total phenolics ranged 5 – 15 mg/100 g, while total flavonoids ranged 2 – 6 mg/100 g (Narutal Red exhibited 23 mg/100g and 13,5 mg g^{-1}, respectively). Non-processed rice types (e.g. Natural Rice, Indian Wild Rice etc.) contained substantially higher amount of biologically active compounds than peeled rice. High amount of individual phenolic compounds was measured in Natural Red and Indian Wild Rice, The main phenolic compounds identitified in hydrolyzed rice samples as aglycons were ferulic acid, catechins and chlorogenic acid.

Because of the relatively high content of biologically active compounds and worldwide consumption it can be concluded that rice (predominantly natural) belongs to important sources of complex phenolics recommended for modern human nutrition.

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4-P29

DETERMINATION OF ACTIVE COMPOUNDS IN INSTANT BABY TEAS

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According to recent knowledge, as the best way of nutrition for infants to 6 months of age is considered breast milk. If the children can´t be breastfed, replacements feeding dairy products are available in the market. In this case, however, infants need extra liquid, because different viscosity and density of alternative food and breast milk can evoke thirst even full saturation. Infant and toddler drinks must meet strict hygiene requirements, but content of biological active compound and other substances is regulated partially or poorly.

The most common products are granulated instant drinks and fruit and herbal teas. All these products contain significant amount of active compounds, e.g. polyphenolic compounds, flavonoids, anthocyanins, vitamins etc. This work was focused on determination of active vitamin, provitamin and phenolic compounds, saccharides, pH and other parameters.

Reducing and total saccharides were analysed by UV-VIS spectophotometric methods. Total phenolic and flavonoid content was measured spectophotometrically too. Individual mono- and disaccharides were analysed by RP-HPLC/RI acetonitrile and water mixture as a mobile phase.

Individual catechins and flavonoids were analysed by RP-HPLC/UV-VIS using methanol:water as mobile phase for catechin elution and acetonitril:methanol:water:phosphoric acid for flavonoid separation. Both separations were done in isocratic mode. Individual phenolics were identified by HPLC/PDA/ESI-MS. Quantitative analysis was performed using external calibration.

Herbal teas and fruit teas contain substantially higher amount of analyzed biologically active compounds when compared with instant beverages. The content of total phenolics was about 6 times higher and total flavonoids about 4 times higher in herbal teas. This finding is also in agreement to the antioxidant capacity, which was significantly higher in herbal teas than in instant beverages.

Opositely, granular drinks contains very high simple saccharide concentration, which, in combination with acidic pH, it can cause milk teeth decay. The content and number of measured saccharides correspond to declared values.

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4-P30
INTERCONTINENTAL DISCRIMINATION OF PURE ORIGIN ROASTED COFFEES BASED ON THEIR METAL CONTENT

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Two species of genus *Coffea* have acquired worldwide economic importance: *Coffea Arabica* (approximately 90% of the world coffee production), and *Coffea Canephora* (approximately 9% of production), usually known as *Arabica* and *Robusta* varieties, respectively[1,2]. Coffee beans used in the blends for commercial coffee industry come from a wide range of geographical areas and may have varied chemical and organoleptic properties. The producers must select among the different materials to produce the best quality brand because there is a great interest in the commercial qualification of coffee[2]. Analytical methods should assess the nutritional value, sensorial properties, safety and authenticity in cases of fraudulent or accidental mislabelling[3].

In this study, the nutritionally relevant mineral elements (Ca, Mg, Na, K, Fe, Mn and P) of pure origin espresso coffees representing the main coffee producers: Africa (Kenya, Mussulo and Ethiopia); South America (Colombia and Brazil); Central America (Honduras, Cuba, Mexico and Guatemala); Asia (China, India and Timor) and Oceania (New Guinea) were analyzed by high-resolution continuum source flame atomic absorption spectrometry (except P that was determined by a standard spectrometric method). The obtained mineral concentrations were correlated with the geographic area of production applying pattern recognition techniques such as principal component analysis and cluster analysis. Samples from Oceania, Central and South America formed clusters clearly separated due to their significantly different chemical compositions. Samples from Africa and Asia regions presented similar patterns of distribution but both were well separated from the other groups. The results indicated that the mineral contents of Ca, Mg, Na, K, Fe, Mn and P may be used to characterize, differentiate and identify the geographic origin of pure origin roasted coffees.

REFERENCES

1. Martín M., Pablos F., González A.: Anal. Chim. Acta *358*, 177 (1998).
2. Martín M., Pablos F., González A.: Food Chem. *66*, 365 (1999).
3. Grembecka M., Malinowska E., Szefer P.: Sci. Total Environ. *383*, 59 (2007).

4-P31
EVALUATION OF HEAVY METAL CONCENTRATIONS IN ARMS OF *OCTOPUS VULGARIS*

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The commercial significance of cephalopods to world fisheries is of relatively recent, but growing importance[1]. Like several other cephalopods species the common octopus, *Octopus vulgaris*, is part of the traditional diet of coastal communities in southern Europe. In Portugal, octopus has a high economic, social and cultural value.

Cephalopods are an excellent source of some essential elements, however given the morphological and biological characteristics associated to their habitat, some contaminant metals may accumulate in their issues[2]. Toxic metals can be bioaccumulated and retained in cephalopods and consequently passed on the predators, thus potentially increasing the contaminant load in higher trophic levels, including humans[1]. Therefore contaminant levels in cephalopods are also of direct concern to public health.

To evaluate the safety concerning the levels of cadmium, lead, arsenic and chromium in muscle tissues of *Octopus vulgaris*, fresh samples were purchased at local Oporto retail establishments (NW Portugal). Microwave-assisted digestion was performed and a high resolution continuum source graphite furnace atomic absorption spectrometry procedure was optimized for sensitive and accurate quantification of the metallic elements. The detected concentrations of cadmium (except in one sample) and lead in muscle tissues are generally below the maximum levels recommended by the European Commision Regulation (1,0 mg kg^{-1} ww for Cd and Pb). Concerning Cr and As mean values, no comparison can be made with EC Regulation since it does not provide a legal limit for these elements. Estimation of the non-carcinogenic and carcinogenic health risks by the target hazard quotient and target carcinogenic risk, established by the U.S. Environmental Protection Agency was also performed. From a public health perspective, this study also provides consumers a better knowledge of contamination problems associated to this species.

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REFERENCES

1. Pierce G., Stowasser G., Hastie L., Bustamante P.: Ecotoxicol. Environ. Saf. *70*, 422 (2008).

2. Lourenço H., Anacleto P., Afonso C., Ferraria V., Martins M., Carvalho M., Lino A., Nunes M: Food Chem. *113*, 1146 (2009).

4-P32

POLYANILINE MICROARRAY ELECTRODES FOR CADMIUM ANALYSIS

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In recent years, modern voltammetric techniques are gaining increasing importance in environmental control. Analytical voltammetry is being significantly influenced by using microelectrodes, since they exhibit several attractive and important properties such as small size, low background charging and high current density[1]. The current response of a microelectrode is directly proportional to the active area. One possibility of creating high active electrode area on a miniaturized sensor is by polymerization. Recently, electropolymerized polymer films have received considerable attention in the preparation of sensors. During the last years, aniline polymer has aroused much interest, because the conducting redox polymer film shows good tranducing properties and high stability and reproducibility, being widely used in the preparation of biosensors[2].

In the present work, a polyaniline based sensor was developed for cadmium determination in seafood sample digests. The screen printed carbon electrode (SPCE) surface was sprayed with a commercial deodorant at a distance of *ca.* 200 mm during 8 s and dried. The modified electrode was then electropolymerized with 0,1 mol dm^{-3} aniline solution in 0,5 mol dm^{-3} H_2SO_4. Scanning electron microscopy was used to evaluate the modified electrode surface. The electrochemical characteristics of the sensor were studied by cyclic and square-wave voltammetry. The optimised stripping procedure in which preconcentration of cadmium was achieved by deposi-ting at 1,20 V (*vs*. Ag/AgCl) produced a well-defined anodic peak at *ca.* -0,7 V at pH 4,6. Working range, detection and quantification limits of the polymerized microarrays were also determined and compared with those reached with the unmodified electrode. The polymerized microarray electrodes were successfully applied to quantify cadmium in seafood sample digests.

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REFERENCES

1. Huang X., O'Mahony A., Compton R.: Small *5*, 776 (2009).
2. Yin T., Wei W., Zeng J.: Anal. Bioanal. Chem. *386*, 2087 (2006).

4-P33

ANTIOXIDANT ACTIVITY OF BARLEY IN REGARD TO THE QUALITATIVE PARAMETERS OF MALT

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The aim of the study is to establish the total antioxidant activity of the grain samples and malt and subsequently evaluate the effect on the qualitative parameters of malt. The grain and malt of six spring barley (*Hordeum vulgare* L.) malting varieties treated by a zinc fertilizer during two growth stages (DC 31 and 55) were used as the source material. The extracts were obtained by mashing 25 g of homogenized grist with the supplement of 225 ml of distilled water at the temperature of 45 °C for the duration of 15 minutes. By the means of the TEAC method (Trolox Equivalent Antioxidant Capacity), the antioxidant activity was spectrophotometrically determined on the basis of the samples capacity to eliminate coloring caused by synthetic radicals DPPH (1,1-diphenyl-2-(2,4,6-trinitrophenyl) hydrazyl). The acquired results could be of a practical help in selection and preference for concrete malting barley varieties, and for malt and beer production.

This work was carried out with the support of the Research Centre for Study of Extract Compounds of Barley and Hop VC 1M0570.

4-P34

INFLUENCE OF DIFFERENT KINDS OF SPICES ON ACRYLAMIDE CONTENT IN BUCKWHEAT GINGER CAKES

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Acrylamide is considered as probably carcinogenic to humans[1]. Acrylamide has formed during thermal processing in carbohydrate-rich and protein-low plant foods at high temperatures and low moisture conditions associated with frying, baking, and roasting[2,3]. There are many reports how to reduce acrylamide formation in foods, including selecting foodstuffs, removing acrylamide precursors, adapting processing conditions, and adding food ingredients[4].

The aim of this study was to assess the impact of selected kinds of spices (clove, cinnamon, allspice, white pepper, anise, star anise, coriander, fennel, nutmeg, cardamom, vanilla, ginger and mix of spices) in buckwheat ginger cakes on acrylamide formation.

The results showed that acrylamide content was significantly reduced in ginger cakes with nutmeg, fennel, anise and clove addition. The minimal decrease of acrylamide content was found in ginger cakes with vanilla, cardamom, white pepper, ginger and mix of spices. In contrast, acrylamide content in the ginger cakes with cinnamon and coriander increased. No change of acrylamide content was observed in ginger cakes with star anise.

The final acrylamide content was probably influenced by chemical constituents with antioxidant capacity in particular kinds of spices. The effects of selected spices and their chemical constituents on the final acrylamide content will be studied consecutively.

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REFERENCES

1. Casado F. J., Sánchez A. H., Montaño A.: Food Chem. *119,* 161 (2010).
2. Tareke E., Rydberg P., Karlsson P., Törnqvist M., Eriksson S.: Chem. Res. Toxicol. *13*, 517 (2000).
3. Hidalgo F. J., Delgado R. M., Zamora R.: Food Chem. *122,* 596 (2010).
4. Ou S., Shi J., Huang C., Zhang G., Teng J., Jiang Y., Yang B.: J. Hazard. Mater. *182,* 863 (2010).

4-P35
DECLINE IN ACRYLAMIDE EXPOSURE BY ADOPTION OF MITIGATION TOOLS

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Acrylamide is known as a probably carcinogenic compound and its occurrence has been proven in many staple foods. Since the estimation of acrylamide exposure of high-school students revealed that their exposure was close to the tolerable level of daily acrylamide intake – 2,6 $\mu g\ kg^{-1}$ of body weight[1,2] – the effort to mitigate acrylamide is especially valuable.

Various tools for reduction of acrylamide level in particular foodstuffs summarized in The CIAA Acrylamide Toolbox (2009) are recommended[3]. The choice of the appropriate alternative depends on the expected effect of the specific intervention in food processing on the final quality of food product. Exerted efforts in food processing alterations should be balanced with an adequate impact on a decrease of acrylamide exposure.

In close co-operation with domestic producers of cereal based foodstuffs which belong to the highest contributors of acrylamide intake, several tools of acrylamide reduction were successfully tried: an application of asparaginase pretreatment in biscuits, pastry and pancakes production; a partial replacement of ammonium raising agent with sodium salt in gingerbread production; a support of leavening in bread making; an application of natural antioxidants present in spices in honey cake production. Based on our survey of consumption habits of Slovak and Czech high-school students the adoption of mentioned tools could be reflected in a decrease of acrylamide burden of this highly exposed group up to 30 %.

This contribution is the result of the project implementation "The Centre of Excellence for Contaminants and Microorganisms in Foods" supported by the Research & Development Operational Programme funded by the ERDF. This work was also supported by APVV under the contracts No. LPP 0310-09 and SK-PL-0051-09.

REFERENCES

1. Ciesarova Z.: AgroFood Ind. Hi-Tech, in press.
2. Tardiff R. G., Gargas M. L., Kirman C. R., Carson M. L., Sweeney L. M.: Food Chem. Toxicol. *48,* 658 (2010).
3. http://www.ciaa.be/documents/brochures/ac_toolbox_20090216.pdf [cit. 2011-28-04].

4-P36
ACRYLAMIDE FORMATION IN BREAD INFUENCED BY MODIFICATIONS IN COMPOSITION

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Acrylamide is a health hazardous compound that is formed in bread upon baking as a consequence of asparagine degradation in the presence of reducing sugars. Acrylamide was determined predominantly in bread crust in significant correlation with brown colour[1,2].

Presented study was focused on monitoring of acrylamide content in bread according to baking technology and recipe modifications in addition of different flour, bread improvers and concentration of dried yeast.

It was determined that potato flour addition in recipe significantly promoted acrylamide formation. On the other hand, the lowest acrylamide content was observed in bread with graham flour. Addition of calcium inorganic salts slightly lowered acrylamide content in bread up to 10 %. Processing conditions, baking temperature and yeast activity were proved to be the most significant factors of acrylamide formation in bread.

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REFERENCES
1. Marková L., Kukurová K., Ciesarová Z., Bednáriková, A., Šimko, P., Behan, T., Kravec, J.: Chem. Listy *104*, 660 (2010).
2. Mustafa A., Fink M., Kamal-Eldin A., Rosén J., Andersson R., Áman P.: Food Chem. *112*, 767 (2009).

4-P37

NANOENCAPSULATION OF BEER PHENOLICS

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Polyphenols constitute ubiquitous groups of plant metabolites with many biological activites. They form an integral part of both human and animal. Unfortunately, the concentrations of polyphenols that appear effective in vitro are often of an order of magnitude higher than the levels measured in vivo. Plant phenolics exhibited limited stability and conditioned solubility and some of them have unpleasant taste.

Benefit effect of beer is caused by many natural substances in sufficient quantity and ratio; the most important are beer phenolics originated mainly from hops and malt. Except positive health effect, phenolics complicate beer technology by turbidity formation.

In this work encapsulation of beer phenolics into nanoparticles was tested. Individual phenolics (quercetine, gallic acid and xanthohumol), malt and hop extract were encapsulated into liposome and nanoparticles. Two different liposome methods were tested: the thin film evaporation and sonication. The nanoprecipitation technique based on PLGA and PEG-5000 was used for gallic acid and xanthohumol entrapment. Total phenolics and antioxidant activity were measured spectrophotometrically. Individual phenolics were measured by HPLC/PDA/ESI-MS (Thermo Fisher Scientific, USA). Beer phenolics were separated on Ultra Aqueous C18 column using gradient elution with acetonitrile-methanol-water mixture. Stability of particles as well as encapsulated phenolic was tested at different pH and temperature.

Multilamellar vesicles were obtained by thin film evaporation, while small unilamellar vesicles were formed by sonication, which can be combined with extrusion technique. Sonication exhibited lower range of encapsulating efficiency than film evaporation (60-70% and 80%, respectively). Ethanolic solutions of phospholipids exhibit high encapsulation efficiency and the liposomes made in the presence of ethanol had a relatively small size (about 140 - 180 nm, DLS). Liposomes were relatively stable at pH ranging from 4,0 to 7,0.

The nanoencapsulation efficiency reached 92% with the particle diameter being about 90-100 nm (DLS). Stability of encapsulated phenolics increased for several times when compared with the pure actives, and possessed more effective antioxidant activities. Thus, studied techniques and encapsulated beer phenolics could be useful to functional beverages and foods.

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4-P38

POLYMORPHISM IN *LOXA* GENE AND PHENOTYPE CHARACTERISTICS IN SEVERAL VARIETIES OF CZECH SPRING BARLEY

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Lipoxygenases (LOX, linoleate:oxygen oxidoreductase, EC1.13.11.1 2) are non-heme iron- dioxygenases that catalyze hydroperoxidation of fatty acids. In plants, the hydroperoxide are further metabolized into lipid-breakdown products as 2-nonenal. In barley and malt, LOX directly influences final sensory quality of beer.

The objective of this work is to study polymorphism in LoxA gene in 21 parent varieties of Czech spring barley with different lipoxygenase activity - varieties used for Czech beer technology as well as some collection varieties with previously described lower LOX activity. DNA was extracted from barley leaves by DNeasy Plant Mini Kit (Quiagen). The DNA concentration and purity was analyzed using agarose gel electrophoresis. 8 PCR markers were proposed for *LoxA* gene amplification. PCR products were purified and sequenced. The LOX activity was analyzed by UV spectrometry at 234 nm. The 2-nonenal was analyzed by gas chromatography and antioxidant activity was analyzed by Randox kit.

Some of parent varieties substantially differed in LOX activity. Among these only several were used for breeding into F2 generation. Parent varieties KM 2008 and Marthe exhibited the best results with regard to extremely low thermostability of LOX from KM 2008 and excellent yields and malting characteristics of Marthe variety. Both varieties differed in point mutation (G/A) in exone 3 of *LoxA* gene, which was found in variety KM 2008 only. Thus, co-dominant CAPS marker was proposed for allele differentiation. PCR products were cleaved by NaeI endonuclease specific to GCCGGC sequence present in standard allele only. Thus, PCR product at size 593 bp was obtained in standard allele, while 662 bp was found in mutant allele.

To verify association of *LoxA* mutation with phenotype, LOX activity in 40 mutant and 40 standard plants was measured. In plants with mutant allele 5–10x lower LOX activity was found. LOX activity was connected with trans-2-nonenal level as well as with antioxidant status.

Further breewing of LOX less hybrids could lead to obtaining new varieties with reduced LOX activity. These varieties will be suitable for Czech beer technology.

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4-P39

DETERMINATION OF SULPHUR AMINO ACIDS IN BARLEY, MALT AND BEER

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Sulphur-containing amino acids, a natural part of barley, malt and beer, are precursors of volatile sulphur substances. These volatile sulphur substances can unfavourably affect sensory properties of beer even in very low concentrations.

Determination of amino acids in food is one of the most labored analytical food analyses. A method used for the amino acid analysis must be sufficiently accurate and sensitive; it must be also sufficiently fast and experimentally undemanding. The method of gas chromatography was used to determine sulphur-containing amino acids (methionine and cysteine) in barley, malt and beer. Before the analysis, sulphur-containing amino acids were derived and volatile N(O,S)-ethoxycarbonyl propyl esters were subsequently analyzed using the gas chromatography with a selective flame photometric detector[1-3]. Following validation parameters were achieved: LOQ 1,6, $R^2 = 0,99974$, RSD 15 %.

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REFERENCES

1. Hušek P., Matucha P., Vránová A., Šimek P.: J. Chromatogr. B *789*, 311 (2003).
2. Myung S., Kim M., Min H., Yoo E., Kim K.: J. Chromatogr. B *727*, 1 (1999).
3. Hušek P.: J. Chromatogr. B *717*, 57 (1998).

4-P40

DEVELOPMENT OF CAPILLARY ZONE ELECTROPHORESIS METHOD FOR MONITORING OF SELECTED ORGANIC ACIDS IN FERMENTED FOODS

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During the processing of food matrix by microorganisms, great number of various metabolites is formed. Simple organic acids (such as acetic acid, lactic acid etc.) belong among the most important ones. Therefore, monitoring of their production during fermentation process provides valuable information for control of biotechnological processes. Moreover, organic acids have a significant impact on sensory properties, stability of food and are an indicator of microbial contamination[1].

The link between the different fermentation processes is, for example, lactic acid. Lactic acid is usually formed during fermentation of grape must as well as in the production of fermented dairy products. Lactic acid shows a different perspective on the nature of organic acids. They are either desirable (for the production of milk fermented products), or undesirable – as an indicator of contaminant microflora (lactic fermentation of grape must, undesirable milk souring).

Determination of organic acids requires a sensitive analytical method. Capillary zone electrophoresis (CZE) is a suitable method for the determination of these acids, because acids are easily ionizable and CZE is an ecological and economical method, have low exigencies to quantity of samples. Method is fast, accurate and sensitive to low concentrations of analytes. This work is focused to develop a sensitive method for the determination of selected low molecular weight organic acids by capillary zone electrophoresis in wines, wine must and products of lactic fermentation[2].

The aim of work is to find the optimal separation system for the rapid routine determination of selected acids. The acids were separated under slightly alkaline conditions and detected by directly by UV detector (214 nm). The capillary length was 60 cm.[3]

REFERENCES

1. Mato I., Suárez-Luque S., Huidobro J. F.: Food Res. Int. *38,* 1175 (2005).
2. Apichai S., Pattana T., Rodjana B., Supalax S.: LWT *40,* 1741 (2007).
3. Castiñeira A., Peña R. M., Herrero C., García-Martín S.: J. Food Compos. Anal. *15*, 319 (2002).

4-P41

PRODUCTION OF ENZYME COCTAILS FOR HYDROLYSIS OF COMPLEX WASTE SUBSTRATES

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Lignocellulose is the major component of biomass, comprising around half of the plant matter produced by photosynthesis. It consists of three types of polymers, cellulose, hemicellulose and lignin that are strongly intermeshed and chemically bonded by non-covalent forces and by covalent cross-linkages. Only a small amount of the cellulose, hemicellulose and lignin produced as by-products in agriculture, forestry or food industry is used, the rest is being considered as a waste. On the other side, these materials could represent very attractive substrates for various biotechnological processes; nevertheless, they must be turned into simple substances utilizable by yeasts and other biotechnologically employed microorganisms.

This work was aimed at production and characterization of enzyme cocktails able to hydrolyze lignocellulose and other complex waste materials coming from forestry and food industry. Enzymes were produced by fungal strains *Aureobasidium pullulans*, *Fusarium solani* and white-rot fungi *Phanerochaete chrysosporium*. In particular, we focused on production of cellulase, amylase, xylanase, lipase, protease and ligninolytic enzymes: laccase, manganese- dependent peroxidase and lignin peroxidase. The cultivations were carried out in submersed mode in mineral medium supplemented by waste co-substrates such as wheat bran, corn bran, rice bran and oat bran, sawdust, rice and apple fiber.

Composition of the enzyme mixtures depended on the co-substrate used as well as on the time of cultivation. The highest activity of cellulase, amylase and xylanase were observed during the first period of cultivation (up to the 7th day of cultivation), after that their activity started to decrease which was followed by an increase of ligninolytic enzymes activity. Generally, the presence of waste co-substrate in the medium strongly enhanced expression of the hydrolytic enzymes. The only exception was *Aureobasidium pullulans* which produced high amount of cellulase and xylanase in basal mineral medium without any co-substrate. Finally, produced enzyme cocktails were applied on selected wastes (wheat bran, apple fiber etc.) and their hydrolysis was observed.

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4-P42

DEPENDENCE OF BIODEGRADATION BEHAVIOR OF POLYCAPROLACTONE FILM ON THE WAY OF ITS PREPARATION

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Poly(ε-caprolactone) (PCL), a member of the family of aliphatic polyesters, is one of a few synthetic polymers which was proved to be biodegradable. Since environmental degradation of PCL can occur due to the action of microorganisms frequently present in an ecosystem, this aliphatic polyester represents a suitable candidate for the production of biodegradable packaging materials. The degradation rate of PCL is known to be significantly dependent on various parameters, such as molecular weight and its distribution, crystalline morphology, the presence of catalytic residues or un-reacted monomer, type of microorganism or mixed cultures used and degradation conditions, etc.

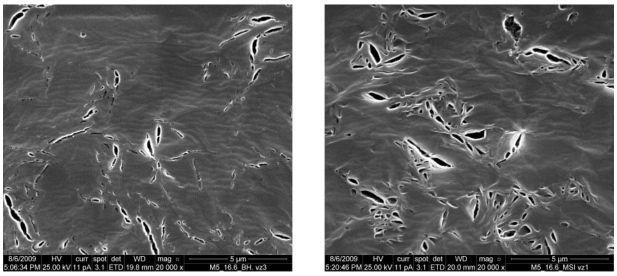

Figure 1. SEM micrographs showing the PCL surface (melt-pressing) after 42-day degradation by *Bacillus subtilis* in nutritious (BH) medium (left one), in mineral medium (right one)

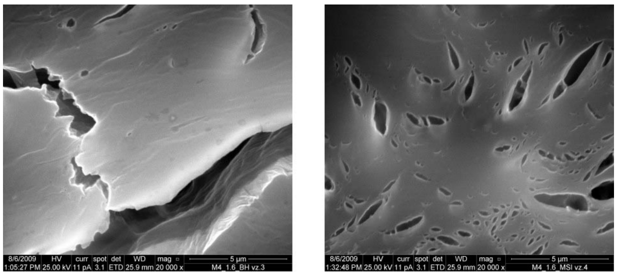

Figure 2. SEM micrographs showing the PCL surface (solution casting) after 42-day degradation by *Bacillus subtilis* in nutritious (BH) medium (left one), in mineral medium (right one)

The aim of this work was to study the degradation behaviour of poly(ε-caprolactone) films, obtained by two different methods; melt pressing and casting in chloroform, by the action of bacterial strain *Bacillus subtilis* CCM 1999. PCL films were aged in mineral (MS) and brain heart infusion medium (BH) at 29 ± 1 °C and pH = 7 for 42-day degradation period. The appearance of randomly oriented cracks on the surface of incubated PCL films accompanied with a slight decrease in molecular weight was observed after degradation runs.

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4-P43

COMPARISON OF CONTENT OF PHENOLICS AND BITTER SUBSTANCES IN BEERS WITH DIFFERENT ALCOHOL CONTENT

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Beer is one of the most popular beverages in the all over the world. It is available in numerous flavors and fashions. Beer is famous not only for its freshness and a bitter taste, but it has also a lot of healthy properties such as elimination of toxic and waste materials out of the body thanks its diuretic effect, prevention osteoporosis thanks potassium and sodium content or anticancer properties thanks polyphenolic compounds.

Total of 30 samples of beers with different degree of alcohol content were tested. Total phenolics, total flavonoids, bitter substances and antioxidant activity were determined spectrophotometrically. Basic brewing characteristics were masured by pycnometry. Identification and quantitative analysis of individual phenolics was performed using on-line HPLC/PDA/ESI/MS analysis (Thermo Fisher Scientific). Beer phenolics were separated on Ultra Aqueous C18 column using gradient elution with acetonitrile/ 1% acetic acid.

The highest content of total phenolics was determined for 11° beer "Černá Hora". Chlorogenic acid was found as the most abundant beer flavonoid in 12° Budvar Buweiser. It was proved, that iso-acids exhibit about 65,44 – 93,37 % of the total bitter substances. These components are responsible for the main part of beer bitterness. Alcohol free beers contained in general lower amount of phenolics except "Bernard free". When compared with foreign beers, in Czech beers higher level of majority of active compounds was detected and specific distribution of individual derivatives was found as well.

Results show, that composition of active substances is not directly dependent on ethanol content, the main factor for beer quality is probably used technology. Some types of alcohol free beer could serve as beverage with very positive effect on human health.

This work was supported by projects 2B08057 (NPV II) and FR 2953/G4/2011 of the Czech Ministry of Education.

4-P44

COMPARATIVE ANALYSIS OF LIPOPHILIC COMPOUNDS IN EGGS OF ORGANIC ISA BROWN AND ARAUCANA HENS

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Hen eggs represent a rich source of important nutrients, including lipids and carotenoids. The composition of hen eggs can be manipulated be genetic selection or by nutritional manipulation[1].

The aim of this study was to compare the fatty acids, cholesterol and carotenoids content of egg yolk of ISA Brown and Araucana hens grown in free range housing system. Hens received the same organic food, with no supplements and water *ad libitum*. Fatty acids and cholesterol were analyzed by GC-FID and carotenoids were quantified by RP-HPLC-PDA.

The major fatty acids in egg yolk were palmitic acid – 23,5 % and 25,6 %; oleic acid – 40,5 and 40,2 %; linoleic acid – 14,8 % and 13,9 %; and stearic acid – 9,4 % and 9,6 % in ISA Brown and, respectively in Araucana hens. Araucana eggs showed a higher content of saturated fatty acids and a lower content of unsaturated fatty acids. The results are in agreement with previously reported data[2,4]. The total cholesterol content was 14,7 mg g^{-1} yolk for ISA Brown and 12,7 mg g^{-1} for Araucana hens. Since the hens received the same diet, the fatty acids and the cholesterol content are influenced by genetic factors.

Carotenoids were separated and quantified by HPLC analysis. The major carotenoids were lutein and zeaxanthin which accounts more than 75 % of total carotenoids. β-cryptoxanthin, β-carotene and β-apocarotenal were present in small amounts in both samples Lutein was found at 1,8 mg/100 g in ISA Brown and at 1,75 mg/100g in Araucana. Zeaxanthin reached 0,97 mg/100 g and respectively 0,87 mg/100 g. The high content of zeaxanthin is related to the high proportion of *Zea mays* in hens food, since the profile of carotenoids in eggs is strictly influenced by the diet[1,3]. Organic eggs of both breeding represents very good sources of highly bioavailable lutein and zeaxanthin, pigments which are correlated with lower risk of age-related macular degeneration.

REFERENCES

1. Chung H. Y., Rasmussen H. M., Johnson E. J.: J. Nutr. *134*, 1887 (2004).
2. Millet S., De Ceulaer, Van Paemel M., Raes K., De Smet S., Janssens G.P.J.: Br. Poul. Sci. *47*, 294 (2006).
3. Van Ruth S., Alewijn M., Rogers K., Newton-Smith E., Tena N., Mirko B., Koot A.: Food Chem. *126*, 1299 (2011).
4. Simčic M., Stibilj V., Holcman A.: Food Chem. *125*, 873 (2011).

4-P45

COPPER, SELENIUM SUPPLEMENTED YEAST BIOMASS - A SOURCE OF MICROELEMENTS

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Yeast can accumulate of selenium and copper. After an absorption in gastro-intestinal tract, copper is bound to albumin. Selenium is incorporated into organic selenium-containing compounds, mainly selenomethionine[1,2].

Production strain *Saccharomyces cerevisiae* 612 was cultivated under aerobic conditions for 48 hours with 0; 10; 20; 30; 40 and 50 mg l^{-1} of sodium selenite and with 0; 20; 40; 60; 80; 100 µg $CuSO_4$ 100 mg l^{-1}, respectively. The medium (YPD) contained (in g l^{-1}): yeast extract 10; peptone 20; glucose 35. The Se content in yeast cells was found increased with increasing concentration of sodium selenite in the medium[3] and the highest value of Cu was reached using 100 µg of copper sulphate in the medium[4] (Table I).

Table I, Biomass yield and total selenium and copper content in *Saccharomyces cerevisiae* strain 612

Na_2SeO_3	Content of Se	$CuSO_4$	Content of Cu
[mg l^{-1}]	[mg kg^{-1}]	[µg l^{-1}]	[mg kg^{-1}]
0	2,38	0	3,46
10	121	20	7,2
20	463	40	11,2
30	1068	60	14,8
40	1675	80	16,8
50	1741	100	21,6

This study confirmed the possibility of accumulation of selenium and copper by *Saccharomyces cerevisiae*. Se, Cu enriched biomass can be potentially used in human nutrition.

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REFERENCES

1. Skalická M., Koréneková B., Kožárová I.: Potravinárstvo *1*, 64 (2009).
2. Yin H., Fan G., Gu Z.: Food Sci. Technol. *43,* 666 (2010).
3. Poláková A., Szabová E., Urminská D., Šillerová S.: Potravinárstvo *1*, 76 (2011).
4. Šillerová S., Poláková A., Urminská D., Szabová E.: Potravinárstvo *1*, 85 (2011).

4-P46

EFFECTS OF EXTRACTION CONDITIONS ON THE ANTIOXIDANT ACTIVITY OF BUCKWHEAT AND SPELT FLOURS

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Cereals and pseudocereals (including spelt and buckwheat) play an important role in maintaining good health. Flours are significant source of bioactive compounds, e.g. dietary fibre, vitamins, minerals and last but not least phenolic compounds that are frequently associated with the health benefits of cereals. Functional foods are specially designed products with modified content of compounds of interest, most frequently antioxidants. To do so, it is important to optimize the extraction process leading to their isolation, characterization of their properties and to find conditions suitable for their application is a way promising their maximum efficiency in new matrix. The present study was focused on comparison of five different extractions solvent systems used to prepare extracts from spelt and buckwheat flours, i.e., absolute ethanol, 50 % ethanol in water (v/v), distilled water, 20% acetone in water (v/v) and 50% acetone in water (v/v). Antioxidant and radical-scavenging activities of extracts were tested involving several commonly assay, i.e. $^{\bullet}$DPPH and ABTS$^{\bullet+}$ by means both UV-VIS and EPR spectroscopy. Total phenolic content (TPC) was evaluated using Folin-Ciocalteau assay. Besides them, CIE Lab color coordinates of all samples were assessed under standardized conditions. As regards the results obtained, significant differences in TPC content were found especially between extracts prepared to absolute ethanol and 50% acetone. For both, spelt and buckwheat extracts, 50% acetone extract revealed the highest content of phenolic compounds compared to the other extraction systems. Antioxidant activity asessed using the ABTS$^{\bullet+}$ test decreased in the order: 50% Acetone > 50% EtOH > 20% Acetone > H_2O > EtOH, whereas in case of $^{\bullet}$DPPH assays, antioxidant activity decreased in order: 50% Acetone > 20% Acetone > 50% EtOH > H_2O > EtOH. Results obtained indicate that the extraction solvents significantly alter the antioxidant activity of spelt and buckwheat flour extracts. In summary, absolute ethanol is the least effective solvent for the extraction of phenolic compounds from both types of flours, followed by distilled water. On the contrary, 50% acetone could be recommended as a solvent suitable to prepare antioxidant extracts from spelt and buckwheat flours with maximum polyphenols yield.

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4-P47

ELEMENTAL COMPOSITION AND TOTAL ANTIOXIDANT ACTIVITY OF CZECH WINES DERIVING FROM GRAPES GROWNED BY DIFFERENT AGRICULTURE METHODS

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The aim of this pilot study is to compare the influence of the agriculture on elemental content of vineyard components (soil, leaves and grapes) and wines. In addition, radical scavenging activity of wines from ecological and integrated production was determined.

The determination of 22 elements (Li, Be, Na, K, Ca, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, As, Se, Sr, Mo, Cd, Sn, Ba, Tl, Pb – selected by essentiality and possible toxicity)[1] in extracts of soil, leaves, grapes and wine samples collected from vineyards of Ivaň (belongs to wine region Mikulov, bigest wine region in Czech republic) was performed. For this purpose, inductively coupled plasma-mass spectrometry has been used. For the determination of elements influenced by polyatomic interferences, collision cell technology on He/H mode has been used. The ^{115}In was used as internal standard.

Main purpose of this elemental analysis was comparision of vineyards, treated by different agriculture methods - ecological (according to international federation of organic agriculture movements) and integrated. Leaves and grapes were from two varieties of Vitis vinifera - Pinot noir and Sauvignon blanc. For comparsion of wines, only Pinot noir 2010 samples from ecological and integrated production were analysed. All samples were from the same winegrower so we get rid of influence of different technologies of treating and winemaking.

Microwave assisted extraction was used for preparation of samples of leaves and grapes. Soil samples were extracted by 2M nitric acid (thermal assisted).

Antioxidant properties of Pinot noir cleret samples from ecological and integrated production were investigated by Electron paramagnetic resonance spectroscopy, using stable radicals 1,1-diphenyl-2-picrylhydrazyl (DPPH)[2]. For evaluation EPR spectra, Origin and MicromathScientist software were used. As reference technique UV-VIS spectroscopy (734nm) using ABTS system was chosen[3].

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REFERENCES

1. Passias P. N., Farmaki E. G., Thomaidis N. S., Piperaki E. A.: Food Anal. Method. *3*, 195 (2010).
2. Stasko A., Polovka M., Brezova V., Biskupic S., Malik F: Food Chem. *96*, 185 (2006).
3. Blanda G., Ceretani L., Bendini A., Cardinali A., Lercker A.: Eur. Food Res. Technol. *227*, 191 (2008).

4-P48

EFFECT OF SELECTED INORGANIC SALTS ON ACRYLAMIDE CONTENT AND SENSORY PROPERTIES OF BREAD

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Sensory evaluation of food, especially, odour, taste, and colour, has been always used as criteria in selection of food. Cooking, baking and roasting are applied to make food more palatable, digestible and microbiologically safe.

In order to improve some aspects of dough behaviour and final bread quality, some additives are commonly used. The choice of „bread improvers" depends on legislative limits and expected impact on product properties. The presented work is focused on the comparison of the effect of seven different inorganic salts commonly used as improvers on acrylamide content in bread and parallelly on its sensory properties.

There were examined KH_2PO_4, NaH_2PO_4, $Na_2H_2P_2O_7$ and $Na_4P_2O_7$ – each one of them in concentration of 2500 mg/kg of bread mix; and $CaCl_2$, Ca-Lactate and NH_4Cl - each one of them in concentration of 3000 mg kg^{-1} of bread mix. Sensory properties such as appearance of bread loaf, surface, colour and crispness of crust, porosity of crumb and rise in volume were scored in scale from 1 to 5, and odour and taste from 0 to 100. Obtained results were evaluated by methods of multivariate statistical analysis. There was observed positive effect of $CaCl_2$ addition on sensory quality and simultaneously on 10 % decrease of acrylamide content in bread. Individual applications of KH_2PO_4, NaH_2PO_4, $Na_4P_2O_7$ and NH_4Cl resulted in negative perception of some sensory attributes of evaluated bread while content of acrylamide was always increased significantly.

Acrylamide possible mitigation strategy in a bread crust was studied with respect to colour characteristics measured in CIEXYZ, CIEL*a*b*, CIEL*u*v*, CIEL*C*H° and HunterL*a*b* colour spaces, too. From all the examined colour variables only 7 significantly correlated with acrylamide content. Colour characteristics b* from CIELab and HunterLab colour space affirmed their relation to acrylamide creation, but the most intensive correlation with acrylamide was found for hue value v* derived from CIEL*u*v* colour space. Results showed that the addition of inorganic salts hasn't expected considerable elimination impact in the bread crust, but creation of acrylamide in a home bread maker's conditions was especially low in comparison to industrial ones. This gives an additional study on potentiality of acrylamide reduction in some industrial foods.

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4-P49

COMPARISON OF VOLATILE AROMA-ACTIVE COMPOUNDS AND COLORIMETRIC CHARACTERISTICS OF ORGANIC AND CONVENTIONAL WINES

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There are fundamental differences in organic and conventional production practices, but limited information is available detailing how various practices influence the nutritional and sensorial quality of products. Seventeen organic (12 white and 5 red) and twenty nine conventional (19 white and 10 red) varietal high-quality dry wines with alcohol content 11-13% (vol.) with attributes "late harvest" or "special selection" were sourced from three white wine varieties (Chardonnay, Pinot blanc and Traminer red) and two red wine varieties (Pinot noir and Cabernet Sauvignon). All the samples were originated from the same very close vineyard sub-region (Modrý Kameň) in middle Slovakian vineyard region.

Colour trichromatic measurements were made in a Shimadzu UV-3600 spectrophotometer, using 10 mm path length quarzglas cels. The whole transmission visible spectrum (380-780 nm) was recorded and illuminat D65 (daylight source) and 10° observer (perception angle of a human observer) were used as standard conditions. Colour values of conventional and organic wines were calculated by using the Shimadzu Special Edition software ColorLite v.3.1.16. Colour characteristics were expressed by more than twenty colour parameters in CIEXYZ, CIEL*a*b*, CIEL*u*v*, CIEL*C*H° and HunterL*a*b* colour spaces. Principal component analysis performed to all the colour data was not able to distinguish organic from conventional wines but canonical discriminant analysis correctly classified more than 90% of the wine samples according to their affiliation to organic and conventional viticulture.

The methods of GC/MS and GC/FID were used for study of volatile aroma-active compounds in bio and conventional produced wines of variety Chardonnay with attributes “dry, late harvest”. The aim of our work was study of qualitative and quantitative differences in profiles of “volatiles”, distinguishing organic wine production practice from conventional one.

On the grounds of obtained results is evident that used kind of wine production does not affect the qualitative profile of volatiles apart from α-pinene and sorbic acid. α-Pinene was not detected in the conventional sample of wine, on the contrary, sorbic acid was not detected in the organic wine. It is possible that ascorbic acid is present in conventional wine in synthetic form, although, this substance exists in natural form, too, but it does not occur in Chardonnay. Concerning the relative contents of analysed wine volatile aroma-active compounds, dominant constituents are: 3-methyl–butanol, 2-phenylethanol, linalool and diethyl succinate. While the relative content of 3-methyl–butanol has not changed significantly with wine production practice, different situation is concerning the 2-phenylethanol and linalool. Their summary content increased markedly from approx. 12% to approx. 21% in conventional sample of wine. The content of diethyl succinate has not changed with used wine production practice, practically. It is obvious, that kind of wine production does not have the significant influence on relative content of esters and alcohols of organic acids, except to two esters: ethyl 2-hydroxy-3-phenylpropanoate and benzyl salicylate.

There is the interesting situation in evaluation of terpenes and their oxidized derivates, too. Some of them, such as β-pinene, p-cymene, 1,8-cineole and D-limonene significantly decreased their contents (from 2 to 17times) in conventional samples of Chardonnay wine.

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4-P50

SELECTED CHARACTERISTICS OF POULTRY MECHANICALLY SEPARATED MEAT (MSM)

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The MSM is obtained by removing remaining meat from bones using mechanical means, where the normal structure of the muscle fiber is mostly lost. The MSM technologies work from a few bars pressure to high pressure separation. MSMs are mostly intended as a part of food for human consumers. The EU total amount of MSM produced was about 700 000 tones a year 2007 (77% of high pressure, 23% low pressure MSM) and is growing. Poultry is the source of 88% of MSM, while 12% part is pig. EU regulation obliges producers the calcium content of MSM not exceeds 100mg/100g[1]. USDA has set more stringent rules. At least 98% of the bone particles have their size ≤ 0,5 mm, no bone particle is > 0,85 mm, bone solids content is ≤ 3%, protein content is ≥ 14% and fat content is ≤ 30% of MSM used in the formulation of meat food products with some limitations e.g. MSM is prohibited in junior, baby and toddler food[2].

The aim of this study was to obtain and compare selected characteristics of common poultry MSM produced by low and high pressure technology from different stuff and to screen MSM for the content and size of bone fragments.

Values of selected characteristics of poultry MSM were dependent on the way of separation and the stuff used[3]. The bone fragments were expected in the higher pressure products in contrast to the low pressure MSM but not of 3 mm large-size (Tab.1). USDA measures fulfills only MSM from hand chops as for the size of bone particles. The higher pressure MSMs were also short in proteins[3]. The content of calcium and/or bone solids meets both USDA and EU legislature. The bone fragments of low pressure MSM of 1-1,9 mm size were proved. This finding needs a confirmation and an organoleptic study of the food prepared from such MSM.

Table I, Bone fragments in poultry MSM #(count/25g)[3]

MSM	Fragments mg kg^{-1}	1 - 1,9 mm #	2 - 2,9 mm #	>3 mm #
Lima, higher pressure				
Backs	481	10	7	2
Thighbone	429	12	7	1
Neck	458	13	6	1
Breastbone	965	16	5	2
Mixture	516	11	5	2
Baader 601, Low pessure				
Sidebone	135	2	0	0
Articular capsule	89	3	0	0
Hand chops	20	0	0	0
Mech. Chops	138	6	0	0
Mixture	143	1	0	0

REFERENCES

1. COM/2010/0704 final, Brussels, 2.12.2010.
2. USDA. Mechanically Separated (Species), Title 9, Section 319.5, 319,6 p. 297. Office of the Federal Register, MD, USA 2003.
3. Čížková S.: Diplomová práce. Vysoké učení technické v Brně, Brno 2011.

4-P51

GROWTH AND PRODUCTION PROPERTIES OF RED YEASTS USING WASTE GLYCEROL AND LIPIDS AS CARBON SOURCE

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Carotenogenic yeasts are ubiquitous due to its worldwide distribution in terrestrial, freshwater and marine habitats, and have ability to colonize a large variety of substrates. For this reason, various waste materials can be used as cheap substrates for its cultivation. In this work several red yeast strains (*Sporobolomyces roseus, Rhodotorula glutinis, Rhodotorula rubra, Rhodotorula aurantiaca, Cystofilobasi-dium capitatum*) were enrolled into a comparative screening study. Each strain was cultivated at optimal growth conditions and in medium with glycerol and lipid sources alone and in mixture with glucose. Obtained pigments and ergosterol were analyzed by HPLC/PDA/MS/ESI device. Extracellular lipase activity was measured spectrophotometrically.

Red yeasts prefer glucose as the main carbon source and produced biomass in optimal condition was similar in all tested strains Furthermore, most strains were able to utilize pure as well as waste glycerol to produce significant amounts of biomass and pigments. The maximum pigment yields were reached in a media with glycerol: glucose mixture 3:1. Using glycerol as carbon source, similar carotenoid pigments as in glucose medium were produced; the main pigments were beta-carotene, torulene and torularhodin.

Cultivations on oil medium, growth was reduced and the colour of the cultures was unusual. Extracellular lipase activity in red yeast medium was relatively low. Higher activity was found in all tested *Rhodotorula* strains, mainly in *R.aurantiaca*. Pigment composition was slightly changed using oil medium; higher amount of xanthophylls was found and very low level of beta carotene was formed in these conditions. Thus, it can be concluded that using oil, as carbon source for cultivation of red yeast is not recommended.

At the current annual production capacity (9,8 billion liters of biodiesel), 980 million liters of glycerol/yr are produced compared to a demand of only 216 million liters/yr. This fact implies that waste glycerol after transesterification can be used as medium component, which can induce carotenoid rich biomass production and influence its final quality.

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4-P52

PARTIAL PURIFICATION AND CHARACTERIZATION OF α-GALACTOSIDASE FROM *CRYPTOCOCCUS LAURENTII*

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The yeasts of genus *Cryptococcus* are widely occurring pathogenic yeasts. The pathogen has a capsule composed of complex polysaccharides that function in virulence in a manner analogous to those of the classical encapsulated bacteria. During the process of budding and release and degradation of capsular polysaccharides, the capsule undergoes several changes probably as a consequence of transglycosylating and hydrolytic activities of enzymes.The aim of this study was the partial purification and characterization of α-galactosidase from *Cryptococcus laurentii* CCY 17-3-29 which may play a role in capsule modifications.

The cells were grown on medium with lactose as a C-source to the stationary phase. After homogenization they were undergone fractional centrifugation. α-Galactosidase was purified from the cytosole (CF) and after extraction from the membranes (EMF), too. The purification path was represented by liquid chromatography on CM-Sephadex C-50, Sepharose-Concanavalin A and Superdex 75 or Superose 12 columns. Enzyme was purified 101 fold from CF and 105 fold from EMF, respectively, but three different protein zones were still observed both by SDS PAGE and IEF PAGE for CF and even more for EMF. According to liquid chromatography, the band with Mr 130 kDa and Ip 4,2 can correspond to major form of α-galactosidase from CF, but structural analysis (MALDI TOF/TOF) of these bands failed because of low concentration of proteins (silver-staining). The reason was the decrease of enzyme solubility observed during purification.

Substrate for activity assay and the method were optimized. Enzyme obtained from both fractions was characterized by its pH optimum, temperature optimum, pH stability and thermal stability. The kinetic analysis was provided as well. α-Galactosidase showed not only the ability to release galactosyl group from substrate but possessed transglycosylation activities, too. The reverse hydrolysis was not observed.

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4-P53

TRANSGLYCOSYLASES – GLYCOSYLHYDROLASES WHICH DO NOT LIKE WATER OR ENZYME NOMENCLATURE VERSUS CAZY

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Classification of enzymes in Enzyme nomenclature is based on the reaction they catalyze. Glycosyltransferases (EC 2.4) transfer generally sugar from oligosaccharide or a high-energy compound (donor unit modified with nucleotide) to another carbohydrate molecule as an acceptor. Some of these enzymes have also hydrolytic activity which can be regarded as a transfer of a glycosyl group from the donor to water. Glycosylhydrolases (EC 3.2) catalyze the hydrolytic cleavage of glycosidic bonds. But this requires not only the removal of a particular glycosyl group from substrate but likewise the transfer of this group to suitable acceptor - water. In principle, all hydrolytic enzymes may be classified as transferases, since hydrolysis itself can be regarded as a transfer of a specific group to water as an acceptor. Probably this was the reason to classify a special group of enzymes, transglycosylases, as glycosyltransferases. In this case, the catalysis is realized in two stages. In the first one, they cleave polysaccharides forming smaller units, what is typical for hydrolases. Then the fragment with newly created reducing end is transferred to another saccharide (transglycosylation) instead of water (hydrolysis).

According to CAZy (Carbohydrate-active enzymes database) based on structurally-related catalytic and carbohydrate-binding modules (or functional domains) of enzymes that degrade, modify, or create glycosidic bonds, these enzymes are clearly positioned in glycoside hydrolase families. Glycosyltransferase group is according this classification formed solely from enzymes utilizing donors modified with nucleotides. Transglycosylases of the plant and fungal cell wall are enzymes taking part in the modification of polysacchrides and their incorporation into the cell wall. These enzymes enable the cell walls to behave as dynamic structures, e.g. they enable the expansion and remodeling of the cell wall during cell growth without its weakening. The best-known enzyme from this group is plant xyloglucan endotransglycosylase (XET, EC 2.4.1.207 or member of GH 16 family). A mechanism of transglycosylation, the character of its bi-bi reaction, the discovery of unspecific XETs changing the concept in the realization of cross-linking of cell wall polysaccharides as well as the development of specific methods for transglycosylase assays will be explained on this model. Identification of other types of transglycosylases in plants will be discussed as well as the transglycosylating activities and the responsible enzymes in the cell walls of yeasts and fungi.

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4-P54

EFFECT OF CONCENTRATION OF BIOMASS ON BIOSORPTION CAPACITY

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The scientists have been dealing with the possibility and techniques of adjustment of water pollution by heavy metals for several decades. At the present time, there exist numerous chemical and physical techniques. The main disadvantages of these techniques are low effectiveness at lower concentrations and a production of secondary waste[1]. Therefore the attention is oriented to search for simple, effective, cheap and ecological method, such as biosorption, wich is based on the bond of metals onto cellular structure[2,3] (microbial or vegetable cells).

The aim of this work is the study of the biosorption of heavy metal (copper, lead and zinc) on thermophilic bacteria of the genus *Geobacillus (G. thermodenitrificans* CCM 2566 *and G. thermocatenulatus* CCM 2809). The optimal value of pH was determined as 5 for copper and zinc and 4 for lead.

The 20 ml solution of metal with concentration 0,5 g l^{-1} (Cu^{2+}, Zn^{2+}), resp. 1 g l^{-1} (Pb^{2+}) was mixed with dry biomass with concentration 0,5; 1; 2 and 3 g l^{-1}. The mixture was incubated at 25 °C and 100 rpm for 12 hours. The biomass was harvested by centrifugation at 6000 rpm for 10 min. The supernatant was used for complexometric titration.

By the use of biomass G. thermodenitrificans we were able to reach the following results. For Cu^{2+} ions the adsorption capacity was highest, using concentration of biomass at 0,5 g l^{-1} (56,9 mg g^{-1}). The adsorption capacity rapidly decreased with increased concentrations and at certain value stops decreasing completely, around the value of 20 mg g^{-1}. Similarly for Zn^{2+} ions, the highest adsorption capacity is for concentration of biomass at 0,5 g l^{-1} (14,6 mg g^{-1}) and slowly decreases at the capacity of 7,4 mg g^{-1}. For Pb^{2+} ions, the decrease is almost linear to the concentration of biomass of 2 g l^{-1}, i.e. from 109,5 mg g^{-1} to 52,7 mg g^{-1}.

The adsorption capacity for Cu^{2+} ions of the *G. thermocatenulatus* is highest at the lowest concentration of biomass (64,7 mg g^{-1}), then it steeply decreases and at concen-tration of biomass of 1 g l^{-1} does not change. In case of Zn^{2+} ions, we can see a moderate drop with the increasing concentration with the range of 20,6 – 12,8 mg g^{-1}. For Pb^{2+} ions, the decrease is slow, down from adsorption capacity of 105,9 mg g^{-1} to 54,2 mg g^{-1}.

Affinity of metals to bacteria was determined in the order Pb > Cu > Zn. The results show, that *G. thermocatenulatus* has better adsorption capabilities than *G. thermodenitrificans*.

REFERENCES

1. Volesky B.: Hydrometallurgy *59*, 203 (2001).
2. Volesky B.: Water Res. 41, *4017* (2007).
3. Vijayaraghavan N., Yun Y. S.: Biotechnol. Adv. *26*, 266 (2008).

4-P55

YEAST *SACCHAROMYCES CEREVISIAE* AS A SOURCE OF BIOGENIC SUBSTANCES

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Yeasts have been tapped as sources of vitamins, antioxidants and as supplements for their nutraceutical or health-promoting attributes[1]. The composition of three yeast strains biomass of *Saccharomyces cerevisiae*, i.e. Kolín, 612 and Gyöng was studied.

The yeasts were grown on YPD medium containing 10 g l^{-1} yeast extract, 20 g l^{-1} peptone and 35 g l^{-1} glucose with natural pH 6,6. Results confirmed a significant content of protein in obtained biomass, while the concentration of glucose and fat was relatively low[2] (Table I).

The biomass of yeast is relatively high in protein. The high concentration of glucose and low content of lipid[3] was not observed in our study.

The analysis of amino acids composition showed the presence of all essential amino acids. The results confirmed the high quantitave and qualitative value of proteins of *S. cerevisiae*. The yeast biomass contained five times the cereal protein content. The biological value of these proteins is higher thanks to the presence of sulphur amino acids[4].

Table I, Content of protein, glucose and lipid in *S.cerevisiae* [g kg^{-1}]

Compound	strain Kolín	strain 612	strain Gyöng
Protein	489,7	458	422
Glucose	0,4	7,83	15,51
Lipid	58,8	54,6	53,1

The biomass of yeast is characterized by high content of vitamins, particularly of the type B, such as strain Kolín, which had a concentraion of vitamin B_2 20,6 mg kg^{-1}.

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REFERENCES

1. Abbas C. A., in the book: *Yeasts in food and beverages*, chapt. 10, p. 286. Springer-Verlang, Berlin 2006.
2. Šillerová S., Poláková A., Urminská D., Szabová E.: Potravinárstvo *1*, 88 (2010).
3. Swanson K. S., Fahey G. C. J.: Nutritional biotechnology in the feed and food industries. Preoceeding of Alltech's 20th Annual Symposium. Kentucky, USA 475 (2004).
4. Ürgeová E., Marecová M.: Nova Biotechnologica *3*, 153 (2003).

P56

THE PHENOLIC CONTENT AND ANTIOXIDANT ACTIVITY OF VARIETAL SLOVAK RED WINES

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Antioxidants are specific substances, which oxidate themselves and in this way they protect other sensitive bioactive food components against destruction. At the same time they restrict the activity of free radicals and change them to less active forms. Large group of antioxidants are polyphenols, which affect sensory properties of fruit and vegetables, like colour (anthocyans), taste (flavonoids) or odour (phenols)[1]. The signiticant source of polyphenolic compounds are wines. Polyphenols (in optimal amount and combination), found maily in red wines, have very complex (positively) effects on organism.

The aim of this work was to determine chosen antioxidant properties of the most sold brand red wines – Blaufränkisch and St. Laurent, originating from different Slovakia vineyard regions. Total polyphenols contents was determined with phenol Folin-Ciocalteau´s reagent[2], the content of total anthocyanins was determined by pH differential method[3] and antioxidant activity was determined using DPPH stable free radical[4]. All three parameters were determined by UV-VIS absorption spectrometry.

The determined total polyphenol contents in observed wines were within the interval 1668 – 2734 mg l^{-1} (Blaufränkisch), respectively 1409 – 3276 mg l^{-1} (St. Laurent). Total anthocyanin contents in wines were within the interval 156,9 – 450 mg l^{-1} (Blaufränkisch), resp. not detectable – 460,1 mg l^{-1} (St. Laurent). Antioxidant activity in wines was within the interval 80,2 – 86,0 % (Blaufränkisch), respectively 71,0 – 84,1 % (St. Laurent). The results of measured properties (total polyphenol contents, total anthocyanin contents and antioxidant activities) of the wines originating from different vineyard Slovak regions are not statisticaly significant. The only one exception is total polyphenol contents in wine – St. Laurent, where statisticaly significant differences were foud in wines originating from South-slovak and Little Carpathian vineyard regions.

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REFERENCES

1. Vollmannová A., Tomáš J., Urminská D., Melicháčová S., Krížová L.: Czech J. Food Sci. *27*, 248 (2009).
2. Faitová K., Lachman J., Pivec V., Hejtmánková A., Dudjak J., Šulc M. 2003. *Vitamíny 2003 - Přírodní antioxidanty a volné radikály, Pardubice, 15-17 Sept. 2003,* Proceedings, p. 167 (Poster no. 20).
3. Lapornik B., Prošek M., Wondra A. G.: J. Food Eng. *71*, 214 (2005).
4. Brand-Williams W., Cuvelier M. E., Berset, C.: Lebensm. Wiss. Technol. *28*, 25 (1995).

4-P57

SCREENING OF *LACTOBACILLUS* STRAINS FOR BACTERIOCIN GENES

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Bacteria of the genus *Lactobacillus* are a major part of the lactic acid bacteria group. Many species of *Lactobacillus* are known to produce bacteriocins. Bacteriocins are antibacterial proteins that kill or inhibit the growth of other bacteria including pathogenic bacteria. The potential use of bacteriocin producing strains as probiotics and bioprotective agents has recently received increased attention.

The aim of the study was screening of 45 *Lactobacillus* strains for bacteriocin genes using polymerase chain reaction (PCR) with specific primers and using DNA/DNA hybridization with specific probes. Strains were isolated from healthy breast-fed babies and collected in Czech Collection of Dairy Microorganisms (CCDM, Tábor, Czech Republic). All strains were cultivated in MRS medium at 37 °C for 2 days aerobically or anaerobically. DNAs were isolated using fenol-chloroform extraction and ethanol precipitation. Purificated DNAs were amplified using PCR and screened for the occurence of genes for gassericin K7A, gassericin K7B, acidocin A, acidocin B, gassericin A, gassericin T and lactacin F[1]. DNA/DNA hybridization with specific probes labeled with digoxigenin followed by imunological detection of hybridisation products were used for detection of some above mentioned bacteriocins. Specific probes were prepared using PCR and DNA of *Lactobacillus gasseri* K7 or *Lactobacillus johnsonii* CCM 2935.

Specific PCR products were detected for gassericin K7A (6 strains), gassericin K7B (5 strains), acidocin A (1 strain), acidocin B (1strain), gassericin T(6 strains), gassericin A (2 strains) and lactacin F (0 strain) using PCR and specific primers. The results were confirmed for gassericin K7A and gassericin K7B using DNA/DNA hybridization with exception of one strain.

We detected the occurrence at least one bacteriocin gene in DNA of 12 new isolated strains of *Lactobacillus* using PCR. The production of proteinaceous antimicrobial substances by these strains will be tested using agar-spot test and agar diffusion test.

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REFERENCES

1. Majhenič A. Č, Matijašić B. B., Rogelj I.: J. Dairy Res. *79*, 199 (2003).

4-P58

COMPARISION OF DGGE PROFILES OF DNA ISOLATED FROM A DAIRY PRODUCT BY DIFFERENT ISOLATION METHODS

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The aim of this work was to evaluate the influence of the DNA isolation method on the characterization of the microbial community of a dairy product by DGGE.

The total and cultivable microbial community of a dairy product was assessed. The total DNA was isolated from 1 ml of the dairy product. To analyse the cultivable microbial community of the dairy product, two approaches were used: i) cultivation of a sample of the dairy product in MRS broth, followed by the extraction of DNA from the biomass after centrifugation and ii) cultivation of a sample of the dairy product on MRS plates and followed by the extraction of DNA from the isolated colonies.

For DNA isolation the following methods were used: PowerSoil DNA Isolation Kit (MoBio), Genomic DNA Purification Kit (Fermentas), the method described by Meng et al.[1], isolation by magnetic particles MPG Uncoated MCPG0502[2]. For DNA quantification Quibit Flourometer was used.

DNA isolated by different methods was amplified with the universal bacterial primers F338GC, R518[3] in PCR reaction (94 °C 5min, 30 cycles of 92 °C 30s, 55 °C 30s, 72 °C 30s and final extention 72 °C 30min). The PCR products were run in DGGE (gradient 6-9% acrylamide, 30-58% of dena-turant) and the profiles were compared.

DNA was successfully isolated from all three matrices by all the DNA isolation methods. However, the higher amount of DNA was obtained when the Genomic DNA Purification Kit (Fermentas) was used. Nevertheless, the quality of DNA was sufficient for PCR amplification, irrespectively of the DNA isolation method used.

The DGGE profiles based on the diversity of 16S rRNA bacterial gene sequence revealed two major bands, regardless to the extraction method.

All tested methods were suitable to isolate DNA from a probiotic dairy product. DGGE proved that there was no significant difference between the methods.

1. Meng X. C., Pang R., Wang C., Wang LQ.: J. Dairy Res. *77*, 498 (2010).
2. Rittich B., Španová A., Šálek P., Němcová P., Trachtová Š., Horák D.: J. Magnetism Magn. Mat. *321*, 1667 (2009).
3. Muyzer G., de Waal E. C., Uitterlinden A. G.: Appl. Environ. Microbiol. *59*, 695 (1993).

4-P59

OCCURENCE OF LACTIC ACID BACTERIA IN GRAPE MUST DURING ALCOHOLIC FERMENTATION

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Winemaking can be summarized as the biotransformation of must into wine, which is carried out principally by *Saccharomyces cerevisiae* strains during the primary, i.e. alcoholic, fermentation. A secondary fermentation, the so-called malolactic fermentation (MLF), assures biodeacidification that is also often encouraged, since it improves wine stability and quality. Malolactic fermentation usually takes place after alcoholic fermentation, but may also occur simultaneously with the primary fermentation. During the fermentation, lactic acid bacteria metabolize malic acid to form lactic acid and CO_2[1]. Strain of lactic acid bacteria isolated from wine belong to the genera *Lactobacillus*, *Oenococcus* and *Pediococcus*, although MLF is largely controlled by *Oenococcus oeni*[2].

The aim of this work was to monitor the total number of lactic acid bacteria occurring in grape must during wine production. The study was performed on the white wine grape variety Sauvignon from both organic and integrated vineyards. The isolation of pure cultures of lactic acid bacteria from mixed cultures and subsequently their identification by polymerase chain reaction (PCR) was also subject of the thesis. Of the total number of 24 purified bacterial strains isolated during the alcoholic fermentation, 22 strains were identified into the genus *Lactobacillus* by the genus-specific PCR and the remaining 2 strains as species *Oenoccocus oeni* by the species-specific PCR. Subsequently, from 22 bacterial strains of the genus *Lactobacillus* 9 strains were classified as *Lbc. paracasei*, 7 strains as *Lbc. plantarum*, and 1 strain as *Lbc. Fermentum*. The remaining 5 strains have not been identified into species yet.

Then, the analysis explored the effect of the way of wine growing (organic and integrated) on species representation of lactic acid bacteria in grape must during the primary fermentation. The experimental results show that the method of wine growing had an impact on both the number of viable cells as well as the species representation of lactic acid bacteria during the alcoholic fermentation of grape must. Whereas on the first day of alcoholic fermentation the number of lactic acid bacteria did not differ significantly, in the later stage of primary fermentation lactic acid bacteria developed better in the variety from organic vineyard. Similarly, the species representation varied; in the variety from organic vineyard were isolated all four tested species, while in the variety from integrated vineyard were isolated only two.

REFERENCES

1. Alexandre H., Costello P. J. Remize F., Guzzo J., Guilloux-Benatier M.: Int. J. Food Microbiol. *93*, 141 (2004).
2. Carrete R., Terasa V., Bordons A., Constanti M.: FEMS Microbiol. Lett. *211*, 155 (2002).

4-P60

THE COMPARISON OF CONTENT OF AROMA ACTIVE COMPOUNDS IN SAMPLES OF ELDER BERRIES (*SAMBUCUS NIGRA L.*)

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Black elder (*Sambucus nigra L.*) grows wild in several countries of Europe and is cultivated on a small scale in some European countries. It contains many health promoting substances, which could, in the form of new products, enrich the consumer market. Successful commercial use of elderberry fruits depends especially on their good flavour, which is related to the content of volatile aroma active substances[1].

The aroma of elder flowers[2] and elder berries[1,3] have been characterized before in detail by several authors and more than 100 volatiles have been identified. Jensen et al.[4] divided the aroma compounds of the elderberry into six odour classes: elderberry, flowery, fruity, grassy, agrestic and miscellaneous. The characteristic elderberry odour is related to dihydroedulan, β-damascenone and ethyl-9-decenoate. In the flowery group (Z)- and (E)-rose oxide, nerol oxide, 1-nonanal and hotrienol contribute significantly with elder flower notes, whereas linalool, α-terpineol, benzyl alcohol, 2-phenylethanol and phenylacetaldehyde contribute with flowery notes. The fruity odour of elderberry juice appears to be related to alcohols, aldehydes and esters of lower carboxylic acids and lower alcohols. The grassy group is composed of well-known aliphatic aldehydes and alcohols with typical odors of green grass. 1-octen-3-one and 1-octen-3-ol with mushroom note and 3-hydroxy-2-butanone with creamy, buttery note belong to the agrestic group. Benzaldehyde with its candy, sweet note could be placed to miscellaneous group, also lower carboxylic acids and ketones with creamy, oily or buttery odour[1,3].

The aim of this project was to identify and quantify the volatile aroma active compounds in samples of elderberries grown in Czech Republic. Wild elder and four cultivars of cultivated elderberries (Bohatka, Dana, Albida and Haschberg) were used for analysis. Aroma compounds were extracted by solid phase microextraction method and analysed by GC-FID and GC-MS. In total, 84 aroma compounds were identified and quantified, mainly alcohols, aldehydes and esters. Most of them are well known aroma compounds and have previously been detected in elderberries[1,3]. However, significant differences ($P < 0,05$) in content of single aroma compounds were found among samples, which is in accordance with other authors[1,3].

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REFERENCES

1. Kaack K., Christensen L. P., Hughes M., Eder R.: Eur. Food Res. Technol. *221*, 244 (2005).
2. Kaack K., Christensen L. P., Hughes M., Eder R.: Eur. Food Res. Technol. *223*, 57 (2006).
3. Kaack K.: Eur. Food Res. Technol. *227*, 45 (2008).
4. Jensen K., Christensen L. P., Hansen M., Jørgensen U., Kaack K.: J. Sci. Food Agric. *81*, 237 (2000).

4-P61

RELATIONSHIP BETWEEN VOLATILE COMPOUNDS CONTENT AND SENSORY ATTRIBUTES OF STERILIZED PROCESSED CHEESE

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Pasteurized processed cheese products are cheese-based food produced by comminuting, blending and melting one or more natural cheeses and optional ingredients into a smooth homogenous blend with the aid of heat, mechanical shear and usually emulsifying salts. Optional ingredients which are determined by the product type, include dairy ingredients, vegetables, meats, stabilizers, emulsifying salts, flavours, colours, preservatives and water[1,2]. Dairy ingredients means butter, cottage cheese, milk concentrates (e.g. skimmed milk powder, whey powder, caseinates) etc. Chemical composition of processed cheese depends on a processed cheese type, raw materials used[3] and manufacturing processes[4].

The aim of this project was to monitor changes of the volatile aroma active compounds and several sensory attributes in samples of sterilized processed cheeses. Sterilized cheeses were stored at 6 ± 2 °C, 23 ± 2 °C, 40 ± 2 °C during two years. Pasteurized processed cheeses used for comparison were stored only at 6 ± 2 °C during six month.

Head space solid phase microextraction (HS–SPME) was used for isolation of volatile compounds. Extracted compounds were identified and quantified by followed gas chromatography analysis.

In processed cheeses total of 23 volatile compounds were identified. They included 8 alcohols, 5 aldehydes, 5 ketones, 4 acids and 1 ester. Sterilization heating and consequent storage at higher temperature caused increase in amount of most of the aromatic compounds.

The sensory quality of processed cheese was described by scale test[5]. Differences between cheese flavours were evaluated by Kruskall-Wallis statistical test. The relationship between sensory perception and volatile compound content was determined statistically with Pearson correlation coefficients.

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REFERENCES

1. Guinee T.P., in book: *Encyclopedia of Dairy science (Roginski H., ed.)*. Academic press, p. 411, London 2003.
2. Kristensen D., Hansen E., Arndal A., Trinderup R.A., Skibsted L. H.: Int. Dairy J. *11*, 837 (2001).
3. Buňka F., Hrabě J., Kráčmar S.: Int. Dairy J. *14*, 829 (2004).
4. Kramer A., Szczesniak A.S., in book: *Texture measurements of food*, chapt. 1, p. 5. D. Riedel, Dordrecht 1973
5. Lawless H. T., Heymann H., in book: *Sensory Evaluation of food,* chapt. 7, p. 254. Kluwer Academic/Plenum Publishers, New York 1999.

4-P62

THE CONTENT OF LACTIC ACID BACTERIA IN CHOSEN PROBIOTIC FOODS

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Probiotics, live microbial food supplements that beneficially affect the host by improving its intestinal microbial balance, are quickly gaining interest as functional foods[1].

Microbes have been used for thousands years in foods and drinks, however, relatively recently have undergone scientific scrutiny to examine their purported health benefits.

Some of the claims about a beneficial effect of probiotic consumption include: improving intestinal tract health, enhancing the immune system, enhancing the bioavailability of nutrients, reducing symptoms of lactose intolerance, decreasing the prevalence of allergy, and reducing risk of certain cancers[2]. The mechanisms by which probiotics exert their effects are still speculative, but may involve modifying gut pH, antagonizing pathogens through production of antimicrobial compounds, competing pathogen by binding for receptor sites as well as for available nutrients and growth factors, stimulating immunomodulatory cells, and producing lactase[3].

The widely used probiotics are the lactic acid bacteria, particularly the *Lactobacillus* and *Bifidobacterium* species[1]. However, there are reports on the probiotic potential of *Pediococcus*, *Lactococcus*, *Bacillus* and yeasts[4]. The main criteria for use include the ability to: exert a beneficial effect on the host, survive transit through the intestinal tract, adhere to the intestinal epithelial cell lining, produce antimicrobial substances towards pathogens, stabilize the intestinal microflora. Probiotics must also have good shelf-life in foods, contain a large number of viable cells at the time of consumption and be non-pathogenic and nontoxic[3].

Worldwide, probiotics are currently available in a variety of food products and supplements, particularly in yogurts and fermented milk products, cheeses, ice creams, butter, meat products and others[1]. Studies show an effective dose of 10^9-10^{10} organisms per day, which corresponds to an intake of about a liter of fermented milk per day[3].

The aim of this work was to assess and compare the content of lactic acid bacteria in chosen probiotic foods available in Czech market. Coeuret et al.[1] also demonstrated the need to control lactobacilli present in commercial probiotic food products, because the identity and number of species do not always correspond to those stated on the labels.

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REFERENCES

1. Coeuret V., Gueguen M., Vernoux J. P.: Int. J. Food Microbiol. *97*, 147 (2004).
2. Bengmark S.: Food Technol. Biotech. *48*, 464 (2010).
3. Kopp-Hoolihan L.: J. Am. Diet. Assoc. *101*, 229 (2001).
4. Soccol C. R., Vandenberghe L. P. D., Spier M. R., Medeiros A. B. P., Yamaguishi C. T., Lindner J. D., Pandey A., Thomaz-Soccol V.: Food Technol. Biotech. *48*, 413 (2010).

4-P63

SUBSTRATE SPECIFICITY OF XYLOGLUCAN ENDOTRANSGLYCOSYLASE (XET) FROM GERMINATING SEEDS OF PARSLEY

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Enzyme xyloglucan endotransglycosylase (XET, EC 2.4.1.207) catalyzes cleavage of xyloglucan molecule (XG), after which the strand with the non-reducing end is released while the enzyme remains attached to the other strand and connect strand with newly created reducing end with a non-reducing end of another XG molecule or xyloglucan oligosaccharide (XGOS).

The typical transglycosylation reaction catalyzed by XET between substrate pairs XG/XGOS was already detected and partly characterized in most plants, including parsley. But, in recent years, a new type of transglycosylation reaction was reported, so called heterotransglycosylation, which represents the reaction, where donor and acceptor are polysaccharides of different type. This reaction is in germinating seeds of parsley probably catalysed by nonspecific form of XET, analogous to heterotransglycosylation activities in germinating seeds of nasturtium.

The fluorescently labeled oligosaccharides were used for detection of transglycosylating activities. The incorporation of donor into these acceptors was monitored in protein extracts derived from germinating seeds of parsley by HPLC method. The method of isoelectric focusing (IEF-PAGE) was used for detection of the presence of XET multiple forms.

Number of XETs with different pI in the range from 4,55 to 5,9, was determined by IEF-PAGE in parsley seeds. By HPLC method, already described activities were approved (i.g. XG/ XGOS, XG/pustulooligosaccharides), and also heterotransglycosylase activities by using new, by now unknown donor/acceptor pairs were detected, e.g. XG/laminariooligosaccharides, XG/β(1-3,1-4)-glucan oligosaccharides.

The results suggested the presence of number of XETs with different pI and also a large spectrum of donor/acceptor pairs, whose heterotransglycosylation reaction is catalyzed by nonspecific form of XET presents in germinating seeds of parsley.

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4-P64

HETEROTRANSGLYCOSYLATING ACTIVITIES IN GERMINATING SEEDS OF NASTURTIUM

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Enzyme xyloglucan endotransglycosylase (XET, EC 2.4.1.207) catalyzes cleavage of β-(1-4) glycosidic bonds of xyloglucan molecule (XG). Cleavage chain of XG containing newly created reducing end is transferred on hydroxyl group of C-4 glucosic unit of non-reducing end of XG acceptor molecule or xyloglucan oligosacharides (XGOS).

Besides typical XET-catalysed transglycosylation between substrate pairs XG/XGOS, new type of the transglycosylation reaction, called heterotransglycosylation, was described in germinating seeds of nasturtium, introducing reaction where acceptor and donor are polysaccharides of different type. These activities were assigned to nonspecific majority form of XET. The presence of other types of transglycosylases except XET, have not been confirmed on molecular level in any plants yet.

Fluorescently labeled oligosaccharides are used for detection of transglycosylating activities. The incorporation of donor to these acceptors was monitored in protein extracts derived from germinating seeds of nasturtium by means of HPLC method.

By HPLC method all described heterotransglycosylating activities except the incorporation of XG to pustulooligosaccharides (PUOS) were approved and also new activities on by now undocumented donor/acceptor pairs were detected. From seven types of polysaccharides, XET was able to accept as donor substrate except XG, also hydroxyethylcellulose and laminarin which were incorporated into fluorescent labeled XGOS. Even more oligosaccharides were able to act as acceptor substrate of this enzyme.

The incorporation of fluorescently labeled oligosaccharides into the primary cell wall was confirmed by microscopy.

The results suggest the existence of even more donor/acceptor pairs, whose reaction is catalyzed by nonspecific majority form of XET from germinating seeds of nasturtium.

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4-P65

INCORPORATION OF PUSTULO-OLIGOSACCHARIDES TO PLANT CELL WALL STRUCTURES

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Pustulan is a typical polysaccharide of the yeast cell wall. Its presence in the plant cell walls was not described. By contrast, incorporation of xyloglucan chains (XG) to sulforhodamine labeled pustulooligosaccharides (PUOS-SR) by an action of unspecific xyloglucan endotransglycosylase (XET, EC 2.4.1.207) from germinating seeds of nasturtium was detected using dot blot method.

The aim of this work was to verify this result using methods of HPLC with fluorescent detector for reaction mixtures composed from protein extract with appropriate donor substrate (unlabeled polysaccharides) and PUOS-SR as an acceptor and fluorescent microscopy for the incorporation of PUOS-SR to the cell structures of nasturtium germinating seeds.

No reaction between XG and PUOS-SR was observed by HPLC. The other described reactions, e.g. between substrate pairs XG/xyloglucan oligosaccharides, XG/cello- oligosaccharides, XG/xylo-oligosaccharides, XG/laminari- oligosaccharides and XG/1,3-1,4-gluco-oligosaccharides were confirmed. From these reasons, the reaction between PUOS-SR and eight other polysaccharides was proved. The only interaction was detected between substrate pairs hydroxyethylcellulose (HEC) /PUOS-SR and laminarin/ PUOS-SR, two known components of the cell wall of yeasts.

Whereas PUOS-SR were not incorporated in to the cell wall of germinating nasturtium, the microscopy confirmed these results. Surprisingly, PUOS-SR were built in plazmalema indicating the presence of suitable naked polysaccharide in this structure.

These results suggest the importance of use of more pararel methods for verifying of new-found heterotransglycosylating activities in plant extracts as well as a presence of an unknown enzyme in germinating nasturtium seeds with the ability to incorporate HEC and laminarin in to PUOS-SR.

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4-P66
PRODUCTION OF CELLULASE AND POLYGALACTURONASE ON PLANT ORIGIN WASTE

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Production of cellulase and polygalacturonase by *Aspergillus niger* and *Aureobasidium pullulans* was studied in submerged (SmF) and solid state fermentation (SSF) systems. Substrates used in fermentation systems were mandarin peels and grape pomace.

With *Aspergillus niger* used on grape pomace as a sole carbon source, cellulase production was detected after 72 hours in SSF and after 24 hours in SmF systems. The activity of cellulase per gram of substrate was higher in a submerged than in a solid state fermentation system. The longer time for higher polygalacturonase production was necessary in submerged fermentation systems and polygalacturonase activity was higher in SmF. The SSF fermentation with mandarin peels as a sole carbon source was similar, the highest detected activity of cellulase was determined after 72 hours. Different production of polygalacturonase was observed on mandarin peels in SmF systems. A comparison of enzyme productivities on grape pomace and mandarin peels showed that polygalacturonase activity per gram of substrate is highest in SmF system with mandarin peels as a sole carbon source.

With *Aureobasidium pullulans* used on grape pomace as a sole carbon source, cellulase production was detected after 48 hours in SmF and SSF fermentation systems. The activity of cellulase per gram of substrate was higher in solid state system than in a submerged fermentation system. Longer time for higher polygalacturonase production was necessary in both fermentation systems. Polygalacturonase activity was higher in SmF. The SSF fermentation with mandarin peels as a sole carbon source was similar, the highest detected activity of cellulase was determined after 48 hours. Different production of polygalacturonase was observed on mandarin peels in SmF systems. A comparison of enzyme productivities on grape pomace and mandarin peels showed that polygalacturonase activity per gram of substrate is highest in SmF system with mandarin peels as a sole carbon source. For both systems and both substrates manganese-dependent peroxidase was detected for the first time.

Differences in the enzyme synthesis by *Aspergillus niger* and *Aureobasidium pullulans* depend on both the substrates used as well as on the fermentation system.

4-P67
MONITORING AND DEGRADATION OF PESTICIDES WITHIN VEGETABLE GROWING IN THE SYSTEM OF INTEGRATED CROP PROTECTION

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Pesticides have become an integral part of modern agriculture, they are chemical substances used primarily to protect crops during the pre-harvest period and storage[1]. Potential adverse effects on non-target organisms, including also humans, is one of the main disadvantage of their application. Therefore, the maximum amount of these substances in food is regulated by the maximum residue limits (MRL). Monitoring of pesticide residues is carried out both at national and international level e.g. within European Union.

Nowadays consumers prefer healthy products of higher quality thus more demands are placed particularly on the production that leads also to minimization of pesticides residues. In this context is used integrated crops management. Aim is such measure, which lead to exclusion of unilateral application of chemical protection.The ultimate goal is to achieve high-quality production of crops by ecologically appropriate and economically acceptable methods.

Within this study will be presented results of analyzes of pesticides residues that are a part of the project focused on the protection of the main species of field vegetables against pests.Monitoring of pesticide residues in samples of vegetables (celery, onion, cauliflower, carrots, lettuce,...) from small farmers was the first part of our study. In none of the sixteen analyzed samples of vegetables were not exceeded MRLs.

The next task was to study the dynamics of pesticide residues from small plot experiments (model treatments of parsley). Selected pesticide preparations (10 active substances) were applied on parsley and samples were taken to analysis of pesticides at weekly intervals (three replicates).The MRL was exceeded only by spinosadin parsley sampled three days after pesticide application (0,028 ± 0,008 mg kg^{-1}, MRL= 0,020 mg kg^{-1}). No other active substances have exceeded the legislation limit. The distribution of analytes in parsley was also determined by comparison of amount of residues in a root and leaves.The greatest amount of pesticides residues was found for azoxystrobin which was present in the parsley root and in the parsley leaves too, but on the other hand, azoxystrobin showed the largest percentage decline over time.

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REFERENCES

1. Alder L., Greulich K., Kempe G., Vieth B.: Mass Spectrom. Rev. *25*, 838 (2006).

4-P68

MOLECULAR IDENTIFICATION OF SELECTED SPECIES OF LACTIC ACID BACTERIA AND BIFIDOBACTERIA IN FOOD ADDITIVES

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Lactic acid bacteria (LAB) are a heterogenous group of immobile, microaerofilic or anaerobic, Gram-positive microorgasnisms with ferment different types of nutrients to produce lactic acid. Nowdays LAB include some species of genus *Lactococcus, Streptococcus, Enterococcus, Lactobacillus, Pediococcus.*Bifidobacteria are an individual group. LAB of genus *Lactobacillus* and bifidobacteria are important in food fermentation, food preservation and as starters for dairy products. Many strains of different species are known as probiotics. Probiotics are „live microorganisms which, when administered in adequate amounts, confer health benefit on the host“[1]. Nowadays, probiotic bacteria are used as food additives in food processing industry. Methods of molecular biology based on DNA amplification can be used for detection of probiotics in complex samples such as food product or food additives.[2]

The aim of this work was to isolate DNA from complex matrices of three batches of 2 probiotic food additives and to detect target bacterial DNA using PCR. Total DNA was isolated from crude cell lysates of food additives Linex® Forte and PangaminBifi Plus. Magnetic nonporous (P(HEMA-*co*-GMA)) particles were used for DNA isolation. These particles are coated by carboxyl groups that inables reversible adsorbtionof DNA[3]. The concentration of isolated DNA was checked by spectrofotometric measurement. Differences among quantity of isolated DNA from three batches of food additives were estimated. Isolated DNA was amplified in genus-specific (with primers specific for genus *Lactobacillus* and *Bifidobacterium*) and species-specific PCRs (with primers specific for *Lactobacillus acidophilus* and *Bifidobacterium-animalis*). Results were compared with declared specification given by producer.

It was shown that declared bacteria are in accord with detected bacteria in both products. It was also shown that species *Lactobacillus acidophilus* and *Bifidobacterium-animalis* are present it both products.

REFERENCES:

1. Giraffa, G., Chanishvili, N., Widyastuti, Y.: Res. Microbiol. *161*, 480 (2010).
2. Bernardeau M., Guguen M., Vernaoux J. P.: FEMS Microbiol. Rev. *30*, 487 (2006).
3. Rittich B., Španová A., Horák D., Beneš M. J., Klesnilová L., Petrová K., Rybnikář A.: Colloid. Surf. B *52*, 143 (2006).

4-P69

THE ROLE OF ENTEROCOCCI IN TYRAMINE FORMATION IN TYPICAL CZECH FERMENTED SAUSAGES HERKULES

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The aim of this study was isolation, identification and characterization of bacteria of the genus Enterococcus from probiotic Czech fermented sausages Herkules and evaluation of their role in biogenic amine tyramine formation during fermentation, ripening and storage of those sausages. Sausages were made at two different producers (A and B). All together, two types of products were made at each producer, according to the traditional recepture: probiotic Herkules, where probiotic strain L. casei 01 was applied and typical traditional Herkules as a control. The sausages were fermented at 15°C for 28 days and as final products storaged at 6,0 ± 0,5 °C during the whole expiration period (21 days after production). In all sausages, numbers of single probiotic cells, numbers of enterococci and concentration of tyramine were monitored during the fermentation and during the whole expiration period of products (storage at 6,0 ± 0,5 °C) . The samples were taken each 7 days for analysis. In probiotic sausages, the numbers of probiotics during the whole analysis were higher than 10^6 KTJ/g of a product. Altogether 380 suspected enterococci isolates were obtained from sausages samples during analysis. Using genus-specific PCR, 370 isolates were identified as the members of the genus Enterococcus. For identification of isolates into the species level, species-specific PCR was used. Among 350 Enterococcus spp. isolates were classified as E. faecium and 20 as E. faecalis. The gene coding for tyrosine decarboxylase was detected in all enterococci isolates. All enterococci strains produced biogenic amine tyramine when cultivated in decarboxylase medium and analysed by HPLC. At both producers, enterococci counts in the non-probiotic sausages were higher in comparison with probiotic sausages during fermentation. Even more, no enterococci were detected in probiotic sausages during the storage period. It seems that tyramine production in dry sausages is higly connected with the presence of enterococci. In probiotic sausages of both producers, the concentration of tyramine was significantly lower than in controls without probiotics. Reducing of enterococci counts connected with lower production of tyramine should probably be caused by probiotic strain L. casei 01 which makes it very usable strain in fermented sausage manufacture.

4-P70

CAROTENOIDS AND FATTY ACIDS PROFILE AND THEIR QUANTIFICATION IN SOME WILD AND CULTIVATED BLUEBERRIES FROM ROMANIA

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Blueberries (*Vacccinium myrtillus*) are often consumed worldwide because their beneficial effects on human health, most of them related to the antioxidant properties. They contain micronutrients, various phytochemicals and antioxidant compounds, represented especially by anthocyanins[1-3]. Carotenoids are lipophilic compounds that can contribute to the overall antioxidant capacity of blueberries. The aim of this study was the identification and quantification of carotenoids by RP-HPLC-PDA and fatty acids using Gas Chromatography (GC) with flame ionization detection (FID) in two wild blueberries and three cultivated species, BlueCrop, Elliot and Duke. The total carotenoids content was generally higher for wild blueberries than in cultivated ones. The main carotenoids identified were lutein and β-carotene. The main fatty acids identified in all samples were the polyunsaturated linoleic (18:2) and linolenic (18:3) acids. The ratio between PUFA/SFA is 8,54 for Wild 2, 10,16 for Wild 1 and between 3,51-6,87 for cultivated blueberries, which indicate a high degree of unsaturation.

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REFERENCES

1. Marinova D., Ribarova F.: J. Food Compos. Anal. *20*, 370 (2007).
2. Mazza G., Cottrell T.: J. Food Compos. Anal. *21,* 249 (2008).
3. Wannes W. A., Mhamdi B., Marzouk B.: Food Chem. *112,* 62 (2009).

4-P71

SEPARATION AND QUANTIFICATION OF CAROTENOIDS FROM GRAPES CULTIVATED IN CONVENTIONAL AND ORGANIC SYSTEMS

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Organic agriculture involve plants which are cultivated without using synthetic pesticides, herbicides or fertilizers and promotes biodiversity, biological cycles and improve the product quality. There are few studies regarding the impact of organic agricultural on plant metabolites level. Among other secondary metabolites, carotenoids play important roles for the total antioxidant activity of grapes (*Vitis vinifera* sp.) and for their nutritional value[1,2]. The aim of this study was to evaluate the carotenoid concentrations in some table and wine grapes cultivated in two different practices: organic and conventional. The carotenoids were separated and quantified using high performance liquid chromatography. The main carotenoids in all grape samples analysed were lutein and ß-carotene. Fetească regală wine variety was found to have the highest carotenoids content in both, conventional and organic cultivation systems, significantly higher than all other varieties. In the case of table grape varieties, the average concentration of ß-carotene was significantly higher in the case of organic culture than in conventional ones.

REFERENCES

1. Oliveira C., Silva Ferreira A. C., Mendez Pinto M., Hogg T., Alves F., Guedes de Pinho P.: J. Agric. Food Chem., *57*, 5967 (2003).
2. Crupi P., Coletta A., Milella R. A., Palmisano G., Baiano A., La Notte E., Antonacci D.: J. Food Sci., *75*, S191 (2010).

4-P72

IDENTIFICATION OF SOYBEAN IN THE FOODSTUFF BY REAL-TIME PCR METHOD

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We used real-time PCR oriented on gene *lec* that coded lectin specified to soy for soybean detection in the food. On this target sequence was directed PCR system with primers Le2F and Le2Rbis and Le2P Taqman type probe. In the GenBank database, there has been no homologous sequence in related species. The ability for amplification of DNA of all samples was verified by system TR03/04/Pb. We determined inclusiveness and exclusiveness of the system and we found detection limit (2,75 pg) and the practical detection limit (0,02%). The whole system designed for the detection of soy in food products was tested on real food samples from the normal distribution network. The results were generally consistent with data on the content of soy foods in their packaging.

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4-P73

TARTRAZINE DETERMINATION FROM MUSTARD SAMPLE BY HPLC, TLC-PHOTODENSITOMETRY AND TLC- DIGITAL PROCESSING OF IMAGES

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Mustard seeds are known for long time ago, being used in traditional medicine due to them composition rich in bioactive compounds, minerals and vitamins inclusive riboflavine. Nowadays, mustard seeds are used to prepare one of the most popular food product – the mustard. From commercial reason the product is colored with natural colorants such as riboflavin and β-carotene or synthetic dyes such as tratrazine. The Romanian state regulation accepts tartrazine in this kind of foods in quantities bellow 300 mg kg^{-1}.

The food dye was extracted from solid matrix by ultrasound assisted extraction (UAE) with a methanol - ammonia mixture. The extract was further purified by SPE. Recovery determination was carried out on mustard samples spiked with tartrazine at different concentration levels. Good recoveries (R>95%) and low relative standard deviation were obtained.

Tartrazine determination was performed by using two chromatographic methods TLC and HPLC. Calibration curves method was used for quantitative determination.

An extensive study for quantitative evaluation of dyes was developed by comparing the photodensitometric results with those obtained by using different software (Image Decipher-TLC, Sorbfil TLC) for digital processing and evaluation of the chromatographic plates.

LOD and LOQ values for the synthetic dye were also determined for each method and compared. The methods were applied for tartrazine determination in various mustard samples purchased from a local market.

4-P74

THE POTENTIALITIES OF MULTI-ELEMENT COMPOSITION AND ^{87}Sr/^{86}Sr RATIO OF WINES AS TRACES OF WINE PROVENANCE

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A suitable method for determination of the Sr isotope ratio ^{87}Sr/^{86}Sr[1] in wines by inductively coupled plasma MS (ICP-MS) was developed to be used as a fingerprint technique for differentiation of Murfatlar and Dealu Mare-Valea Calugareasca wine regions from Romania.

To corectly distinguish between wine and soil samples originating from a specific region, a combination of ^{87}Sr/^{86}Sr and multielement data (Sr, Mn, Cr, Pb, Ni, Ag, Cd and Cu) was obtained and compared. Elements can be considered as good indicators of wine origin, since they are not metabolized or modified during the vinification process[2]. The mineral element content and the stable isotopes ratio have been shown to be useful in the determination of the geographical elaboration of data with a discrimination of 100%[3]. While the metal content is able to differentiate with great accuracy wines according to the geographical origin, the stable isotope rations is a useful indicator to differentiate the year of production.

REFERENCES

1. Suhaj M., Korenovska M.: Acta Aliment. Hung. *34*, 393 (2005).
2. Arvanitoyannis I. S., Katsota M. N., Psarra E. P., Soufleros E. H., Almeida C. M. R., Vasconcelos M. T. S. D.: J. Anal. Atom. Spectrom. *16*, 607 (2001).
3. Giaccio M., Vicentini A.: J. Commodity Sci. Technol. Quality *47*, 267 (2008).

P75

DETERMINATION OF DILL ESSENTIAL OIL COMPOSITION USING MODERN EXTRACTION TECHNIQUES

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The essential oils are aromatic, volatile liquids extracted from plant parts such as leaves, flowers, fruits, seeds, roots, barks and stems. The essential oils have many applications: therapeutic, in perfume and cosmetic industry[1-5]. The objective of this work was to extract, separate and determine the essential oils from indigenous dill (*Anethum graveolens*), using various modern extraction techniques.

Three different techniques (maceration, ultrasonic and microwave-assisted solvent extraction) were used for extraction of essential volatile oils from dill. The extraction was performed using various organic solvents, like: hexane, diethyl ether, ethanol, and mixtures of the solvents in 1:1, v/v ratio. The extracts were analyzed by HPTLC technique and the fingerprint information was obtained. The GC-FID method was used to characterize the extraction efficiency and to identify the essential oils. The most efficient extraction technique was maceration, but unfortunately requires a longer period of time. By the two modern extraction methods (ultrasonic and microwave-assisted solvent extraction), an amount comparable to that obtained by maceration was acquired, but in much shorter time, which is a great advantage. In dill extracts the following components were identified: α-phellandrene, β-phellandrene, dill ether, limonene, carvone, myristicin, apiole, α-pinene, β-ocimene, myrcene, *p*-cymene, and other oil constituents.

REFERENCES

1. Ortan A., Popescu M. L., Gaita A. L., Dinu-Pirvu C., Campeanu G.: Rom. Biotech. Lett. *14*, 4342 (2009).
2. Jirovetz L., Buchbauer G., Stoyanova A. S., Georgiev E. V., Damianova S. T.: J. Agric. Food Chem. *51*, 3854 (2003).
3. Stavri M., Gibbons S.: Phytother Res. *19*, 938 (2005).
4. Bakkali F., Averbeck S., Averbeck D., Idaomar M.: Food Chem. Toxicol. *46*, 446 (2008).
5. Aburjai T., Natsheh F. M.: Phytother Res. *17*, 987 (2003).

4-P76

LOW-LEVELS SPECIFICATION OF ELEMENTS IN DRINKING WATERS USING ICP-MS

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Trace metals concentration in water is a major component in the determination of water quality. In this work a rapid and accurate quantitative method for the determination of trace amounts of metals in some spring and mineral waters was successfully developed by using an inductively coupled plasma mass spectrometry. Method validation consisted of testing the parameters of linearity, precision, accuracy, limit of detection, limit of quantification and uncertainty of measurement. For drinking water quality all the results have been compared with standards. The validated method has been used for determination of selected metals (Al, Cr, Mn, Cu, Zn, Pb, Cd, Fe and Se) in four spring waters in order to assess any toxicological problem.

4-P77

METALLOTHIONEINS UPREGULATION IN LIVER OF THE MOUSE *MUS MUSCULUS* UNDER CADMIUM EXPOSURE

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The presence of metals in environmental issues generally provokes upregulation of different metal-containing biomolecules, especially metallothioneins conventionally considered as a metal scavenger agent in the exposed organisms. Metallothioneins are low molecular proteins (about 7 kDa) enriched in cysteine groups that easily complex metals such as Cu, Zn, Cd and Hg, for this reason they have been used as conventional biomarkers in environmental studies.

Metallomic studies of the free-living mouse *Mus spretus* have been proposed for environmental pollution assessment[1] but usually is difficult to interpretate the changes of protein expression caused by the presence of contaminants. For this reason exposure experiments have been performed in the laboratory mouse *Mus musculus*, genetically close to *Mus spretus*, that can be used as model organism.

Several complementary chromatographic techniques (SEC and AEC) have been coupled to ICP-MS to trace the presence of Cd-, Cu- and Zn-bound biomolecules in exposure experiment to Cd performed on *Mus musculus*. Exposure experiment was carry out by subcutaneous injection of a growing dose of 0,1 to 1,0 mg of Cd per Kg of body weight per day. Several soubgroups was sacrified in different days and the liver was extracted for the study. A control group injected with ultrapure water was used as reference.

It is remarkable the presence of a Cd-peak at about 7kDa, at the same retention time that the standard of metallothionein-I, which increase with Cd exposure. This peak was collected and separated by SEC and purified by AEC-ICP-MS, which results in different peaks containing Cu, Zn and Cd in different ratio that can be correlated with MT isoforms. Further studies for the identification of these isoforms by MALDI-TOF-MS have been performed.

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REFERENCES

1. González-Fernández M., García-Sevillano M. A., Jara-Biedma R., García-Barrera T., Vioque A., López-Barea J., Pueyo C., Gómez-Ariza J. L.: J. Anal. At. Spectrom. *26*, 141 (2011).

4-P78

ENZYMATIC AND ANTIOXIDANT ACTIVITY CHARACTERISTIC OF THE VARIETIES *TRITICUM AESTIVUM,* L. AND *TRITICUM DURUM,* L.

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In this work we evaluated the protein-carbohydrate complex, water activity, antioxidant activity and polyphenol content of durum wheat *(Triticum durum L.)*** and winter wheat *(Triticum aestivum L.)**. Both wheat are important raw materials for the production of flours for the bakery and pasta industry. Slovak varieties Karpatia* and Pentadur** were evaluated. From the Central European region the Serbian varieties: Etida*, Vojvodina*, Pobeda*, Renesansa*, Rusija*, NS Dur ** and Durumko** were evaluated.

Optimal activity of alpha-amylase according to averaging values of falling number showed varieties Etida (212s), Pobeda (214s) Renesansa (277s). The highest values of crude protein were measured in varieties Karpatia and Durumko above 13,50%. The content of insoluble protein (gluten) was above average high in variety Rusija 36,31%. The highest Zeleny index was measured in variety Karpatia. In the Slovak varieties Karpatia and Pentadur we measured the highest values of starch. For the others varieties it ranged from 54,82% - 60,64%.

Water activity measured at 25 °C was with regard to varieties uniform. The highest values were measured in varieties Pobeda and Pentadur (0,506/0,503). Variety Renesansa showed the highest value of antioxidant activity by DPPH method (48,5%) and variety Durumko 46,0%. Values of polyphenols content ranged from 0,117 to 0,477 mg of catechin/g sample, in all varieties.

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4-P79

THE ANTIOXIDANT EFFECT OF HERBAL EXTRACTS IN COMPARISION WITH SYNTHETIC ANTIOXIDANTS.

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Herbal extracts are considered as a good sources of antioxidant compounds[1,2]. This work describes antioxidant effect of 10 kinds of herbs. Two different antioxidant assays were used – test based on deoxyribose oxidation (dR) and DPPH method. Deoxyribose assay showed the antioxidant effect of selected extracts expossed as the inhibition of formation of oxidative products of deoxyribose from 3,97 to 50,05 %. The DPPH method values ranged from 6,47 to 73,80 %. Extract of *Prunus spinosa* L., *Euphrasia rostkoviana* Hayne were the best antioxidants at DPPH method. *Prunus spinosa* L. and *Bellis perennis* L. extracts showed the strongest antioxidant effect determined by deoxyribose assay. Antioxidant effect of herbal extracts was also compared to synthetic antioxidants (BHT, ascorbic acid and phenolic acids). Herbal extracts can be utillised in selected combinations to improve sensory and functional properties of some kinds of beverages.

REFERENCES

1. Hinneburg I., Damien Dorman H. J., Raimo Hiltunen R.: Food Chem. *97*, 122 (2006).
2. Proestos C. H., Boziaris I. S., Kapsokefalou M., Komaitis M.: Food Technol. Biotechnol. *46*, 151 (2008).

4-P80

ANALYSIS OF PHYTOESTROGENS IN MILK SAMPLES

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The feedingstuffs containing soy, legumes and (or) red clover-are a source of isoflavones which can be excreted into milk of farm animals, thus transferred into the human food chain. Isoflavones are biologically active compounds classified as phytoetsrogens because their structures resemble to mammalian steroidal estrogens and they have a weak affinity for the estrogens receptors. Many animal and human clinical as well as epidemiological studies demonstrate the association of soybean isoflavones with the prevention of human cancers, cardiovascular diseases, osteoporosis and the postmenopausal symptoms.

To learn more on the fate of isoflavons in cows, feeding experiments were established. In the first phase of our study, suitable analytical method had to be developed. The performance characteristics achievable in two instrumental systems consisting of high / ultra high performance liquid chromatography (HPLC/UPLC) coupled to tandem mass spectrometer (MS/MS) were evaluated, one of the key crite-rium was low limit of quantification (LOQ) to enable control of both parent compounds and their transformation products. In this respect, UPLC coupled to 5500 Q-TRAP MS (Applied Biosystems) analytical system provided the best results, moreover it enabled reduction of analysis time to only few minutes.

Using this system, the concentration of isoflavones in milk samples, at the time of feeding by isoflavones based diet, their levels were determined to range between 8,8 – 59,6 ng ml^{-1} for daidzein, 20,6 – 237,0 ng ml^{-1} for genistein, 2,1 – 38,0 for equol and 2,1 – 38,4 ng ml^{-1} for glycitein. These experiments have shown an interesting way how to increase dietary intake of plant phytoestrogens through animal products.

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4-P81

RAPID ALCOHOL DETERMINATION IN SPIRIT DRINKS AND WINES USING MULTIVARIATE CALIBRATION WITH PRINCIPLE COMPONENT REGRESSION OF NIR DERIVATIVE SPECTRA

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The detection and quantification of alcohols with high sensitivity, selectivity and accuracy is required in many different areas[1,2]. The food, beverage (wine, beer and spirits) require simple, fast and economic analytical methods in order to control fermentation processes and the quality of final products. Many analytical methods have been developed during the years for the determination of ethanol. Although some of these methods are precise and reliable, they are complex, time consuming and require previous separations processes (distillation, pervaporation), expensive instrumentation and trained operators. A simple method using NIR transmision spectrometry and multivariate calibration was performed for verification of ethanol concentration in fruit spirit drinks and in organic and conventional wines.

The transmission spectra of spirits and wines were measured in a range 1370 – 1580 nm in the 1 mm quartz cell (QX-Suprasil, Hellma) by Shimadzu UV-3600 spectrometer (Kyoto, Japan). Ethanol values were calculated by using the Shimadzu Special Edition software Panorana Quantify, v.3.1.16.0 (LabCognition, GmbH & Co. KG) enabling multivariate calibration by method of principle component regression (PCR). All spectra were smoothed with the Savitzky-Golay method and transformed to the first derivative forms to develop the PCR model because derivatives of orders higher than two are more sensitive to noise and have not been shown to have an advantage in calibration. Multivariate calibration resulted with correlation coefficients (R^2 0,9999) and with the standard error of cross-validation (SECV 0,46%). The content of ethanol in sixty eight fruit spirits was found at average concentration of 49.4% with the standard deviation 0,42%. In the case of alcohol determination in seventeen organic and twenty eight conventional wines a compliance was found between the declared and determined alcohol content while maximum difference did not exceed 1%.

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REFERENCES
1. Bűning-Pfaue H.: Food Chem. *82*, 107 (2003).
2. Gonzáles-Rodríguez J., Pérez-Juan P., Lugue de Astro M. D.: Talanta *59,* 691 (2003).

4-P82

MULTIVARIATE EVALUATION OF BREAD COLOUR CHANGES AFFECTED BY SOME POTENTIAL ADDITIVES FOR ACRYLAMIDE MITIGATION

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Acrylamide possible mitigation strategy in a potato bread crust with some additive inorganic salts application was studied with respect to colour evaluation by multivariate statistics. Impact of CaCl2, Ca^{2+} lactate, NH4Cl, NaH2PO4, KH2PO4, Na2H2P2O7 and Na4P2O7 additives on acrylamide creation in potato bread crust was evaluated and colour differences measured by 30 colour characteristics in CIEXYZ, CIEL*a*b*, CIEL*u*v*, CIEL*C*H° and HunterL*a*b* colour spaces. The reflection spectra were recorded using a Shimadzu UV-3600 spectrophotometer (Kyoto, Japan) with Large Integrating Sphere Assembly LISR 3100 (Shimadzu, Kyoto, Japan) enabling reflection measurements from the surface of defined upper layer of dried and grounded bread crust samples. Each crust sample was measured at six different position on ground crust sample surface and the whole visible reflection spectrum (380–770 nm) was recorded. Colour values were calculated by using the Shimadzu Special Edition software ColorLite v3.1.16. Relative changes in colour of bread crust were not very intensive for human sensorial observation, perception and recognition. From all the examined colour variables only 7 significantly correlated with acrylamide content. Colour characteristics b* from CIELab and HunterLab colour space affirmed their relation to acrylamide creation, but the most intensive correlation with acrylamide was found for hue value v* derived from CIEL*u*v* colour space. Results showed that the addition of inorganic salts hasn't expected considerable elimination impact in the bread crust, but creation of acrylamide in a home bread maker's conditions was especially low in comparison to industrial ones. This gives an additional study on potentiality of acrylamide reduction in some industrial foods.

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4-P83

DETERMINATION AND COMPARATIVE STUDY OF SOLUBLE OXALATE IN GRAIN AMARANTH MUTANT LINES

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Grain amaranth (Amaranthus spp.) is a widely know pseudocereal with interesting nutritional characteristics including proteins, well suited to human nutritional needs. Its highly nutritious gluten-free grain make amaranth a good alternative to cereals or legumes and can be used in gluten-free diet. However, there are also naturally occurring anti-nutrients like oxalate that represent potential risk factor for kidney stone development and its presence in food lowers calcium and magnesium availability[1-4].

Soluble oxalate with potentially dietary injurious implications for human health were determined in some amaranth gamma-irradiation induced mutant lines by capillary isotachophoresis and compared to their reference genotypes Amaranthus cruentus Ficha and hybrid K-433 during the cultivation period 2006-2010. Factor and canonical discriminant analysis demonstrated significant differences among reference and mutant lines. Kruskal-Wallis one-way analysis of variance and multiple comparisons with t distribution confirmed statistical significant differences ($p \leq 0{,}01$) among compared amaranth genotypes. Five out of nine tested lines were identified as variants with the significant and long-term lower soluble oxalate concentration in comparison to respective reference amaranth genotypes. Detailed analysis of other biochemical traits in these lines showed also improved nutritional quality of the grains over the control samples. Therefore, this plant material might be a good matrix in the further breeding programme of this crop.

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REFERENCES
1. Hönow R., Hesse A.: Food Chem. *78*, 511 (2002).
2. Chai W., Liebman M.: J. Food Comp. Anal. *18*, 723 (2005)
3. Judprasong K., Charoenkiatkul S., Sungpuag P., Vasanachitt K., Nakjamanong Y.: J. Food Comp. Anal. *19*, 340 (2005).
4. Lilbert B., Franceschi V. R.: J. Agric. Food Chem. *35*, 926 (1987).

4-P84

CADMIUM, LEAD AND MERCURY CONTENTS IN FISHES

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Fish meat is a perfect foodstuff which is up to standard of rational nourishment. It is source of healthy and good digestible material rich on proteins, minerals and vitamins. Fish muscles especially back and lateral muscles are the most important parts of fish organism consumed for escellent chemical composition. Proteins in fish meat are rich on high aminoacids content. The content of fish fat is usually low with the high proportion of unsaturated fatty acids. Also minerals and B, A and D vitamins are very important components of this foodstuff. According to rational nourishment the fish meat should be consumed minimal 2-3 times weakly.

Our research was focused on analysis of bottomn sediments in water reservoir Kolinany from the aspect of Cd, Hg and Pb contents, the determination of observed heavy metal contents in different parts of carp body and the evaluation of hygienic status and suitability of fish meat for the human consumption[1,2]. Our results confirmed hygienic harmless of bottom sediments in water reservoir Kolinany. The Cd, Pb and Hg contents in sediments represent no risk their input into the fish organisms. The Cd content in fish meat is lower than maximal available amount given by legislative norms, but in selected parts of fish organism such as skin, gills and fins the Cd hygienic limit is 2,9 – 6,6 times exceeded. The Pb content in fish meat is under the hygienic limit, only in skin, gills and fins is the content of this heavy metal 1,31- 2,64 higher then maximal available value given by legislative. Fish skin, gills and fins belong to the non cosumed parts of fish body by people. The Hg content in fish meat is also lower than hygienc limit. The highest Hg content was observed in fish muscles (0,0544 mg kg^{-1}) and the lowest one in fish gonads (0,0058 mg kg^{-1}). The results of Cd, Pb and Hg content determination in carp body confirmed that fish muscles belong to suitable foodstuffs for the human consumption.

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REFERENCES
1. Andreji J., Stráňai I., Massányi P., Valent M.: J. Environ. Sci. Health *40*, 899 (2005).
2. Andreji J., Stráňai I., Massányi P., Kačániová M.,Valent M.: J. Environ. Sci. Health *41*, 1071 (2006).

4-P85
CONCENTRATIONS OF COMPOUNDS SULPHUR IMMISIONS IN ATMOSPHERE IN RELATION TO EVALUATION OF AGRICULTURAL CROPS YIELD DAMAGES

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In this work we have tried to highlight the issue, which is in our modern times very actual for the quality and condition of the air and ground atmosphere in the Upper Nitra region. This area belongs to very polluted areas in Slovakia. The biggest air polluter in the region Nováky is the chemical company Duslo Šaľa in Zemianské Kostoľany.

Measurements of ground concentrations of SO_2 compounds immisions and their local gradient were carried out in 2006 - 2009 on sites Bystričany, Diviaky nad Nitricou and PD Močenok. These mentioned sites are localities with important agricultural production from the standpoint of atmosphere pollution as well as hygienic state of grown agricultural raw materials and commodities. Average annual concentrations were evaluated to possible decline of agricultural crops yield by determining of factor for calculation of its real coefficient of reducing. Results of measurements were expressed as local gradient representing for the whole period on site Bystričany 19,5 mg, Diviaky 19,1 mg and Močenok in average 16,5 mg. m^{-2} d^{-1} SO_2. Values of gradient were calculated on content concentration: Bystričany 25,0 µg, Diviaky 24,8 µg and Močenok 21,4 µg m^{-3} SO_2.

For finding of possible reducing of agricultural commodities yield in plant production under real immisions conditions, the coefficient was calculated for correction of table value of the coefficient of yield reducing for tested crops. It represented factor in average for the whole monitored period in locality Bystričany 0,606, Diviaky nad Nitricou 0,617 and PD Močenok 0,534.

Approximation of the results in practice derives from characteristics of surveyed localities from the standpoint of level of their contamination by industrial immisions of sulphur and afterwards in gaining of calculated coefficients for calculation of real size of agricultural production reducing and size of compensation of arose of damages.

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4-P86
CAN BE CRANBERRIES RISKY?

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At present the interest in cranberries as fruit with excellent beneficial effect on the human health is increased. Cranberries (*Vaccinium vitis – idaea* L.) which are rich on anthocyanins, polyphenols, flavonoids and other components with high antioxidant activity belong to the healthiest foodstuffs. The aim of this work was to determine the content of selected heavy metals and bioactive compounds in wild cranberries collected from two different localities (West Tatras, Gelnica) of the Slovak Republic and to compare them with properties of three cultivated cranberry cultivars (Koralle, Ida and Runo Bielawskie) from research centre Kriva. The total content of anthocyanins (TA) was assessed by modified method of Lapornik et al. (2005). The determination of total polyphenol content (TP) was realised according Lachman et al. (2003) using Folin-Ciocalteu reagent. Antioxidant activity (AA) was determined by the method of Brand-Williams (1995) using DPPH (2,2-diphenyl-1-picrylhydrazyl). The used analytical method was spectrophotometry (Shimadzu UV/VIS-1240). The content of heavy metals was determined by AAS method (VARIAN AA 240 FS) after microwave decomposition (MARS X-press) of cranberry samples. The highest content of bioactive compounds was determined in medium early cultivar Ida (TA 875,41 mg kg^{-1}, TP 1803,68 mg kg^{-1}, AA 86,48%), the lowest one in wild cranberries from Gelnica (TA 326,93 mg kg^{-1}, TP 1457,11 mg.kg^{-1}, AA 77,85%). The Pb content was in all analysed cranberry samples 8 – 11 times higher than maximal available value given by Food Codex of Slovak Republic. The highest Pb content was determined in cv. Runo Bielawskie, the lowest one in cv. Ida. Also hygienic limit given by legislative for Cd content was in all investigated cranberry samples exceeded (1,6 times in cv. Koralle till threefold in cv. Runo Bielawskie). In two cranberry samples also Zn content was higher than hygienic limit (cv. Ida 14%, cv. Runo Bielawskie 20%).

The obtained results confirmed the suggestion that cranberries are the excellent source of bioactive compounds. On the other hand it is necessary to monitor risky element contents in this fruit, because cranberries can be also risky from the aspect of high contents of heavy metals.

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4-P87

AROMA ACTIVE COMPOUNDS IN SEVERAL TYPES OF PROCESSED CHEESE ANALOGUES

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Cheese analogues may be classified as cheese substitutes or imitations, which partly or wholly substitute or imitate cheese and in which milk fat, milk protein or both are partially or wholly replaced by non-milk-based components, principally of vegetable origin[1]. Nowadays they are widely used in the food industry as an ingredient for prepared foods such as pizza, lasagne, cordon blue products etc. They are being used increasingly due to their cost-effectiveness, attributable to the simplicity of their manufacture, which is almost identical with the classic manufacturing of processed cheese, and to the replacement of selected milk ingredients by cheaper vegetable products. Ingredients such as rennet casein, vegetable oils or fats, salts, acids and flavourings are generally used in the manufacture of imitation cheese[1,2]. However, replacement of milk fat by vegetable fats causes typical textural defects (especially worse melting properties) and insufficient flavour of the product. On the other hand, it contributes to a more favourable ratio of saturated and unsaturated fats and to the reduced cholesterol content. In the Czech Republic, cheese analogues form about 10% of the total production of processed products.

The flavour of cheese is derived from a number of aroma active compounds. From the chemical point of view they are mostly volatile compounds, including alcohols, aldehydes, ketones, fatty acids, esters, lactones, terpenes etc. Lipids, lactose and proteins are the main precursors for formation of these compounds[3].

The aim of this study was to identify and quantify aroma active compounds in cheese analogues containing various types of vegetable fats (coconut fat, palm oil, sunflower oil). Aroma compounds were isolated by solid-phase microextraction and analysed by gas chromatography. This method is simple and fast and minimizes thermal, mechanical, and chemical modifications of the sample. In total, 33 aroma compounds were identified and quantified, 6 aldehydes, 7 ketons, 13 alcohols, 3 esters and 3 fatty acids. Most of them are well known aroma compounds. However, significant differences ($P < 0,05$) in content of single aroma compounds were found among samples of cheese analogues.

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REFERENCES

1. Guinee T. P., Carić M., Kaláb M., in the book: *Cheese: Chemistry, Physics and Microbiology*, p. 349., Elsevier Applied Science, London 1997.
2. Bachmann H. P.: Int. Dairy J. *11*, 505 (2001).
3. Sunesen L. O., Lund P., Sorensen J., Holmer G.: Lebensm. Wiss. Technol. *35*, 128, (2002).

4-P88

THE CONTENT OF AROMA COMPOUNDS IN FERMENTED MILK PRODUCTS

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Fermented milk products are dairy foods that have been fermented with lactic acid bacteria such as *Lactobacillus*, *Lactococcus*, *Leuconostoc* and *Bifidobacterium*. The fermentation process increases the shelf-life[1], improves the digestibility[2] and contributes to the taste and aroma (flavour) of the product. The bases of flavour are the aroma active compounds, which are created during fermentation process and thermal treatment of foods from their precursors[3,4]. The understanding of conditions and regularities of aroma compounds creation and their influence on sensory perception of flavour is necessary for production of high-quality products.

Technological process of production consists of several steps. The fermentation phase, where not only characteristic taste and aroma, but also structure and consistence of product are developed, is the most important. Taste and aroma active compounds arise by enzymatic decomposition of lactose, proteins and fat in milk[5].

The anaerobic transformation of saccharides to lactic acid is the basic biochemical pathway during production. The produced lactic acid imparts fine refreshing acid taste to these products and extends their durability. It also precipitates milk proteins, which are then easier to digest and improves utilization of calcium, phosphorus and iron[2]. The proteolytic activity of lactic acid bacteria changes physico-chemical properties of casein, which influences rigidity, texture and viscosity of coagulate. Arisen peptides and free amino acids are precursors for enzymic reactions producing taste compounds. Similarly some aroma compounds can arise by lipolytic activity. Released fatty acids are precursors for methyl ketones, alcohols, lactones and esters, which also impart characteristic flavour to product[5].

The aim of this work was to identify and quantify several aroma compounds in various types of fermented milk products using SPME-GC-MS method and to judge their influence on flavour of products. In total 18 types of products (7 types of flavoured creamy yogurts, 3 types of drinking yogurts, 4 types of acidophilic milk, 2 types of kefirs and 2 types of clabbered milk) with various flavourings were analysed.

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REFERENCES

1. Condurso C., Vernera A., Romeo V., Ziino M., Conte F.: Int. Dairy J. *18*, 819 (2008).
2. Pohjanheimo T., Sandell M.: Int. Dairy J. *19*, 459 (2009).
3. Kailasapathy K.: Food Sci. Technol. *39*, 1221 (2006).
4. Nongonierma A. B., Springett M., Le Quere J. L., Cayot P., Voilley A.: Int. Dairy J. *16*, 102 (2006).
5. Roginski H., Fuquay J., Fox P.: *Encyclopedia of Dairy Science*. Academic Press London 2003.

4-P89

VITAMINS IN YEAST *SACCHAROMYCES CEREVISIAE*

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Yeast cells are known as an important source of proteins, vitamins and minerals. The yeast *Saccharomyces cerevisiae* is rich in B group vitamins. Various strains of this yeast are used in the baking, alcohol, and brewing industries, as well as for obtaining vitamin preparations. Among the vitamins synthesized by yeast are thiamine (vitamin B_1), pyridoxine (B_6) and riboflavin (B_2)[1,2]. The content of vitamin B_1, B_2, B_6 and

ß-carotene of three yeast strains of *S. cerevisiae*, i.e. Kolín, 612 and Gyöng was studied.

The yeasts were grown on YPD medium containing 10 g l^{-1} yeast extract, 20 g l^{-1} peptone and 20 g l^{-1} glucose. HPLC/DAD method for the determination of vitamins B_1, B_2 and B_6 was used. The content of ß-carotene was realized by UV/VIS spectrophotometry. Results of the analysis of vitamins in obtained biomass differ according to the used yeast strain[3] (Table I).

Table I
Content of vitamins in yeast *S. cerevisiae* [mg kg^{-1}]

Vitamin	strain Kolín	strain 612	strain Gyöng
Vitamin B_1	1,8	<1	<1
Vitamin B_2	20,6	5,1	5,6
Vitamin B_6	<1	<1	<1
ß-carotene	<1	<1	<1

The biomass of yeast is generally characterized by high content of vitamins, particularly of the type B, such as strain Kolín, which had a concentraion of vitamin B_2 20,6 mg kg^{-1}.

The yeast usually contains 4 – 8 mg of riboflavin in 100 mg biomass[4] what was not confirmed by our study.

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REFERENCES

1. Abbas Ch. A., in the book: *Yeasts in food and beverages*, chapt. 10, p. 315. Springer-Verlang, Berlin 2006.
2. Abramov, S. A., Kotenko, S. Ts., Ramazanov, A. S., Islamova, F. I.: Appl. Biochem. Microbiol. *4*, s. 385 (2003).
3. Šillerová, S., Poláková, A., Urminská, D., Szabová, E.: Potravinárstvo *1*, 88 (2010).
4. Ürgeová, E., Marecová, M.: Nova Biotechnologica *3*, 153 (2003).

4-P90

DYNAMICS OF THE CONTENT OF CAROTENOIDS AND LYCOPENE IN TOMATO FRUIT (*LYCOPERSICON ESCULENTUM* MILL.) DURING STORAGE AND AFTER PROCESSING

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The aim of our work was to evaluate the changes of carotenoids and lycopene content in 7 varieties of tomato fruits (*Lycopersicon esculentum* MILL.): Báb, Eskort, Premium, Rodik, Roti, Šampion and Žiara. Changes in carotenoids and lycopene content were evaluated during storage and in thermally processed tomato juice. Petroleum ether was used as extraction solvent and extracts of carotenoids and lycopene were analysed by UV-VIS spectrophotometry Jenway at wavelenght of 445 a 472 nm.

Experiments showed that carotenoids and lycopene content increased during storage of tomato fruits at storage temperature 21 °C of varieties Báb, Šampion, Žiara, Rodik and Roti. In terms of raising carotenoids and lycopene content at storage temperature 5 °C had tomato fruits less suitable conditions comparing to storage temperature 21 °C. Content of carotenoids was decreasing at varieties: Báb, Šampion, Rodik, Roti and Premium.

After heat treatment of tomato fruits to produce tomato juice carotenoids content increased at a Žiara and Premium varieties, while varieties of the Báb, Šampion, Rodik, Roti and Eskort, the heat treatment reduced the content. Lycopene content increased only in the variety Eskort, when other varieties are reduced, but this does not indicate less utilization of this important nutrient in the human body.

The work shows that the dynamics of changes in contents of carotenoids and lycopene affects the variety and method of storage and processing.

1. Arab L.: Exp. Biol. Med. *227*, 894 (2002).
2. Belitz, H. D., Grosch, W., Schieberle P.: *Food chemistry*. 3. Rev. Ed. Berlin, Springer Verlag, 2004.
3. Bohm, V., Bitsch, R.: Eur. J. Nutr. *38*, 118 (1999).
4. Ollanketo, M., Hartonen, K., Riekolla, M. L.: Eur. Food Res. Technol. *121*, 561 (2001).
5. Stacewicz-Sapuntzakis M., Bowen P.: Biochim. Biophys. Acta *1740*, 202 (2005).

4P-91

DETERMINATION OF RESIDUAL FORMIC ACID IMPURITY IN HYALURONATE OLIGOSACCHARIDES

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In the past few years, intensive research has been focused on the explanation of the role of hyaluronate oligosaccharides in a diverse range of physiological and diseases processes[1]. It is important to use well-characterized highly purified hyaluronate oligosaccharides, because some undesired biological activities could occur due to the presence of minor components. One of the major impurities remaining in hyaluronate oligosaccharides is formic acid which comes from the mobile phase during chromatographic separation of oligosaccharides.

For the determination of short-chain formic acid in various matrices many methods have been developed including gas[2] and liquid[3] chromatography, colorimetric[4] and enzymatic[5] methods. Some of these techniques are limited by the need for derivatization, a large amount of sample needed for analysis and by an inability to achieve a low detection limit.

A fast, reliable capillary electrophoretic method for analysis of formate as a component of hyaluronate oligosaccharides was optimalized applying indirect UV detection. A rapid separation was achieved using a fused-silica capillary, 2,6-pyridine dicarboxylic acid as the background electrolyte and CTAB additive in order to turn the direction of electroosmotic flow. Detection and quantification limits of formate were determined as 0,8 ppm and 2,5 ppm, respectively. These limits are about 20 times lower when compared to commonly used ion-exclusion chromatography evaluation. The presented method can be used for verification of the suitability of hyaluronate oligosaccharides for pharmaceutical applications.

REFERENCES

1. Stern R., Asari A. A., Sugahara K. N.: Eur. J. Cell Biol. *85*, 704 (2006).
2. Lou D. W., Lee X., Pawliszyn J.: J. Chromatogr. A *1201*, 228 (2008).
3. Fisher K.: Anal. Chim. Acta *465*, 157 (2002).
4. Sleat R., Mah R. A.: Appl. Environ. Microbiol. *47*, 884 (1984).
5. Ortega, F., Ballesteros M., Centenera J. I., Domínguez E.: J. Pharm. Biomed. Anal. *8*, 991 (1990).

4-P92

IDENTIFICATION OF BARLEY VARIETIES RECOMMENDED FOR CZECH BEER

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The distinctive nature of České pivo (Czech Beer) is imparted by a number of factors, particularly by the use of high quality Czech brewing raw materials and specific technology. The Czech Republic is perhaps the only country in the world where the brewing experts distinguish whether a new malting barley variety is suitable for export malt or for maintaining the sensory character of the national beverage. Barley varieties suitable for Czech beer are characterized by a lower wort attenuation and resistance to malt overmodification during germination. The varieties recommended by the Research Institute of Brewing and Malting, Plc. for production of beer with the Protected Geographical Indication are Radegast, Blaník, Aksamit, Bojos, Malz, Advent, Calgari, and Tolar.

These varieties were identified with prolamin proteins of barley grain, so-called hordeins. Hordeins were analyzed using the method of vertical gel electrophoresis on polyacrylamide gel. The method is based on high degree of polymorphism of hordeins of barley or malt produced from it which can be after extraction from grain electrophoretically separated. Spectra of separated hordeins are specific for the particular barley varieties.

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REFERENCES

1. Kosař, K., Psota, V., Mikyška, A.: Czech J. Genet. Plant Breed. *40,* 137 (2004).

4-P93

DEVELOPMENT OF A FOCUSED MICROWAVE-BASED EXTRACTION METHOD FOR THE DETERMINATION OF ARSENICALS IN FOOD SAMPLES

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The extraction of arsenic species from food samples can be carried out by using several analytical tecniques but ultrasonic bath has became the method of choice in a great number of works. Most recently, the use of ultrasonic probe has been introduced for speciation. With the later, the transmision of acustic energy in the medium is direct since the probe is instroduced in the sample. The power can be amplified and for this reason the transmited energy can be 100 fold higher than those obtained with a bath.

On the other hand, in spite of the good performance of traditional microwave ovens, the use of focused microwave radiation could be advantageous because of the better safety conditions due to the operation at atmospheric pressure and simultaneous control of different heating programs for different samples owing to the possibility of operating each reaction vessel independently. Focused microwave assisted extraction at atmospheric pressure has given satisfactory results for the quantitative extraction of PAHs, polychlorobiphenyls and pesticides from environmental matrices[1-4] with advantages of security, easy manipulation and sample preparation automatization. Microwave irradiation is often used instead of conventional heating because the solvent is rapidly heated in a reproducible way. Moreover, the selective interaction with polar molecules allows local heating and an improvement of extraction efficiency.

In this work, we have developed a method for the speciation of arsenic in *Anemonia sulcata* by focused microwave extraction followed by HPLC-ICP-MS and HPLC-QqQ determination. *Anemonia sulcata* is a marine organism very appreciated in Spain that is usually eaten muffled and fried with olive oil. The results obtained show that the use oficrowave extraction allows very good recoveries for the majority of the arsenic species in only five minutes. The proposed method simplifies the sample preparation that usually constitutes a key step in speciation.

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REFERENCES

1. H. Budzinski, A. Papineau, P. Baumard, P. Garringes.: C.R. Acad. Sci. *321*, 69 (1995).
2. H. Budzinski, A. Papineau, P. Baumard, P. Garringes, S. Wise.: PAC Journal., *9*, 225 (1996).
3. H. Budzinski, M. Letellier, K. Le Menach, P. Garringes.: J Chromatogr. *837*, 187 (1999).
4. M. Letellier, H. Budzinski, J. Bellocq, J. Connan, Organic Geochem. *30*, 1353 (1999).

4-P94

TOTAL ANTIOXIDANT STATUS, LIPIDS AND PROTEINS OXIDATION LEVELS IN SUBCLINICAL MASTITIS COW'S MILK

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Bovine mastitis is a major disease affecting dairy cattle worldwide. In mastitis and other inflammation processes affecting the mammary tissue, the number of somatic cell in milk increases, especially the polymorphonuclear cells (PMN). Antibacterial activity of these cells is mediated, in part, through reactive oxygen species (ROS). An excess of ROS and the absence of optimal amounts of antioxidants are leading to oxidative stress[1].

The aim of this study was to evaluate the antioxidant/oxidant status in milk of cows with subclinical mastitis using 3 parameters: (1) total antioxidant status (TAS), (2) levels of malondialdehyde (MDA) a degradation product of lipid peroxidation, and (3) levels of proteins oxidation. Results were compared with those obtained in another group of healthy cows. Subclinical mastitis diagnose was realized using electrical conductivity method and also by determining the number of somatic cells in milk (SCC).

The levels of SCC, TAS, lipids peroxidation (MDA) and proteins oxidation were significantly higher in subclinical mastitis milk compared to those from healthy cows (with mean values of 11,39 ± 5,61 μmol l^{-1} for TAS; 70,46 ± 5,45 nmol ml^{-1} for MDA and 0,557 ± 0,091 μmol ml^{-1} for DNPH), due to the exacerbation of oxidative stress in subclinically inflammed mammary gland. From toxicological point of view, consumption of milk containing products derived from oxidation of fatty acids, cholesterol and proteins should be avoided because they have various adverse biological effects. Thus, these oxidation products are considered cytotoxic and even mutagenic and carcinogenic[2].

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REFERENCES

1. Andrei S., Matei S., Fit N., Cernea C., Ciupe S., Bogdan S., Groza I.: Romanian Biotechnological Letters, in press
2. Sieber R.: Intern. Dairy Journal *15*, 191 (2005).

4-P95

DIAGNOSIS AND MONITORING BY GC/MS

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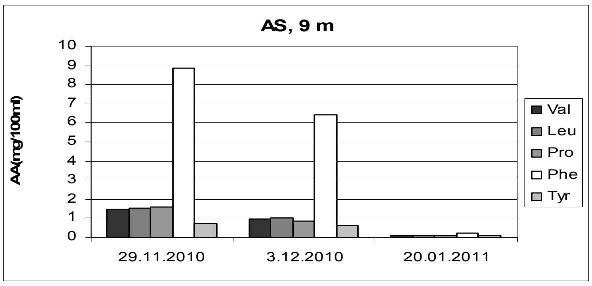
Phenylketonuria (PKU) and maple syrup urine disease (MSUD) are metabolic diseases arised from inherited errors in enzymes pathways with symptoms due to the buildup of a biosynthetic precursor or product that the body cannot excrete. In mammals, phenylalanine (Phe) is metabolized in tyrosine (Tyr) in the liver. PKU is caused by the deficiency of phenylalanine hydroxylase[1]. The enzyme defect leads to pattern of plasma amino acids with increase of Phe and decrease of Tyr. These inborn errors can cause major intellectual disturbances, but neonatal diagnosis and appropriate treatment can be helpful. Gas chromatography-mass spectrometry (GC/MS) method is the best choice for some inborn error of metabolism diseases diagnosis, being a cheaper but a very precise method[2-7].

A method for diagnosis of these inborn error diseases of metabolism by GC/MS in selected ion monitoring (SIM) mode is presented. Labeled methionine was used as internal standard. The method uses a drop of blood blotted onto a sample card, an aliquot is extracted for analysis and peak profile is used to diagnose metabolic errors. Validation of the method showed good values for the different parameters of validation studied as linearity, precision, accuracy, day-to-day reproducibility, LOD and LOQ.

Quantitative analysis of five amino acids: valine (Val), leucine (Leu), proline (Pro), phenylalanine (Phe) and tyrosine (Tyr), in blood samples, by using blood spots[1] or 0,5 ml blood[8] gave similar results. The method only takes a few minutes to perform and requires minimal sample preparation. The accurate assay of the amino acids by using this minim invasive method, and ^{15}N-labeled methionine as internal standard could be used for neonatale screening for phenylketonuria and other aminoacidemias. The monitoring of the five amino acids levels in patients and the influence of treatment on their levels is presented.

Due to the low cost, the GC-MS method is suitable for applications in all countries. The high benefits of the early diagnosis and treatment are strong arguments for the neonatal screening of metabolic disorder. Newborn screening for PKU relies on Phe detection in the blood from filter paper. The classical bacterial inhibition assay (BIA) used for the diagnosis of PKU is a semi-quantitative method giving false positive results. More precise methods, such as the MS/MS technique, were developed, but they have the disadvantage of high price and less affordable equipment. By comparison, the proposed GC/MS method is simple, inexpensive, easily operated and high-speed technique.

In scheme 1 can be seen the effect of treatment on phenylalanine and other amino acids level in the case of a nine months PKU patient.



Scheme 1. The amino acid values evolution during treatment for a 9 months PKU patient

The GC/MS method is rapid and proper for diagnosis and monitoring the PKU and MSUD diseases.

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REFERENCES

1. Deng C., Li N., Wang X., Zhang X., Zeng J.: Rapid Commun. Mass Spectrom. *19*, 647 (2005).
2. Phillips M., Cataneo R. N., Cheema T., Greenberg J.: Clin. Chim Acta. *344*, 189 (2004).
3. Culea M., Cozar O., Ristoiu D.: JMS *41*, 1594 (2006).
4. Nagy N., Takats Z., Pollreisz F., Szabo T., Vekey K.: Rapid Commun. Mass Spectrom. *17*, 983 (2003).
5. Deng C., Yin X., Zhang L., Zhang X.: Rapid. Commun. Mass Spectrom. *19*, 2227 (2005).
6. Jouvet P., Hubert P., Saudubray J. M., Rabier D., Man N. K.: Pediatric Res. *58*, 278 (2005).
7. Shen X., Deng C., Wang B., Dong L.: Anal. Bioanal. Chem. *384*, 931 (2006).
8. Culea M., Hachey D. L.: Rapid Commun. Mass Spectrom. *9*, 655 (1995).

4-P96

LEUCINE TRANSMEMBRANAR TRANSPORT STUDY IN THE HUMAN RED CELL

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A study of the leucine transport in human erythrocytes is presented. The present experiment involves the measuring of the outward flux of leucine from erythrocytes into a leucine-free medium. The cells were adjusted to a haematocrit of 20%.

Samples containing transported amino acids were purified on Dowex 50W-W8 exchange resin, eluted with NH_4OH 4M and derivatized. In order to obtain N-trifluoro-acetyl n-butyl ester derivatives, a two step derivatization was made: esterification at C terminal edge and trifluoroacetylation at the N terminal edge. The measurements were performed by gas chromatography-mass spectrometry (GC-MS) using a Trace GC equipped with a Rtx-5MS capillary column (30m x 0,25 mm, 0,25 µm film thickness) and coupled to a Trace DSQ ThermoFinnigan quadrupole mass spectrometer. The GC temperature program for amino acids separation was: 50 °C (1 min), rising at 100 °C at a rate of 20 °C/min, then rising at 310 °C at a rate of 30 °C/min, 310 °C (2 min). A selected ion monitoring (SIM) mode was used for higher sensitivity with three orders of magnitude. The ions selected for leucine and for the internal standard, ^{15}N-leucine, were m/z 182 and m/z 183, respectively. Glycine was measured by selecting the ions m/z 154 and m/z 155, the last one for ^{15}N-Glycine, internal standard. Helium was used as carrier gas at a flow rate of 1 ml/min. The interface and the EI ion source were kept at 250°C.

The method was validated for the small values of the amino acid transmembranar transport, by appling the methods to known amounts of amino acid, adding the same quantity of ^{15}N-Leucine, as internal standard. The isotope dilution GC/MS technique was applied and two types of calculations were compared, by using regression curve and matrix calculation. The efflux of leucine (Leu) was calculated by using the regression curve obtained as the ratio of Leu and the internal standard amounts versus Leu amounts[1-3]. High leucine erythrocyte content seems to be easily exchanged, even at low temperatures.

The aim of this work was to study the influence of different parameters as time, temperature and electromagnetic field on the leucine transmembranar transport.

Table I. The leucine values for efflux study at 4°C

Time (min)	0	5	10	15	20	25	30
Leu (μg ml^{-1}) RBC	0	0	1,18	2,16	3,24	8,24	10,78

Table II. The leucine values for efflux study at 21°C

Time (min)	0	5	10	15	20
Leu (μg ml^{-1}) RBC	0	6,09	12,10	30,38	33,3

Table I and II present the amino acid efflux values transported in vitro at 4°C and at room temperature of 21°C, respectively. The values are reported to the red blood cell volume (RBC).

Leucine transport is faster in comparison with that of glycine. A significant transport can be observed also at low temperature (4°C).

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REFERENCES

1. Culea M., Neamtu S., Palibroda N., Borsa M., Nicoara S.: J. Mol. Struct. *348*, 377 (1995).
2. Hoare D. G.: J. Physiol. *221*, 331 (1972).
3. Darmaun D., Froguel P., Rongier M., Robert J. J.: J. Appl. Physiol. *67*, 2383 (1989).

LIST OF PLENARY LECTURES

PL1 METABOLOMICS: A CHALLENGING TOOL FOR THE ASSESSMENT OF THE ENVIRONMENTAL IMACTS ON FOOD CHAINS
Jana Hajšlová, Lukáš Václavík, Tomáš Čajka, Jana Pulkrabová and Vladimír Kocourek

PL2 ENGINEERING ADVANCED MATERIALS THROUGH PRE-STRESSED DOUBLE NETWORK POLYMER SYSTEMS
Alan J. Lesser

PL3 WATER IN THE CELL
Martin Chaplin

PL4 THE DEVELOPMENT OF SILICATE MATERIALS FOR BIOMEDICAL APPLICATIONS
Martin T. Palou, Gabriela Lutišanová, Jana Kozánková and Ján Lokaj

PL5 OXIDATION OF MARINE PHOSHOLIPIDS
Turid Rustad, Revilija Mozuraityte, Ivar Storrø and Vera Kristinova

PL6 NEW TRENDS IN ALUMINIUM ELECTROLYSIS
Ján Híveš and Pavel Fellner

LIST OF LECTURES ACCORDING TO THE SECTION

1. Physical & Applied Chemistry

1-IL1 CHARGE TRANSER MASS SPECTROMETRY: FROM PROTON TRANSER (PTR-MS) TO ELECTRON TRANSFER (ET-MS) IONIZATION MASS SPECTROMETRY AND THEIR APPLICATIONS
Tilmann D. Märk, Bishu Agarwal, Kurt Becker, Achim Edtbauer, Stefan Haidacher, Gernot Hanel, Eugen Hartungen, Stefan Jaksch, Alfons Jordan, Simone Jürschik, Christopher Mayhew, Lukas Märk, Fredrik Petersson, Hans Seehauser, Ralf Schottkowsky and Philipp Sulzer

1-KL1 QUANTUM MODEL OF HYDROGEN ATOM
Osmera Pavel

1-L1 ELECTROPHORETIC DEPOSITION OF THIN ORGANIC FILMS FOR SOLAR ENERGY CONVERSION PURPOSE
Ivaylo Zhivkov, Daniela Mladenova, Patricie Heinrichová, Imad Ouzzane, Martin Vala and Martin Weiter

1-L2 FADING OF INKJET PRINTED DIGITAL PHOTOGRAPHS AND METHODS FOR ITS EVALUATION
Michal Veselý and Petr Dzik

1-L3 EPR STUDY OF ANTIOXIDATIVE EFFECT OF MELATONIN IN VIVO
Pavel Stopka, Jana Křížová, Jan Mareš, Richard Rokyta, Michael Anděl, Vlasta Rychterová, Kateryna Deykun, Jana Jurčovičová, Andrea Štofková, Martina Škurlová, Jaroslav Pokorný, Josef Kroupa, Christina Minářová

1-L4 STUDY ON HYALURONAN INTERACTIONS WITH L-LYSINE AND 6-AMINOCAPROIC ACID
Martin Chytil, Martin Trojan, Jan Zeman, Miloslav Pekař

1-L5 OPTICAL PROPERTIES OF DIKETO-PYRROLO-PYRROLES FOR ORGANIC ELECTRONICS APPLICATIONS
Martin Vala, Martin Weiter, Patricie Heinrichova, Imad Ouzzane

1-L6 RADICAL PRODUCTS GENERATED BY THE OXIDATION OF SOME SELECTED TYPES OF SECONDARY AMINES
Ladislav Omelka and Lenka Šafaříková

1-L7 ANALOGICAL MODELING AND NUMERICAL SIMULATION OF STATE PARAMETERS FOR A THERMAL EXPLOSION
Mihaela-Ligia Ungureşan , Francisc Vasile Dulf, Eva-Henrietta Dulf

1-L8 DIFFUSION OF ORGANIC DYES IN AQUEOUS SOLUTIONS AND AGAROSE GELS STUDIED BY METHOD OF HORIZONTAL DIFFUSION CELLS
Petr Sedláček, Jiří Smilek, Martina Klučáková

1-L9 ON THE THERMODYNAMIC CONSISTENCY OF RATE CONSTANTS IN MASS ACTION RATE EQUATIONS
Miloslav Pekař

1-L10 SOLUTION PROCESSED TITANIA COATINGS FABRICATED BY INKJET MATERIAL DEPOSITION
Petr Dzik, Michal Veselý

1-L11 FRACTIONAL MODEL OF THE (^{13}C) ISOTOPE SEPARATION COLUMN
Dulf Eva-Henrietta, Dulf Francisc, Szasz Csaba

2. Materials Chemistry

2-IL TiO_2 THICK FILMS SUPPORTED ON RETICULATED MACROPOROUS Al_2O_3 FOAMS AND THEIR PHOTOACALALYTIC ACTIVITY IN WATER PURIFICATION
Melinda Vargová, Gustav Plesch, Ulrich F. Vogt, Karol Jesenák and Michal Gorbár

2-L1 DISTRIBUTED PARAMETER MODEL OF THE 2-ETHYL-HEXENAL HYDROGENATION PROCESS
Roxana Both, Eva-Henrietta Dulf And Clement Festila

2-L2 SOL-GEL PHASE TRANSITIONS OF TEMPERATURE-SENSITIVE BIODEGRADABLE COPOLYMERS
Lenka Michlovská, Lucy Vojtová, Ludmila Mravcová, Ivana Chamradová and Josef Jančář

2-L3 TOWARDS THE DEVELOPMENT OF NEW MATERIALS BASED ON HYALURONIC ACID
Gloria Huerta-Ángeles, Eva Přikopová, Jiří Kislinger, Jan Sokolov, Martin Pravda, Miloslav Pekař and Vladimír Velebný

2-L4 POLYMORPHISM OF 3,6-BIS-(4'-PYRIDYL)-2,5-DIHYDRO-PYRROLO[3,4-C]PYRROLE-1,4-DIONE , MATERIAL FOR HYDROGEN SENSOR
Ota Salyk and Jan Vynuchal

2-L5 CHANGES OF MORPHOLOGY OF COPOLYMER POWDER PP-IC AT 210°C AND AT VARIOUS ANNEALING TIMES
Jiří Sadílek, Jaroslav Kučera, Zdeněk Buráň and Klára Smolná

2-L6 OPTIMIZATION OF CROSS METATHESIS REACTION OF OLEOCHEMICALS WITH ETHYL ACRYLATE
Mudassar Abbas and Christian Slugovc

2-L7 DEVELOPMENT AND CHARACTERIZATION OF NOVEL SOLAR CELLS FOR ORGANIC PHOTOVOLTAICS
Martin Weiter, Patricie Heinrichová, Martin Šedina, Imad Ouzzane, Karol Flimel and Martin Vala

2-L8 STRUCTURE ANALYSIS OF NANOMATERIALS ON A LABORATORY SAXS SYSTEM
Peter M. Worsch, Heimo Schnablegger and Otto Glatter

2-L9 DEVELOPMENT OF SANDWICH STRUCTURE OF CO-EXTRUDED POLYPROPYLENE SHEETS
Jaroslav Kučera, Josef Křivánek, Ladislav Pospíšil and Oldřich Šuba

2-L10 EVALUATION OF MECHANICAL PROPERTIES OF PLASMA POLYMER FILMS USING CONVENTIONAL AND CYCLIC NANOINDENTATION TECHNIQUES
Erik Pálesch, Vladimír Čech, Rutul Trivedi and Lukáš Hoferek

2-L11 KINETICS OF FORMATION OF SELF-ASSEMBLED MONOLAYERS
Adam Babik, Josef Zemek, Rutul Trivedi, Jan Mistrik and Vladimír Čech

2-L12 THE SOL-GEL PROCES IN MANUFACTURE OF REFRACTORY MATERIALS
Lukáš Tvrdík, Karel Lang, Petr Ptáček, Lenka Nevřivová and Pavel Kovář

2-L13 RESORBABLE IMPLANTS FOR HARD TISSUE REGENERATION
Lucy Vojtová, Josef Jančář, Alois Nečas, Ladislav Plánka, Robert Srnec, Eva Prosecká, Martin Plecner, Evžen Amler, Leoš Křen and Jana Hlučilová

2-L14 DEVELOPMENT OF FIBRE-REINFORCED GEOTHERMAL CEMENT COMPOSITES FOR APPLICATION IN PRETENTIOUS HYDROTHERMAL CONDITIONS
Ľubomír Bágeľ, Martin Palou and Vladimír Živica

2-L15 MOLECULAR MODELLING – STRUCTURE AND PROPERTIES OF (UN)SATURATED N-HETEROCYCLIC CARBENES
Eva Kulovaná, Lukáš Richtera, Soňa Hermanová and Josef Jančář

2-L16 HIGHER THERMAL EFFICIENCY FOR THE 2-ETHYL-HEXENAL HYDROGENATION PROCESS
Roxana Both, Eva-Henrietta Dulf and Clement Festila

2-L17 SYNTHESIS AND APPLICATION OF SURFACE MODIFIED IRON OXIDE MAGNETIC NANOPARTICLES FOR REMOVAL OF POLLUTANTS
Shahab Shariati, Mohammad Farajib and Fatemeh Shariati

2-L18 FIVE MECHANISMS OF IRON CORROSION IN UNTREATED WATERS
Pavel Novák, Milan Kouřil, Šárka Msallamová and Jan Stoulil

3. Environmental Chemistry & Technology

3-KL METALLOMIC STUDY OF THE FREE-LIVING MOUSE MUS SPRETUS FOR POLLUTION ASSESSMENT
José Luis Gomez-Ariza, Tamara García-Barrera, Macarena Gonzalez-Fernandez, Miguel Angel García-Sevillano, Rocío Jara-Biedma,

3-L1 REFLECTION OF UNDERLYING ROCKS IN CHEMISTRY OF UNDERGROUND AND SURFACE WATERS
Stanislav Škoda, Jan Váchal, Miroslav Tesař, Radka Váchalová, Miroslav Dumbrovský, Veronika Sobotková

3-L2 WATER – PHYTOPLANKTON – FISH TRANSFER OF SOME HEAVY METALS IN THE DANUBE AQUATIC ECOSYSTEMS OF THE IRON GATES NATURAL PARK
Marius Lucian Matache, Petre Capotă, Maria Patroescu, Alin Tudorache, Laurenţiu Rozylowicz

3-L3 INFLUENCE OF WATER EROSION PROCESSES ON SEDIMENT AND NUTRIET TRANSPORT FROM SMALL AGRICULTURAL CATCHMENT AREA
František Pavlík, Miroslav Dumbrovský, Jana Konečná, Jana Podhrázská

3-L4 PERFLUORINATED COMPOUNDS LEVELS IN WILD FISH FROM THE CZECH REPUBLIC
Petra Hradkova, Veronika Hlouskova, Jan Poustka, Ondrej Lacina, Jana Pulkrabova, Jana Hajslova

3-L5 BIOACCUMULATION OF SOME ORGANOCHLORINE PESTICIDES IN BIOLOGICAL SAMPLES FROM THE LOWER PRUT FLOOD PLAIN NATURAL PARK
Marius Lucian Matache, Iulia Gabriela David, Carmen Hura, Laurenţiu Rozylowicz, Gabriel Chişamera, Diana Onose

3-L6 ASSESSING HEAVY METAL CONTAMINATION IN THE VICINITY OF GOLD MINE, LOEI PROVINCE, THAILAND
Tepwitoon Thongsri, Pornsawan Visoottiviseth and Pahol Kosiyachinda

3-L7 EFFECT OF LAND CONSOLIDATION AND WATERSHED CONSERVATION MANAGEMENT ON WATER QUALITY IN HUBENOV WATER RESOURCE
Miroslav Dumbrovský, Jana Uhrová, František Pavlík, Jan Váchal, Radka Váchalová, Miroslav Tesař

4. Food Chemistry & Biotechnology

4-IL MASS SPECTROMETRY AND SDAP FOR IDENTIFICATION OF PR-PROTEINS/TYPE I ALLERGENS FROM PLANTS
Eva Stratilová and Pavel Řehulka

4-L1 FOOD SAFETY FROM PERSPECTIVE OF MICROBIOLOGIST
Michal Slaný, Petra Vašíčková and Ivo Pavlík

4-L2 CHARACTERIZATION OF MAGNETIC AND NON-MAGNETIC SOLID POLYMER MICROSPHERES
Štěpánka Trachtová, Alena Španová, Bohuslav Rittich and Daniel Horák

4-L3 NEW INTEGRATED STRATEGY FOR QUICK ANALYSIS OF PERSISTENT ORGANOHALOGEN CONTAMINANTS AND POLYCYCLIC AROMATIC HYDROCARBONS IN FOOD
Kamila Kalachová, Jana Pulkrabová, Tomáš Čajka and Jana Hajšlová

4-L4 ARSENIC SPECIATION IN RICE: SURVEY OF THE CZECH MARKET
Jana Navratilová and Peter Šimko

4-L5 ORGANIC AND CONVENTIONAL WINES CHARACTERISATION BY MODERN SPECTROSCOPIC TECHNIQUES
Blanka Tobolková, Martin Polovka and Milan Suhaj

4-L6 OPTIMISATION OF GC/MS MULTIRESIDUE METHOD FOR DETERMINATION OF PESTICIDES IN FRUIT AND VEGETABLE
Petr Dohnal, Adam Vavrouš, Vladimír Kocourek, Tomáš Čajka and Jana Hajšlová,

4-L7 QUANTIFICATION OF FATTY ACIDS IN BUFFALO MILK AND CHEESE USING GC-FID
Aurelia Coroian, Cristian Tudor Matea, Cristian Ovidiu Coroian, Dan Cristian Vodnar, Monica Trif and Constantin Bele

4-L8 MULTIRESIDUE ANALYSIS OF MORE THAN 300 PESTICIDES IN FOOD OF PLANT ORIGIN USING LC-MS/MS: EVALUATION OF QUECHERS PROCEDURES
Jana Urbanová, Radka Křížová, Vojtěch Hrbek, Ondřej Lacina, Vladimír Kocourek and Jana Hajšlová

4-L9 ANALYSIS OF FLAVONOID GLYCOSIDES AFTER HYDROLYSIS WITH HPLC-DAD-ELSD METHOD FOCUSED ON IDENTIFICACTION OF SACCHARIDES
Věra Javorková and Jiří Pazourek

4-L10 NON-ISOTHERMAL KINETICS OF BENZO[A]PYRENE PHOTOOXIDATION IN NON-POLAR FOOD MATRIX
Peter Šimko, Emil Kolek, Božena Skláršová, Alena Bednáriková and Peter Šimon

4-L11 DETERMINATION OF BIOLOGICALLY ACTIVE COMPOUNDS IN FLAXSEED (LINUM USITATISSMUM L.)
Hana Novotná, Věra Schulzová, Marie Bjelková and Jana Hajšlová

4-L12 AMINO ACIDS DETERMINATION IN DRY FERMENTED SALAMI
Monica Culea, Elena Horj, Diana Florescu, Andreea Iordache, Levente Bara and Codrean Pop

4-L13 PRODUCTION, ISOLATION AND APPLICATION OF POLYHYDROXYALKANOATES – BIODERADABLE ALTERNATIVE TO PETROCHEMICAL PLASTICS
Stanislav Obruča, Marie Staňková, Ondřej Šnajdar, Ludmila Mravcová and Ivana Márová

4-L14 ANALYTICAL APPROACHES TO ANALYSIS OF MYCOTOXINS IN FEEDSTUFFS; OCCURRENCE IN REAL LIFE SAMPLES
Milena Zachariášová, Zbyněk Dzuman, Šárka Nováková and Jana Hajšlová

4-L15 DEVELOPMENT AND VALIDATION OF A RAPID METHOD FOR THE ANALYSIS OF PESTICIDE RESIDUES IN TEA USING GC–MS
Adam Vavrouš, Tomáš Čajka, Vladimír Kocourek, Petr Dohnal and Jana Hajšlová

4-L16 PHYTOSTEROL CONTENT OF SOME ROMANIAN WILD AND CULTIVATED BERRIES POMACE
Dulf Francisc-Vasile, Sanda Andrei, Unguresan Mihaela-Ligia and Andrea Bunea

4-L17 DETERMINATION OF POLYCYCLIC AROMATIC HYDROCARBONS (PAHS) IN SEAFOOD USING GC-MS: A COLLABORATIVE STUDY
Lucie Drábová, Jana Pulkrabová, Kateřina Maštovská, Vladimír Kocourek and Jana Hajšlová

4-L18 TYRAMINE CONTENT AND ENTEROCOCCI ISOLATED FROM THE SLOVAK EWE´S CHEESES AS A POTENTIAL SOURCE OF BIOGENIC AMINES
Lucia Poľáková, Krina Trivedi, Renáta Karpíšková, Marta Dušková, Zuzana Dičáková, Juraj Gallo, Lenka Vorlová and Eva Dudriková

4-L19 LEAD, CADMIUM, AND ARSENIC IN EWE´S MILK AND BLOOD SERUM OF EWES AND THEIR LAMBS AS CHEMICAL HAZARD ON FARMS IN EAST SLOVAKIA
Eva Dudriková, Miroslav Húska, Viera Spišáková, Zuzana Dičáková, Lucia Poľáková, Juraj Gallo and Katarína Fatrcová-Šrámková

4-L20 ANALYSIS OF NON-TRADITIONAL FRUITS (*HIPPOPHAE RHAMNOIDES* AND *CORNUS MAS*)
Jitka Cetkovská, Milena Vespalcová, Pavel Diviš, Jaromír Pořízka

LIST OF POSTERS ACCORDING TO THE SECTION

1. Physical & Applied Chemistry

1-P1 INVESTIGATIONS OF THERMOCHEMICAL PARAMETERS OF COMPLEXES OF THE FIRST ROW TRANSITION METAL HALIDES WITH N-DONOR LIGANDS
Abdul Majeed Khan, P. O. Dunstan, Ali Riaz

1-P2 THE CALCULATION OF FRAGMENTATION ENERGY OF ORGANOMETALLIC COMPOUNDS BY USING AB INITIO METHOD
Amer Al Mahmoud Al Sheikh, Jan Žídek and František Krčma

1-P3 CHARACTERISATION OF FATTY ACIDS IN HUMIC ACIDS EXTRACTED FROM PHYSICALLY MODIFIED SOUTH-MORAVIAN LIGNITE
Anna Čtvrtníčková, Laurent Grasset and Jiří Kučerík

1-P4 INSIGHTS INTO LIGNITE STRUCTURE FROM CHEMICAL DEGRADATION METHODS
Leoš Doskočil, Laurent Grasset and Miloslav Pekař

1-P5 DEVELOPMENT ORGANIC SOLAR CELLS BASED ON CONJUGATED POLYMERS
Patricie Heinrichová, Petr Dzik, Ivaylo Zhivkov, Daniela Mladenova and Martin Weiter

1-P6 INFLUENCE OF THE MODIFICATION OF HUMIC ACIDS ON THEIR REACTIVITY
Michal Kalina, Martina Klučáková and Michaela Smítalová

1-P7 DENSITOMETRY OF HYALURONAN AND HYALURONAN WITH CTAB
Andrea Kargerová and Miloslav Pekař

1-P8 STABILITY OF METAL-HUMIC COMPLEXES
Kristýna Nováčková, Jiří Kislinger and Martina Klučáková

1-P9 CONFORMATIONAL CHANGES IN AQUEOUS SOLUTIONS OF HUMIC ACIDS
Martina Klučáková Andrea Kargerová and Kristýna Nováčková

1-P10 UTILIZATION OF IN-ELECTRODE COULOMETRIC TITRATION IN HUMIC RESEARCH
Martina Klučáková and Jana Viktorinová

1-P11 EFFECT OF COMPOST APPLICATION ON PROPERTIES OF SOIL HUMIC SUBSTANCES
Martina Klučáková, Vojtěch Enev, Lubica Pospíšilová

1-P12 EFFECT OF METHYLATION ON HUMIC ACIDS
Soňa Konečná, Laurent Grasset and Martina Klučáková

1-P13 DYNAMIC TENSIOMETRY OF HYALURONAN-SURFACTANT SYSTEMS
Milan Herzog, Jitka Krouská and Miloslav Pekař

1-P14 DETERMINATION OF CARBOHYDRATES AND VOLATILE COMPOUNDS AT ASH WOOD PREHYDROLYSIS
Marta Laurová

1-P15 MONITORING OF HISTORICAL BOOKS PROPERTIES
Iveta Čabalová, František Kačík and Marta Laurová

1-P16 HYDROLYSIS OF RECYCLED FIBERS
Viera Kučerová, Katarína Jaďuďová, Lenka Halajová and Marta Laurová

1-P17 PHOTOCATALYTIC INACTIVATION OF THE YEAST ON TITANIUM DIOXIDE THIN LAYERS
Michaela Lipenská, Michal Veselý, Petr Dzik and Maria Veselá

1-P18 A STUDY OF THE CHEMISTRY OF PLASMA JET INTERACTION WITH SOLUTIONS
Lucie Němcová, František Krčma, Colin Kelsey, Bill Graham, Jenish Patel and Davide Mariotti

1-P19 EDXS AND FTIR SRATR SPECTROSCOPIC CHARACTERIZATION OF SOIL HUMIC SUBSTANCES
Lubica Pospíšilová and Jan Hladký

1-P20 PULSE MODE IN PLASMA POLYMERIZATION OF HEXAMETHYLDISILOXANE
Lucie Blahová, Michal Procházka and František Krčma

1-P21 PLASMATREATMENT OF CORROSION LAYERS FROM IRON IN RF LOW PRESSURE HYDROGEN PLASMA
Věra Sázavská, František Krčma, Drahomíra Janová and Martin Zmrzlý

1-P22 CHARACTERIZATION OF PHTHALOCYANINE DERIVATES FOR APPLICATION IN ORGANIC PHOTOVOLATIC
Martin Šedina, Imad Ouzzane, Karol Flimel, Martin Vala and Martin Weiter

1-P23 THE INFLUENCE OF SUBSTRATE AND PRIMER LAYER ON VARIOUS CHARACTERISTICS OF PRINTED CONDUCTIVE COMPONENTS
Lucie Tylšová and Tomáš Syrový

1-P24 INFLUENCE OF PH ON SYSTEM CONTAINING ANIONIC POLYELECTROLYTE AND CATIONIC OR NONIONIC SURFACTANT
Jana Hejná, Filip Mravec, Tereza Halasová and Miloslav Pekař

1-P25 DYNAMICS OF HYALURONAN WATER SOLUTIONS AS AFFECTED BY MOLECULAR SIZE
Alena Průšová, Pellegrino Conte, Jiří Kučerík and Giuseppe Alonzo

1-P26 DECOMPOSITION OF VOC IN SURFACE DIELECTRIC BARRIER DISCHARGES COMBINED WITH IN SITU PHOTOCATALYZIS
Jana Vyhnalíková, František Krčma, Radka Balaštíková and Petr Dzik

1-P27 CHEMICAL CROSSLINKING OF LIGNITE HUMIC ACIDS
Zdeněk Cihlář And Jiří Kučerík

1-P28 LIGHT FASTNESS OF HISTORICAL TRANSPARENCIES
Silvia Káčerová, Michal Veselý and Petr Dzik

1-P29 EPR STUDY OF N-CENTERED RADICALS PRODUCED BY THE OXIDATION OF SOME MONO-, BI- AND TRIFUNCTIONAL SECONDARY AMINES
Lenka Šafaříková and Ladislav Omelka

1-P30 THE STUDY OF CHEMICAL REACTIONS BETWEEN NITROGEN AND METHANE BY USING ATMOSPHERIC PRESSURE DC GLOW DISCHARGE
Lucie Polachova, Jonathan S. Watson, Gabriel Horvath, Nigel J. Mason, Frantisek Krcma, Miroslav Zahoran, Stefan Matejcik

1-P31 INFLUENCE OF VARNISHES ON INKJET PRINT LIGHTFASTNESS
Eva Štěpánková, Michal Veselý and Petr Dzik

1-P32 FREE RADICALS IN THE NAILS AFFECTED BY FUNGI MYCOSIS (ONYCHOMYCOSIS)
Pavel Stopka

1-P33 SYNTHESIS, SPECTROSCOPIC AND THERMODYNAMIC STUDIES OF THE ADDUCTS OF FIRST ROW TRANSITION METAL(II) BROMIDES WITH 1,2,3,4-TETRAHYDROQUINOLINE
Abdul Majeed Khan and Pedro Oliver Dunstan

2. Materials Chemistry

2-P1 SYNTHESIS AND SPECTRAL CHARACTERIZATION OF SOME NEW P-TERT-OCTYL CALIX[4]ARENE DERIVATIVES
Alina Saponar, Elisabeth-Jeanne Popovici, Ecaterina Bica and Gabriela Nemes

2-P2 HUMIC ACIDS AS THE ADDITIVES IN POLYPROPYLENE
Barbora Bakajová, Jiří Tocháček, Lukáš Běťák and Jiří Kučerík

2-P3 PRACTICAL ASPECTS OF BULK POLYMERIZATION OF STYRENE
Lukáš Běťák, František Kučera, Andrea Pulančíková

2-P4 ADSORPTION OF TEXTILE ACID DYES IN THE PRESENCE OF A CATIONIC SURFACTANT ON MESOPOROUS MATERIALS AL-MCM-41
Boukoussa Bouhadjar, Hamacha Rachida, Morsli Amine, Bengueddach Abdelkader

2-P5 GEOPOLYMERS – EARTH FRIENDLY BUILDING MATERIAL BASED ON ALKALI ACTIVATION OF INDUSTRIAL BY-PRODUCTS
Lukáš Buzek, František Šoukal, Jan Koplík, Jiří Másilko, Lenka Jaskowiecová

2-P6 EFFECT OF pH ON PHOTOCATALYTIC ACTIVITY OF TITANIUM DIOXIDE SUSPENSIONS PREPARED VIA HYDROTHERMAL METHOD
Marcela Černá, Chantal Guillard, Eric Puzenat, Michal Veselý and Petr Dzik

2-P7 THEORETICAL BACKGROUND OF GYPSUM DEHYDRATION TO ALPHA-GYPSUM IN CHLORIDE SOLUTIONS
Karel Dvořák, Marcela Fridrichová and Dominik Gazdič

2-P8 EFFECT OF THE RAW MATERIALS ON THE PROPERTIES OF TERBIUM DOPED YTTRIUM TANTALATE LUMINESCENT MATERIALS
Ecaterina Bica, Elisabeth-Jeanne Popovici, Marius Morar, Lucian Barbu-Tudoran and Emil Indrea

2-P9 THE STOPPED-FLOW TECHNIQUE FOR 1-ALKENE POLYMERIZATION
Adam Hoza, Miroslav Skoumal, Igor Cejpek and Jan Kratochvíla

2-P10 ANISOTROPIC FILM CONSTRUCTION USING PLASMA NANOTECHNOLOGY
Lukáš Hoferek and Vladimír Čech

2-P11 PHYSICAL CONDITIONS AFFECTING RHEOLOGICAL PROPERTIES OF THERMOSENSITIVE COPOLYMERS
Ivana Chamradová, Lucy Vojtová, Lenka Michlovská and Josef Jančář

2-P12 THE DISTRIBUTION OF P_2O_5 IN THE STRUCTURE OF THE CLINKER MINERALS
Tomáš Ifka, Martin T. Palou and Zora Bazelová

2-P13 PHYSICAL-CHEMICAL PROPERTIES OF PRECURSORS FOR YAG:CE TYPE PHOSPHORS OBTAINED BY WET-CHEMICAL SYNTHESIS
Ioana Perhaita, Elisabeth-Jeanne Popovici, Laura Muresan, Marius Morar and Adrian Cadis

2-P14 LEACHABLE POLLUTANTS OF SECONDARY RAW MATERIALS
Lenka Jaskowiecová, Tomáš Opravil, Jan Koplík, Lukáš Buzek and Martin Repka

2-P15 INCREASES IN WATER RESISTANCE OF MACRO-DEFECT-FREE COMPOSITES
Jiří Másilko, František Šoukal, Lukáš Buzek, Jan Koplík, Martin Repka

2-P16 APATITE-LAYER FORMATION ON LS_2-BASED GLASS-CERAMICS IN SBF AND DMEM SOLUTION
Gabriela Lutišanová, Martin T. Palou and Jana Kozánková

2-P17 IMMOBILIZATION OF HEAVY METALS IN ALUMINOSILICATE MATRIXES
Jan Koplík, Lukáš Buzek, František Šoukal, Lenka Jaskowiecová and Jiří Másilko

2-P18 BIOMEDICAL ELASTOMERIC POLYURETHANES BASED ON POLY(ETHYLENE GLYCOL)
Vojtěch Kupka, Lucy Vojtová, Radka Bálková and Josef Jančář

2-P19 LUMINESCENT MATERIALS ACTIVATED WITH RARE EARTH IONS WITH GARNET STRUCTURE
Laura Muresan, Elisabeth- Jeanne Popovici, Ecaterina Bica and Dan Silipas

2-P20 PHYTO-TOXICITY OF SINGLE-WALLED CARBON NANOTUBES ON PLANT GROWTH
Matea Cristian Tudor, Bele Constantin, Razvan Stefan and Iancu Cornel

2-P21 STUDY OF MECHANICAL AND BALLISTIC PROPERTIES OF TEXTILE MATERIALS USED IN BALLISTIC APPLICATIONS
Regina Mikulíková, Richard Klement, Jan Křesťan, Aleš Dvořák, Josef Krátký and Stanislav Rolc

2-P22 INFLUENCE OF THE ENVIRONMENT ON POLYMER ALUMINO-SILICATE MORTARS ON THE BASIS OF ALKALI-ACTIVATED FLY ASH
Nguyen Anh Duong and František Škvára

2-P23 THERMODILATOMETRIC ANALYSIS OF KAOLIN
Magdaléna Nosková, Petr Ptáček, František Šoukal, Tomáš Opravil, Jaromír Havlica, Jiří Brandtštetr

2-P24 EFFECT OF ORGANOTITANATE CROSSLINKING AGENT ON GELATION OF POLYVINYL ALCOHOL
Martin Repka, František Šoukal, Jiří Másilko, Lenka Jaskowiecová, Radek Straka and Michaela Withová

2-P25 OPTICAL AND ELECTRICAL PROPERTIES OF THIN LAYERS OF HUMIC SUBSTANCES
Miroslava Špérová, Martin Weiter and Jiří Kučerík

2-P26 STUDY OF USABILITY OF SANDS FOR PREPARATION OF AEROCONCRETE IN HYDROTERMAL CONDITIONS
Radek Straka, Tomáš Opravil and Jan Koplík

2-P27 MOLECULAR SIMULATION STUDIES OF ADSORPTION AND SELECTIVITY OF CO_2 ON ZEOLITIC IMIDAZOLATE FRAMEWORK-78
Suntharee Phuangjumpee , Oraphan Saengsawang and Supot Hannongbua

2-P28 HYDRATION OF CARBOXYMETHYL CELLULOSE OF DIFFERENT MOLECULAR WEIGHT STUDIED BY SEVERAL DSC APPROACHES
Tomáš Mlčoch and Jiří Kučerík

2-P29 ECOTOXICOLOGICAL EVALUATION OF CHOSEN FLY ASHES UTILIZABLE IN BUILDING MATERIALS
Kristýna Urbánková, Zuzana Pavlitová Letková

2-P30 ANALYSIS OF CREEP BEHAVIOR OF HIGH DENSITY POLYETHYLENE
Robert Válek, Eva Nezbedová and Jaroslav Hell

2-P31 THE STUDIUM OF THE EFFECT OF SELECTED MINERAL ADMIXTURES ON THE HYDRATION OF PORTLAND CEMENT BY ISOPERIBOLIC CALORIMETRY
Pavel Šiler and Josef Krátký

2-P32 PYROLYSIS OF POLYMER COMPOUNDS
Veronika Veľková, Vladimír Lalík and Tatiana Bubeníková

2-P33 THE CONTENT OF POLYCYCLIC AROMATIC HYDROCARBONS IN MOTOR OIL AND HYDRAULIC OIL
Veronika Veľková, Eva Výbohová and Tatiana Bubeníková

2-P34 DETERMINATION OF HYDROGEN CYANIDE FROM POLYURETHANE IN BUILDING INSULATION MATERIALS
Vladimír Lalík, Veronika Veľková, Anton Osvald and Miroslav Vanek

2-P35 ELASTOMERIC POLYURETHANES FILLED BY BIODEGRADABLE POLYHYDROXYBUTYRATE
Lucy Vojtová, Vojtěch Kupka, Jan Žídek, Stanislav Obruča, Ivana Márová and Josef Jančář

2-P36 PROSPECTIVE NOVEL TRIAZOLE-BASED ALUMINIUM COMPLEXES AS CATALYSTS FOR POLYMERIZATION OF CYCLIC ESTERS
Zdeňka Vyroubalová, Soňa Hermanová, Monica M. Moya Cabrera and Jocelyn Alcántara

2-P37 VISCOELASTIC PROPERTIES OF COLLAGEN I
Nina Tomečková and Josef Jančář

2-P38 MECHANICAL PROPERTIES OF CEMENT PASTE IN RELATION ON THE HYDRATION DEGREE
Jaromír Wasserbauer, Bernahard Pichler and Martin Zmrzlý

2-P39 THIXOTROPY, AGING AND RHEOLOGICAL PROPERTIES OF CEMENT PASTES
Michaela Wirthová, Martin Zmrzlý and Jaromír Wasserbauer

2-P40 WDS ANALYSES OF CONVERSION COATINGS ON MAGNESIUM ALLOYS
Martin Zmrzlý, Jakub Tkacz, Jaromír Wasserbauer and Michaela Wirthová

2-P41 DONOR-ACCEPTOR COMPLEXES OF TETRAHYDROFURAN WITH SULFUR TRIOXIDE AND SELENIUM TRIOXIDE
Lukáš Richtera, Eva Kulovaná, Jiří Toužín

2-P42 PROCESSING OF MONOFILAMENTS FROM BIODEGRADABLE POLYSACCHARIDE MATERIALS
Jiří Běťák, Jolana Ščudlová, Pavel Klein, Ilona Matějková, David Mašek and Vladimír Velebný

2-P43 SYNTHESIS OF POLYVINYLALCOHOL-HYDROXYAPATITE COMPOSITE MEMBRANES
Zuzana Balgová and Martin Palou

2-P44 UTILIZATION OF FLUID BED FLY ASH IN THE PREPARATION OF RAW MATERIAL BASE FOR PORTLAND CEMENT BURNING
Dominik Gazdič, Marcela Fridrichová and Karel Dvořák

2-P45 IMPROVEMENT OF CORROSION RESISTANCE OF EXTRUDED AZ61 MAGNESIUM ALLOY
Aneta Němcová, Jakub Tkacz, Martin Zmrzlý and Bohumil Pacal

2-P46 INFLUENCE OF THE UREA ON CORROSION PROCESS OF ALUMINIUM ALLOYS
Eva Novotná, Aneta Němcová, Bohumil Pacal and Jaromír Tulka

2-P47 RELATIONSHIPS BETWEEN REACTIVITY AND MOLECULAR ELECTROSTATIC POTENTIAL OF NITROAROMATIC ENERGETIC MATERIALS
Michal Pexa and Zdeněk Friedl

2-P48 INDUSTRIAL POLLUTANTS PERMEATION RESISTANCE OF NANOSTRUCTURED FILMS
Radek Přikryl, Vladimir Obšel, Ondřej Sedláček and Michal Procházka

2-P49 PRODUCTION TECHNOLOGY OF DENTAL COMPOSITE PREPREG
Radek Přikryl and Přemysl Menčík

2-P50 ANALYSIS OF ORGANIC PHASE CHANGE MATERIALS AS LATENT HEAT STORAGE MEDIUM
Milan Ostrý, Radek Přikryl and Pavel Charvát

2-P51 CORROSION DEGRADATION OF MAGNESIUM ALLOYS AS A NEW DEGRADABLE BIOMATERIALS
Miroslava Horynová, Helena Doležalová Weissmannová, Pavel Doležal and Pavel Gejdoš

2-P52 CONVERSION COATINGS ON MAGNESIUM ALLOYS' SURFACE
Jakub Tkacz and Martin Zmrzlý

2-P53 CONSTRUCTION OF ELINGHAM´S DIAGRAM FOR THE PRESENTATION OF THERMODYNAMIC DATA ABOUT THERMAL STABILITY OF HYDRATES AND HYDROXIDES
Petr Ptáček, Magdaléna Nosková, František Šoukal, Tomáš Opravil, Jaromír Havlica and Jiří Brandtštetr

2-P54 SYNTHESIS OF β-SPODUMENE GROG FOR LAS CERAMICS
Helena Krejčová, Petr Ptáček, Tomáš Opravil, František Šoukal and Jaromír Havlica

2-P55 HYDROLYSIS OF (UN)SATURATED N-HETEROCYCLIC CARBENES
Eva Kulovaná, Lukáš Richtera, Soňa Hermanová and Josef Jančář

2-P56 MOLECULAR SIMULATION STUDIES OF ADSORPTION AND SELECTIVITY OF CO_2 ON ZEOLITIC IMIDAZOLATE FRAMEWORK-78
Suntharee Phuangjumpee, Oraphan Saengsawang and Supot Hannongbua

2-P57 CHARACTERIZATION OF POLY[2-METHOXY-5-(3′,7′-DIMETHYLOCTYLOXY)-1,4-PHENYLENEVINYLENE] ELECTROPHORETIC SUSPENSIONS USED FOR THIN FILM DEPOSITION
Daniela Mladenova, Ivaylo Zhivkov, Imad Ouzzane, Martin Vala and Martin Weiter

2-P58 PHYTO-TOXICITY OF SINGLE-WALLED CARBON NANOTUBES ON PLANT GROWTH
Matea Cristian Tudor, Bele Constantin, Razvan Stefan and Iancu Cornel

3. Environmental Chemistry & Technology

3-P1 Se (IV) AND $MeHg^+$ DETERMINATION BY HPLC-ICP-MS IN NATURAL WATERS AFTER HOLLOW FIBER PRECONCENTRATION
Fernando Moreno, Tamara García-Barrera and José Luis Gómez-Ariza

3-P2 A NEW APPROACH FOR DETERMINATION OF POP'S IN WATER BY HF-LPME
Jose Manuel Manso Sayago, Tamara García-Barrera and José Luis Gómez-Ariza

3-P3 ISOPROSTANES AS MARKERS OF OXIDATIVE STRESS
Martina Bolechová, Josef Čáslavský and Milada Vávrová

3-P4 OXIDATIVE STRESS ASSESSMENT OF SALMON EGGS
Martina Bolechová, Angela Squier, Josef Čáslavský, Kenneth Boyd and Milada Vávrová

3-P5 SEASONAL DYNAMICS OF HEAVY METALS IN VEGETATION IN CONSTRUCTED WETLANDS FOR WASTEWATER TREATMENT
Tereza Březinová and Jan Vymazal

3-P6 DEVELOPMENT AND OPTIMISATION OF SPE/CZE METHOD FOR THE ANALYSIS OF NON-STEROIDAL ANTI-INFLAMMATORY DRUGS FROM WATER
Lukáš Čapka, Petr Lacina and Milada Vávrová

3-P7 SOIL TO PLANT TRANSFER OF METALS IN BAIA MARE AREA, NW ROMANIA
Cristina Mihali, Gabriela Oprea, Angela Michnea, Marin Senila and Cecilia Roman

3-P8 HEAVY METALS CONTAMINATION OF SELECTED BRNO CITY LOCALITIES
Andrea Debnárová, Helena Doležalová Weissmannová and Jan Brzobohatý

3-P9 THE ISSUE DETERMINATION OF ORGANOTIN COMPOUNDS IN THE ENVIRONMENT
Andrea Debnárová and Milada Vávrová

3-P10 MONITORING OF DEAR CONTAMINATION BY ORGANOHALOGEN COMPOUNDS BASED XENOBIOTICS
Petra Doušová, Milada Vávrová and Josef Čáslavský

3-P11 DECONTAMINATION OF SOIL POLLUTED BY PETROLEUM WASTES
Barbora Badalíková and Jaroslava Bartlová

3-P12 SPRAY SCRUBBER SYSTEM FOR REMOVING CO_2 EMISSIONS FROM THE AIR
Josef Kalivoda and Tomáš Svěrák

3-P13 THE STUDY OF THE PRECONCENTRATION OF PLATINUM GROUP METALS ON MODIFIED SORBENTS AND THEIR DETERMINATION BY ICP-OES
Renata Komendová and Lumír Sommer

3-P14 PHOSPHORUS SORPTION – COMPARISON OF THE SORPTION CHARACTERISTICS OF FILTRATION MATERIALS
Michal Kriška

3-P15 THE USE OF COMPREHENSIVE TWO-DIMENSIONAL GAS CHROMATOGRAPHY WITH MASS SPECTROMETRIC DETECTION FOR THE ASSESSMENT OF DRUG-RESIDUE CONTAMINATION OF WASTEWATER AND SURFACE WATER
Petr Lacina, Ludmila Mravcová and Milada Vávrová

3-P16 CHROMIUM REMOVAL FROM WATER BY SYNTHETIC GOETHITE
Lenka Oroszová, Miroslava Václavíková and Katarína Štefušová

3-P17 METHOD OF SEQUENTIAL EXTRACTION OF HEAVY METALS FROM SOLID ENERGETIC WASTE PRODUCTS
Helena Doležalová Weissmannová, Alena Kořínková and Andrea Debnárová

3-P18 SPATIAL DISTRIBUTION OF METALS IN TOP SOILS AND CONTAMINATION ASSESSMENT METHODS
Helena Doležalová Weissmannová, Petr Chovanec, Silvie Mihočová, Lukáš Kováč, Jan Brzobohatý and Milan Marek

3-P19 MODIFIED DIGESTION METHOD OF TOTAL PHOSPHORUS DETERMINATION IN RIVER SEDIMENTS
Marcela Lagová and Jitka Malá

3-P20 EFFECT OF TEMPERATURE ON TREATMENT OF HUMIC WATER BY DIFFERENT COAGULANTS
Jana Leskovjanová and Petr Dolejš

3-P21 COMPARISON OF BIOLOGICAL RESPONSE OF THE CLAM *SCROBICULARIA PLANA* FROM DIFFERENT ENVIRONMENTAL AREAS BY SEC-UV-ICP-MS
Amanda Gago-Tinoco, Macarena González-Fernández, Tamara García-Barrera, Julian Blasco, Maria Joao Bebiano and Jose Luis Gómez-Ariza

3-P22 SPECIATION OF ARSENIC METABOLITES IN THE FREE-LIVING MOUSE MUS SPRETUS FROM DOÑANA NATIONAL PARK USED AS BIOINDICATOR FOR ENVIRONMENTAL POLLUTION MONITORING
Miguel Angel García-Sevillano, Macarena González-Fernández, Rocío Jara-Biedma, Tamara García-Barrera, Amalia Vioque-Fernández, Juan López-Barea, Carmen Pueyo And Jose Luis Gómez-Ariza

3-P23 NOVEL APPROACHES TO ANALYSIS OF 3-CHLOROPROPANE-1,2-DIOL DIESTERS IN PALM OIL EMPLOYING LIQUID CHROMATOGRAPHY–MASS SPECTROMETRY AND DIRECT ANALYSIS IN REAL TIME–MASS SPECTROMETRY
Eliška Moravcová, Lukáš Václavík, Ondřej Lacina, Vojtěch Hrbek, Kateřina Riddellová and Jana Hajšlová

3-P24 ANALYSIS OF TAR GENERATED FROM BURNING BIOMASS
Ludmila Mravcová, Milada Vávrová, Andrea Magdechová, Martin Lisý and Marek Baláš

3-P25 SYNTHETIC AROMATIC SUBSTANCES IN WASTEWATERS
Milada Vávrová, Petra Tylichová, Libor Zouhar, Josef Čáslavský, Michaela Stoupalová, Vladimír Večerek

3-P26 *PROBLEMS WITH THE SPECTROPHOTOMETRIC AND ELECTROTHERMAL AAS DETERMINATION OF TRIVALENT CHROMIUM IN WATERS*
Jana Nevrlá and Lumír Sommer

3-P27 MONITORING OF PRESENCE OF SELECTED HEAVY METALS IN WELL WATERS
Beáta Piršelová, Roman Kuna and Katarína Milecová

3-P28 RESPONSE OF SOYBEAN ROOTS TO IONS OF CADMIUM AND ARSENIC
Patrik Mészáros, Beáta Piršelová and Ildikó Matušíková

3-P29 RESPONSE OF SOYBEAN ROOTS TO CADMIUM, NITROGEN STRESS AND THEIR COMBINATIONS
Yievgeniia Golovatiuk, Patrik Mészáros, Beáta Piršelová and Ildikó Matušíková

3-P30 EVALUATION OF BIOAVAILABILITY OF TOXIC METALS IN AQUATIC SYSTEM BY USING PASSIVE SAMPLERS CHEMCATCHER
Michaela Šebková and Josef Čáslavský

3-P31 DETERMINATION OF POLYBROMINATED DIPHENYLETHERS AND PERFLUORINATED COMPOUNDS IN SOIL AND MATRICE FROM FIREPLACES
Milada Vávrová, Ilona Vondráčková, Ludmila Moravcová, Michaela Stoupalová and Vladimír Večerek

3-P32 EVALUATION OF CONTENT OF PCB AND PBDE IN PLANT BIOINDICATORS
Ilona Vondráčková, Milada Vávrová, Ludmila Mravcová and Bohuslava Tremlová

3-P33 DUMP WATER QUALITY AND WETLAND VEGETATION STRUCTURE
Jiřina Ticháčková and Jan Vymazal

3-P34 SIMULTANEOUS DETERMINATION OF METAL TRACES BY ICP-MS IN ENVIRONMENTAL WATERS USING SPE PRECONCENTRATION ON DIFFERENT POLYMERIC SORBENTS
Zuzana Holubová and Lumír Sommer

3-P35 SUITABILITY OF EXTRACTION METHODS FOR DETERMINATION OF DRUG RESIDUES IN THE RIVER SEDIMENT
Petra Ženatová, Milada Vávrová and Petra Dadáková

3-P36 APPLICATION OF VOLATILE COMPOUNDS DISTRIBUTION FOR THE ASSESSMENT OF THE CZECH BEER AUTHENTICITY
Petra Doušová, Josef Čáslavský

3-P37 MOBILE RAMAN SPECTROMETER IN SERVICE OF FIRE BRIGADE UNITS
Helena Mičánková, Josef Čáslavský, Pavel Kukleta

3-P38 ECOTOXICOLOGICAL EVALUATION OF SEDIMENTS
Helena Zlámalová Gargošová, Zuzana Dobešová

3-P39 ECOTOXICITY OF SELECTED PESTICIDES
Helena Zlámalová Gargošová, Lucie Winklerová

3-P40 MATRICES FROM FIRE PLACES - THEIR POSSIBLE ECOTOXICITY
Helena Zlámalová Gargošová, Adéla Pasírbková, Milada Vávrová

3-P41 ELIMINATION OF RESIDUES OF ESTROGENS DURING DRINKING WATER TREATMENT
Zuzana Olejníčková, Jaroslav Mega and Zdeněk Šimek

3-P42 THE CONTENT OF COOPER AND CHROMIUM IN FISH TISSUE FROM THE WATER RESERVOIRS IN GOLIANOVO AND KOLÍŇANY
Juraj Tóth, Ján Tomáš, Július Árvay, Radovan Stanovič, Daniel Bajčan and Dominika Bončíková

3-P43 THE CONTENT OF HEAVY METALS IN SEDIMENTS FROM THE THE WATER RESERVOIRS
Juraj Tóth, Tomáš Tóth, Peter Lazor, Dominika Bončíková, Alena Vollmannová and Juraj Miššík

3-P44 CONTENTS OF CADMIUM AND NICKEL IN SOILS AND PLANT MATERIAL AFTER BIOSLUDGE APPLICATION IN VPP KOLÍŇANY
Juraj Tóth, Tomáš Tóth, Janette Musilová and Judita Bystrická

3-P45 CONTENT OF RISK ELEMENTS IN SOIL AND WHEAT AROUND THE CHEMICAL FACTORY OF EAST SLOVAKIA
Radovan Stanovič, Ján Tomáš, Alena Vollmannová, Peter Lazor, Julius Árvay, Juraj Miššík and Juraj Tóth

3-P46 THE TOLERANCE OF SELECTED GRASS SPECIES TO PETROLEUM STRESS
Anna Gogoláková, Jana Urbánková and Peter Štrba

3-P47 CHLOROPHYLL FLUORESCENCE RESPONSE OF CORN SEEDLINGS TO LEAD STRESS
Anna Gogoláková and Peter Štrba

3-P48 CHLOROPHYLL FLUORESCENCE RESPONSE OF PISUM SATIVUM TO LEAD STRESS
Anna Gogoláková, Monika Bagóová and Peter Štrba

3-P49 EFFECT OF CADMIUM AND ARSENIC IONS ON THE CONTENT OF PHOTOSYNTHETIC PIGMENTS IN LEAVES OF GLYCINE MAX (L.) MERILL.
Terézia Dobroviczká, Beáta Piršelová and Ildikó Matušíková

3-P50 CHLOROPHYLL FLUORESCENCE IN LEAVES OF SOYBEAN AS INDICATOR OF CADMIUM STRESS
Terézia Dobroviczká, Beáta Piršelová, Anna Gogoláková and Ildikó Matušíková

3-P51 CONTAMINATION LEVEL OF UPPER FLOW RIVER NITRA ALLUVIAL AREA AFFECTED BY LOCAL INDUSTRIAL ACTIVITY
Radovan Stanovič, Július Árvay, Alena Vollmannová and Michal Kujovský

3-P52 CHIRAL ANALYSIS OF DRUG RESIDUALS IN WASTE WATERS
Dagmar Svobodová and Josef Čáslavský

3-P53 DETERMINATION OF SYNTHETIC MUSK COMPOUNDS IN WASTE WATER
Libor Zouhar, Milada Vávrová, Kristýna Kubíčková and Ludmila Mravcová

3-P54 DETECTION OF POLYCHLORINATED BIPHENYLS IN AIR AT JAMES ROSS ISLAND, ANTARCTICA
Daniela Mácová, Josef Čáslavský and Daniel Nývlt

3-P55 DETERMINATION OF SURFACTANTS BY CAPILLARY ZONE ELECTROPHORESIS
Hana Finsterlová and Milada Vávrová

3-P56 SIM – GC/MS DETERMINATION OF BTEX IN AMBIENT AIR
Diana Florescu, Andreea Iordache, Elena Horj, Roxana Elena Ionete, Ioan Stefanescu and Monica Culea

3-P57 TRACE OF HEAVY METALS IN FISH TISSUES, SEDIMENT AND WATER FROM DANUBE RIVER IN ROMANIA
Diana Florescu, Andreea Iordache, Claudia Sandru, Roxana Ionete and Monica Culea

3-P58 INVESTIGATION AND MONITORING OF AMBIENT POLLUTANTS OF ATMOSPHERE FROM IN SITU COLLECTED SAMPLES IN AN INDUSTRIAL AREA
Diana Florescu, Andreea Iordache, Felicia Bucura , Marius Constantinescu, Gili Saros, Roxana Ionete and Monica Culea

3-P59 EFFECT OF POLYCICLIC AROMATIC HYDROCARBONS ON THE ENVIRONMENT OF ERBIL CITY
Pakhshan I. Azez

3-P60 SPATIAL AND TEMPORAL DISTRIBUTION OF PHOSPHORUS IN COASTAL MARINE SEDIMENT FROM THE NORTHWESTERN ARABIAN GULF
Majed Bahloul

3-P61 CLUSTER ANALYSIS APPLIED TO ENVIRONMENTAL SEDIMENT DATA IN SULAIBIKHAT BAY, KUWAIT
Hassan Alshemmari, Khalid Al-Matrouk, Majed Al Wadi and Saif Udn

4. Food Chemistry & Biotechnology

4-P1 GAS CHROMATOGRAPHIC-MAS SPECTROMETRIC DETERMINATION OF LOW-MOLECULAR PRODUCTS FORMED DURING EXPOSITION OF POLYCAPROLACTONE TO *ASPERGILLUS*-LIPASE *Valerie Helánová, Jiřina Omelková, Stanislava Voběrková, Soňa Hermanová and Lucie Kulštejnová*

4-P2 OPTIMIZATION OF LIPASE PRODUCTION BY *BACILLUS SUBTILIS*
Stanislava Voběrková, Jiřina Omelková, Soňa Hermanová, Radka Slavíčková and Lukáš Richtera

4-P3 STUDY OF CHANGES IN ANTIOXIDANT LEVELS IN SEASONAL BERRY FRUITS DURING LONG-TERM STORAGE BY FREEZING
Kateřina Duroňová, Markéta Matějková and Ivana Márová

4-P4 ANALYSIS OF POLAR AND NONOPLAR LIPIDS IN THE SURFACE LAYERS OF LONG-STORED APPLES IN THE ATMOSPHERE WITH DIFFERENT OXYGEN CONTENT
Kateřina Duroňová, Milan Čertík and Ivana Márová

4-P5 COMPARISON OF SACCHAROMYCES STRAINS ISOLATED FROM TWO TYPES OF WINE
Hana Šuranská, Dana Vránová, Kamila Augustová, Renata Vadkertiová and Jiřina Omelková

4-P6 ANTIOXIDANT AND ANTIMICROBIAL PROPERTIES OF MONOFLORAL BEE POLLEN USABLE IN HUMAN NUTRITION
Katarína Fatrcová-Šramková, Janka Nôžková, Miroslava Kačániová and Magda Máriássyová

4-P7 POLYUNSATURATED FATTY ACIDS IN FLAX (*LINUM USITATISSIMUM* L.) GENOTYPES WITH HIGH AND LOW CONTENT OF ALFA-LINOLENIC ACID
Marie Bjelková, Janka Nôžková and Katarína Fatrcová-Šramková

4-P8 CONTENT OF POLYUNSATURATED FATTY ACIDS IN LINSEED GENOTYPES (*LINUM USITATISSIMUM* L.) AND COVERING OF RECOMMENDED NUTRITIONAL AMOUNTS BY CONSUMPTION OF LINSEED
Janka Nôžková, Marie Bjelková and Katarína Fatrcová-Šramková

4-P9 MONITORING OF YEASTS POPULATION IN ECOLOGICAL AND INTEGRATED PRODUCED MORAVIAN WINES
Hana Šuranská, Dana Vránová, Ivana Jiříková, Lenka Procházková, Renata Vadkertiová and Jiřina Omelková

4-P10 ISOLATION OF DNA FROM PROBIOTIC PRODUCTS USING SOLID CARRIERS
Ondřej Bonczek, Štěpánka Trachtová, Bohuslav Rittich and Alena Španová

4-P11 FUNCTIONALIZED SINGLE-WALLED CARBON NANOTUBES FOR LIPASE IMMOBILIZATION
Bele Constantin, Bunea Andrea, Matea Cristian, Razvan Stefan and Octavian Negrea

4-P12 CONCANAVALIN A LECTIN-BASED AFFINITY CHROMATOGRAPHY FOR ENRICHMENT AND DETERMIANTION OF GLYCOPROTEINS IN BARLEY MALT
Dagmar Benkovská, Dana Flodrová and Janette Bobáľová

4-P13 THE USE OF ENZYMES CONTAINED IN THE WASHING POWDERS TO THE PREPARATION OF THE CRUDE LYZATES OF *LACTOBACILLUS* CELLS FOR MAGNETIC DNA ISOLATION
Robert Čuta, Michaela Fričová, Alena Španová and Bohuslav Rittich

4-P14 OPTIMALIZATION AND VALIDATION OF SPME-GC-FID METHOD FOR DETERMINATION OF SUSPECTED ALLERGENS IN SELECTED COSMETICS AND FOOD SAMPLES
Radka Divišová, Eva Vítová, Jana Zemanová, Blanka Loupancová and Kateřina Sklenářová

4-P15 USE OF DESCRIPTIVE SENSORY METHODS FOR EVALUATION OF VARIOUS TYPES OF FOODS
Radka Divišová, Eva Vítová, Blanka Loupancová, Kateřina Sklenářová and Libor Babák

4-P16 TRACKING CHANGES IN THE GENOME OF CAROTENOGENIC YEASTS ANALYZED BY TWO DIFFERENT GEL ELECTROPHORESIS TECHNIQUES – PFGE AND DGGE
Terezie Dvořáková, Andrea Háronikvá, Kristína Jankeje, Martina Kubáčková and Ivana Márová

4-P17 DETERMINATION OF IRON (II), IRON (III) AND TOTAL IRON IN SOME β-THALASSEMIA PATIENTS USING DIFFERENT ANALYTICAL TECHNIQUES
Nabil A. Fakhre and Dashty K. Ali

4-P18 ALTERNATIVE PROCEDURE FOR PREPARITON OF *LACTOBACILLUS* CELL LYSATES
Michaela Fričová , Robert Čuta, Alena Španová and Bohuslav Rittich

4-P19 ANALYSIS OF PHYSICO CHEMICAL PROPERTIES OF COLLAGEN ISOLATED FROM DIFFERENT ANIMAL TISSUES
Živan Gojkovič, Stanislav Obruča, Jiří Kučerík, Miloslav Pekař and Ivana Márová

4-P20 FAST METABOLOMIC APPROACH FOR ALZHEIMER DISEASE DIAGNOSIS BASED ON HIGH RESOLUTION MASS SPECTROMETRY
Raúl González Domínguez, Tamara García Barrera and Jose Luis Gómez Ariza

4-P21 USE OF THE MICROALGAE CHLORELLA SOROKINIANA FOR THE BIOTECHNOLOGICAL PRODUCTION OF IODINE AND SELENIUM FUNTIONAL FOOD
Verónica Gómez-Jacinto, Tamara García-Barrera, Inés Garbayo-Nores, Carlos Vilchez-Lobato and Jose Luis Gómez-Ariza

4-P22 CAROTENOIDS-ENRICHED BIOMASS PRODUCTION BY RED YEASTS CULTIVATED ON VARIOUS WASTE SUBSTRATES HYDROLYZED BY MICROBIAL HYDROLASES
Andrea Hároniková, Siniša Petrik, Terezie Dvořáková, Alena Pospíšilová, Ivana Márováa and Milan Čertík

4-P23 FERMENTATIVE HYDROGEN PRODUCTION BY BACTERIAL STRAIN *CLOSTRIDIUM TYROBUTYRICUM* S5.
Maria Chroboková, Alena Španová, Bohuslav Rittich, Barbora Ürgeová, Kristýna Turková and Miloš Dvořák

4-P24 IMPACT OF ELEVATED CO_2 CONCENTRATION ON KEY ENZYME OF PHOTOSYNTHESIS – RUBISCO
Miroslav Hrstka and Libor Babák

4-P25 CHARACTERIZATION OF POLYGALACTURONASES PRODUCED BY *GEOTRICHUM CANDIDUM*
Kateřina Illková, Zuzana Zemková, Jiřina Omelková, Dana Flodrová, Dagmar Benkovská, Renáta Vadkertiová and Eva Stratilová

4-P26 FATTY ACIDS DETERMINATION IN BIOLOGICAL SAMPLES BY GC-MS
Andreea Iordache, Diana Florescu, Elena Horj and Monica Culea

4-P27 MD SIMULATION ON MUTANT NEURAMINIDASE OF INFLUENZA B VIRUS AND ITS INHIBITOR
Jiraporn Tengrang, Thanyada Rungrotmongkol and Supot Hannongbua

4-P28 DETERMINATION OF PHENOLIC GLYCOSIDES AND AGLYCONS IN SELECTED KINDS OF RICE
Andrea Lichnová, Radka Valentová and Ivana Márová

4-P29 DETERMINATION OF ACTIVE COMPOUNDS IN INSTANT BABY TEAS
Andrea Lichnová, Martina Jašková and Ivana Márová

4-P30 INTERCONTINENTAL DISCRIMINATION OF PURE ORIGIN ROASTED COFFEES BASED ON THEIR METAL CONTENT
Marta Oliveira, Simone Morais, Sandra Ramos and Cristina Delerue-Matos

4-P31 EVALUATION OF HEAVY METAL CONCENTRATIONS IN ARMS OF *OCTOPUS VULGARIS*
Marta Oliveira, Simone Morais and Cristina Delerue-Matos

4-P32 POLYANILINE MICROARRAY ELECTRODES FOR CADMIUM ANALYSIS
Marta Oliveira, Paula Venda, Subramanian Viswanathan, Simone Morais and Cristina Delerue-Matos

4-P33 ANTIOXIDANT ACTIVITY OF BARLEY IN REGARD TO THE QUALITATIVE PARAMETERS OF MALT
Vít Mareček, Radim Cerkal, Tereza Hudcová and Pavel Dostálek

4-P34 INFLUENCE OF DIFFERENT KINDS OF SPICES ON ACRYLAMIDE CONTENT IN BUCKWHEAT GINGER CAKES
Lucie Marková, Zuzana Ciesarová, Kristína Kukurová, Henryk Zieliński, Danuta Zielińska and Alena Bednáriková

4-P35 DECLINE IN ACRYLAMIDE EXPOSURE BY ADOPTION OF MITIGATION TOOLS
Zuzana Ciesarová, Kristína Kukurová and Lucie Marková

4-P36 ACRYLAMIDE FORMATION IN BREAD INFUENCED BY MODIFICATIONS IN COMPOSITION
Kristína Kukurová, Lucie Marková, Alena Bednáriková, Zuzana Ciesarová

4-P37 NANOENCAPSULATION OF BEER PHENOLICS
Ivana Márová, Stanislav Obruča, Andrea Lichnová, Zorka Cihlářová, Kateřina Pařilová and Miloslav Pekař

4-P38 POLYMORPHISM IN *LOXA* GENE AND PHENOTYPE CHARACTERISTICS IN SEVERAL VARIETIES OF CZECH SPRING BARLEY
Ivana Márová, Kateřina Duroňová, Jarmila Milotová, Kateřina Pařilovř and Renata Mikulíková

4-P39 DETERMINATION OF SULPHUR AMINO ACIDS IN BARLEY, MALT AND BEER
Renata Mikulíková, Zdeněk Svoboda, Karolína Benešová and Sylvie Běláková

4-P40 DEVELOPMENT OF CAPILLARY ZONE ELECTROPHORESIS METHOD FOR MONITORING OF SELECTED ORGANIC ACIDS IN FERMENTED FOODS
Miloš Dvořák, Milena Vespalcová and Bohuslav Rittich

4-P41 PRODUCTION OF ENZYME COCTAILS FOR HYDROLYSIS OF COMPLEX WASTE SUBSTRATES
Stanislav Obruča, Petra Matoušková, Ivana Márová, Andrea Lichnová, Alena Pospíšilová and Milan Čertík

4-P42 DEPENDENCE OF BIODEGRADATION BEHAVIOR OF POLYCAPROLACTONE FILM ON THE WAY OF ITS PREPARATION
Soňa Hermanová, Jiřina Omelková, Stanislava Voběrková, Radka Bálková, Lukáš Richtera and Ludmila Mravcová

4-P43 COMPARISON OF CONTENT OF PHENOLICS AND BITTER SUBSTANCES IN BEERS WITH DIFFERENT ALCOHOL CONTENT
Kateřina Pařilová, Radana Měřínská, Pavla Benešová, Kateřina Duroňová and Ivana Márová

4-P44 COMPARATIVE ANALYSIS OF LIPOPHILIC COMPOUNDS IN EGGS OF ORGANIC ISA BROWN AND ARAUCANA HENS
Pintea Adela, Andrei Sanda, Dulf Francisc, Bunea Andrea and Matea Cristian

4-P45 COPPER, SELENIUM SUPPLEMENTED YEAST BIOMASS - A SOURCE OF MICROELEMENTS
Anežka Poláková, Silvia Šillerová, Blažena Lavová, Dana Urminská and Eva Szabová

4-P46 EFFECTS OF EXTRACTION CONDITIONS ON THE ANTIOXIDANT ACTIVITY OF BUCKWHEAT AND SPELT FLOURS
Blanka Tobolková, Martin Polovka and Milan Suhaj

4-P47 ELEMENTAL COMPOSITION AND TOTAL ANTIOXIDANT ACTIVITY OF CZECH WINES DERIVING FROM GRAPES GROWNED BY DIFFERENT AGRICULTURE METHODS
Jaromír Pořízka, Pavel Diviš and Jiřina Omelková

4-P48 EFFECT OF SELECTED INORGANIC SALTS ON ACRYLAMIDE CONTENT AND SENSORY PROPERTIES OF BREAD
Jana Sádecká, Milan Suhaj, Kristína Kukurová, Zuzana Ciesarová, Lucie Marková, Renáta Belková and Ivan Ostrovský

4-P49 COMPARISON OF VOLATILE AROMA-ACTIVE COMPOUNDS AND COLORIMETRIC CHARACTERISTICS OF ORGANIC AND CONVENTIONAL WINES
Jana Sádecká, Milan Suhaj, Emil Kolek and Ivan Ostrovský

4-P50 SELECTED CHARACTERISTICS OF POULTRY MECHANICALLY SEPARATED MEAT (MSM)
Silvie Čížková and Jan Šalplachta

4-P51 GROWTH AND PRODUCTION PROPERTIES OF RED YEASTS USING WASTE GLYCEROL AND LIPIDS AS CARBON SOURCE
Siniša Petrik, Ivana Márová, Andrea Hároniková and Iveta Kostovov

4-P52 PARTIAL PURIFICATION AND CHARACTERIZATION OF α-GALACTOSIDASE FROM *CRYPTOCOCCUS LAURENTII*
Csilla Mészárosová, Soňa Garajová, Pavel Řehulka, Nadežda Kolarova and Eva Stratilová

4-P53 TRANSGLYCOSYLASES – GLYCOSYLHYDROLASES WHICH DO NOT LIKE WATER OR ENZYME NOMENCLATURE VERSUS CAZY
Eva Stratilová and Zuzana Zemková

4-P54 EFFECT OF CONCENTRATION OF BIOMASS ON BIOSORPTION CAPACITY
Petra Šupinová, Libor Babák, Miroslava Zichová and Radka Burdychová

4-P55 YEAST *SACCHAROMYCES CEREVISIAE* AS A SOURCE OF BIOGENIC SUBSTANCES
Silvia Šillerová, Dana Urminská, Anežka Poláková, Blažena Lavová and Eva Szabová

4-P56 THE PHENOLIC CONTENT AND ANTIOXIDANT ACTIVITY OF VARIETAL SLOVAK RED WINES
Tomáš Tóth, Daniel Bajčan, Alena Vollmannová, Pavol Trebichalský, Ladislav Lahučký and Juraj Miššík

4-P57 SCREENING OF *LACTOBACILLUS* STRAINS FOR BACTERIOCIN GENES
Kristýna Turková, Bohuslav Rittich and Alena Španová

4-P58 COMPARISION OF DGGE PROFILES OF DNA ISOLATED FROM A DAIRY PRODUCT BY DIFFERENT ISOLATION METHODS
Jana Tvrdíková, Barbora Ürgeová and Olga C. Nunes

4-P59 OCCURENCE OF LACTIC ACID BACTERIA IN GRAPE MUST DURING ALCOHOLIC FERMENTATION
Markéta Valicová, Jiřina Omelková, Štěpánka Trachtová and Alena Španová

4-P60 THE COMPARISON OF CONTENT OF AROMA ACTIVE COMPOUNDS IN SAMPLES OF ELDER BERRIES (*SAMBUCUS NIGRA L.*)
Eva Vítová, Milena Vespalcová, Blanka Loupancová, Radka Divišová, Kateřina Sklenářová and Marcela Korhoňová

4-P61 RELATIONSHIP BETWEEN VOLATILE COMPOUNDS CONTENT AND SENSORY ATTRIBUTES OF STERILIZED PROCESSED CHEESE
Eva Vítová, Blanka Loupancová, Radka Divišová, Kateřina Sklenářová and Miroslav Fišera

4-P62 THE CONTENT OF LACTIC ACID BACTERIA IN CHOSEN PROBIOTIC FOODS
Eva Vítová, Blanka Loupancová, Radka Divišová, Kateřina Sklenářová and Libor Babák

4-P63 SUBSTRATE SPECIFICITY OF XYLOGLUCAN ENDOTRANSGLYCOSYLASE (XET) FROM GERMINATING SEEDS OF PARSLEY
Zuzana Zemková, Dana Flodrová, Soňa Garajová, Vladimír Farkaš and Eva Stratilová

4-P64 HETEROTRANSGLYCOSYLATING ACTIVITIES IN GERMINATING SEEDS OF NASTURTIUM
Zuzana Zemková, Dana Flodrová, Jiří Šalplachta, Ivan Zelko, Renáta Vadkertiová, Vladimír Farkaš and Eva Stratilová

4-P65 INCORPORATION OF PUSTULO-OLIGOSACCHARIDES TO PLANT CELL WALL STRUCTURES
Zuzana Zemková, Dana Flodrová, Ivan Zelko, Renáta Vadkertiová and Eva Stratilová

4-P66 PRODUCTION OF CELLULASE AND POLYGALACTURONASE ON PLANT ORIGIN WASTE
Miroslava Zichová, Kateřina Illková, Kamila Habáníková and Jiřina Omelková

4-P67 MONITORING AND DEGRADATION OF PESTICIDES WITHIN VEGETABLE GROWING IN THE SYSTEM OF INTEGRATED CROP PROTECTION
Vojtěch Hrbek, Jana Urbanová, Vladimír Kocourek and Jana Hajšlová

4-P68 MOLECULAR IDENTIFICATION OF SELECTED SPECIES OF LACTIC ACID BACTERIA AND BIFIDOBACTERIA IN FOOD ADDITIVES
Kristýna Riegelová, Alena Španová, Kristýna Turková and Bohuslav Rittich

4-P69 THE ROLE OF ENTEROCOCCI IN TYRAMINE FORMATION IN TYPICAL CZECH FERMENTED SAUSAGES HERKULES
Radka Burdychová, Libor Babák and Petra Šupinová

4-P70 CAROTENOIDS AND FATTY ACIDS PROFILE AND THEIR QUANTIFICATION IN SOME WILD AND CULTIVATED BLUEBERRIES FROM ROMANIA
Andrea Bunea, Dumitriţa Rugină, Raluca Pop, Adela Pintea, Sanda Andrei, Claudiu Bunea and Constantin Bele

4-P71 SEPARATION AND QUANTIFICATION OF CAROTENOIDS FROM GRAPES CULTIVATED IN CONVENTIONAL AND ORGANIC SYSTEMS
Claudiu Bunea, Cristian Matea, Nastasia Pop, Anca Babeş, Francisc Dulf and Andrea Bunea

4-P72 IDENTIFICATION OF SOYBEAN IN THE FOODSTUFF BY REAL-TIME PCR METHOD
Jiří Šmíd, Zuzana Godálová, Lubica Piknová and Tomáš Kuchta

4-P73 TARTRAZINE DETERMINATION FROM MUSTARD SAMPLE BY HPLC, TLC-PHOTODENSITOMETRY AND TLC- DIGITAL PROCESSING OF IMAGES
Simona Corduta Cobzac, Cristian Tudor Matea, Constantin Bele and Octavian Negrea

4-P74 THE POTENTIALITIES OF MULTI-ELEMENT COMPOSITION AND ^{87}Sr/^{86}Sr RATIO OF WINES AS TRACES OF WINE PROVENANCE
Andreea Iordache, Cezara Voica, Diana Florescu, Roxana Ionete and Monica Culea

4-P75 DETERMINATION OF DILL ESSENTIAL OIL COMPOSITION USING MODERN EXTRACTION TECHNIQUES
Manuela Stan, Maria-Loredana Soran, Codruta Varodi, Ildiko Lung

4-P76 LOW-LEVELS SPECIFICATION OF ELEMENTS IN DRINKING WATERS USING ICP-MS
Andreea Iordache, Diana Florescu, Roxana Ionete and Monica Culea

4-P77 METALLOTHIONEINS UPREGULATION IN LIVER OF THE MOUSE *MUS MUSCULUS* UNDER CADMIUM EXPOSURE
Rocío Jara-Biedma, Macarena González-Fernandez, Miguel Angel García-Sevillano, Tamara García-Barrrera, Juan López-Barea, Carmen Pueyo and José Luis Gómez-Ariza

4-P78 ENZYMATIC AND ANTIOXIDANT ACTIVITY CHARACTERISTIC OF THE VARIETIES *TRITICUM AESTIVUM,* L. AND *TRITICUM DURUM,* L.
Ján Mareček, Karolina Mocko, Eva Ivanišová, Miriam Líškován and Andrea Mendelová

4-P79 THE ANTIOXIDANT EFFECT OF HERBAL EXTRACTS IN COMPARISION WITH SYNTHETIC ANTIOXIDANTS
Ján Mareček, Eva Ivanišová, Marián Tokár and Štefan Dráb

4-P80 ANALYSIS OF PHYTOESTROGENS IN MILK SAMPLES
Ivana Sosnovcová, Věra Schulzová, Anna Hurajová, Ludmila Křížová and Jana Hajšlová

4-P81 RAPID ALCOHOL DETERMINATION IN SPIRIT DRINKS AND WINES USING MULTIVARIATE CALIBRATION WITH PRINCIPLE COMPONENT REGRESSION OF NIR DERIVATIVE SPECTRA
Milan Suhaj

4-P82 MULTIVARIATE EVALUATION OF BREAD COLOUR CHANGES AFFECTED BY SOME POTENTIAL ADDITIVES FOR ACRYLAMIDE MITIGATION
Kristína Kukurová, Zuzana Ciesarová, Renáta Belková and Milan Suhaj

4-P83 DETERMINATION AND COMPARATIVE STUDY OF SOLUBLE OXALATE IN GRAIN AMARANTH MUTANT LINES
Hricová Andrea, Suhaj Milan, Kečkešová Monika and Gajdošová Alena

4-P84 CADMIUM, LEAD AND MERCURY CONTENTS IN FISHES
Tomáš Tóth, Alena Vollmannová, Ján Tomáš, Jaroslav Andreji, Peter Lazor, Juraj Miššík, Juraj Tóth and Daniel Bajčan

4-P85 CONCENTRATIONS OF COMPOUNDS SULPHUR IMMISIONS IN ATMOSPHERE IN RELATION TO EVALUATION OF AGRICULTURAL CROPS YIELD DAMAGES
Tomáš Tóth, Peter Lazor, Ján Tomáš and Juraj Tóth

4-P86 CAN BE CRANBERRIES RISKY?
Radovan Stanovič, Alena Vollmannová, Dana Urminská, Lívia Krížová, Ján Daniel and Michal Medvecký

4-P87 AROMA ACTIVE COMPOUNDS IN SEVERAL TYPES OF PROCESSED CHEESE ANALOGUES
Kateřina Sklenářová, Eva Vítová, Radka Divišová and Blanka Loupancová

4-P88 THE CONTENT OF AROMA COMPOUNDS IN FERMENTED MILK PRODUCTS
Kateřina Sklenářová, Blanka Loupancová, Radka Divišová, Eva Vítová and Libor Babák

4-P89 VITAMINS IN YEAST *SACCHAROMYCES CEREVISIAE*
Silvia Šillerová, Dana Urminská, Anežka Poláková, Blažena Lavová and Eva Szabová

4-P90 DYNAMICS OF THE CONTENT OF CAROTENOIDS AND LYCOPENE IN TOMATO FRUIT (*LYCOPERSICON ESCULENTUM* MILL.) DURING STORAGE AND AFTER PROCESSING
Ján Mareček, Andrea Mendelová and Alena Andrejiová

4-P91 DETERMINATION OF RESIDUAL FORMIC ACID IMPURITY IN HYALURONATE OLIGOSACCHARIDES
Tereza Ehlová, Veronika Leierová, Martina Hermannová, Daniela Šmejkalová and Vladimír Velebný

4-P92 IDENTIFICATION OF BARLEY VARIETIES RECOMMENDED FOR CZECH BEER
Renata Mikulíková, Zdeněk Svoboda, Karolína Benešová and Sylvie Běláková

4-P93 DEVELOPMENT OF A FOCUSED MICROWAVE-BASED EXTRACTION METHOD FOR THE DETERMINATION OF ARSENICALS IN FOOD SAMPLES
Manuel Contreras-Acuña, Tamara García-Barrera and José Luis Gómez-Ariza

4-P94 TOTAL ANTIOXIDANT STATUS, LIPIDS AND PROTEINS OXIDATION LEVELS IN SUBCLINICAL MASTITIS COW'S MILK
Sanda Andrei, Sorana Matei, Adela Pintea, Andrea Bunea, Dumitrita Rugina, Razvan Stefan and Ioan Stefan Groza

4-P95 DIAGNOSIS AND MONITORING BY GC/MS
Elena Horj, Diana Florescu, Andreea Iordache, Cornelia Mesaros, Romana Vulturar and Monica Culea

4-P96 LEUCINE TRANSMEMBRANAR TRANSPORT STUDY IN THE HUMAN RED CELL
Elena Horj, Diana Florescu, Andreea Iordache, Silvia Neamţu and Monica Culea

AUTHOR INDEX

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