

PROMOTE

Konference **Nano Ostrava 2011** byla podpořena v rámci projektu "Podpora zapojení pracovníků VaV do projektů mezinárodní vědecko-výzkumné spolupráce," (PROMOTE), registrační číslo CZ.1.07/2.3.00/09.0131.
Uvedený projekt je financovaný z prostředků Evropského sociálního fondu a státního rozpočtu České Republiky v rámci OP Vzdělávání pro konkurenceschopnost.









INVESTICE DO ROZVOJE VZDĚLÁVÁNÍ

Dear Conference participants, Visitors and Colleagues,

We welcome you to the second international conference **Nano Ostrava 2011**. The symposium will be held at VŠB–TU of Ostrava, one of the oldest Czech universities. The University has more than 24 thousand students who study at seven University faculties. From the number, about 1100 students are from abroad.

The conference has been organized under cooperation of scientists from the Nanotechnology Centre (VŠB–TU of Ostrava), Institute of Physics (VŠB–TU of Ostrava), Institute of Geonics AS CR and PROMOTE project (Support of RTD workers` participation in international projects of scientific and research cooperation - CZ.1.07/2.3.00/09.0131).

After the resounding success of the 1st meeting, for which we thank the participants, we hope to surpass our earlier success this year. The meeting is a platform to facilitate open discussions between scientists, students and representatives from companies with a vested interest in furthering the development of nanomaterials and nanotechnology.

There will be several sessions, where invited lecturers and presenters from the industry who will focus on areas of importance to nanomaterials and nanotechnology.

1. Tools for preparation and characterization of nanomaterials
2. Physical properties of nanostructures
3. Nanotechnology for environment
4. Advances in nanomaterials
5. Environmental and health risks of nanomaterials
6. Nanomaterials of clay minerals and zeolites
7. Communication in the Research and Technology Development

We wish you successful and fruitful conference – and enjoy Ostrava!

Organizing committee of NOM 2011

Organizing Committee:

Jaromír Pištora (Conference Chairman)
Nanotechnology Centre, VŠB-Technical University of Ostrava, Czech Republic

Karla Barabaszová
Nanotechnology Centre, VŠB-Technical University of Ostrava, Czech Republic

Gražyna Simha Martynková
Nanotechnology Centre, VŠB-Technical University of Ostrava, Czech Republic

Daniela Plachá
Nanotechnology Centre, VŠB-Technical University of Ostrava, Czech Republic

Sylva Holešová
Nanotechnology Centre, VŠB-Technical University of Ostrava, Czech Republic

Vladimíra Plačková
VŠB-Technical University of Ostrava, Czech Republic

Eva Plevová
Institute of Geonics AS CR, Ostrava, Czech Republic

Pavel Konečný
Institute of Geonics AS CR, Ostrava, Czech Republic

Klára Dluhošová
University of Ostrava, Czech Republic

Lukáš Straňák
University of Ostrava, Czech Republic

Academic Committee:

Jaromír Pištora (Conference Chairman)
Nanotechnology Centre, VŠB-Technical University of Ostrava, Czech Republic

Hatem Akbulut
Sakarya University, Turkey

Radim Blaheta
Institute of Geonics AS CR, Ostrava, Czech Republic

Pavla Čapková
Nanotechnology Centre, VŠB-Technical University of Ostrava, Czech Republic

Yafei Lu
Beijing University of Chemical Technology, Beijing, China

Mark Rümmeli
Institute for Solid State Research, IFW Dresden, Germany

Jan Šubrt
Institute of Inorganic Chemistry AS CR, Prague, Czech Republic

Marta Valášková
Nanotechnology Centre, VŠB-Technical University of Ostrava, Czech Republic

Welcome from NANO Ostrava 2011 Organizing Committee Chair

On behalf of NANO Ostrava 2011 - Organizing Committee, I extend my hearty welcome to you all to the 2nd NANO Ostrava. You may be aware that this specialized conference in the field of nanotechnology is being organized in the land of ancient European civilization or more known as Moravian civilization. About 24,000 years ago this civilization started becoming more specialized in this land. Today we have gathered here to continue this old tradition with modern technology.

Ostrava is the third largest city in the Czech Republic and thanks to its economical, cultural, educational and political importance belongs among well-known places in our small country in the heart of Europe".

VŠB - TU Ostrava is a modern technical university with a large scale of technical and economic fields of study. Located in the heart of the industrial Northern Moravia region, the university represents a resource of technical, educational and scientific activities and development not only for the region of Northern Moravia but for the whole country.

Our Program Committee has prepared an interesting and informative technical program outlining current progress and activities in many research areas of nanotechnology. Over 100 papers from 10 countries have been accepted and scheduled for the 3-day conference.



Jaromír Pištora

Chairman, NANO Ostrava 2011

Sponsors

The organizers would like to thank the following companies and Moravian-Silesian region for generously sponsoring this meeting:

Moravian-Silesian region



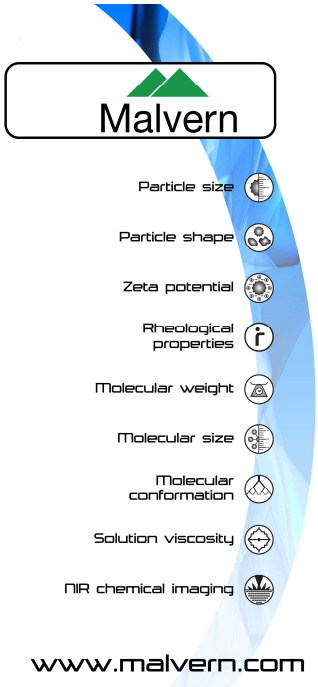
Anamet, s.r.o.



Kurt J. Lesker, company



Malvern Instruments Ltd



Měřící technika - morava



Nicolet CZ, s.r.o.



Specion, s.r.o.



Univerzita Palackého v Olomouci



Vitrum, s.r.o.



Table of content

Invited Lectures:

***A. Bachmatiuk**, R. G. Mendes, I. Ibrahim, D. Placha, G. Simha Martynkowa, B. Büchner, M. H. Rümmeli*: Synthesizing carbon nanotubes and graphene from oxide catalysts 1

M. Cada: Optical functionalities for nanophotonic devices 2

Mohamed EL Mansori: Multiscale manufacturing of micro/nano structured surfaces 3

***Ivo Safarik**, Katerina Horska and Mirka Safarikova*: Magnetically responsive biocomposites for xenobiotics removal 4

Ivo Vávra: Properties of metallic nanoparticles and nanocomposites prepared by vacuum technologies 5

Štefan Višňovský: Optics in magnetic nanostructures 6

Oral Presentations:

Topic 1 - Tools for preparation and characterization of nanomaterials

***Y. Aydin**, M. O. Guler, O. Cevher, D. Gultekin, H. Akbulut*: The Effect of Oxygen Partial Pressure on the Formation of Nano Crystalline Zinc Oxide Films 7

***O. Cevher**, M. O. Guler, D. Gultekin, H. Akbulut*: Effect of Process Parameters on the Growth of SnO_2:Sb Thin Films Prepared by R.F. Magnetron Sputtering 8

***M. O. Guler**, H. Akbulut*: The Effect of Oxygen Partial Pressure on the Thin Film Stress of SnO_2 Coatings 9

A. Vítek, ***R. Kalus***: Calorimetry of gas-phase atomic and molecular clusters 10

L. Matějová, R. Fajgar, Z. Matěj, Y. Maléterová, O. Šolcová: Metal oxide aerogel 11

***Morozová M.**, Klusoň P., Veselý M., Dzik P., Šolcová O.*: The ink-jet printed nanostructured TiO_2 film electrode 12

***S. Novák**, R. Hrach, M. Švec*: Computer study of morphology and electrical properties of (nano)composites 13

Deniz Gultekin, ***Ubeyd Tocoglu**, Mehmet Oguz Guler, Ozgur Cevher, Hatem Akbulut*: Investigation of Al doping effects on the ZnO thin films produced by sol gel spin coating 14

***J. Tokarský**, T. Andrýsek, P. Čapková*: Molecular modeling of gel-based emulsions with cyclosporine A 15

Topic 2 - Physical properties of nanostructures

***F. Staněk**, M. Lesňák, J. Pištora, D. Ciprián*: Prism coupling for planar structures with magnetic ordering 16

*Deniz Gultekin, **Ubeyd Tocoglu**, Mehmet Oguz Guler, Ozgur Cevher, Hatem Akbulut*: Characterization of SnO_2 nanostructured thin film coatings prepared by precipitation method 17

***J. Vlček**, M. Lesňák, J. Pištora*: Magneto-optical magnetic field sensor: theoretical study 18

***Koki Watanabe** and Yoshimasa Nakatake*: Electromagnetic scattering from imperfectly periodic array of circular cylinders 19

Topic 3 - Nanotechnology for environtment

***A. Baciu**, A. Pop, A. Remes, F. Manea, G. Burtica*: Non-enzymatic electrochemical determination of glucose on Ag-doped zeolite-carbon nanotube composite electrode 20

***A. Matlochová**, D. Plachá*: Adsorption of polycyclic aromatic hydrocarbons by carbon nanotubes 21

***S. Motoc**, F. Manea, A. Pop, R. Pode, G. Burtica*: Determination of ibuprofen in water using Ag-doped zeolite-expanded graphite composite electrode 22

***A. Schröfel**, G. Kratošová, M. Šafaříková, I. Šafařík, M. Natšinová, V. Holišová*: Biosynthesis of silver and gold nanoparticles using diatoms and their direct application in catalysis.....24

Topic 4 - Advances in nanomaterials

***H. Akbulut**, H. Gül, A. Alp*: Process Parameters Optimizations for Oxide and Carbide Nano Particle Reinforced MMCs Coatings by Electro co-deposition 25

***R. Dvorský**, J. Trojková, J. Luňáček, K. Piksová, K. Klouda*: Preparation of nanocomposite resistive material with modified silicon nanopatricles $Si(C_{60})$ in tin metal matrix 26

***H. Gül**, A. Alp, H. Akbulut*: Possibility of Producing Ni/Al_2O_3-SiC Hybrid Nanocomposites by Electro-codeposition with double surfactant addition effect 28

***J. Chvojka**, D. Lukáš*: Special collector for deposition oriented nanofibres 29

***R. Karslıoğlu**, M. Uysal, H. Akbulut*: Effect of Sintering Current Density on the Production of MWCNT Reinforced Bronze Matrix MMCs 30

***H. Köse**, A. O. Aydın, H. Akbulut*: Synthesis and characterization of SnO_2 nano-sized anode materials stabilized by tartaric acid 31

***E. Košťáková**, J. Grégr, P. Pokorný, L. Mészáros, M. Chotěbor*: Carbon nanostructures produced by CCVD method 32

***P. Mikeš**, P. Pokorný, R. Charvát, D. Lukáš*: Electrospinnig as a source of X-rays.....33

***L. Sitek**, J. Foldyna, J. Klich, P. Martinec, D. Nováková*: Precursors and carriers of nanoparticles prepared by water jet disintegration.....34

***P. Suchomel**, L. Kvítek, A. Panáček, R. Prucek*: Two-step preparation of silver nanoparticles 35

***M. Uysal**, G. Aydın R. Karslıoğlu, A. Alp, H. Akbulut*: Electro co-deposition of Sn/MWCNT Nanocomposites on the Copper Substrates: Effect of Current Density 37

***Miroslav Vlček**, Sigmund Schroeter, Karel Pálka, Zdeněk Černošek, Jana Holubová*: Nanostructuring of chalcogenide glasses - methods and their applications 38

Topic 5 - Environtmental and health risks of nanomaterials

***Kateřina Dědková**, Aleš Panáček, Virgínia Aragón Fernández, Robert Prucek, Libor Kvítek*: Study of Antibacterial Activity of silver NPs against animal pathogens 39

R. Kucerova, P. Fecko, B. Lyckova, ***L. Koval**, L. Kovalova, L. Kucerova*: Application of biodegradation of PAU, PCB and NEL soil samples from the hazardous waste dump in Pozd'átky (Czech Republic) 41

***J. Kukutschová**, V. Matějka, K. Malachová, J. Dvořáčková, P. Filip*: Non-exhaust nano- and micro-particulate emissions from road tradic and its potential environmental and health impact42

***L. Kvitek**, A. Panacek, R. Prucek, J. Soukupova, M. Kolar*: Biological activity of silver nanoparticles 43

Topic 6 - Nanomaterials of clay minerals and zeolites

***J. Drahokoupil**, Pavel Hrabánek, A. Zikánová, M. Kočiřík*: X-ray diffraction study of MFI zeolite membrane.....45

***V. Matějka**, P. Kovář, M. Šupová, K. Mamulová Kutláková, K. Kočí*: Clay minerals as a support for photocatalytically active nanoparticles.....46

***P. Praus**, O. Kozák*: ZnS and CdS nanoparticles on montmorillonite and their application for photocatalysis 47

Poster Presentations:

***Jan Berger**, Jan Proška, Filip Novotný, Radek Fajgar*: Cicada wings as biotemplate and their modifications 48

***E. Bretšnajdrová**, L. Svoboda, J. Zelenka*: Comparison of different methods for evaluation of shape, size and particle size distribution of selected nanomaterials.....49

***Zdeněk Bureš**, Miroslav Vlček*: Preparation and stabilisation of nanogold and nanosilver.....50

***Robert Czochara**, Rafał Pokrop, Grzegorz Litwinienko*: Synthesis, thermal stability and antioxidant properties of selected C_{60} derivatives.....52

***G. Kratosova**, A. Schrofel, V. Plasek, V. Holisova, M. Natsinova*: Biosynthesis of silver and gold nanoparticles using *Bryophytes* 53

***S. Krejcikova**, K. Koci, L. Obalova, L. Capek and O. Solcova*: Optimized Doped Titania for Photocatalysis Applications 54

*Agnieszka Krogul, **Magda A. Kroteń**, Jadwiga Skupińska, Grzegorz Litwinienko*: DSC, TGA and X-ray analysis of $PdCl_2(X_nPy)_2$ complexes 55

***M. Macháčková**, J. Tokarský, and P. Čapková*: Molecular modeling of polymeric drug carriers for cyclosporine A 56

***Piotr Piotrowski**, Magdalena A. Kroteń, Grzegorz Litwinienko*: Synthesis and studies of antioxidant properties of selected fullerene C_{60} derivatives 57

***M. Pospíšil**, P. Kovář, E. Káfuňková, K. Lang, F. Kovanda*: Molecular simulations of LDH intercalated with porphyrin anions 58

***P. Praus**, R. Dvorský, O. Kozák*: Precipitation of ZnS nanoparticles and their deposition on montmorillonite 59

M. Valášková: Characterization of vermiculite structure using X-ray powder diffraction necessary to their use in nanocomposites 60

*Dana Rosická, **Jan Šembera***: Numerical study of magnetic forces between zero-valent iron nanoparticles and their aggregates 61

***L. Štolcová**, J. Proška, F. Novotný, M. Procházka, I. Richter*: Fabrication of SERS substrates using self-assembled nanostructures 62

***A. Zolanvar**, H. Sadeghi, S. Ranjbaran, M. Moslemi, F. Kaj*: X-ray diffraction and stress dependence of magnetic properties of Fe nanometer thickness thin films 63

***J. Matoušek**, I. Hovorková, R. Bačovská*: Zero-valent nano-size iron particles in reductive dehalogenation of persistent organic pollutants in aquatic environment 64

***E. Plevová**, V. Valovičová, Z. Navrátilová, L. Vaculíková, G. Simha Martynková*: Thermal behaviour of monmorillonites modified with organic cations 65

***M. Rakap**, S. Özkar*: Hydrogen generation from the hydrolysis of sodium borohydride and ammonia-borane using hydroxyapatite-supported cobalt(0) nanoclusters as Catalyst 66

***J. Wiener**, Vejsadová, A. Chládová*: Photocatalytic activity of TiO_2 nanoparticles tested on yeasts 67

*S. Ntaka, **J. Wiener***: Sorption-filtration process using Polyamide 6 Nanofibres as a sorbent to remove dye from wastewater 68

*H. Gül, **A. Alp**, H. Akbulut*: The effect of surfactant concentration on Ni/Al_2O_3-hBN hybrid composite coatings by electro co-deposition 69

***Vijay Baheti**, A. M. Rehan Abbasi, Jiri Militky*: Influence of ball milling conditions on particle size distributions of fly ash 70

***K. Barabaszová**, M. Valášková., P. Kalivoda*: Ultrasonic treatment of vermiculite particles ...71

***E. Bulut**, A. Örnek, M. Özacar*: Doping effect of Fe^{3+} and Ni^{2+} cations on synthesis of spinel $LiM_xMn_{2-x}O_4$ cathode active nanoparticles for rechargeable lithium ion batteries 72

***E. Bulut**, A. Örnek, M. Özacar*: Effect of calcination temperature on synthesis of $LiMn_2O_4$ cathode active nanoparticles for rechargeable lithium ion batteries 74

***B. Cigánová**, J. Wiener*: Plasma treatment of polyamide fibers and nanofibers 76

***M. Karlíková**, L. Kvítek, A. Panáček, R. Prucek, M. Kolář*: Study of preparation of composite materials containing silver nanoparticles 77

***R. Karslıoğlu**, M. Uysal, M. Sancar, H. Akbulut*: Coating Ni-Co-MWCNT Metal Matrix Nanocomposites by Electrodeposition.....79

***H. Köse**, A. O. Aydın, H. Akbulut*: Fabrication of nano-sized SnO_2 powder for anode of Li-ion battery by sol-gel method 80

***M. Kušnerová**, J. Valíček, M. Zeleňák, M. Harničárová, P. Hlaváček, S. Hloch, M. Řepka, V. Kuběna*: Prediction of change of energy embodied in materials in ECAP technology 81

***J. Lang**, V. Matějka, J. Vontorová*: Preparation of multilayer TiO_2 films 82

Xiaoning Zhao, Shicheng Qi, ***Yafei Lu***: Synthesis and characterization of carborane polyesters83

***K. Luberda-Durnaś**, V. Matějka, K. Mamulková-Kutlaková, W. Łasocha*: Simple methods of synthesis ZnS nanoparticles.....84

***M. Najari**, M. Kasiri, H. Tiznobaik, S. Niakan*: Review: Enhancement of the Interfacial Strength between Carbon Fiber and Matrix in Composites by Growing Carbon Nanotubes on Carbon Fiber.....85

***A. Örnek**, E. Bulut, M. Özacar*: Effect of different carbon sources on a particle size of $LiFePO_4$/C nanocomposite cathode active material for lithium-ion batteries 87

A. Örnek, E. Bulut, ***M. Özacar***: Effect of temperature on synthesis nano $LiFePO_4$/C cathode active material for lithium-ion batteries 89

F. Dogan, ***M. Ozacar**, H. Akbulut, M. O. Guler*: Effect of the Temperature on SnO_2 Thin Films Deposited by APCVD 91

***A. M. Rehan Abbasi**, V. K. Baheti, Jiri Militky*: Production of Nanoparticles of Polypyrrole by Ball Milling.....93

***M. Samlíková**, S. Holešová*: Preparation of organoclays with antibacterial properties.....95

***M. Sivera**, L. Kvitek, R. Prucek, A. Panacek*: Study of Silver Nanoparticles Stabilization Performed by Gelatin 96

***S. Šustek**, L. Svoboda, J. Zelenka, K. Zetková, J. Kára*: Modification of hydrogel using collagen and nanostructured clay 98

***J. Zdrálková**, M. Valášková*: Impact of mechanical and chemical treatments on talc structure99

***K. Gröplová**, S. Rebilasová, P. Peikertová, V. Matějka, J. Kukutschová*: Quartz/TiO_2 composite and its acute aquatic toxicity.....100

***E. Gryčová**, J. Seidlerová, P. Štrympl*: Chemical and physical properties of salivary gland stones.....101

P. Danihelka,V. Mička, K. Lach, ***V. Hase****, D. Kupka*: Nanoparticles emissions from molten metals 103

P. Danihelka, K. Lach, V. Mička, ***V. Hase***: Nanoparticles in the Ostrava urban atmosphere .. 104

P. Majorová, ***J. Seidlerová****, G. Simha Martynková*: Delamination of phylosilicates 105

S. Holešová*, P. Pařík, M. Ludwig*: Clay supported 2-phenylimidazole derivatives for catalysis of Henry reaction..... 106

P. Hrabánek*, A. Zikánová, J. Drahokoupil, M. Matějková, I. Jirka, L. Brabec, V. Fíla, B. Bernauer, M. Kočiřík*: In-situ synthesis of oriented zeolite MFI layers 107

M. Reli, O. Kozák, P. Praus, ***K. Kočí****, L. Obalová*: Nanocomposit ZnS-MMT for photocatalytic reduction of CO_2 109

K. Karásková, T. Kulová, P. Kustrowski, ***L. Obalová****, K. Jirátová, F. Kovanda*: The alkali metals as the promoters in Co-Mn-Al mixed oxide..... 110

Synthesizing carbon nanotubes and graphene from oxide catalysts

A. Bachmatiuk[1], R. G. Mendes[1], I. Ibrahim[1], D. Placha[2], G. Simha Martynkowa[2], B. Büchner[1], M. H. Rümmeli[1]

[1]IFW Dresden, P.O. Box 270116, 01171 Dresden, Germany

[2]Nanotechnology Centre, VSB Technical University of Ostrava, 17. listopadu 15, 708 33 Ostrava – Poruba, Czech Republic

a.bachmatiuk@ifw-dresden.de

Metal free carbon nanostructures are desirable materials for numerous potential applications in composites, drug delivery and electronics. Within the realm of Si technology it is well known that metals can reduce chip lifetime. Hence, the use of non-metallic catalysts for sp^2 carbon nanostructures is desirable for silicon compatibility and also composites since metals can degrade their performance. Recently various investigations have successfully implemented ceramic catalyst particles, for example, SiO_2, ZrO_2, SiC, MgO or Al_2O_3. Thus far our research has shown that some oxides reduce through carbothermal reduction, while other oxides remain stable when exposed to CVD processes. Graphitisation is possible from both, however the mechanisms behind sp^2 carbon formation appears to be fundamentally different between the two. In the case of reducible oxides, carbon dissolution and precipitation seem likely. Graphitisation over irreducible oxides is an entirely surface based process.

Keywords: carbon nanostructures, oxide catalysts, graphene formation

Optical functionalities for nanophotonic devices

M. Cada

Dalhousie University, Canada

michael.cada@dal.ca

Photonic devices utilizing nonlinear optics have been studied, designed and implemented over the last several decades. In 1875 J. Kerr showed that a dc electric bias could induce birefringence in optically isotropic media. The birefringence is proportional to the square of the dc bias. This Kerr effect can be described by a third-order nonlinear susceptibility and has traditionally been associated with all-optical responses. Various functionalities have been demonstrated such as all-optical switching, optical bistability, four-wave mixing, or soliton generation.

Although some photonic devices have been conceptually successful, material problems still exist. Optical crystals possessing strong linear electro-optic effects are not compatible with device integration technologies. The scalability has some fundamental limitations due to material and technological issues. The third-order nonlinearity, on the other hand, exists in a wide variety of commonly used materials that can be integrated and have already been used in microelectronics and for optical waveguide components.

The fundamental problem with the third-order nonlinearity is that this effect is very weak in most materials. When a figure of merit of device functionality is considered, the linear electro-optic materials offer superior operational parameters. The promise of the development of new materials that fall under the umbrella of nanotechnology is quite attractive. Structures, materials and devices are being developed on the nanometer scale, thus promising a potential to open a new world of scalability and integration.

It is well known that reducing the size of the optical materials structures to a nanoscale (diameter smaller than the Bohr exciton in a bulk crystal of the same material) leads to significant (orders of magnitudes) enhancements of the third-order optical susceptibility due to the confinement effect. It is thus believed that the electro-optic and all-optical devices based on the third-order nonlinearity may offer an attractive alternative solution for integrated opto-electronics and nanophotonics.

In this work, a different use of the third-order non-linearity is considered. An interplay between the electro-optic and all-optical Kerr effects is suggested as a functional phenomena exploitable in novel photonic devices. It can only be realized with the help of the quadratic, as opposed to a more frequently used linear electro-optic, effect. Two illustrative examples are discussed, namely an electrically controlled multistable switch (ECMS) with memory, and an electrically tunable Bragg grating (ETBG).

Keywords: Optical nonlinearity, Kerr effect, photonic devices, nanostructured materials

Multiscale manufacturing of micro/nano structured surfaces

Mohamed EL Mansori

Arts et Métiers ParisTech, France

Manufacturers are searching for honing and finishing processes that enable engine builders to achieve the tough emissions standards looming on the horizon. The major issue is to hone hole of cylinder liner sizes down into the millionths. The liner surface is in fact the “original” micro-nano scale structured surface; designed with a deterministic pattern of high aspect ratio features and anisotropy of surface properties. It comprises alternating flat plateaus (bearing regions) and deep valleys for lubricant, transportation. To manufacture such a functional surface, abrasive honing is the process of choice. The objective of this presentation is to present a robust methodology that explores the required surface characteristics, based on the premise that an intimate connection exists between the abrasive wear mechanisms prevailing during honing and the multi-scale modification of topographical signatures formed as a result of those mechanisms.

Keywords: Nanofinishing, Multiscale Manufacturing, Functional Surfaces

Magnetically responsive biocomposites for xenobiotics removal

Ivo Safarik, Katerina Horska and Mirka Safarikova

Institute of Nanobiology and Structural Biology of GCRC AS CR, Czech Republic

ivosaf@yahoo.com

Magnetic nano- and microparticles have already found many important applications in various areas of biosciences, medicine, biotechnology, environmental technology etc. These smart materials exhibit different types of response to external magnetic field. In most cases they can be described as composite materials, where the magnetic properties are caused by the presence of iron oxides nano- or microparticles. Such materials can be efficiently separated from difficult-to-handle samples and targeted to the desired place, applied as contrast agents for magnetic resonance imaging or used to generate heat during exposure to alternating magnetic field.

In addition to magnetic composites based on synthetic or inorganic materials, a large amount of magnetic biocomposites has been developed and used. In many cases magnetic fluids (containing iron oxides nanoparticles) or micrometer-sized magnetic particles can be used to convert diamagnetic biological materials (e.g., individual biopolymers, complex polysaccharides of plant origin, microbial and algae cells, etc.) into their magnetic derivatives.

Biosorption of different types of xenobiotics using a variety of biological materials represents emerging possibility for the reduction of environmental pollution. Biosorption has several advantages compared with conventional techniques, namely low cost of the biosorbents (majority of them is made from abundant or even waste material). In addition, large variability of biological materials and their pretreatment enables to select an appropriate biosorbent exhibiting at least partial selectivity in xenobiotics adsorption. In order to improve manipulation of biosorbents, their magnetic derivatives can be used. In small scale, magnetic biocomposites have been successfully used as adsorbents for the removal of both organic and inorganic xenobiotics, such as dyes, pesticides, heavy metal ions and radionuclides. A recently published book chapter (Safarik, I., Horska, K., Safarikova, M.: Magnetically responsive biocomposites for inorganic and organic xenobiotics removal. In: Microbial Biosorption of Metals (Kotrba, P., Mackova, M., Macek, T., Editors), Springer, 2011, pp. 301-320) offers an extensive list of described magnetic biocomposites and their applications.

Magnetic modification of biological materials enables to prepare stimuli-responsive materials with interesting properties. Despite the fact that currently magnetically responsive biocomposites have been tested only in laboratory experiments, there is a great potential for their large-scale applications in the near future.

Keywords: biosorption, biocomposites, magnetic separation, xenobiotics

Properties of metallic nanoparticles and nanocomposites prepared by vacuum technologies

Ivo Vávra

Institute of Electrical Engineering, Slovak Academy of Sciencies, Dúbravská cesta 9, 84104 Bratislava, Slovak Republic

elekvavr@savba.sk

Nanomaterials are playing a substantial role in overall materials research and applications. Nowadays, there exist lot of nanomaterial production technologies, and many of them are able to deliver tens of kilograms of a nomaterial per day. The applications span from mechanics, optics, electronics, and biomedicine to cosmetics and blue-coloured drinks. In many cases the nanomaterial is in fact a nanocomposite with nanoparticles dispersed in a proper matrix. Regarding the nanoparticle shape, one encounters tubes, rods, spheres (even the hollow spheres), cubes, octahedrons, ribbons or many dendritic forms. Regarding the electrical conductivity, the nanoparticles can be metals, semiconductors or insulators.

Nanomaterials suitable for sensorics are intensively researched. The reason is that the surrounding analyte can easily influence a nanoparticle state because of a high ratio of the number of surface/volume atoms. This is the main reason why nanomaterials exhibit high sensitivity to gases, heavy-metal ions or viruses. A change in the state of nanoparticles can be manifested itself in variation of electrical, optical, magnetic or several other parameters.

In this contribution, a review of metallic nanoparticles and nanocomposites and related vacuum technologies will be given.

Vacuum evaporation and vacuum sputtering: Theses methods are widely used in microelectronic industry for the deposition of thin films. There are lot of various modifications of these methods. Some innovative features are still being developed. One of them is vacuum deposition on the liquid surface. Metallic nanoparticles can be deposited on the surface of ionic liquids (IL). The nanoparticles will be formed on top of an ionic liquid, and then they sink into the IL volume which prevents them from coagulation. The result is a colloid containing metallic particles of small size dispersion.

Liquid-Phase Pulsed Laser Ablation (LP-PLA): this advanced method uses a target immersed in a liquid. The ablated material condenses in a liquid, or rather on the inner side of the bubbles that are formed simultaneously with a metal vapour in the moment of the laser pulse incidence. One of the advantages of the method is that after each pulse the metal nanoparticles remain dispersed in the liquid. After many pulses, a sufficiently concentrated colloid is obtained. If needed, it is possible to make a nanocomposite polymer film by mixing the colloid with a polymerizing solution. Though the pioneering experiments with LP-PLA were performed about 20 years ago, the dominant technique became PLA in vacuum. Nowadays, there is a renaissance of the former method because new materials are needed that cannot be achieved by PLA in vacuum.

In conclusion, the problem of alloy metallic nanoparticles preparation will be discussed.

Optics in magnetic nanostructures

Štefan Višňovský

Charles University of Prague, Faculty of Mathematics and Physics, Ke Karlovu 3,121 16 Prague, Czech Republic

Stefan.Visnovsky@mff.cuni.cz

Magnetic nanostructure finds practical use in spin electronics, magnetophotonic devices, sensors of weak magnetic fluxes, as well as in medicine. The response of a magnetic nanostructure to polarized waves provides information on electronic structure, spin ordering, chemical profile and interface nature. The approach is illustrated on (1) 1D structures formed by ferromagnetic Fe, Ni, and Co sandwiched between metals and dielectrics, (2) 1D magnetic oxide structures. The spectral range of concern covers the region from 1 to 5 eV accessible with quartz optics. The explanation of the spectroscopic results makes use of physical models and material tensors of constituents.

Keywords: magnetic nanostructures, magnetophotonic devices

The Effect of Oxygen Partial Pressure on the Formation of Nano Crystalline Zinc Oxide Films

Y. Aydin, M. O. Guler, O. Cevher, D. Gultekin, H. Akbulut

Department of Metallurgical and Material Engineering, Sakarya University, TURKEY

guler@sakarya.edu.tr

In this present work, zinc oxide films were obtained via thermal evaporation followed by R.F. plasma oxidation onto sodium silicate glasses by using high purity zinc targets. The oxidation was performed with O_2/Ar ratio of 12.5/87.5, 25/50 and 50/50 respectively for 1 hour and the effect of oxygen gas pressure on the zinc oxide film formation was investigated. The phase composition, mean grain size and texture orientation of the deposited films was investigated by X-ray diffraction (XRD) techniques and the surface morphology of the deposited films were investigated by Scanning Electron Microscopy (SEM) as well as Atomic force Microscopy (AFM). The resistivity of the oxidized films was also analyzed with 4 – probe resistivity measurement tests.

Keywords: Nano crystalline ZnO, thermal evaporation, plasma oxidation, electrical resistivity

Effect of Process Parameters on the Growth of SnO_2:Sb Thin Films Prepared by R.F. Magnetron Sputtering

O. Cevher, M. O. Guler, D. Gultekin, H. Akbulut

Department of Metallurgical and Material Engineering, Sakarya University, Turkey

ocevher@sakarya.edu.tr

In this work, antimony doped tin oxide (SnO_2:Sb) thin films have been deposited on glass substrates using a radio frequency (r.f.) magnetron sputter process with a antimony doped tin oxide (SnO_2:Sb) target in a mixed oxygen/argon gas environment. Process parameters such as oxygen partial pressure and r.f. power have been studied. The films were deposited for 1h in pure Ar and a mixture of Ar and O_2 environment with O_2/Ar ratio of 5/95 and 10/90. The sputtering deposition was performed by using R.F. powers of 75 W, 100 W and 125 W, respectively. The crystallographic structure of the sample was determined by X-ray diffraction (XRD). The average surface roughness (R_a) measured with atomic force microscopy (AFM). The electrical resistivity of the deposited films was measured by the four-point-probe method.

Keywords: SnO_2:Sb, thin films, r.f. magnetron sputtering

The Effect of Oxygen Partial Pressure on the Thin Film Stress of SnO_2 Coatings

M. O. Guler, H. Akbulut

Department of Metallurgical and Material Engineering, Sakarya University, TURKEY

guler@sakarya.edu.tr

In this study, tin oxide films were deposited onto silicon wafer substrates via radio frequency (r.f.) magnetron sputter process in a mixed oxygen/argon gas environment. The sputtering deposition was performed with O_2/Ar ratio of 0/100, 0.2/99.8, 0.5/99.5, 1/99, 2/98, 5/95 and 10/90 respectively. The thicknesses of the deposited films were fixed at 1.5 mm. The structural properties of as-deposited films were analyzed by X-ray diffraction (XRD) and surface morphology of the deposited films was evaluated by Scanning Electron Microscopy (SEM). Residual stresses in the films were evaluated using the Stoney equation after deposition by using a non-contact surface profiler.

Keywords: SnO_2, rf sputtering, thin films stress, Stoney's equation

Calorimetry of gas-phase atomic and molecular clusters

A. Vítek[1], R. Kalus[2]

[1]University of Ostrava, Department of Physics, Czech Republic; [2]VŠB – TU Ostrava, Department of Applied Mathematics, Czech Republic

Rene.Kalus@vsb.cz

The importance of calorimetric studies of nano- and subnanoparticles will be discussed with the main emphasis on detecting and analyzing structural and/or phase changes in these systems induced by thermal excitations. First, a brief overview of experimental methods available in the present-date cluster science will be given together with a few examples of state-of-the-art measurements. Second, theoretical methods for calorimetric simulations (and beyond) will be outlined, technical and methodological details will be briefly discussed, and some technical problems of numerical modeling of gas-phase clusters calorimetry mentioned and solutions proposed. Finally, the performance and practical use of theoretical methods will be demonstrated by a couple of examples of our own work in the field, including homogeneous rare-gas clusters (either neutral or ionic), pure water clusters, protonated water clusters, and high-pressure water clusters doped by a methane molecule. The question of how numerical simulations can provide a deeper insight into the nature of phase changes in clusters (going, thus, beyond purely calorimetric studies) will also be briefly addressed.

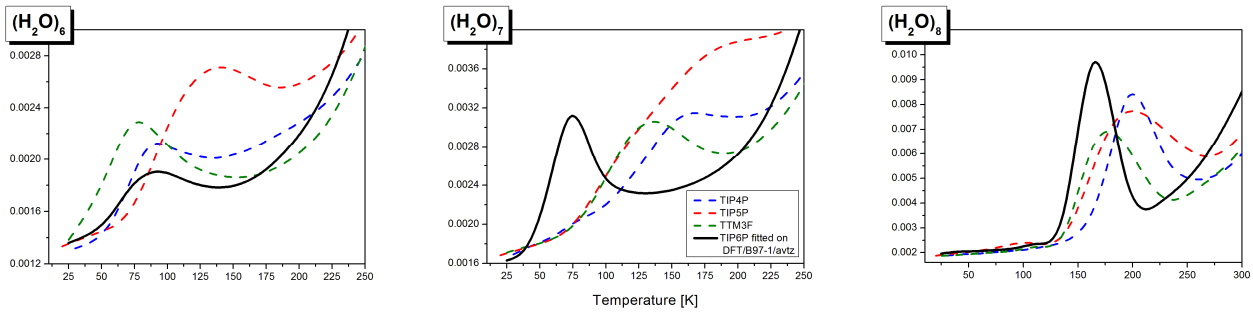


Figure 1: Heat capacity profiles for selected small water clusters calculated using a parallel-tempering Monte Carlo method [1] and a state-of-the-art interaction model based on recent density functional theory calculations (black curves) [2] and compared with data obtained for selected empirical interaction models [3].

Keywords: heat capacity, atomic and molecular clusters, phase changes in clusters, Monte Carlo

[1] R. H. Swendsen and J. S. Wang, *Phys. Rev. Lett.* **57** (1986) 2607.
[2] A. Vítek, R. Kalus, and I. Paidarová, *PCCP* **12** (2010) 13657.
[3] W. L. Jorgensen and J. J. Tirado-Rivers, *J. Am. Chem. Soc.* **110** (1988) 1657; M. W. Mahoney and W. L. Jorgensen, *J. Chem. Phys.* **112** (2000) 8910; G. S. Fanourgakis and S. S. Xanteas, *J. Chem. Phys.*, **128** (2008) Art. No. 074506.

Metal oxide aerogel

L. Matějová[1], R. Fajgar[1], Z. Matěj[2], Y. Maléterová[1], O. Šolcová[1]

[1]Institute of Chemical Process Fundamentals of the ASCR, v.v.i., Czech Republic;

[2]Charles University in Prague, Czech Republic

matejova@icpf.cas.cz

Aerogels are very attractive and promising materials owing to their high total pore volume, specific surface area and variable and adjustable porosity as well in many research areas. Nowadays, aerogel's main applications are based on their adsorption efficiency and low weight relative to their high porosity. Therefore, aerogels are mainly used as heavy metals adsorbents in waste water treatment technologies or additives into building materials. However, their application potential in catalysis/photocatalysis is markedly limited by structural properties of synthesized aerogels, namely amorphousness. Aerogels commonly prepared by sol-gel technique in combination with supercritical drying using supercritical carbon dioxide as extraction solvent show amorphous character. In this case the required crystallinity is reached only by subsequent thermal treatment.

Therefore, in this study the extraction technique opening up the possibility to prepare crystalline aerogels was developed and thoroughly investigated. Moreover, the first catalytic activity tests in AO7 azo-dye photooxidation over synthesized aerogels were performed. Pressurized fluid extraction using combination of subcritical water and super/subcritical methanol was applied to prepare a group of metal oxides - TiO_2, ZrO_2 and mixtures TiO_2-ZrO_2 and TiO_2-CeO_2. The effect of extraction temperature and pressure, as well volume of extraction solvent on textural properties, structural properties and purity of the final porous aerogels was studied. Rigid organogels used for extractions were prepared by the sol-gel process controlled in the reverse micellar environment of the nonionic surfactant Triton X-114 in cyclohexane at ambient temperature. The molar ratio of cyclohexane: Triton X-114: water: alkoxide was kept constant at 11: 1: 1: 1 for single oxide aerogels. In the case of mixed oxide aerogels the molar ratios of individual metal precursors were changed in the range from 0 to 1. Titanium (IV) isopropoxide, 70 wt-% zirconium (IV) propoxide in propan-1-ol and cerium (III) nitrate hexahydrate were used like metal precursors.

Nitrogen physisorption at -196°C, helium pycnometry, high-pressure mercury porosimetry, X-ray diffraction, Raman spectroscopy, organic elementary analysis and UV-Vis were used as characterization tools. Moreover, textural and structural properties of synthesized aerogels were compared with those of chemically identical but conventionally treated – calcined xerogels.

The financial support of the Grant Agency of the Czech Republic (104/09/P290) and of the Czech Academy of Sciences, Program Nanotechnology for Society (KAN400720701) is gratefully acknowledged.

Keywords: metal oxide, aerogel, pressurized fluid extraction, Raman spectroscopy, XRD

The ink-jet printed nanostructured TiO_2 film electrode

Morozová M.[1], Klusoň P.[1], Veselý M.[2], Dzik P.[2], Šolcová O.[1]

[1] Institute of Chemical Process Fundamentals of the ASCR, v. v. i., Czech Republic

[2] Brno Univesity of Technology, Faculty of Chemistry, Czech Republic

morozova@icpf.cas.cz

The high potential of the nanostructured semiconductor oxides in the thin layers form for the utilization as a part of sensors, optical electrodes or solar cells has recently evoked a great interest in the manipulation and optimization of their photoelectrochemical properties. As generally known, these photo-induced properties arise from the semiconductor nature, especially from the ability of the light quantum absorption followed by the charge carrier generation. Titanium dioxide in crystallographic form of anatase is a promising semiconductor widely used in the field of the advanced oxidation processes and the photoelectrochemical sensing due to its excellent efficiency to generate the electron-hole pairs. Nowadays, a variety of deposition techniques and preparation methods of the thin TiO_2 layers created from the liquid precursors exist. This study reports the electrochemical properties of the TiO_2 layers prepared by means of the templated sol-gel method and deposited by the inkjet printing.

Inkjet printing proved to be very elegant and clean technique for the sol deposition. The sol-gel method is based on hydrolysis followed by polycondensation of titanium alkoxide (TIOP) in the core of reverse micelles, where a small amount of water is involved. The micelles are formed by surfactant Triton X 102 in the nonpolar environment of cyclohexane. Prepared sol is loaded into a modified office inkjet printer equipped with the piezoelectric print head. Then small droplets are ejected from the print head and fall onto the conducting ITO glass. The glass plates with printed layers were dried in an oven at 110° C for 30 min and then the samples were thermally treated by calcination at 450° C for 4 hours. The sol deposition and patterning was performed by an adapted experimental inkjet printer Fujifilm Dimatix 2830.

The structure properties of the prepared layers were studied by series of characterization methods involving AFM, SEM, XRD, Raman and UV-Vis. Photo-induced electrochemical behaviour of TiO_2 electrode was investigated by potentiodynamic methods in a three-electrode electrochemical cell containing 0.1 M solution of sodium sulphate which was used as an electrolyte. A saturated Ag/AgCl electrode and a platinum plate were used as the reference and the auxiliary electrodes, respectively. The exposed area of the working TiO_2/ITO electrode was irradiated by monochromatic light and the photoelectrochemical reaction was monitored.

This work was supported by the Grant Agency of the Czech Republic, projects No. 203/08/H032 and of the Czech Academy of Sciences, Program Nanotechnology for Society (KAN400720701) is gratefully acknowledged.

Keywords: Ing-jet printing, Photoelectrochemical, thin TiO_2 layers, sol-gel

Computer study of morphology and electrical properties of (nano)composites

S. Novák[1], R. Hrach[1, 2], M. Švec[1]

[1]J. E. Purkinje University, Department of Physics, Czech Republic; [2]Charles University, Department of Surface and Plasma Science, Czech Republic

Stanislav.Novak@ujep.cz

Composite and nanocomposite metal/dielectric films belong to a group of very interesting novel materials for their mechanical, electrical, and optical properties. We can distinguish three types of the systems depending on metal volume fraction:
films consisting of isolated metal particles in dielectric matrix,
high metal volume fraction films, and
films near a percolation threshold which form a transition stage among the two mentioned above.

We present a computer study of the electrical and morphological properties of the thin (nano)composite films using self-made software analytical tools. Main attention is devoted to a relationship between the electrical properties and the morphology of the films depending on their metal volume fraction, especially near a percolation threshold. The composite structures are generated with given properties by several models [1-4]. Then the structures are studied by various methods of mathematical morphology to obtain structural parameters. The Monte Carlo method and the theory of percolation were used for the study of electrical properties. An ohmic or tunnelling type of conductivity is supposed in the films. Evaluated current paths inside the composite structure form the so-called fuzzy clusters below and especially close to the percolation threshold. Their study brings valuable additional information about the electrical properties depending on the type of composite structure. The results of the morphological and transport analysis are compared, discussed, and searched for information about the processes even in graded composites and for various shapes of inclusions.

[1] R. Hrach, S. Novák, M. Švec, Appl. Surf. Sci. 252 (2006) 5516-5520.
[2] S. Novak, R. Hrach, M. Svec, V. Hrachova, Thin Solid Films 518 (2010) 4537-4541.
[3] S. Novak, R. Hrach, Vacuum 84 (2010) 174-178.
[4] R. Hrach, M. Švec, S. Novák, D. Sedlák, Thin Solid Films 459 (2004) 174-177.

Keywords: Composite films, Modelling, Morphology, Electrical properties, Fuzzy clusters

Investigation of Al doping effects on the ZnO thin films produced by sol gel spin coating

Deniz Gultekin[1], Ubeyd Tocoglu[1], Mehmet Oguz Guler[1], Ozgur Cevher[1], Hatem Akbulut[1]

[1]Sakarya University, Engineering Faculty, Metallurgy and Materials Department, Esentepe Campus, 54187, Sakarya, Turkey

utocoglu@sakarya.edu.tr

In this paper, undoped, and Al-doped ZnO thin films have been sythesis by sol–gel spin coating method. Effects of Al doping on morphologies, crystal orientation and electrical properties of ZnO films was investigated. Zinc chloride ($ZnCl_2$) and aluminum chloride hexahydrate ($AlCl_3.6H_2O$) have been used as precursors. Both precursors dissolved in ethanol and stirred at 60°C for 2h to form a clear, stable and transparent homogeneous sol. Doping of the Zn sol was obtained by adding 1, 5 and 10 vol.% Al sol. Undoped and Al-doped ZnO thin films have been deposited on ITO coated glass substrates by spin coating method. The formed films were heat treated in a furnace for 1 h at 400 °C. X-ray diffractometer (Rigaku D/MAX 2000) has been used to determine preferred crystal orientation and particle size of the thin films. Film morphologies have been examined by using Jeol JSM 6060 LV scanning electron microscope (SEM) equipped with energy-dispersive spectroscopy (EDS) and electrical resistivity of the oxide films has been studied with four-point probe resistivity measurement (Bellsonics PRO4) device.

Keywords: ZnO thin film, Al-doped ZnO, Sol-gel, Spin Coating

Molecular modeling of gel-based emulsions with cyclosporine A

J. Tokarský[1], T. Andrýsek[2], P. Čapková[1]

[1]Nanotechnology Centre, VŠB-TU Ostrava, Czech Republic; [2]Teva Czech Industries s.r.o., Czech Republic

jonas.tokarsky@vsb.cz

Structure and behavior of amphiphilogel nanoparticles as a drug carrier for cyclosporine A (CsA) have been studied by the molecular modeling using empirical force field. Five atomistic models of a gel-based emulsions (GEM) with various gel compositions (denoted as A, B, D, E, F) have been investigated in order to find a system most similar to a sixth atomistic model (denoted as C) of self-microemulsifying drug delivery system (SMEDDS) taken as an exemplar of CsA delivery system. Structural parameters (i.e. volume fraction, concentration profiles, pervasion of water molecules) and energy characteristics (i.e. non-bond interaction energy between CsA and whole remaining components of a gel nanoparticle, CsA/gel nanoparticle intermolecular non-bond interaction energy, CsA-gel molecule pair interaction energy) of these six models in a waterless form (denoted as A0, B0, C0, D0, E0, F0) and in a water containing form (denoted as A60, B60, C60, D60, E60, F60, where the number means 60 wt. % of water) have been studied in dependence on the composition. In addition, a simple method suitable for the quantitative determination of the degree of agglomeration of CsA/gel nanoparticle is presented in this work. Pair interaction energies were calculated using the Flory-Huggins theory as implemented in the Accelrys Materials Studio 4.2 modeling environment.

Molecular dynamics revealed that CsA/gel nanoparticles in models D60, E60 and F60 exhibit the tendency to create fibrous structures in water environment while CsA/gel nanoparticles in models A60 and B60 are strongly agglomerated creating the clusters. CsA/gel nanoparticle in model C60 is very disordered and incompact.

Further results can be briefly summarized as follows. The percentage of pervaded water for model E60 is the closest to the percentage of pervaded water for model C60. Taking into account the results of pair interactions study the GEM E and the SMEDDS C contain gel molecules having very good miscibility with CsA. Interaction energy between CsA molecule and whole remaining components of a gel nanoparticle for models C and E are closer to each other than to the remaining models A, B, D and F.
Presented results lead to the conclusion that GEM E is the most similar to the SMEDDS C and as such is very promising for further experimental study.

Our approach to the characterization is simple and cheap. Presented structural parameters together with energy characteristics can be used (due to its universality) for arbitrary gel compositions in order to help the experimentalists find the most suitable GEM drug delivery systems.

Keywords: molecular modeling, drug delivery, structure characterization, cyclosporine A

Prism coupling for planar structures with magnetic ordering

F. Staněk, M. Lesňák, J. Pištora, D. Ciprián

Department of Physics

Technical University Ostrava, 17. listopadu 15, 708 33 Ostrava – Poruba, Czech Republic

f.stanek@vsb.cz

Attenuated total reflection (ATR) can provide information about thin film thickness, refractive index, and attenuation in addition to the (surface) prism coupling of an optical wave into and off a waveguide. ATR has been found to give more accurate and precise results than other conventional techniques.

In the presented contribution the prism coupling conditions are determined for magnetic structures with induced anisotropy. The tunnelling phenomenon between coupling prism and inspected object is analyzed for zero gap thickness, ultrathin approximation, and for the case of a gap spacing assumed to be wide enough so that the prism could be considered a weak perturbation to the modes of the free structure. The matrix formalism is an extremely useful for description of isotropic and anisotropic multilayer stacks. The characteristic matrices are derived for prism coupler into structures with magnetic ordering for magneto-optical effects.

The prism – film coupler is located in the central cavity of magnetic yoke. By current switching in the coils we can change the amplitude and magnetic field direction and as result to modulate the induced anisotropy in thin film with magnetic ordering.
Switch positions and fine wire in visual field are recording by the very responsive linear CCD camera. The wire position is allotted of the camera and the diameter is the scale factor.

Keywords: Attenuated total reflection (ATR) magnetic field, prism coupler, thin film

Characterization of SnO_2 nanostructured thin film coatings prepared by precipitation method

Deniz Gultekin[1], Ubeyd Tocoglu[1], Mehmet Oguz Guler[1], Ozgur Cevher[1], Hatem Akbulut[1]

[1]Sakarya University, Engineering Faculty, Metallurgy and Materials Department, Esentepe Campus, 54187, Sakarya, Turkey

utocoglu@sakarya.edu.tr

In this paper, SnO_2 nanostructured thin film coatings have been prepared from nanopowders synthesized by precipitation method. Tin (II) chloride dehydrate ($SnCl_2.2H_2O$) have been used as a precursor. $SnCl_2.2H_2O$ precursor have been dissolved in distille water and stirred at 60°C for 2h. Then solution was cooled to room temperature and gets precipitated around the pH 1. Collected nanosized precipitates have been deposited on glass substrate by drain coating and postheated at 300, 400 and 500 °C. X-ray diffractometer (Rigaku D/MAX 2000) has been used to characterization of coatings. Film morphologies have been examined by using Jeol JSM 6060 LV scanning electron microscope (SEM) equipped with energy-dispersive spectroscopy (EDS).

Keywords: SnO_2 thin film, Sol-gel, Precipitation, Nanopowder

Magneto-optical magnetic field sensor: theoretical study

J. Vlček[1], M. Lesňák[2], J. Pištora[2]

[1]Dept. of Mathematics and Descriptive Geometry, [2]Institute of Physics

VŠB-Technical University of Ostrava, Czech Republic

jaroslav.vlcek@vsb.cz

Magnetic field detection based on various physical principles belongs to relatively known domains. The polarization method having its origin in the Kerr rotation variability is mostly used, although it requires precise measuring of angular changes with considerable demands for evaluating equipment. Since the actually accessible sensing elements allow estimating very small intensity differences (in thousandths of normalized input), one is meaningful to deal with the design of magnetic field sensor constructed on reflectometric principle. By fixed incidence angle, proposed device evaluates intensity changes proportionally on the scale between neutral and magnetically saturated state for given material characteristics (permittivity, Voigt parameter) of magneto-optically active component.

In proposed study, the ATR (attenuated total reflection) system "glass prism – Fe or Co – Au – air" with prism coupling defined by planar interfaces is considered. We aim to find optimized thicknesses of metallic layers with regard to the sensitivity on external magnetic field. To this purpose, corresponding sensitivity factor and sensitivity criterion are introduced and applied.

Keywords: Magneto-optics, Sensor, ATR system, Response factor

Electromagnetic scattering from imperfectly periodic array of circular cylinders

Koki Watanabe and Yoshimasa Nakatake

Fukuoka Institute of Technology, Japan

koki@fit.ac.jp

Periodic structures are widely used in microwave, millimeter-wave, and optical wave regions because of various effects; for example, wave reflection, wavelength or polarization selectivity, mode conversion. Therefore, electromagnetic scattering from periodic structures has been extensively studied, and many analytical and numerical approaches have been developed to analyze the scattering problems. When a plane-wave illuminates a periodic structure, the Floquet theorem asserts that the scattered fields are pseudo-periodic, namely, each field component is a product of a periodic function and an exponential phase factor. This implies that the scattered fields have discrete spectra in the wave number space and the field components can be expressed in the generalized Fourier series expansions. As a result, the analysis region can be reduced to only one periodicity cell. However, when the structural periodicity is collapsed even if locally, the Floquet theorem is no longer applicable and the analysis regions of the spatial-domain approaches have to generally cover all the scattering structure under consideration. On the other hand, the fields in the structure with imperfect periodicity have continuous spectra in the wave number space, and an appropriate discretization is necessary for the spectral-domain approaches.

This paper shows an approach to the electromagnetic scattering from imperfectly periodic array of circular cylinders, in which the structural periodicity is locally collapsed. The present formulation is based on the recursive transition-matrix algorithm (RTMA) with the lattice sums technique. The original RTMA is known as a very effective approach to the scattering from finite number of circular cylinders. It uses the cylindrical-wave expansions to express the field components and the boundary conditions at the cylinder surfaces are derived by Graf's addition theorem. RTMA with the lattice sums technique has been proposed for the scattering problem of a perfectly periodic cylinder array for plane-wave incidence, and a practical computation becomes possible by an integral representation of the lattice sums. The present formulation expands RTMA with the lattice sums technique to the scattering problem of imperfectly periodic cylinder array consisting of infinite number of circular cylinders. The formulation requires an appropriate discretization in the wave number space only inside the Brillouin zone. It is well known that the fields in the periodic structures have infinite number of non-smooth points in the wave number space, which are called the Wood anomalies, and the anomalies do not vanish if the structural periodicity is locally collapsed. However, the Wood anomalies are degenerated to finite number of points in the Brillouin zone, and the appropriate discretization is comparatively easy to consider. This paper also shows a possibility to approach to multilayer structures consisting of periodic and aperiodic layers.

Keywords: electromagnetic scattering, imperfectly periodic structure, recursive transition-matrix algorithm

Non-enzymatic electrochemical determination of glucose on Ag-doped zeolite-carbon nanotube composite electrode

A. Baciu, A. Pop, A. Remes, F. Manea, G. Burtica

Faculty of Industrial Chemistry and Environmental Engineering, „Politehnica" University of Timisoara, Romania

anamaria.baciu@chim.upt.ro

A new electrocatalyst, silver-doped zeolite-multiwalled carbon nanotube-epoxy (Ag-ZCNT) composite electrode was obtained and tested for non-enzymatic oxidation and determination of glucose from aqueous solution. SEM imaging has been used to provide qualitative information about distribution of multiwalled carbon nanotube and Ag-doped zeolite zones and some surface features of this composite electrode. The prepared Ag-ZCNT composite electrode showed good mechanical strength and good electrical conductivity (0.14 Scm^{-1}), which was determined using the standard four probe technique. The direct electrocatalytic oxidation of glucose in alkaline medium at Ag-ZCNT composite electrode was investigated by cyclic voltammetry (CV). The anodic current peaks corresponding to AgO formation increased progressively linearly with glucose concentration during the reverse scanning. Also, linear-sweep voltammetery technique allowed to determine the glucose on Ag-ZCNT also only during reverse scanning obtained directly over the concentration range 0.1 mM – 1 mM glucose. No peak corresponding to glucose oxidation was identified during direct scanning. An example of LSV series recorded by reverse scanning is shown in Figure 1. This modified electrode exhibited electrocatalytic effect towards glucose by dehydrogenation reaction between AgO and glucose allowing its determination in aqueous solution. This sensor exhibited a rapid response, a low detection limit, a good sensitivity, as well as good stability and repeatability. Therefore, this work has demonstrated a simple and effective sensing method for non-enzymatic detection of glucose.

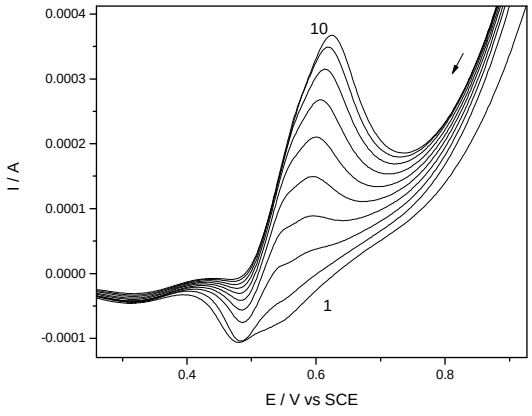


Figure 1. Linear-sweep voltammograms recorded on Ag-ZCNT composite electrode in 0.1 M NaOH supporting electrolyte (curve 1) and in the presence of various glucose concentrations: 2-0.4 mM, 3-0.8 mM, 4-1.2 mM, 5-1.6 mM, 6-2 mM, 7-2.4 mM, 8-2.8 mM, 9-3.2 mM, 10-3.6 mM; the arrow marks the scanning direction

Keywords: electrochemical sensor, glucose, non-enzymatic determination, Ag-doped zeolite-carbon nanotube composite electrode

Adsorption of polycyclic aromatic hydrocarbons by carbon nanotubes

A. Matlochová[1], D. Plachá[2]

[1]Institute of Geological Engineering; [2]Nanotechnology Centre, VŠB – TU of Ostrava, Czech Republic

adela.matlochova@vsb.cz

Carbon nanotubes (CNTs) were discovered by Sumio Iijima in 1991 and since that time they are having widespread potential applications in medicine, electronics, optics, cosmonautics etc. Due to their favourable properties potential environmental applications of CNTs as superior sorbents have been suggested for removal of chemicals such as heavy metals or organic compounds from water. CNTs are fullerene-related structures which consist of rolled graphene sheets. Manufactured thin multi-walled carbon nanotubes (MWCNTs) used in this study are produced via the catalytic carbon vapor deposition process. Their properties include high surface area about 250-300 $m^2.g^{-1}$, small size, average diameter of just 9,5 nm and high aspect ratio.

In this research adsorption of polycyclic aromatic hydrocarbons (PAHs) - phenanthrene and fluoranthene onto the MWCNTs in aqueous media was investigated. PAHs were selected due to their significant concentrations in the aquatic environment, high toxicity and persistence. Both single-solute sorption and competitive sorption was examined using solid phase extraction technique and gas chromatography with FID. Linear isotherms were used to describe the adsorption process of PAHs by the CNTs. Advantages of sorption on MWCNTs comprise high efficiency, high adsorption capacity, low consumption of sorbent and relatively short sorption time.

Although nanomaterials seem to be superior adsorbents, there are some recent studies which highlight their potential risks to human beings and ecosystems if nanoparticles are released to the environment. Therefore, it is important to continue study the behavior of carbon nanotubes in waters for evaluating their environmental impact.

The work has been done in connection with project Energy Units for Utilization of non Traditional Energy Sources, reg. no. CZ.1.05/2.1.00/03.0069 supported by Research and Development for Innovations Operational Programme financed by Structural Founds of Europe Union and from the means of state budget of the Czech Republic; and with project VaV: FR-TI1/389 – program TIP.

Keywords: Adsorption, Carbon nanotubes, Polycyclic aromatic hydrocarbons, Isotherms

Determination of ibuprofen in water using Ag-doped zeolite-expanded graphite composite electrode

S. Motoc, F. Manea, A. Pop, R. Pode, G. Burtica

Faculty of Industrial Chemistry and Environmental Engineering, „Politehnica" University of Timisoara, Romania

sorina.motoc@chim.upt.ro

Many drug and pharmaceutical residues are present in the environment due to the growing production and use of pharmaceuticals. Ibuprofen (IBP), the third most popular clinically used drug in the world was selected in this study because the detectable levels have been found in water bodies due to its incomplete removal during the wastewater treatment. Several time-consuming and laborious methods for the quantitative determination of IBP have been reported, thus electrochemical methods have attracted interest, because of the fast response, large sensitivities, simple operation, and the possibilities of miniaturization. The electrode material plays the major role in the electroanalysis by the potential window and the electrocatalytic effect towards analyte oxidation/reduction.

In this study the electrochemical detection of IBP in aqueous solution on Ag-doped zeolite-expanded graphite (AgZEG) composite electrode is reported. The electrochemical oxidation and detection of ibuprofen on AgZEG composite electrode was investigated using cyclic voltammetry (CV), differential-pulsed voltammetry (DPV), square-wave volatmmetry (SWV), chronoamperometry (CA) and multiple-pulsed amperometry (MPA). A series of optimization studies were made on the voltammetric and pulsed parameters.Thus, the scan rate of 100 $mV \cdot s^{-1}$, 100 mV pulse amplitude, 5 ms modulation time and 50 mV step potential represent the optimum conditions for DPV application. Also, optimized values of the SWV parameters were determined, i.e., 25 Hz modulation frequency, 200 mV step potential and 100 mV pulse amplitude. No signal enhancement was reached using MPA technique. An example of DPV recorded at various IBP concentrations ranged between 1- 10 $mg \cdot L^{-1}$ is presented in Figure 1. The best electroanalytical performance of AgZEG electrode was achieved for DPV and SWV techniques, and the electrochemical determination methods revealed a good accuracy based on the comparison with spectrophotometric results.

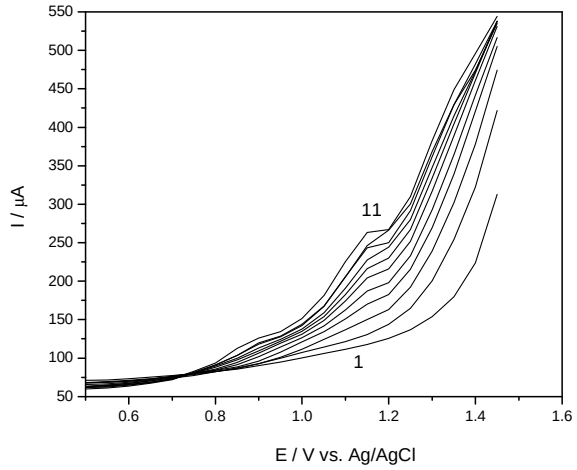


Figure 1. DPV voltammograms recorded on AgZEG composite electrode in 0.1 M Na_2SO_4 supporting electrolyte (curve 1) and in the presence of various IBP concentrations: 1-10 mg·L^{-1} (curves 2-11)

Keywords: pharmaceuticals, water quality, Ibuprofen determination, Ag-doped zeolite-expanded graphite composite electrode

Biosynthesis of silver and gold nanoparticles using diatoms and their direct application in catalysis

A. Schröfel[1], G. Kratošová[1], M. Šafaříková[2], I. Šafařík[2], M. Natšinová[1], V. Holišová[1]

[1]VSB-Technical University of Ostrava, Nanotechnology Centre, Czech Republic

[2]Institute of Nanobiology and Structural Biology, Academy of Science of Czech Republic

adam.schrofel@vsb.cz

Development of reliable and eco-friendly procedures of metallic nanoparticles and nanocomposites synthesis is crucial for success of advancing nanotechnology applications. In present years, increasing attention is paid to a synthesis of composites that either directly appears from or is inspired by the nature. Biosynthesis of metallic nanoparticles by photoautotrophic organisms or their components were also tested and described recently. Diatoms are unicellular photosynthesizing microorganisms belonging into the group of brown algae (division *Chromophyta*, class *Bacillariophyceae*) encased in siliceous cell walls – frustules. Frustule of a diatom is always formed by two valves (epitheca and hypotheca) connected together by circular pieces of silica called girdle bands. The construction material of a frustule is mainly nanostructured amorphous polymerized silicic acid.

Formation of nanoparticles in presence of siliceous frustules likely provides occasions for novel bionanocomposite use. We present experiments leading to a protocol for synthesis of AuNPs, EPS-gold and silica-gold bionanocomposites by biologically driven processes in diatom strain *Diadesmisgallica*. Catalytic properties of resulting nanocomposite were confirmed by reduction of 4-nitrophenol solution in the presence of sodium borohydride.

Keywords: Biosynthesis, Gold,Catalysis, Nanoparticles, Diatoms, 4-paranitrophenol

Process Parameters Optimizations for Oxide and Carbide Nano Particle Reinforced MMCs Coatings by Electro co-deposition

H. Akbulut[1], H. Gül[2], A. Alp[1]

[1]Sakarya University, Engineering Faculty, Department of Metallurgical & Materials Engineering, Esentepe Campus, 54187, Sakarya/TURKEY

[2]Duzce University, Gumusova Vocational School, Department of Metallurgy, 81850, Duzce/TURKEY

akbulut@sakarya.edu.tr

In the present work, Ni/Al_2O_3 and Ni/SiC metal matrix composite (MMC) coatings were prepared from nickel sulphate bath the plating electrolyte containing nano □-Al_2O_3 (80 nm) and SiC (between 100–1000 nm). The coatings were carried out with by direct current (DC) plating method to increase the surface hardness and wear resistance of the electrodeposited Ni. For these purposes, optimization of the deposition parameters for specific applications was thought to investigate such as particle concentration in electrolyte, the effect of surfactant concentration, current density, and bath pH, etc. Al_2O_3 and SiC nano-powders were co-deposited with nickel matrix on the steel substrates. The depositions were controlled to obtain specific thickness (between 50 – 200 µm) and particle volume fraction in the matrix (between 0.03 and 0.12). The characterization of the coatings was investigated by scanning electron microscopy (SEM) and X-ray diffraction (XRD) facilities. The hardness of the resultant coatings was also measured and found to be 280-641 Hv for Al_2O_3 nano composites and 280 – 571 Hv depending on the particle volume in the Ni matrix. The effects of the surfactant on the zeta potential, co-deposition, and distribution of nano ceramic powders in nickel matrix, and tribological properties of composite coatings were investigated. The results showed that the wear resistance of the nano-composites was approximately 2-3.5 times increased compared with unreinforced Ni deposited material

Keywords: Nano composite, Electro-codeposition, zeta potential, wear, friction

Preparation of nanocomposite resistive material with modified silicon nanopatricles Si(C_{60}) in tin metal matrix

R. Dvorský[1], J. Trojková[1], J. Luňáček[1], K. Piksová[2], K. Klouda[3]

[1]VSB-Technical University of Ostrava, CZ, [2]Czech Technical University, CZ

[3]The National Institute for Nuclear, Chemical and Biological Protection, CZ

richard.dvorsky@vsb.cz

The paper describes the preparation of the nanocomposite resistive material formed of very tight solid dispersion of modified Si(C60) nanoparticles embedded into the Sn metal matrix. Fig. 1 depicts the primary size distribution of Si nanoparticles manufactured top-down in the Water Jet Mill disintegrator. Simultaneously there is also the distribution of the same nanoparticles modified by fullerene molecules C60, which shows the the growth in size due to absorption and (presumably) aggregation. These modified Si(C60) nanoparticles were dispersed in demineralised water and by means of controlled lyophilisation transformed into form of microwires and nanowires, which you can see in Fig.2.

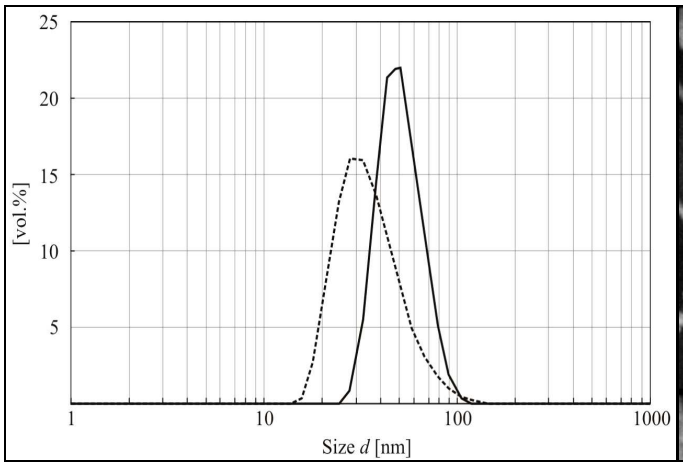


Fig.1 The DLS measured diameter distribution of pure silicon nanoparticles (modus 28 nm, dotted line) and distribution of silicon nanoparticles modified by fulleren

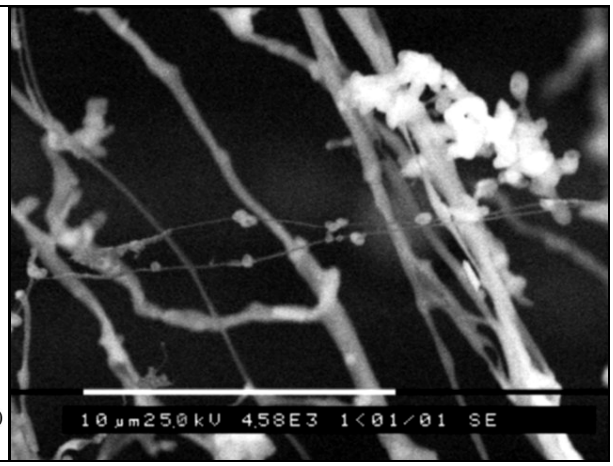


Fig.2 The SEM image of microwires of Si(C_{60}) modified nanoparticles, which are the main dispersion component for the fabrication of nanocomposite with

The depicted fibrous material is further homogenised with various fractions of Sn metal nanoparticles and cold pressed into a form of □9 mm x 3 mm tablets. In the second step of material processing the tablet is exposed to pressure up to 2,5 GPa together with the melting-sintering at the temperatures above the melting point of tin. Due to high pressure molten Sn metal is mostly expelled to the surface of the sample and only the ultrathin Sn metal bonding netting is maintained inside between the Si(C60) nanoparticles. The measurements on the described nanomaterials have shown interesting electrical properties and their perspective application in the area of thermistor elements, promising improvement in sensitivity of temperature sensors.

This research was performed at VŠB-Technical University of Ostrava, sponsored by Research Plan MSM 6198910016 and RMTVC CZ.1.05/2.1.00/01.0040.

Keywords: nanoparticles, silicon, fullerene, metal matrix nanocomposite, resistivity

Possibility of Producing Ni/Al_2O_3-SiC Hybrid Nanocomposites by Electro-codeposition with double surfactant addition effect

H. Gül[a], A. Alp[b], H. Akbulut[b]

[a]Duzce University, Gumusova Vocational School, Department of Metallurgy, 81850, Duzce/TURKEY

[b]Sakarya University, Engineering Faculty, Department of Metallurgical & Materials Engineering, Esentepe Campus, 54187, Sakarya/TURKEY

harungul@duzce.edu.tr

In this study, a nickel sulphate plating bath containing SiC (particle size between 100–1000 nm) and Al_2O_3 (80 nm) nano particles were used. The aim of this work is to obtain hard and wear resistant nano particle reinforced nickel metal matrix composite's (MMCs) on steel surfaces for anti wear applications such as dies, tools and working parts for automobiles and vehicles. The influence of surfactant quantity in electrolyte on particle distribution, microhardness and wear resistance of nanocomposite coatings has been studied. The nickel hybrid composite coatings were characterized by scanning electron microscopy (SEM) analysis before and after wear tests. The depositions were controlled to obtain specific thickness (between 50 – 200 µm) and particle volume fraction in the matrix (between 0.02 – 0.12). The hardness of the coatings was also measured and found to be 291 – 455 Hv depending on the particle volume in the Ni matrix. The effects of the surfactant quantity in electrolyte on co-deposition and distribution of nano particles in nickel matrix and tribological properties of composite coatings were investigated. The tribological behaviors of the electrodeposited hybrid nanocomposite coatings sliding against M50 steel ball (Ø 10mm) were examined by using CSM instrument. All the friction and wear tests were performed under unlubricated condition at room temperature and in ambient air (relative humidity 55–65 %). The results showed that the wear resistances of hybrid nano composites were increased when compared with unreinforced Ni coatings.

Keywords: Nano MMCs, SiC-Al_2O_3 hybrid composite, friction, wear resistance

Special collector for deposition oriented nanofibres

J. Chvojka[1], D. Lukáš[1]

[1]TECHNICAL UNIVERSITY OF LIBEREC, FACULTY OF TEXTILE ENGINEERING,

Department of Nonwovens, Studentská 2, 461 17 Liberec, Czech Republic

jiri.chvojka@tul.cz

The aim of this article is to focus on electrospinning process and particularly on a special collector. The collector, which can be a metal plate, will supply traditional collectors. This special collector is consisted of conductive materials. It is compound from a metal plate. On this plate are 4 parallel conductive wires. The collector rotates while the nanofibrous layers are deposited between the conductive wires. It is possible to change a distance between wires to adjust a length of nanofibres. Special collectors can affect an electrostatical field near the collector and by this way we can control a deposition of nanofibers on the special collector. Nanofibres are highly oriented between conductive wires. The final nanofibrous layer can be used for filtration or as a scaffold in tissue engineering.

Keywords: nanofibres, special collector

Effect of Sintering Current Density on the Production of MWCNT Reinforced Bronze Matrix MMCs

R. Karslıoğlu, M. Uysal, H. Akbulut

Sakarya University Engineering Faculty Department of Metallurgical and Materials Engineering Sakarya 54187, Turkey

rkarslioglu@sakarya.edu.tr

In this work, a bronze matrix (90 wt. % Cu + 10 wt. % Sn) was reinforced with multi-walled carbon nanotube (MWCNT) using mechanical alloying and a subsequent current activated sintering technique. The mechanically ball-milled bronze matrix composite powders reinforced with 1.0 wt. % MWCNT was first cold-compacted under a pressure of 300 MPa. The compacted structure was sintered at atmospheric conditions to nearly a full density within 10 min. using currentactivated sintering, in which the powders were heated by a low voltage and high current source and compressed simultaneously. The samples were sintered at three different applied currents to investigate the effect of current density on microstructural properties. The aim is providing a dense structure and well-bonded MWCNT reinforcements with the bronze matrix for possible high tech. bearing applications. Density measurement, microhardness testing and optical and scanning electron microscopy (SEM) investigations were carried out for microstructural characterization of the hybrid composites. It was pointed out that increasing applied current during the sintering process resulted in obtaining denser, hard homogeneous distribution of MWCNT in bronze matrix.

Keywords: Current Activated Sintering 1,Nanocomposite 2,MWCNT 3, Bronze 4

Synthesis and characterization of SnO_2 nano-sized anode materials stabilized by tartaric acid

H. Köse[1], A. O. Aydın[1], H. Akbulut[1]

[1]Sakarya University, Turkey

hkose@sakarya.edu.tr

Lithium ion batteries have a high energy density and are widely used in energy storage for electrical devices, such as mobile phones and notebook computers. Tin and tin oxide based materials are being used as active anode materials for rechargeable lithium batteries. Tin oxide anodes have different discharge and charge mechanism compared with carbonaceous anodes. Therefore, these materials have attracted much attention due to their large reversible capacity.

SnO_2 nano sized materials can be obtained by different techniques. Among the processing techniques, sol-gel offers advantages such as low cost, low temperatures processing and also precise control of stoichiometry.

In this study, SnO_2 nanocoatings were fabricated by sol-gel method. Sols were prepared from tin (IV) acetate precursor by dissolving in absolute ethanol at room temperature and tartaric acid was used as a stabilizer. Films were coated on the stainless steel substrates using spin coating system. After that, the gel film was dried in an oven at 100 ^oC. Subsequently, dried films were calcinated in a programmable oven with a heating rate of 1 ^oC/min. Firstly, precalcination was performed at 350 ^oC for half an hour, then temperature was increased up to 550 ^oC and the sintered for 2 hours.
SEM, AFM and XRD analysis were performed to characterize nanofilms. The results indicated that the nanocrystalline nature of the SnO_2 films is evidences for applying as an anode electrode for Li-ion batteries.

Keywords: Tin Oxide, sol-gel, nanofilm, anode

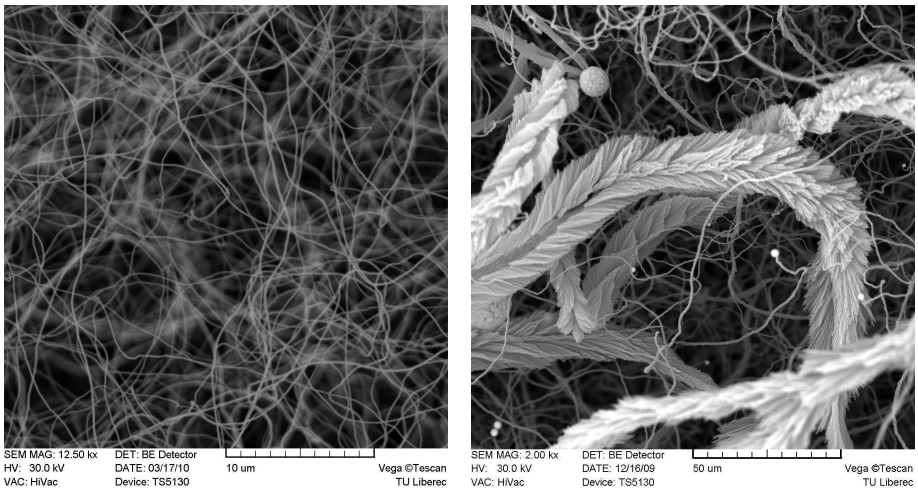
Carbon nanostructures produced by CCVD method

E. Košťáková[1], J. Grégr[1], P. Pokorný[1], L. Mészáros[2], M. Chotěbor[1]

[1]Technical University of Liberec, Czech Republic; [2]Budapest University of Technology and Economics, Hungary

eva.kostakova@tul.cz

The contribution describe simple laboratory set-up of CCVD method for production of carbon nanostuctures. The work introduced several unconventional substrates with integrated catalysts for carbon nanotubes synthesis. These substrates are rocks, metal plates and metal wires.
The main interest was in production of carbon nanotubes, although also other very interesting carbon nanostructures or nanostructured surfaces can be produced by the method. However optimal conditions for production of long multiwalled carbon nanotubes were found. The diameter of these multiwalled carbon nanotubes is bellow one hundred nanometers. There were used Raman spectroscopy, EDS and scanning electron microscopy for study of the produced carbon nanostructures.
The experiments studied also the effect of the used temperature, exposure time, flow of gaseous carbon source and especially the influence of "connections" substrate-catalyst. The work is unique especially by the use of unconventional easily available substrates containing catalysts without demanding special preparation of substrates and catalyst deposition. The unique is also usage of natural gas as gas source of carbon. Of course there are some problems with purity of it, but the presented study shows that it can be used with very well results for such laboratory experiments.



SEM images introduce two examples of carbon nanostructured materials grown from catalyst particles onto special substrate (metal wire in the left side figure and basalt stone in the right side figure) by CCVD method.

The available utilization of these carbon nanotubes and nanostructures on the described unconventional substrates is for example in special active filtration, substrates for catalytic chemical and biochemical reactions.

Keywords: carbon nanotubes, chemical vapor deposition, carbon nanomaterials.

Electrospinnig as a source of X-rays

P. Mikeš[1], P. Pokorný[1], R. Charvát[1], D. Lukáš[1]

[1]Nanoscience Centre, Technical University of Liberec, Czech Republic

petr.mikes@tul.cz

Here we report about unexpected features of electrospinning process called Counterion condensation. Electrospinnig is a process for creation of nanofibers by application of high electric field on the polymeric solution. This process depends on many physical and chemical parameters which have crucial impact on spinnability of polymer, fiber diameter as well as on the generation of X-rays which is the main topic of this work. Several radiographic and spectral measurements were carried for better understanding of this unexpected phenomenon. This work has been proving that we have to include quantum mechanical principles to electrospinning theory.
The first detection of X-rays produced by nanofiber deposition was observed using radiographic film. All samples were for five minutes tested by exposure of Deuterium lamp, HERAEUS D200F, 300W, providing UV light of wavelength interval 160 nm- 400 nm, i.e. 50 eV -120 eV. The syringe with a hypodermic needle was used as a spinning electrode filled by the fresh 12% wt. aqueous solution of poly-vinyl-alcohol (PVA). Grounded bronze grid was used as a collector. Three independent spectral measurements were carried out by using of the SLP Silicon Lithium-drifted [Si(Li)] Planar Low-Energy X-ray Detector. Beryllium window was shielded against the external electrostatic field by grounded bronze grid where nanofibers were collected.

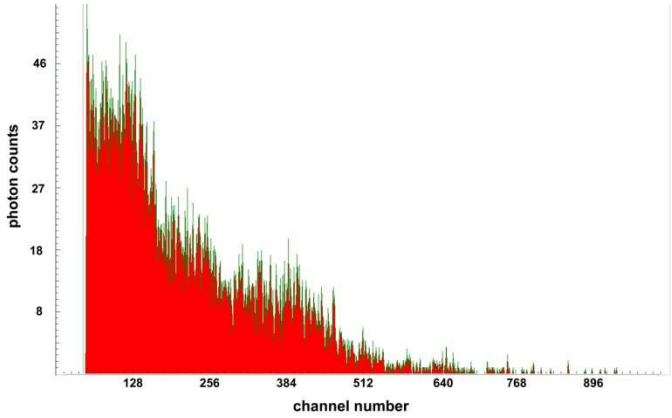


Figure 1. Spectrum of X-ray photons emitted from electrospinning jets. Continuous energy spectrum of X-ray total photon counts accumulated during 150 s, vertical scales, emitted from the PEO electrospinning jets recorded by the unshielded Si-PIN X-Ray Detector. The distance between the needle and the collector was adjusted on 110mm and the voltage was 30 kV.

A massive amplification of the field strength in the vicinity of an electrospinning jet or a nanofibre is due to the effect of “counterion condensation”, which is a well-known effect in polyelectrolytes. The condensation appears, as discussed in, when the dimensionless linear charge density q on a tiny nanofiber-like cylindrical body exceeds its critical value. Counterions “condense” in the immediate vicinity of the cylindrical fiber forming the so-called Manning region.

Keywords: X-rays, nanofibers, electrospinning, cnounterion condensation

Precursors and carriers of nanoparticles prepared by water jet disintegration

L. Sitek, J. Foldyna, J. Klich, P. Martinec, D. Nováková

Institute of Geonics ASCR, v.v.i., Ostrava, Czech Republic

libor.sitek@ugn.cas.cz

Preparation of precursors represents part of synthesis of nanoparticles of metal oxides, for example by heating, chemical treatment, etc. Participation of crystalline structure of the mineral precursor (e.g. with Fe^{+2} in the structure) in the formation process of nanoparticles of oxide represents one of modifiers of the synthesis process. Also some industrial technologies require appropriate nanoparticle carriers with specific surface properties, such as mechanical strength, abrasive resistance, sorptive properties, biocompatibility, etc. The nanoparticle carriers can be used in waste water treatment and gas cleaning, or as contact sorptive-catalytic materials in insulating systems of ecological buildings, indicators of condition of concrete structures and the like.

Carriers allowing better homogenisation of nanoparticles in applied industrial materials are required in case of use of nanoparticles in various technologies. Materials in question are primarily of composite character, such as agglomerated materials, building materials and bonding agents (impacting corrosion resistance, increase in possible insulating effects under thermal action on steel construction of structures, increase in their stress-strain properties, etc.), paints (increase in hardness, abrasive resistance and UV fasteness), bonding agents (plasticity and rheology), cosmetics, etc.

Techniques of micro-milling are used to prepare sub-micron particles. Experiments performed at the Institute of Geonics demonstrated that different milling technologies (such as planetary mill, ball mill, milling by pressure air and disintegration by high-speed water jet) produce not only various distribution of particles in the final product of disintegration, but they differ primarily in various yield of micron and sub-micron particles. Resulting product also contains particles that are disturbed intensively in a various degree even at the level of crystalline structure of the disintegrated material. Milling technology affects also morphology and specific surface of particles. Method of disintegration of material (precursors, aggregate systems, as well as nanoparticle carriers) is crucial for synthesis of nanoparticles from precursors, their dispersion during separation processes and usage of nanoparticles with carriers in various applications. Range of experiments performed at the Institute of Geonics since 1995 proved that disintegration technologies based on high-speed water jets could represent very effective tool for the above mentioned purposes. The main objective of this work is to show possibility of water jet technology for preparation of desired precursors and/or carriers. The results of disintegration of mica and kaolin using water jet are presented.

The article was written in connection with the project of the Institute of clean technologies for mining and utilization of raw materials for energy use, reg. no. CZ.1.05/2.1.00/03.0082 supported by Research and Development for Innovations Operational Programme financed by Structural Founds of Europe Union and from the means of state budget of the Czech Republic. The work was supported also by the Academy of Sciences of the Czech Republic, project No. AV0Z30860518.

Keywords: water jet, particle disintegration, precursors of nanoparticles, carriers of nanoparticles.

Two-step preparation of silver nanoparticles

P. Suchomel, L. Kvítek, A. Panáček, R. Prucek

Department of Physical Chemistry, Faculty of Science, Palacky University, 771 46 Olomouc, Czech Republic

The reduction of Ag (I) compounds is the most frequently applied method for the silver nanoparticles preparation [1]. There are many various compounds that can be reduced to silver nanoparticles. For example AgNO3, [Ag(NH3)2]+, etc. were used [2][3]. This work presents a new method of silver nanoparticles preparation based on synthesis of silver bromide nanoparticles and their reduction to metal silver nanoparticles.

The silver colloidal dispersions were obtained by two-step preparation method. The first step was based on preparation of the colloidal dispersions of silver bromide nanoparticles. Prepared silver bromide nanoparticles were reduced by sodium borohydride in the second step. The effective diameter of silver bromide nanoparticles and silver nanoparticles were determined by using dynamic light scattering and transmission electron microscopy techniques.

The colloidal dispersions of silver bromide nanoparticles were prepared by reaction of silver nitrate and potassium bromide solutions mixed in various ratios. The first preparation way was based on the various concentration of potassium bromide in a reaction system with constant silver nitrate concentration. In the second case, the various concentration of silver nitrate and constant concentration of potassium bromide were used. The KBr/AgNO3 ratios were 2, 5, 10, 20 and 50 in the both cases. The various concentration of KBr was 2·10-3, 5·10-3, 1·10-2, 2·10-2 and 5·10-2 mol·dm-3 in the presence of 1·10-3 mol·dm-3 AgNO3 and the various concentration of AgNO3 was 1·10-3, 5·10-4, 2·10-4, 1·10-4 and 5·10-5 mol·dm-3 in the presence of 2·10-3 mol·dm-3 KBr. The effective diameter of prepared silver bromide nanoparticles strictly depends on a silver nitrate and potassium bromide ratio (Fig. 1).

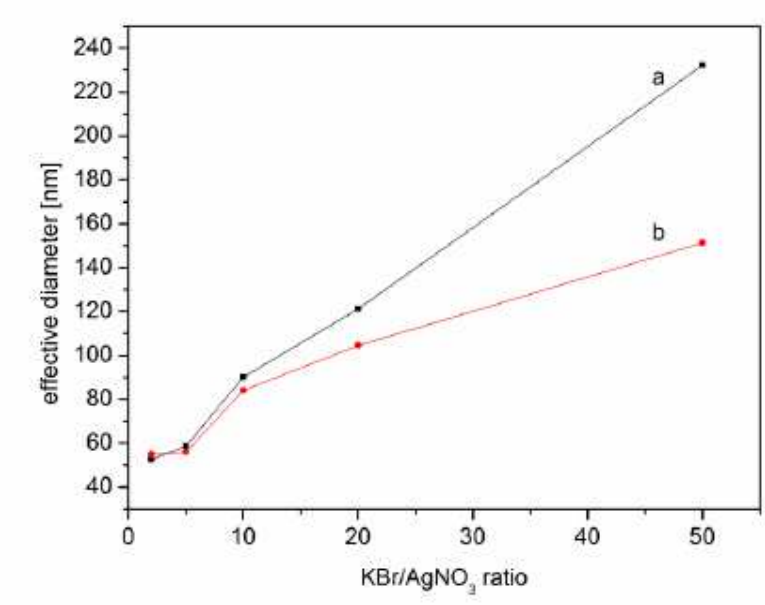


Fig. 1 The influence of KBr/AgNO3 ratio on the effective diameter of prepared silver bromide nanoparticles. (a) 1·10-3 mol·dm-3 AgNO3, various KBr concentration (b) 2·10-3 mol·dm-3 KBr, various concentration of AgNO3

Synthesized silver bromide nanoparticles were reduced by sodium borohydride to silver nanoparticles. The concentration of sodium borohydride was 1·10-2 mol·dm3 in the reaction system. Prepared silver nanoparticles were characterized by DLS and also by TEM techniques. The reduction of silver bromide colloidal dispersions leaded to silver nanoparticles with size between 29 and 50 nm (Fig. 2, Fig. 3).

	$KBr/AgNO_3$ ratio	2	5	10	20	50
$1 \cdot 10^{-3}$ mol·dm^{-3} $AgNO_3$	d AgBr	52,6	58,6	90,0	121,1	232,2
	d Ag	47,3	37,4	28,7	166,0	939,0
$2 \cdot 10^{-3}$ mol·dm^{-3} KBr	d AgBr	54,9	55,9	84,0	104,5	151,4
	d Ag	45,0	42,7	40,0	37,9	50,4

Fig. 2 The effective diameter of prepared silver bromide nanoparticles and silver nanoparticles measured by DLS.

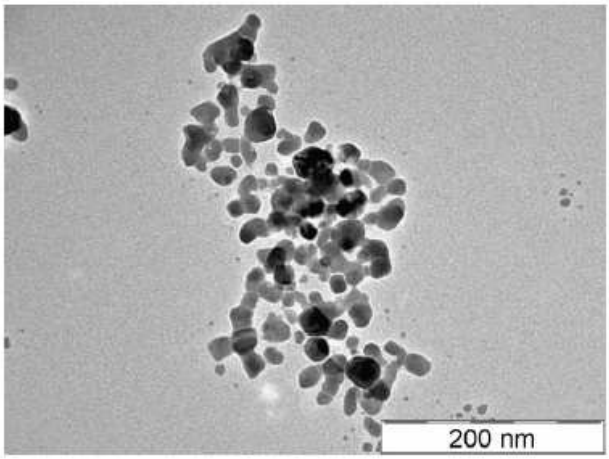


Fig. 3 TEM image of silver nanoparticles prepared by mixing of 1·10-3 mol·dm-3 AgNO3 and 2·10-3 mol·dm-3 KBr solutions.

The results demonstrate preparation of silver bromide nanoparticles with various effective diameters. Obtained results show, that effective diameter of prepared silver bromide nanoparticles highly depend on a reaction compounds ratio. The reduction of silver bromide nanoparticles enable preparation of silver nanoparticles with effective diameter of tens nm.

This work has been supported by the Operational Program Research and Development for Innovations - European Social Fund (CZ.1.05/2.1.00/03.0058), by the Ministry of Education of the Czech Republic (1M6198959201) and by Internal Grant of Palacky University Olomouc (PrF_2011_020).

[1] Z. Khaan, S. A. Al-Thabaiti, A. Y. Obaid, A.O. Al-Youbi, *Colloids and Surfaces B: Biointerfaces*, 2011, 82, 513–517
[2] I. Sondi, D. V. Goia, E. Matijević, *J. Colloid Interface Sci.*, 2003, 260, 75-81
[3] A. Panáček, L. Kvítek, R. Prucek, at al., *J. Phys. Chem. B*, 2006, 110, 16248-16253

Keywords: Nanoparticles, Silver, Silver Bromide, Preparation, Reduction

Electro co-deposition of Sn/MWCNT Nanocomposites on the Copper Substrates: Effect of Current Density

M. Uysal, G. Aydın R. Karslıoğlu, A. Alp, H. Akbulut

Sakarya University Engineering Faculty Department of Metallurgical and Materials Engineering Sakarya 54187, Turkey

mehmetu@sakarya.edu.tr

In recent years, Sn based anode materials are becoming to apply for Li-ion batteries because of their good retention of discharge capacity compared with traditional carbon based electrodes. However, large volume increase and subsequent damage of the Sn based electrodes leaded to limitation for commercial applications. Introducing of MWCNTs into Sn based anode materials is expected to increase cycle life and discharge capacity for possible Li-ion battery application. In this study, MWCNTs were first suspended in the electrolyte and current density-deposition time parameters were investigated for providing high amount of MWCNT co-deposition with Sn matrix. The activation parameters of MWCNTs for homogenous suspension in the electrolyte were also studied. The systematic studies were extended to optimize the deposition time and current density. The resultant nano composites were characterized by scanning electron microscopy (SEM). It was found increasing deposition time resulted in increasing MWCNTs in the deposited Sn matrix. MWCNTs were found to be homogeneously distributed and well-bonded with Sn matrix.

Keywords: Electro co-deposition 1, nano Sn-MMCNT MMCs 2,current density 3, Li-ion batteries

Nanostructuring of chalcogenide glasses - methods and their applications

Miroslav Vlček[1], Sigmund Schroeter[2], Karel Pálka[1], Zdeněk Černošek[1], Jana Holubová[1]

[1]Faculty of Chemical Technology, University of Pardubice, 532 10 Pardubice, Czech Republic

[2]Institute for Physical High Technology, D-07745 Jena, Germany

Miroslav.Vlcek@upce.cz

Chalcogenide glasses, nonoxide glasses in which O is replaced by S, Se or Te, are very promising materials for different photonic applications due to their unique properties and functionalities. Replacement of oxygen by heavier chalcogen atom results in the reduction of average bond strength. The weaker covalent bonds in chalcogenide glasses cause significantly lower rigidity and lower softening temperatures, which are usually between 120 – 320 ^{o}C. Thus many of chalcogenide glasses are photosensitive to exposure with suitable radiation (VIS, UV, X-ray, e-beam, flux of ions,…), which can cause transformation in their internal structure and/or reaction with the environment (O2 from air, metal,…). These photostructural changes/reactions results in significant changes of optical and physico-chemical properties which can be exploited for fabrication of nanostructures in these high refractive index and IR transparent materials.

In this paper, we give a review of nanostructuring methods and possible applications of these materials in extremely high-resolution lithography and the fabrication of diffractive optical elements suitable for applications in VIS and IR spectral region.
Bulk samples of chalcogenides were synthesised from 5N purity elements in a rocking furnace and thin films (thickness d = 300 – 1000 nm) were deposited by vacuum thermal evaporation (starting pressure $6x10^{-6}$ Pa) using resistance- heated silica crucible by deposition rate about 1 nm/s. Photoinduced structural changes induced by different radiation sources were studied by Raman and IR spectroscopy, as well as by Conventional and Modulated Differential Scanning Calorimetry for better understanding of phenomena essence. On glasses with significant photo structural changes the kinetics of their selective dissolution in various alkaline solvents were studied. Both, positive and negative selective etching behaviours were found and applied for production of corrugated relief type phase diffractive optical elements with patterns´ size down to about 400 nm (gratings, Fresnel lenses, microlens arrays…). Still finer structures (about 100 nm or even less) were fabricated using electron beam lithography technique with subsequent selective wet etching.

Direct laser writing with focused laser beam (He-Cd laser, Ar laser and/or doubled wavelength) and excimer laser interference lithography (KrF laser) method were used for cost-effective, single step direct fabrication of surface relief gratings as well.
High quality of fabricated DOEs was confirmed using electron microscopy, AFM and by measurements of diffractive efficiency of fabricated DOE.

This work was supported by the grant P204/11/0832 from the Czech Science Foundation and by the grant 0021627501 from the Czech Ministry of Education, Youth and Sports

Keywords: Chalcogenide glass, Diffractive Optical Element, Nanostructure

Study of Antibacterial Activity of silver NPs against animal pathogens

[1]Kateřina Dědková, [1]Aleš Panáček, [2]Virgínia Aragón Fernández, [1]Robert Prucek, [1]Libor Kvítek

[1]Regional Centre of Advanced Technologies and Materials, Department of Physical Chemistry, Faculty of Science, Palacký University Olomouc, Czech Republic

[2]Centre de Recerca en Sanitat Animal, Univestitat Autónoma de Barcelona, Spain

Silver nanoparticles (NPs) constitute one of the major groups of nanomaterials used in many applications due to their unique properties such as catalytic, optical and biological activity. Antibacterial activity of silver NPs is known for very long time. At the present time, the investigation of this phenomenon is intensifying because of the increase of bacterial resistance to antibiotics, caused by their overuse. Properties of silver NPs are dependent on their size and morphology. Antibacterial activity of silver NPs can be used, for example, in medicine for the preparation of special biofilms which cover catheters or prosthesis which may be useful in reducing the risk of infectious complications in patients with indwelling catheters [1].

In our recent studies we reported the controlled preparation of silver NPs using modified Tollens process, which enables to prepare silver NPs with average size ranging from 25 nm to 100 nm. The size of the synthesized silver NPs was controlled by using reducing sacharides with different redox potentials and by different silver ions concentration undergoing reduction. Concentration of silver ions was easily controlled by changing the concentration of complexing agent, ammonia, in the reduction system [2]. Silver NPs with average size of 25 nm prepared via reduction of $[Ag(NH_3)_2]^+$ by maltose exhibited high antimicrobial and bactericidal activity against Gram-positive and Gram-negative bacteria, including highly multiresistant strains such as methicillin-resistant *Staphylococcus aureus* [3].

In this study, silver NPs with average size of 30 nm prepared via reduction of $[Ag(NH_3)_2]^+$ by maltose were involved in antibacterial testing together with silver NPs with average size of 20 nm prepared by reduction of $AgNO_3$ by $NaBH_4$. For the purpose of the antibacterial activity testing of silver NPs, ten different bacterial strains (*Salmonella enteritis LT2, Staphylococcus aureus, Salmonella enteritis, Salmonella typhimurium, Streptococcus dysagalatiae, Enterococcus faecalis, Streptococcus uberis, Escherichia coli, Escherichia coli eae*$^+$ *a Haemophilus parasuis*) were used. *Salmonella enteritis LT2* and *Staphylococcus aureus* were used as referent strains obtained from collection of cultures, others were isolated from animals e.g. cow, rabbit, bird and so on. Antibacterial activity of silver NPs was determined via standard microdilution method, which enables to determine the minimum inhibitory concentration (MIC) of tested substances. Antibacterial activity of silver NPs prepared by Tollens proces was observed for stabilised and non-stabilised silver NPs. For the stabilisation purpose, several polymers and surfactants were used. It was found a correlation between the aggregation stability and enhanced bactericidal activity in the system of the silver NPs modified by gelatine, kasein, SDS, polyvinylpyrrolidine (PVP), polyacrylic acid (PAC) and Tween 80. The lowest MIC equal to 3,375 μg/ml was found for 30 nm sized silver NPs stabilized by 1% (w/w) gelatine against *Haemophilus parasuis*. The lowest MIC equal to 6,75 μg/ml was found for 20 nm sized silver NPs stabilised by 0,035 % sodium salt of polyacrylic acid against *Escherichia coli eae*$^+$.

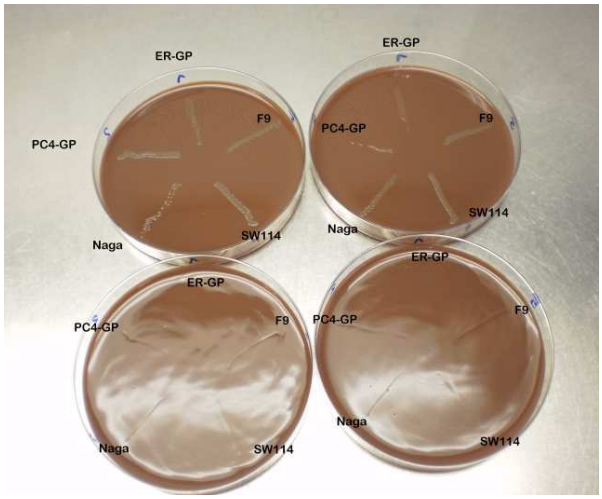


Figure 1. Chocolate plate with five strains of Haemophilus parasuis – overhead plates without silver NPs, at the bottom plates containig silver NPs at the silver concentration equal to 3,375μg/ml

The authors gratefully acknowledge the supports by the Operational Program Research and Development for Innovations – European Social Fund (project CZ.1.05/2.1.00/03.0058). The authors also gratefully acknowledge the supports by the Ministry of Education, Youth and Sports of the Czech Republic MSM6198959218, by the Czech Science Foundation (GAP304/10/1316) and by the internal grant Palacký Univerzity Olomouc (PrF_2011_020).

Keywords: silver, nanoparticles, antibacterial activity

References:

D. Roe, B. Karandikar, N. Bonn-Savage, B. Gibbins and J.-B. Roullet *Journal of Antimicrobial Chemotherapy* , **61** 869–876 (2008)

L. Kvitek, R. Prucek, A. Panacek, *J Mater Chem*, **15**, 1099-1105 (2005).

A. Panáček, L. Kvítek, R. Prucek, M. Kolář, R. Večeřová, N. Pizúrová, V. K. Sharma, T. Nevěčná, R. Zbořil, *J. Phys. Chem B*, **110**, 16248-16253, (2006).

Application of biodegradation of PAU, PCB and NEL soil samples from the hazardous waste dump in Pozd'átky (Czech Republic)

R. Kucerova[1], P. Fecko[1], B. Lyckova[1], L. Koval[1], L. Kovalova[1], L. Kucerova[1]

[1]VSB – Technical university of Ostrava, Institute of environmental engineering, Czech Republic

lukas.koval@vsb.cz

The objective of the project was a laboratory check of biodegradation of soil samples contaminated by PAH(s), PCB(s) and NEL from the hazardous waste dump in the Pozďátky locality. For the laboratory check, pure bacterial cultures of *Rhodococcus Sp*. and *Pseudomonas putida* have been used. It is apparent from the laboratory experiments results that after one-month bacterial leaching, applying the bacterium of *Rhodococcus Sp.* There is a 66 % removal of NEL, a 67 % removal of PAH(s) and a 14 % removal of PCB(s). Applying a pure culture of *Pseudomonas putida* there is a 76 % removal of NEL, a 25 % removal of PAH(s) and a 14 % removal of PCB(s).

Keywords: biodegradation, bacterial leaching, Rhodococcus Sp., Pseudomonas putida

Non-exhaust nano- and micro-particulate emissions from road tradic and its potential environmental and health impact

J. Kukutschová[1], V. Matějka[1], K. Malachová[2], J. Dvořáčková[3], P. Filip[4]

[1]Nanotechnology Centre, VSB- Technical University Ostrava, Czech Republic, [2]Faculty of Science, University of Ostrava, [3]Faculty of Medicine, University of Ostrava, Czech Republic, [4]Center for Advanced Friction Studies, Southern Illinois University Carbondale, USA

Road traffic represents significant source contributing to particulate air pollution, especially by combustion processes, e.g. diesel soot particles. However, there is another source emitting micro- and nano-sized particles. Friction processes associated with braking of automobiles produce wear particles of different size, morphology and chemical composition.
Wear particles generated during the dynamometer tests was collected from the dynamometer chamber. The collected non-airborne debris was compared with ball-milled particles from identical brake lining to assess chemical changes of single components which occur during friction. Particle size distribution of airborne particle fraction was measured in situ and the generated wear particles were collected. The collected fractions and the original bulk material were analyzed using several chemical and microscopic techniques. The experiments demonstrated that the airborne wear particles released were in sizes between 10 nm and 20 µm. The numbers of ultrafine particles (< 100 nm) were by three orders of magnitude larger when compared to the microparticles. A significant release of ultrafine particles was measured when the average temperature of the rotor reached 300°C, the combustion initiation temperature of organics present in brakes. In contrast to particle size distribution data, the microscopic analysis revealed the presence of nanoparticles, mostly in the form of agglomerates, in all captured fractions. The majority of elements present in the bulk material were also detected in the ultrafine fraction of the wear particles [1]. In order to characterize wear particles properly, it is necessary to combine several analytical techniques. The presence of copper and iron oxides as well as carbonaceous components is typical for all collected debris samples [2]. Chemistry of wear debris resembles chemistry of the friction layer. Composition, mutagenic potency and pulmonary toxicity of wear debris and ball-milled particles were also analyzed. Mutagenic potency of initial friction composite and wear particles was evaluated by two *in vitro* bacterial microbioassays (SOS Chromotest, Ames test). Obtained results show potency of wear particles for interacting with DNA after metabolic activation, which indicates the presence of indirect mutagens. The pulmonary toxicity test on rats revealed an acute response of the lung tissue to the ball-milled particles. Further research is necessary to address the role of brake wear particles and potential impact of sub-chronic exposure to wear debris particles [3].

[1] Kukutschová J., Moravec P., Tomášek V., Matějka V., Smolík J., Schwarz J., Seidlerová J., Šafářová K., Filip P.: Environmental Pollution, (2011), 159, 998-1006.
[2] Kukutschová J., Roubíček V., Mašláň M., Jančík D., Slovák V., Malachová K., Pavlíčková Z., Filip P.: Wear, (2010), 268, 86-93.
[3] Kukutschová J., Roubíček V., Malachová K., Pavlíčková Z., Holuša R., Kubačková J., Mička V., MacCrimmon D., Filip P.: Wear, (2009), 267, 807-817.

The study was supported by the Czech Ministry of Education (project number CZ.1.05./2.1.00/01.0040).

Biological activity of silver nanoparticles

L. Kvitek[1], A. Panacek[1], R. Prucek[1], J. Soukupova[1], M. Kolar[2]

[1]Regional Centre of Advanced Technologies and Materials, Department of Physical Chemistry, Faculty of Science, Palacky University, 17. listopadu 12, 77146 Olomouc, Czech Republic

[2]Department of Microbiology, Faculty of Medicine and Dentistry, Palacky University, Hnevotinska 3, 77515 Olomouc, Czech Republic

libor.kvitek@upol.cz

People utilize antibacterial activity of silver very long time, certainly several thousand years. However, silver nanoparticles (NPs), which exhibit very strong bactericidal activity against both gram-positive and gram-negative bacteria including multiresistant strains, are used as effective disinfection agent only from the beginning of the 21th century. They are widely used as antibacterial agents in many things of everyday use, typically as antibacterial modification of fabrics [1]. However, toxicity of silver NPs against living organisms and also in environment is intensively studied because these problems are not solved yet [2].

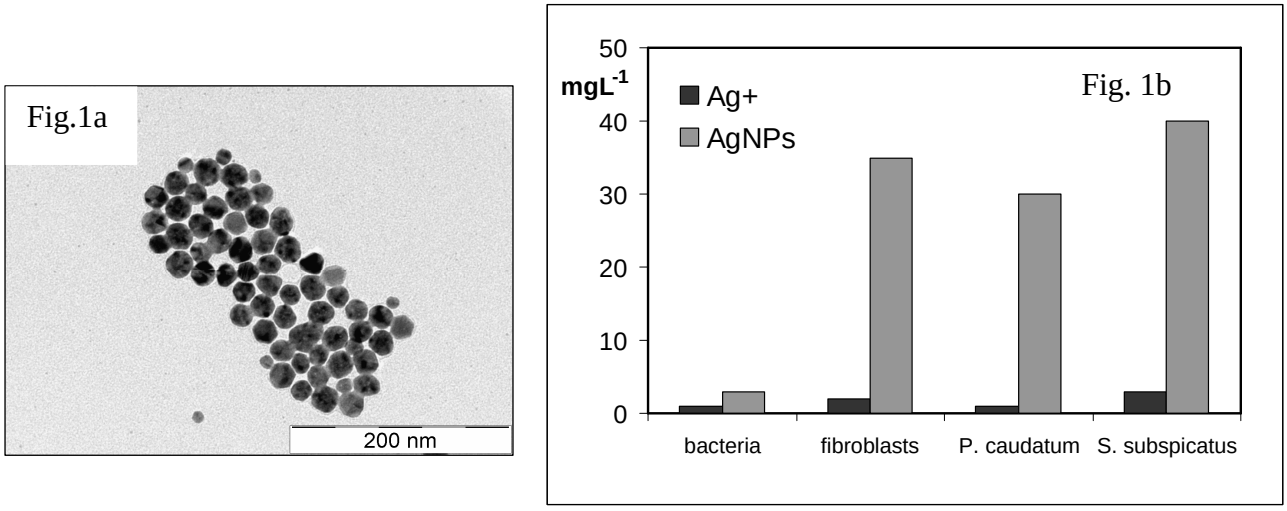


Fig. 1a) TEM image of silver NPs prepared via modified Tollens process; b) toxicity levels of ionic silver and silver NPs for tested microorganisms and human fibroblasts.

Silver NPs with diameter about 25 nm (determined from TEM; see Fig. 1a) can be prepared using simple reduction method based on the Tollens reaction [3]. These NPs show high antibacterial activity at very low concentrations ranging from 1 to 10 mg·l^{-1}, which can be improved by modification of these NPs with surfactants or polymers. Especially antibacterial activity of modified silver NPs is comparable with antibacterial activity of ionic silver. However, silver NPs are not so toxic against mammalian cells in comparison with ionic silver. The experimentally obtained value of acute toxicity for silver NPs is higher than 30 mg·l^{-1} of Ag while the cytotoxic level of ionic silver is much lower (approx. 1 mg·l^{-1}). Moreover, the silver NPs reveal also lower environmental toxicity as have been determined using typical indicator of environmental pollution - the unicellular eukaryotic organism *Paramecium caudatum*. Toxicity level of silver NPs can be considered in this

case higher than 30 $mg \cdot l^{-1}$ while ionic silver still retains its toxicity even at the concentration 0.4 $mg \cdot l^{-1}$. These values unambiguously demonstrate significantly lower toxicity of silver NPs against the eukaryotic organisms with high antibacterial activity, which is comparable with more toxic ionic silver. These values were confirmed via additional experiments where other simple organisms as algae (e.g. *Scenedesmus subspicatus*) or complex organisms as fruit fly (*Drosophila melanogaster)* were used in toxicity tests. However, the effect of sublethal concentrations of silver nanoparticles in environment is so far unknown quantity.

This work has been supported by the Operational Program Research and Development for Innovations - European Social Fund (CZ.1.05/2.1.00/03.0058) and by the Ministry of Education of the Czech Republic (1M6198959201) and also by internal grant of Palacký University in Olomouc (PrF_2011_020).

Reference:
[1] M. Rai, A. Yadav and A. Gade *Biotechnol. Adv.* **27**, 76-83 (2009)
[2] X. Chen and H. Schluesener *Toxicol. Lett.* **176**, 1-12 (2008)
[3] L. Kvitek, R. Prucek, A. Panacek, R. Novotny, J. Hrbac and R. Zboril *J. Mater. Chem.* **15**, 1099-1105 (2005)

Keywords: silver nanoparticles, antibacterial activity, toxicity

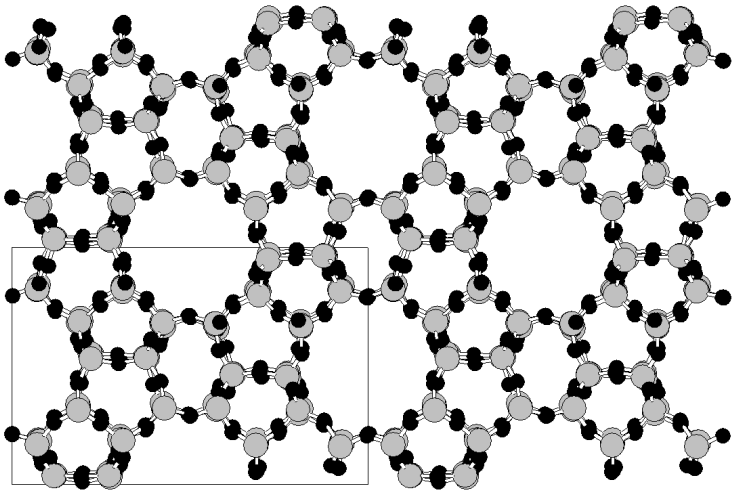
X-ray diffraction study of MFI zeolite membrane

J. Drahokoupil[1], Pavel Hrabánek[2], A. Zikánová[2], M. Kočiřík[2]

[1]Institute of Physics of the ASCR, v.v.i., Czech Republic[2]J. Heyrovský Institute of Physical Chemistry of the ASCR, v.v.i., Czech Republic

draho@fzu.cz

Preparation of preferentially oriented zeolite MFI films is highly desirable due to the potential applications such as high-flux membranes for membrane reactors and selective sensitive sensors. For zeolite MFI (silicalite-1) film, the preferred orientation of crystals is achieved when straight channels are perpendicular to the support surface (*b*-orientation) allowing an easy access for permeating species through the film.



The b-oriented silicalite-1 atomic structure, four unit cells is plotted. Si - ○, O - ●.

The X-ray diffraction study of textured MFI thin layer is complicated by relatively big unit cell and close *a*, *b* lattice parameters that result in overlapping of diffraction peaks. Therefore some non-traditional ways (for x-ray diffraction) have to be used to be able to describe the MFI preferred orientations.

Keywords: *X-ray, Texture, Zeolite, Membrane*

Clay minerals as a support for photocatalytically active nanoparticles

V. Matějka[1], P. Kovář[2], M. Šupová[3], K. Mamulová Kutláková[1], K. Kočí[1]

[1]VŠB-TU Ostrava, [2]ČTCAP a.s., [3]IRSM, AS CR

vlastimil.matejka@vsb.cz

The number of application of nanoparticles has increassing tedency and comprises different industrial areas. One of the most studied utilization of photocatalytic materials comprises photocatalytic degradation of pollutants occuring in living environment. The fixing of the nanoparticles on the surface of clay minerals brings several outstanding advantages including diminishing of possible environmental impact, easier manipulation of the nanoparticles etc. even photodegradation activity of these composites is significantly increased. In this work we foccused on the preparation and characterization of two composites – kaolinite/TiO_2 and vermiculite/CdS. The prepared composites were studied using X-ray diffraction method, the surface morphology was studied using atomic force microscopy and scanning electron microscopy. The photodegradation activity was evaluated by the photodegradation of model organic dye and by photoreduction of carbon dioxide. Both composites show enhanced photoactivity in comparison to bare nanoparticles.

Keywords: TiO_2, CdS, clay minerals, composite.

ZnS and CdS nanoparticles on montmorillonite and their application for photocatalysis

P. Praus, O. Kozák

VŠB-Technical University of Ostrava, Czech Republic

petr.praus@vsb.cz

ZnS and CdS nanoparticles were prepared and deposited on montmorillonite (MMT) in the presence of cetytrimethylammonium (CTA). The band gap energies of nanosize ZnS and CdS were estimated at 3.9 eV and 2.6 eV, respectively. The calculated nanoparticles sizes (diameters) were about 7 nm. Transmission electron microscopy proved the formation of nanoparticles with diameters about 5 nm. Selected area electron diffraction patterns revealed the presence of romboedric ZnS and cubic CdS. The dynamic light scattering method confirmed that the nanoparticles were firmly anchored on the surface of MMT particles. CTA was found to be intercalated into MMT and adsorbed on its external surface.

The prepared ZnS- and CdS-montmorillonite nanocomposites (ZnS-MMT-CTA, CdS-MMT-CTA) contained 6 % of ZnS/CdS and 30 % of CTA. They were used for the photocatalytic reduction of CO_2 providing a considerably high efficiency that several times exceeded the results of commercial TiO_2 Degussa P25. The yields of originating gas products can be arranged in the order: $H_2 >> CH_4 > CO$. Methanol and carbon oxide were also observed in very low concentrations.

Phenol in its aqueous solutions was decomposed by ZnS-MMT-CTA, CdS-MMT-CTA and TiO_2 (P25 Degussa) under ultraviolet light. UV-decomposition without a photocatalyst was performed as well. The prepared nanocomposites were more efficient than the commercial TiO_2 and UV-decomposition. By studying reaction kinetics all the phenol decomposition reactions were found to be of the pseudo-first order. The rates of phenol degradation with ZnS and CdS were comparable and higher than the rates calculated for the degradation with TiO_2 and UV-decomposition. The gas chromatography (GC-MS) and pH measurement results suggested that phenol was oxidized to strong carboxylic acids and further to carbon dioxide.

This work was supported by the Czech Science Foundation (project No. P107/11/1918) and by the Regional Materials Science and Technology Centre (CZ.1.05/2.1.00/01.0040).

Keywords: Nanoparticles, ZnS, CdS, montmorillonite, photocatalysis

Cicada wings as biotemplate and their modifications

Jan Berger[1], Jan Proška[1], Filip Novotný[1], Radek Fajgar[2]

[1] Czech Technical University in Prague, Faculty of Nuclear Sciences and Physical Engineering, Dept. of Physical Electronics, Czech Republic; [2] Academy of Sciences of the Czech Republic, Institute of Chemical Process Fundamentals, Czech Republic

Biomimetics is a modern discipline, which deals with the use of natural structures to create functional materials. Natural patterns can be modified, replicated or mimicked and materials with interesting properties can be created. Surface of cicada's wings is covered with arrays of nanopillars [Fig. 1] and reveals high hydrophobicity. Using chemical vapor deposition (CVD), the chitinous nanopillars were covered by thin layers of SnO_2 or TiO_2. These chemical modifications resulted in reversibly light- and heat-switchable surface wettability. Also, the surface reliefs of wings were negatively replicated by nanocasting into poly(dimethylsulfoxide) (PDMS) and into poly(methylmetacrylate) (PMMA). The PMMA replicas were sputter covered with gold, and silver, respectively. The ultrathin noble metal foils appear to be promising material in research of surface-enhanced Raman scattering (SERS).

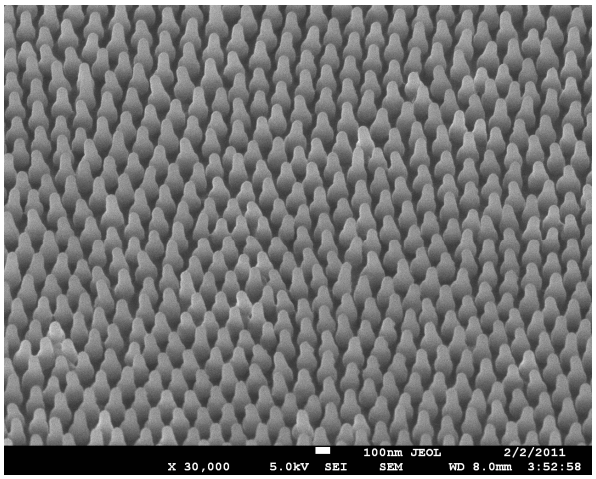


Fig. 1 Dorsal side of cicadas wing with chitinous nanopillars.

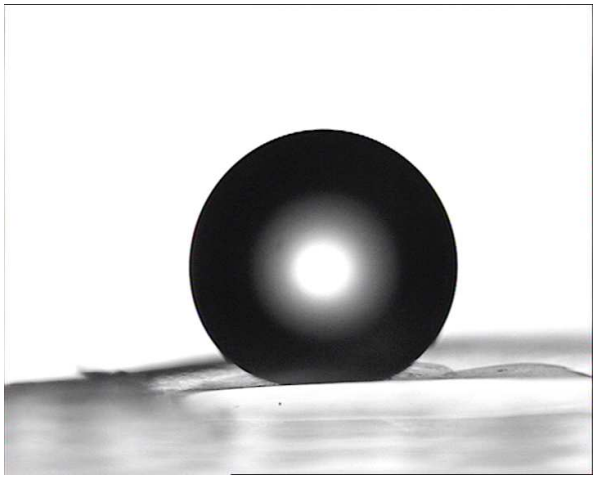


Fig. 2 Drop of water (2 μl) on non-wetting surface of cicadas wing

This work was supported by grants GAAV KAN401220801, Ministry of Education, Youth and Sports, project COST OC09038, and GAČR 203/09/1117.

Keywords: Cicada, biotemplating, nanocasting, wetting, SERS

Comparison of different methods for evaluation of shape, size and particle size distribution of selected nanomaterials

E. Bretšnajdrová[1], L. Svoboda[1], J. Zelenka[2]

[1]University of Pardubice, Czech Republic; [2]Synpo, a. s., Czech Republic

edita.bretsnajdrova@student.upce.cz

At present we can meet nanomaterials and nanotechnology more frequently than ever. Nanoparticles are defined as particulate dispersion or solid particles with a size in the range of 10 – 100 nm and they have unique properties due to their outside and inside structure. With their aid it is possible realize processes, which they are not realizable and achievable by other resources.

A great attention is paid to the study of preparation of nanomaterials applicable in many industrial fields. Except detailed chemical structure, such nanoparticle properties as shape and size distribution are fundamental to the given application. To measure these parameters various methods are used, e.g. acoustic spectrometry, methods based on the light scattering, X-ray disc centrifuge system, transmission electron microscopy (TEM) and atomic force microscopy (AFM).

The ultrasound based technique is suitable for characterizing heterogeneous solid in liquid or liquid in liquid colloidal systems. This method is suitable for concentrated solutions. We have used equipment DT-1200 (Dispersion technology, USA). The technique of laser diffraction is based on the principle that particles passing through a laser beam will scatter light at an angle that is directly related to their size. Particle size distribution measurement was carried out by dynamic light scattering using two types of instruments – Mastersizer 2000 MU (Malvern, United Kingdom) and 90 Plus/BI-MAS (Brookhaven Instruments Corporation, USA). For measurement of particle size the X-ray disc centrifuge system (Brookhaven Instruments Corporation, USA) was used. XDC method allows you to make measurements in either a centrifugal or a gravitational field. A Solver Pro M Atomic Force Microscope (NT-MDT, Russia) was used in tapping mode to produce three-dimensional images of the surface. Second method for measuring size and shape of particles is TEM. We used equipment TEM Tecnai G2 Spirit Twin (FEI, USA). Merits and limitations of the individual methods used to evaluation of nanoparticles of various types are discussed in this work.

This work has been supported by the projects FT-TA3/055 of the Ministry of Industry and Trade of the Czech Republic and has been supported from student grant IGA SGFChT04.

Keywords: Particle size distribution, particle shape, particle size

Preparation and stabilisation of nanogold and nanosilver

Zdeněk Bureš, Miroslav Vlček

Faculty of Chemical Technology, University of Pardubice, Czech Republic

Bures.Zdenek@seznam.cz

In this paper we studied various ways of preparation metal nanoparticles especially silver and gold nanoparticles. Possibility of stabilisation of prepared silver nanoparticles (AgNPs) and gold nanoparticles (AuNPs) was studied as well. Silver and gold nanoparticles can have multiple applications in spectroscopy, surface enhanced Raman scattering, catalysis, medicine, bio-sensing, as well as antistatic and antibacterial materials, or as a cryogenic superconducting materials, etc.

AgNPs were prepared by direct reduction of silver nitrate ($AgNO_3$) in aqueous solution using sodiumborohydride $Na[BH_4]$ as a reducing agent without any stabilizer according the equation (1):

$$\mathbf{2\ AgNO_3 + 2\ Na[BH_4] \rightarrow 2\ Ag^0 + H_2 + B_2H_6 + 2\ NaNO_3} \qquad \mathbf{(1)}$$

Part of aqueous solution of AgNPs was stabilized by PVP+NaCl. Finally AgNPs were prepared by reduction of $AgNO_3$ by $Na[BH_4]$ in the current stabilization by sodium citrate. For all prepared samples (not stabilized, stabilized by PVP+NaCl and/or sodium citrate) absorption spectra were measured. From the shape of absorption bands, the size of prepared AgNPs was determined and from time dependence changes of these spectra stability of AgNPs was checked.

Unstabilized samples of AgNPs aggregate by precipitation of colloidal silver. This is reflected in the absorption spectra as a shift in wavelength absorbent peak to lower values and leads to the absorption of light in the all spectrum. Silver nanoparticles prepared with no stabilization are not suitable for further use due to their instability and aggregation. The dispersions of stabilized nanoparticles show their absorption maxima corresponding to plasmon resonance of silver nanoparticles. Both stabilization using PVP+NaCl and sodium citrate are in principle applicable. Our experiments gave evidence that from these stabilizers PVP+NaCl is better due to stronger sterical effect of PVP. This preservation was proven to be very useful combination for preserving the original characteristics of the prepared nanoparticles, which is of great importance for the preservation and practical applications in many industries.

AuNPs were prepared by reduction tetrachloroauric acid solution $H[AuCl_4]$ by various reducing agents.The obtained nanoparticles were again characterized by time dependence of absorption spectra. In first step we used ascorbic acid, gallic acid and sodium citrate at different concentrations as reductants. We found that the best results give usage of ascorbic acid, but its concentration plays important role as well.

Stability of AuNPs prepared by reduction with ascorbic acid was ensured using different kinds of stabilizers such as polyvinylpyrrolidone (PVP), polyethylenglycole (PEG), polyvinylalcohol (PVA), Tween 20, agar-agar and sodium citrate. As the best stabilizer for stabilizing prepared AuNPs we found PVP and Tween 20. All other stabilizers were not long-acting to keep properties of AuNPs,

due to partial aggregation. Stabilizer Agar-agar is not suitable at all, AuNPs aggregate soon, practically after preparation. The use of citrate as a stabilizer depends on its concentration.

As a conclusion we can state that suitable methods of preparation and long-term stabilisation of AgNPs and AuNPs were found.

Acknowledgement: This work was supported by the grant P204/11/0832 from the Czech Science Foundation and by the grant 0021627501 from the Czech Ministry of Education, Youth and Sports.

Keywords: Metal nanoparticles, Nanosilver, Nanogold

Synthesis, thermal stability and antioxidant properties of selected C_{60} derivatives

Robert Czochara, Rafał Pokrop, Grzegorz Litwinienko

Faculty of Chemistry, University of Warsaw

rczochara@chem.uw.edu.pl

Fullerenes, especially the most popular form C_{60} and carbon nanotubes possess unique properties making them an interesting material of potential applications in many fields of science, industry and biomedicine. From the technological point of view it is very important that fullerenes act like free radical sponge and, therefore, be regarded as efficient radical scavengers.

Because fullerene C_{60} is soluble in limited number of solvents (toluene, benzene, carbon disulfide) we decided to synthesize organic derivatives of fullerene C_{60} containing polar groups i.e.: hydroxyl, nitro and polycyclosulfate groups (Figure 1).

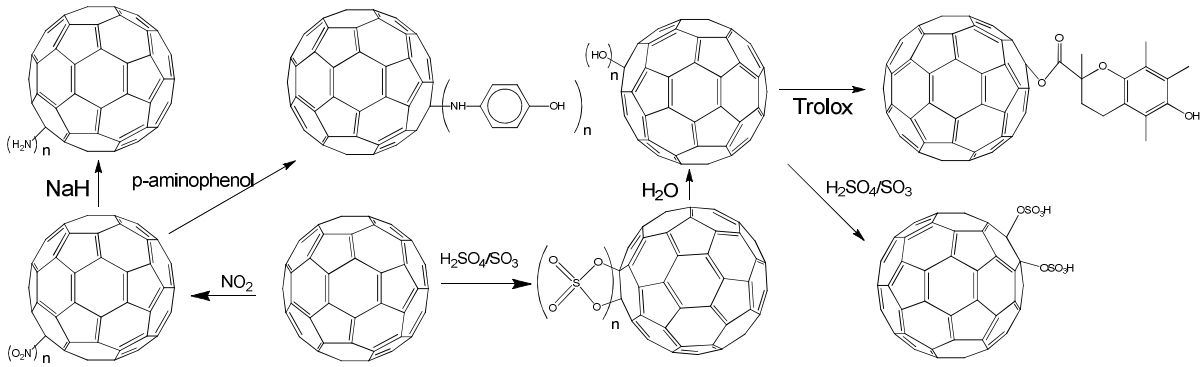


Figure 1. Fullerene C_{60} derivatives studied in this work.

In this work we present the results of $C_{60}(X)_n$ (where X= -OH; -NO_2; -NH_2; -SO_4; -NH-Ph-OH; -Trolox; -SO_3H) characterization by the means of infrared, UV-Vis, NMR, mas spectrometry methods. We decided to carry out the investigation on the thermal stability using thermogravimerty. By the means of the differential scanning calorimetry (DSC) we also carried out the investigation on the potential antioxidant properties during the oxidation of linolenic acid as model of oxidizable organic material.

This work was supported by Ministry of Science and Higher Education, grant N N507452937.

Keywords: Fullerene, Synthesis, Functionalization, Thermal Analysis, Antioxidant

Biosynthesis of silver and gold nanoparticles using *Bryophytes*

G. Kratosova[1], A. Schrofel[1], V. Plasek[2], V. Holisova[1], M. Natsinova[1]

[1]VSB-Technical University of Ostrava, Nanotechnology Centre, Czech Republic

[2]University of Ostrava, Faculty of Science, Czech Republic

gabriela.kratosova@vsb.cz

There is a wide spectrum of possible living organisms for the synthesis of nanoparticles. The biological synthesis has recently gained significant interest due to use of mild experimental conditions (temperature, pressure, and pH). With successive methodological improvement, biological synthesis might acquire some extra advantages such as higher productivity, higher stability of nanoparticles, and lower cost. In last few years, the synthesis of metallic nanoparticles has been reported using bacteria, yeast, fungi, diatoms and higher plants.

Novel biosynthesis experiments with three *Bryophytes* species were conducted using native and mashed moss biomass in the presence of silver ($AgNO_3$) and gold salts ($HAuCl_4$). Fast formation of gold and silver nanoparticles was reported and confirmed by means of SEM, TEM and X-ray diffraction methods.

Results from this study are promising for further applications such as catalysis, antimicrobial applications, biosorption, phytomining, or phytoremediation.

Keywords: Biosynthesis, Gold, Silver, Nanoparticles, Bryophytes

Optimized Doped Titania for Photocatalysis Applications

S. Krejcikova[1], K. Koci[2], L. Obalova[2], L. Capek[3] and O. Solcova[1]

[1]Institute of Chemical Process Fundamentals CAS, Czech Republic

[2]Technical University of Ostrava, Czech Republic

[3]University of Pardubice, Faculty of Chemical Technology, Czech Republic

krejcikova.simona@ icpf.cas.cz

Titania (TiO2) belongs to nanomaterials with enormous application potential in photocatalysis, dye-sensitized photoelectrochemical cells, electrochromic devices, sensors and other applications. TiO2 can be used as a photocatalyst for removing of organic impurities from waste water and air due to its auspicious physical and chemical properties, therefore a great effort has been spent to reveal and improve its photocatalytic activity for practical applications. It is well known that the photocatalytic activity of titania depends on its crystal structure, doping, surface area, surface hydroxyl group *etc*. Much scientific attention has been attracted by the effect of doping. Among various metals a doping effect of silver has been intensively studied owing to high stability of silver and its excellent electrical and thermal conductivity. Moreover silver ability to prevent the backward recombination of a promoted electron (e^-) and a generated hole (h^+) can positively effected the titania photocatalytic activity. This study is focused on preparation and characterization of the specially designed TiO2 and its modified versions (doped by metal) suitable for photodegradations of organic pollutants.

Samples were prepared by sol-gel technique in the reverse micellar environment. The nonionic surfactant Triton X-114, cyclohexane, distilled water and metal precursors were mixed. Thermal treatment (400°C for 4 hours (1°C/min)) was used to form crystalline phase and also to remove all organic impurities.

Surface morfology, texture and crystallinity were thoroughly studied to judge their impact to catalytic properties. Therefore textural and structural characterization of prepared samples were performed by appropriate technique (Nitrogen physical adsorption, X – ray diffraction XRD, Scanning electron microscopy SEM, X – ray fluorescent analysis XRF, elemental analyses and UV – Vis spectroscopy).

The photocatalytic reduction of carbon dioxide and the photocatalytic oxidation of 4- chlorophenol water solution were chosen for the catalyst activity tests and the evaluation of silver doping effect.

The financial support of the Grant Agency of the Czech Republic (104/09/0694 and 203/08/H032) is gratefully acknowledged.

Keywords: Titanium dioxide, Silver, Photocatalysis

DSC, TGA and X-ray analysis of $PdCl_2(X_nPy)_2$ complexes

Agnieszka Krogul, Magda A. Kroteń, Jadwiga Skupińska, Grzegorz Litwinienko

Faculty of Chemistry, University of Warsaw, Poland

akrogul@chem.uw.edu.pl

Square planar palladium(II) complexes with nitrogen donor ligands are of great interest because of their reactivity and potential applications as catalysts in carbonylations [1], polymerizations [2], and other reactions in synthetic organic chemistry. Typical electron donating ligands contain nitrogen: pyridine, bipyridine, phenanthroline and their derivatives, other contains phosphorus and very common P-donor phoshine.

Recently, we have improved $PdCl_2$/Fe/I_2/Py catalytic system (invented in our laboratory [3]) by the use of pyridine derivatives with electron withdrawing (chlorine atoms) or electron donating (methyl groups) substituents [4].

In our current work we examined the impact of electron donating and electron withdrawing effects in the pyridine ring on the on the crystal structure and we studied thermal stability of the whole series of $PdCl_2(X_nPy)_2$ complexes. The following X_nPy ligands were used to form complexes with $PdCl_2$: pyridine, 2-methylpyridine, 3-methylpyridine, 4-methylpyridine, 2,6-dimethylpyridine, 2,4-dimethylpyridine, 3,5- dimethylpyridine, 2-chloropyridine, 3-chloropyridine, 2,6-dichloropyridine, 2,4-dichloropyridine and 3,5-dichloropyridine.

For the first time we characterized crystal structures of: $PdCl_2(3\text{-}MePy)_2$, $PdCl_2(4\text{-}MePy)_2$, $PdCl_2(2,4\text{-}Me_2Py)_2$, $PdCl_2(3,5\text{-}Me_2Py)_2$, $PdCl_2(2\text{-}ClPy)_2$ and $PdCl_2(2,6\text{-}Cl_2Py)_2$ and compared our results with data accessible in the literature for other complexes. In order to study thermal stability of all synthesized complexes Differential Scanning Calorimetry (DSC) and Termogravimetry Analysis (TGA) were applied. We found that $PdCl_2$ compounds with ClPy are less stable than complexes with MePy ligands. During studies of thermal decomposition of $PdCl_2(X_nPy)_2$ we noticed some interesting nano-scale properties of the thermal products and they will be presented and discussed.

Keywords: palladium complexes, substituted pyridines, crystal structure, thermal analysis.

References:

[1] F. Paul, Coord. Chem. Rev. 203 (2000) 269.
[2] J. Skupińska, M. Karpińska, J. Mol. Catal. A. 161 (2000) 69.
[3] G. R. Owen, H. A. Burkill, R. Vilar, A. J. P. Withe, D. J. Williams, J. Organomet. Chem. 690 (2005) 5113.
[4] A. Krogul, J. Skupińska, G. Litwinienko, J. Mol. Catal. A. 337 (2011) 9.

Molecular modeling of polymeric drug carriers for cyclosporine A

M. Macháčková[1], J. Tokarský[1], and P. Čapková[1]

[1]Nanotechnology Centre, VŠB - Technical University of Ostrava, Czech Republic

Design of new pharmaceutical forms with existing drug molecules is motivated by the requirements of better bioavailability, targeted transport, selective and long lasting effect, etc. Common requirements for drug carriers are: zero toxicity, biodegradability and drug-carrier binding suitable for a given purpose (i.e. garrancy of targeted drug release). Biodegradable polymers are good candidates fulfilling the first two conditions and have been intensively studied during recent years first of all as carriers for cancerostatic drugs.

In present work biodegradable polymers have been investigated as drug carriers for imunosupresive drug cyclosporine A (CsA). The main requirement for this new drug formulation was the non-bond interaction between CsA and polymeric chain. Six biodegradable polymers have been chosen for the present study in order to estimate the most suitable candidates for CsA carrier: polylactide (PLA), polycaprolactam (PCL), polychitosan (PCh), polyglycolic acid (PGA), polyethylene glycol (PEG) and polycellulose (PC).

The character of binding between CsA and polymer chain is the key characteristics for a given targeted drug delivery. The investigation of interactions between CsA and polymeric chains is the main subject of the present study, where the main aim is to prepare the supramolecular system with non-bond CsA-polymer interaction, i.e. without covalent bonding between CsA and polymer chain.

Classical molecular dynamics and docking calculations were employed to model and predict CsA-polymer interactions. These interactions were characterized using Flory-Huggins interaction parameter χ, which describes the mutual interactions between CsA and polymeric chain. All calculations have been carried out in Materials Studio modeling environment.

Three different chain lengths for each polymer have been built in order to create one, two and three loops of chain around the CsA molecule. Conformation analysis performed for all polymers led to the optimum chains conformation for docking calculations. All types of biodegradable polymers (chain length: two loops around CsA) have been selected for dynamic simulations in NPT ensemble.

Results led to the following conclusions. Polymers PCh and PC show better miscibility with CsA than others, while PC is more miscible with CsA than PCh. These two chains have similar tertiary structure and exhibit the same twisting. The interaction energies between CsA and polymer (either PCh or PC) are nearly the same and much lower (i.e. stronger interaction) than in case of mixtures containing other polymers. Molecular modeling also revealed that PC chain in CsA-PC mixture is more agglomerated than PCh chain in CsA-PCh mixture. Taking into account the presented results it can be concluded that PC is very promising for further experimental study.

Keywords: Molecular modeling, Drug carriers, Biodegradable polymers

Synthesis and studies of antioxidant properties of selected fullerene C_{60} derivatives

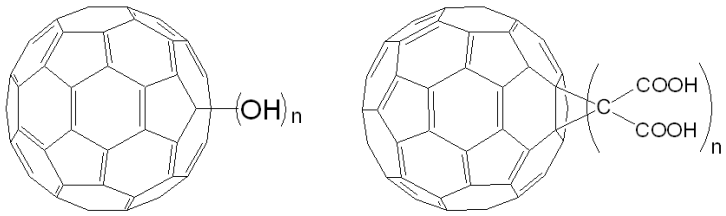
Piotr Piotrowski, Magdalena A. Kroteń, Grzegorz Litwinienko

Faculty of Chemistry, University of Warsaw

ppiotrowski@chem.uw.edu.pl

After their discovery in 1985, fullerenes rapidly became an important object of nanotechnological research leading to new areas of chemistry, physics and materials engineering. However, poor solubility of fullerenes in water sets a limit on their practical applications. Chemical functionalization is one of methods that allow to prepare water soluble derivatives of fullerene. C_{60} cage covalently bonded with chemical groups have different physical and chemical properties.

In our work we synthesized fullerene derivatives with hydroxyl groups and carboxyl groups as malonic acid adducts of C_{60}.



Products were analyzed by FT-IR spectroscopy and thermogravimetry (TG). Since it has been reported in the literature that above-mentioned fullerene derivatives show no cytotoxicity we decided to study their free-radical scavenging activity.

Potential antioxidant properties of these fullerene derivatives were analyzed by differential scanning calorimetry(DSC) during the oxidation of linolenic acid (LNA). Kinetic parameters: pre-exponential factor, reaction rate constant and activation energy for thermal oxidation of pure LNA sample and samples that included LNA with fullerene, fullerenol C_{60} $(OH)_{25,6}$ and malonic acid adducts of fullerene: $C_{60}(C(COOH)_2)_2$, $C_{60}(C(COOH)_2)_3$, $C_{60}(C(COOH)_2)_4$ were determined. Results show that antioxidative stability of LNA increased most after addition of fullerenol $C_{60}(OH)_{25,6}$.

This work was supported by Ministry of Science and Higher Education of Poland, grant N N507452937.

Keywords: Fullerene, Fullerenol, Carboxyfullerene, Antioxidants, Thermal Analysis

Molecular simulations of LDH intercalated with porphyrin anions

M. Pospíšil[1], P. Kovář[1], E. Káfuňková[2, 3], K. Lang[2], F. Kovanda[4]

[1]Charles University in Prague, Faculty of Mathematics and Physics, Prague, Czech Republic

[2]Institute of Inorganic Chemistry, v.v.i, Academy of Sciences, Řež, Czech Republic

[3]Charles University in Prague, Faculty of Sciences, Prague, Czech Republic

[4]Department of Solid State Chemistry, Institute of Chemical Technology, Prague, Czech Republic

pospisil@karlov.mff.cuni.cz

Molecular modelling together with powder X-ray diffraction analysis provide detail information about interlayer space arrangement of Mg-Al layer double hydroxides (LDH) intercalated with [5,10,15,20-tetrakis(4-sulfonatophenyl)porphyrin (TPPS)]. LDH was prepared with the Mg/Al molar ratio 2.

Three different types of initial models were created in order to simulate the experimental XRD patterns of LDH with a TPPS loading of 70–80%. Type 1 represent single-phase systems with a 100% TPPS loading in the interlayer space, Type 2 represent the coexistence of two phases corresponding to the total exchange from 75 to 92%, Type 3 represent models with the coexistence of both TPPS and NO^{3-} anions in the same interlayer space. Results show in according to the energy characteristics and simulated XRD compared with experimental that the Type 2 is the most probable arrangement of TPPS. There are layers saturated only with TPPS anions and others are filled only with original NO^{3-} anions in the LDH interlayer.

Keywords: LDH, porphyrin, molecular simulation

Precipitation of ZnS nanoparticles and their deposition on montmorillonite

P. Praus, R. Dvorský, O. Kozák

VŠB-Technical University of Ostrava, Czech Republic

petr.praus@vsb.cz

ZnS nanoparticles were prepared by the precipitation of zinc acetate with sodium sulphide in aqueous solutions. The originated colloidal dispersions were stabilized by cetyltrimethylammonium (CTA) cations to prevent an unlimited growth of the nanoparticles. The concentration of CTA was set to be higher than its critical micellar concentration to form micelles, in which ZnS nanoparticles were assumed to be encapsulated. Then, Na-rich montmorillonite (MMT) was added into the dispersions and the resulting ZnS-CTA-MMT agglomerates were filtered, dried and stored for next applications.

The band-gap energy of ZnS nanoparticles (3.9 eV) was determined from their UV spectra using the Tauc equation for direct semiconductors. Consequently, it was used for the calculation of the nanoparticles size by means of the Brus equation describing the quantum size effect. In this manner, the theoretical size (diameter) of ZnS nanoparticles was estimated at 7 nm. The dynamic light scattering method was used for the measurement of the nanoparticles hydrodynamic diameter *d* directly in aqueous dispersions. Their moduses for CTA and ZnS-CTA micelles were determined at 3 nm and 16 nm, respectively (Fig. 1).

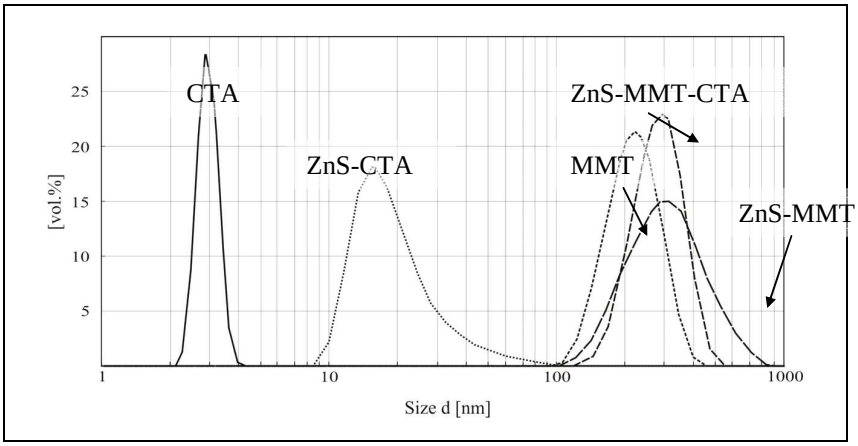


Figure 1 Particle size distributions by volume.

These findings indicate that ZnS nanoparticles were incorporated inside CTA micelles. The positively charged ZnS-CTA micelles adsorbed themselves to the negatively charged surface of MMT particles forming stable agglomerates with the hydrodynamic diameter of 298 nm (modus). For comparison, ZnS and MMT (without CTA) formed the aggregates with the similar modus diameter of 306 nm. The particles size distributions are demonstrated in Figure 1.

This work was supported by the Czech Science Foundation (project No. P107/11/1918) and by the Regional Materials Science and Technology Centre (CZ.1.05/2.1.00/01.0040).

Keywords: Nanoparticles, ZnS, CdS, montmorillonite, micelles

Characterization of vermiculite structure using X-ray powder diffraction necessary to their use in nanocomposites

M. Valášková

Nanotechnology centre, VŠB – Technical University of Ostrava, Czech Republic

marta.valaskova@vsb.cz

Vermiculite is a secondary mineral formed primarily alteration of mica and less commonly by alteration of amphibole, chlorite, olivine, pyroxene or other clay minerals. Structural unit contains the 2:1 layer plus any material in the interlayer. Substitution of metal cations with similar size and lower valence, such as Si^{4+} - Al^{3+}, Al^{3+} - Mg^{2+} creates a net negative layer charge of 2:1 layers. The positive charge deficiency is compensated by exchangeable cations K^{+}, Na^{+}, Ca^{2+}, Mg^{2+} located in the interlayer space between the parallel 2:1 layers. These cations are hydrated and only weakly held by the negative layer charge. Vermiculites show negative layer charge $0.6<x<0.9$ eq/$(Si,Al)_4 O_{10}$, arising mostly from the tetrahedral substitution Al^{3+} for Si^{4+}.

The pretreatment of vermiculites for following their usage in nanocomposites (e.g., as new forms of vermiculite carrier of functional metal nanoparticles or organic molecules; vermiculite nanofiller into polymer matrix) represents several specific techniques (e.g. exfoliation and delamination, intercalation with inorganic and organic species, acid leaching, thermal treatment.).

The stability of different states of hydration or intercalation can be studied using the X-ray powder diffraction (XRD) method during low-rate temperature variation and can be related to the results of thermogravimetry. The changes in the interlayer space influence the manner of neighboring layers in the stacking layer sequences and sort vermiculite structure to the ordered or semi-ordered.

Author is grateful to the Ministry of Education of the Czech Republic (project GA ČR 210/11/2215).

Keywords: vermiculite, nanocomposite, X-ray powder diffraction, structural characterization

Numerical study of magnetic forces between zero-valent iron nanoparticles and their aggregates

Dana Rosická[1], Jan Šembera[1]

[1]Institute of New Technologies and Applied Informatics, Technical University of Liberec, the Czech Republic

jan.sembera@tul.cz

Zero-valent iron nanoparticles are being studied and used in frame of groundwater remediation technologies such as permeable reactive barriers and in-situ reagent. Their main advantages are that they would be able to migrate in porous medium and rapidly react in-situ due to their huge specific reaction surface. If not modified they rapidly aggregate and lose these properties so many surface modifications are being suggested to stabilize them. One of the reasons of rapid particle aggregation is strong and long-range attractive magnetic forces among particles. The core of spherical particles is formed by zero-valent iron and its shell is composed from iron oxides, especially magnetite. The contribution deals with examination of magnetic forces among bare particles without any surface modification.

The magnetic field around an aggregate is dependent on aggregate structure and on structure of particles constituting the aggregate. We suppose that the magnitude of polarization vector of each single particle is the same and they differ only in directions of vector of polarization. In the presented work the difference between composite magnetic field of aggregates with randomly distributed and structured particle distribution will be shown.

This result was realized under the state subsidy of the Czech Republic within the research and development project "Advanced Remedial Technologies and Processes Centre" 1M0554 - Programme of Research Centres supported by Ministry of Education and within the research project FR-TI1/456 "Development and implementation of the tools additively modulating soil and water bioremediation" - Programme MPO-TIP supported by Ministry of Industry and Trade.

Keywords: magnetic forces, nanoparticles, aggregation

Fabrication of SERS substrates using self-assembled nanostructures

L. Štolcová[1], J. Proška[1], F. Novotný[1], M. Procházka[2], I. Richter[1]

[1] Czech Technical University in Prague, Faculty of Nuclear Sciences and Physical Engineering, Department of Physical Electronics, Czech Republic

[2] Charles University, Faculty of Mathematics and Physics, Division of Biomolecular Physics, Czech Republic

lucie.stolcova@centrum.cz

The biggest disadvantage of Raman spectroscopy, very small cross section of Raman scattering, can be overcome by considerable enhancement of the local electromagnetic field due to excitation of localized surface plasmon resonances in metal colloidal particles or structured metal surfaces. This effect, surface-enhanced Raman scattering (SERS), has great potential for applications in biology, medicine and diagnostics but commercial fabrication of the so-called SERS substrates remains a challenge.

We describe here several ways to fabricate highly reproducible SERS substrates using self-assembled structures. We focus on different geometries that can be achieved with metal-coated self-assembled submicron polystyrene spheres.

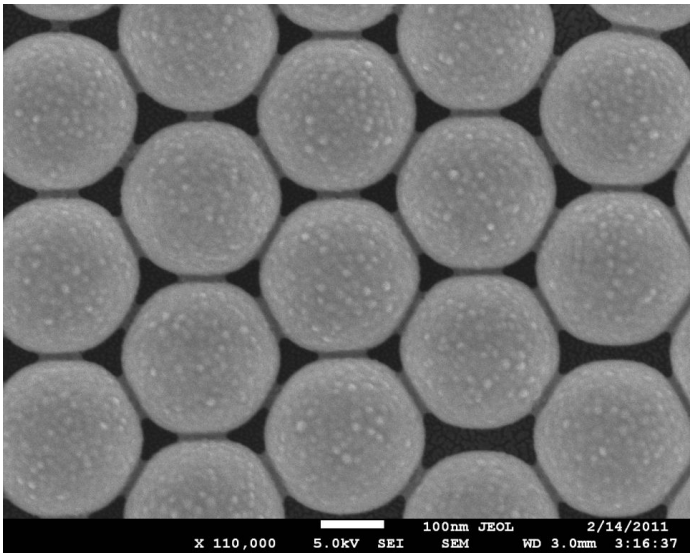


Figure 1. Self-assembled polystyrene spheres (∅ = 266 nm) coated with 20 nm of gold

Keywords: SERS, reproducible SERS substrates, self-assembly

This work was supported partially by grants of GAAV KAN401220801, Ministry of Education, Youth and Sports, project COST OC09038, and MŠMT MSM0021620835.

X-ray diffraction and stress dependence of magnetic properties of Fe nanometer thickness thin films

A. Zolanvar, H. Sadeghi, S. Ranjbaran, M. Moslemi, F. Kaj

Dept. of Physics, Faculty of Science, Arak University, Arak38156-8-8349, Iran

a-zolanvar@araku.ac.ir

Following the general trend of materials sciences towards nano-sized materials, the thin-film community is increasingly concerned with layers with thicknesses that do not exceed a few nanometers. Such ultrathin layers exhibit fascinating properties, such as very high yield stresses and new magnetic properties. The structure of these very thin layers is, however, expected to be highly inhomogeneous because of the presence of a surface and an interface in their close vicinity. We have grown sputtered thin films of iron at various argon sputtering gas pressures, as well as for several thicknesses, and studied the magnetic characteristics of the films. The magnetic characteristics of the films were examined at room temperature using an AGFM. Films grown at different pressures have significantly different microstructure and stress. Films grown at higher chamber pressures exhibit lower saturation magnetization than those deposited at lower sputtering pressures. Films of various thicknesses exhibited less stress in thicker films resulting from microstructural changes. Films deposited at higher argon sputtering gas pressures possess larger grains in a rougher surface.

X-ray diffraction is the method of choice to study lattice parameters in materials because of its very high accuracy. One widely used method for studying elastic strains in bulk or thin film materials is the $\sin^2\psi$ method. It relies on the measurement of the position of X-ray lines diffracted from lattice planes with different orientations (ψ) with respect to the sample normal. If one considers a single-crystal thin film of a cubic material under an equal biaxial state of strain, the ε(strain) *versus* $\sin^2\psi$ plot is linear and yields the stress and the stress-free lattice parameter a_0. It is known that the presence of different crystallographic textures in elastically anisotropic materials may yield oscillatory ε*versus* $\sin^2\psi$ plots. These results have important practical consequences concerning the determination of strains by conventional XRD techniques in ultrathin films. The results indicate that a relative difference of 0.1% between the average lattice parameter.

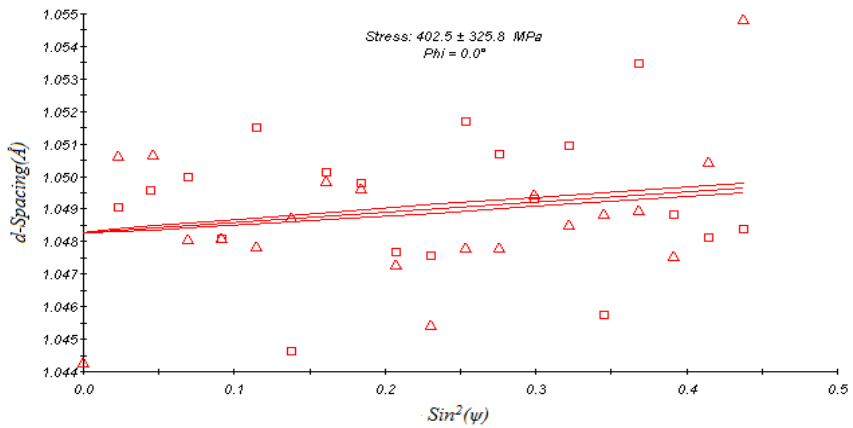


Fig1. d-spacing as a function of $\sin^2\psi$ for Fe film with 50 nm thickness.

Keywords: Fe thin films; Magnetic *properties;* stress; $\sin^2\psi$ method

Zero-valent nano-size iron particles in reductive dehalogenation of persistent organic pollutants in aquatic environment

J. Matoušek, I. Hovorková, R. Bačovská

Masaryk University, Faculty of Science,

Centre for Research of Toxic Compounds in the Environment, Brno, Czech Republic

matousek@recetox.muni.cz

Persistent toxicants, especially persistent organic pollutants (POPs) addressed by the Stockholm Convention (2001) contaminate not only sites of production, storage, use and unintentional releases but much wider areas due to long-range trans-border transport through air and water. In spite of prohibition of use of some of intentionally produced compounds (such as chlorinated pesticides and polychlorinated biphenyls) for a couple of decades ago, they still can be found in inland surface and ground waters, soil, biota, food chains and human beings.

Beside other techniques, reductive dehalogenation was suggested for decontamination of POPs, using metallic reagents, *inter alia* iron.

New possibilities of this reaction are offered by extremely reactive nano-size zero-valent iron particles. First experience of using this material for cleaning surface and ground waters contaminated with various chlorinated and other pollutants is presented, further prospects are discussed.

Keywords: Persistent organic pollutants, Decontamination, Nano-size zero-valent iron

Thermal behaviour of monmorillonites modified with organic cations

E. Plevová[1], V. Valovičová[1], Z. Navrátilová[2], L. Vaculíková[1], G. Simha Martynková[3]

[1]Institute of geonics, AS CR, v.v.i, Ostrava, Czech Republic

[2]Department of Chemistry, Faculty of Sciences, University of Ostrava, Czech Republic

[3]Centre of Nanotechnology, Technical University of Ostrava, Czech Republic

eva.plevova@ugn.cas.cz

The montmorillonites were modified by alkylammonium cations. Modified montmorillonites were prepared by sorption procedure. The X-ray powder diffraction was used to calculate the values of the basal spacing of the modified montmorillonites to confirm the intercalation of organic cations into the montmorillonite structure. Thermal behaviour of the modified montmorillonites was investigated by simultaneous thermogravimetry and differential thermal analysis. In the case of the modified clays, the shape of thermal curves generally corresponded to the original clays, but the thermal effects exhibited the different temperature values and peak intensities. The biggest changes in temperature values were found for dehydratation and recrystallization process. With increasing concentration of surfactants, the amount of adsorbed water in montmorillonite decreased. The decrease of the amount of free or interlayer water results from the replacement of hydrated cations by surfactant as well as from improvement of hydrophobic properties. Contrariwise with increasing of surfactant concentration, the temperature of exotherm effects connected to recrystallization and transformation also increased.

Keywords: montmorillonites, alkylammonium cations, intercalation, thermal analysis

Hydrogen generation from the hydrolysis of sodium borohydride and ammonia-borane using hydroxyapatite-supported cobalt(0) nanoclusters as Catalyst

M. Rakap, S. Özkar

Middle East Technical University, Department of Chemistry, Ankara, Turkey

rakap@metu.edu.tr

Hydroxyapatite-supported cobalt(0) nanoclusters were prepared at room temperature following a two step procedure: Hydroxyapatite was first added to the aqueous solution of cobalt(II) nitrate to replace Ca^{2+} ions in the lattice of HAP by Co^{2+} ions. Then, upon addition of sodium borohydride, cobalt(II) ions were reduced to obtain the cobalt(0) nanoclusters while catalyzing the hydrolysis reaction of sodium borohydride at room temperature. The prepared hydroxyapatite-supported cobalt(0) nanoclusters were isolated as solid materials and characterized by XPS, TEM, SEM, and N_2 adsorption-desorption technique. The hydroxyapatite-supported cobalt(0) nanoclusters were found to be highly active, long-lived, and reusable catalyst in the hydrolysis of alkaline sodium borohydride and ammonia-borane providing 25600 and 7400 turnovers, respectively. Catalytic hydrolysis of alkaline sodium borohydride and ammonia-borane was found to be first order with respect to the catalyst concentration, but zero order with respect to the substrate concentration. Activation energies for the hydrolysis of alkaline sodium borohydride and ammonia-borane in the presence of hydroxyapatite-supported cobalt(0) nanoclusters were found to be 53.0 and 50.0 kJ/mol, respectively. These findings make the hydroxyapatite-supported cobalt(0) nanoclusters a promising candidate to be used as catalyst in developing highly efficient portable hydrogen generation systems using sodium borohydride or ammonia-borane as solid hydrogen storage material.

$$NaBH_4(aq) + 2H_2O(l) \xrightarrow{\text{catalyst}} NaBO_2(aq) + 4H_2(g)$$

$$H_3NBH_3(aq) + 2H_2O(l) \xrightarrow{\text{catalyst}} NH_4^+(aq) + BO_2^-(aq) + 3H_2(g)$$

Keywords: cobalt, nanoclusters, hydroxyapatite, hydrogen, sodium borohydride, ammonia-borane

Photocatalytic activity of TiO_2 nanoparticles tested on yeasts

J. Wiener[1], Vejsadová[1], A. Chládová[1]

[1]Technical University of Liberec, Czech Republic

jakub.wiener@tul.cz

Yeasts

Yeasts are eukaryotic micro-organisms classified in the Fungi, with the 1500 species currently described. The yeast species have been used in production of alcoholic beverages for thousands years by fermentation – the metabolism of carbohydrates by certain species of yeast under low-oxygen or anaerobic conditions. It is also extremely important as a model organism in modern cell biology research, and is one of the most thoroughly researched eukaryotic microorganisms.

Photocatalysis

In actual time is a photoactivity of nanoparticles TiO_2 in front of scientific interests, on the score of its general using in the area of innovate environment. Titanium dioxide belongs to mostly used photocatalysers. Photocatalytic effect on TiO_2 makes it possible at the low temperature oxidative decomposing of organic structures and also bacillus. The final product of these reactions is theoretically broking of all organic materials to elemental inorganic components.

Experiments and results

In this study is tested a novel method of biocide activity of TiO_2 nanoparticles. As a model organism were used yeasts, which have suitable properties for standard testing of biocide activity. Used yeasts (*Saccharomyces cerevisiae)* are nontoxic, quickly growing and easy to detect. The main advantage is that experiments it is not necessary to realize in biological lab. The results obtained on yeasts are practical, because the anti-yeasts activity is sometimes required on common products (for example textiles). Standard tests of biocidic activity are based on bacteria (such as *Staphylococus aureus*), but the tests must be realized in special bacteriological lab.

I our novel test method of photocatalysis is measured the volume of CO_2 produced by fermentation process in system containing yeasts. The rate of CO_2 production is a criterion of yeasts activity. The influence of photocatalysis on the yeasts activity was tested under UV-A light for different times.

Conclusion

The activity of yeasts was decreased by UV irradiation if the system contains nanoparticles of TiO_2. Presented method can be used for standard testing of biocide properties of TiO_2 nanopartocles.

This paper is supported by: 1M0553 - Výzkumné centrum TEXTIL II (MŠMT)

Keywords: TiO_2, yeast, photocatalyses, test

Sorption-filtration process using Polyamide 6 Nanofibres as a sorbent to remove dye from wastewater

S. Ntaka[1], J. Wiener[1]

[1]Technical University of Liberec, Czech Republic

jakub.wiener@tul.cz

The aim of this study was to examine the use of polyamide 6 nanofibers as a sorbent material for removal of dye on textile wastewater. Simulated wastewater of two acid dyes namely C.I. Blue 113 and C.I. Blue 41 was used for experiment test with simulated concentration of 10 mg L^{-1} ,for sorption-filtration process electrospun polyamide 6 with surface density 5 g $m^{-2}$was used as the sorbent material and the process was performed under vacuum pressure. The experimental result showed that the presented study on polyamide 6 nanofibers has a great potential to be used for dye removal, since it was able to absorb dye at room temperature and high sorption was observed on C.I. Blue 113 dye with the experimental equilibrium reach after the 7^{th} cycle for both experiments.

Keywords: Sorption-filtration process; Sorbent; electrospun polyamide 6 nanofibers; Wastewater; Dye removal

The effect of surfactant concentration on Ni/Al_2O_3-hBN hybrid composite coatings by electro co-deposition

H. Gül[a], A. Alp[b], H. Akbulut[b]

[a]Duzce University, Gumusova Vocational School, Department of Metallurgy, 81850, Duzce/TURKEY

[b]Sakarya University, Engineering Faculty, Department of Metallurgical & Materials Engineering, Esentepe Campus, 54187, Sakarya/TURKEY

harungul@duzce.edu.tr

In the present work, Ni/Al_2O_3-hBN hybrid metal matrix composite (MMC) coatings were deposited from a modified Watt's type electrolyte containing nano-Al_2O_3 particles and hBN flakes by direct current (DC) plating method. It was aimed to develop optimum surfactant concentration for obtaining non-agglomerated particles and high volume fraction of ceramic particles in the deposited Ni matrix. Since Ni coatings have low surface hardness for the applications that both corrosion and wear resistant are expected, Ni hybrid composite coatings were considered to overcome difficulties for dies, tools, automobile parts, microdevices and bearing applications. Al_2O_3 nano particles with average particle size of 80 nm were co-deposited with micron sized flake-like h-BN particles and nickel matrix on the steel substrates. For the determination of the deposited layer properties micro hardness, wear resistance and friction coefficient were measured and scanning electron microscopy (SEM), equipped with EDS, was used for characterization. The hardness of the coatings was found to be 317 – 372 Hv depending on the particle volume fraction in the Ni matrix. The specific thickness and particle volume fraction in the deposited composite coatings were measured about between 50 – 200 µm and 0.02 – 0.12, respectively. The effect of ceramic particles in nickel matrix on the tribological performances of the hybrid coatings produced with different surfactant concentrations was investigated. The tribological tests were performed by a reciprocating ball-on disk apparatus sliding against M50 steel ball (Ø 10 mm). All the friction and wear tests were performed under unlubricated condition at room temperature and in ambient air (relative humidity 55–65 %). The results showed that the wear rate of the nano composite coatings could be reduced to such low values of $20x10^{-4}$ mm^3/Nm.

Keywords: Electro-codeposition, nano MMC, Al_2O_3-hBN hybrid coatings, wear resistance

Influence of ball milling conditions on particle size distributions of fly ash

Vijay Baheti, A.M. Rehan Abbasi, Jiri Militky

Depart. of Textile Materials, Technical University of Liberec, Czech Republic.

vijaykumar.baheti@gmail.com

Nanotechnology has become the buzz-word in recent years. Materials at the nanoscale have enhanced mechanical, electrical, magnetic, optical and chemical properties to a level that cannot be achieved by large scale materials. A variety of ways have been reported to synthesize nano level materials such as plasma arcing, chemical vapor deposition, electro deposition, sol-gel synthesis, high energy ball milling etc. Among these methods high energy ball milling has advantages of being simple, relatively inexpensive to produce, applicable to any class of materials and can be easily scaled up to large quantities. In the milling process, powder particles are subjected to a severe plastic deformation due to the repetitive compressive loads arising from the impacts between the balls and the powder. The purpose of this study is to determine optimum milling conditions for preparation of nanoparticles with known size distributions and fly ash was chosen as raw material for milling. The effect of milling time, ball size, direction of rotation and state of milling in dry or wet condition were selected as milling parameters to decide the size reduction of particles.

The reduction in particle size of fly ash from micron level to the nano level was carried out using a high-energy planetary ball mill of Fritsch Pulverisette 7, Germany in a sintered corundum bowl of 80 ml capacity using zirconia balls of 10 mm Φ and 3 mm Φ sizes under both dry and wet conditions for total duration of 5 hours keeping the reverse rotation of bowl OFF and ON. The ball mill was loaded with ball to powder weight ratio of 10:1. The rotation speed of the planet carrier was 850 rpm. However it was observed in case of dry milling that material starts to stick to the surface of bowl due to role played by surface forces below particular size reductions and thus results in longer milling time to achieve the nanoscale dimensions. While this phenomenon of sticking to the bowl surface not encountered during wet milling of fly ash in deionised water. So wet milling was decided for grinding the fly ash to nanodimenisions.

Wet milling was carried out by adding 6 g of fly ash in 20 ml of deionised water with same milling conditions as mentioned previously. The milled sample powder was taken out at a regular interval of every 2 hours of milling. Then this mixture of fly ash in deionised water was diluted to obtain the dispersion of 0.5% fly ash in deionised water for characterisation of particle size measurement on Coulter N4 Plus, Fritsch Particle size analysette 22 and Malvern Zetasizer nano based on different principles like light diffraction and brownian motion of particles. However Malvern Zetasizer based on quasi elastic light scattering found to be precise and accurate to measure the size of particles down to nanoscale as compared to other methods which overestimated the size of particles.

Keywords: Ball Milling, Quasi Elastic Light Scattering, Particle Size Distribution

Ultrasonic treatment of vermiculite particles

K. Barabaszová, M. Valášková., P. Kalivoda

Nanotechnology Centre, VŠB-Technical University of Ostrava, Czech Republic

karla.barabaszova@vsb.cz

Preparation of small phyllosilicate particles requires reduction of their diameter. In industry, the mechanical wet and dry grinding combination procedures are usually utilized to this purpose. The acquired particles lateral size and particle thickness obtained from millings allow their many important applications such as coating, lightweight additive, isolation, etc. Short-time grinding of clay particles (phyllosilicates) produces considerable particle size reduction but extended grinding lead to an intense structural degradation of the lamellar shape and progressive amorphization accompanied with formation of hard agglomerates.

Ultrasonic treatment (ultrasonication) in industry is traditionally used for agglomerates separation and particle size reduction. This method is interesting for preparing of nanometric clay particles. Reduction of the particle size using treatment by high power ultrasound is an alternative method that avoids to undesirable effects. It is known that clay minerals particles and crystal structure upon ultrasound treatment change physical and chemical properties which consequently affect properties of their nanocomposites matrices.

Important are also conditions for the use of ultrasound, which may affect the clay particles contamination. The resulting heterogeneity of structural defects and chemical contamination may unfavourably change material properties, thus influencing the properties of composites manufactured from defined phyllosilicates particles.

Ultrasonic treatment has positive influence on the defined clay particles preparation but negative in the case of their particle size analyses. Is known, during laser diffraction analysis is traditionally used the ultrasonication. This procedure is realized in dispersing unit with different liquid medium (distilled water or denatured alcohol) and is responsible for individual particle stabilizing and particle agglomerates destruction. Monitoring the exposure to ultrasound on the structural and chemical properties of vermiculite particles is of great importance for the characterization of their size. Aim of this work is to study the effect of ultrasound treatment in comparison to the grinding procedure on the morphology and structural changes of vermiculite particles.

The shape of vermiculite particles after ultrasonic treatment has been studied using scanning electron microscopy (SEM) and atomic force microscopy (AFM). Structural phase analysis has been performed using X-ray powder diffraction (XRD) method and infrared spectroscopy (FTIR). The specific surface area and porosity were determined using gas adsorption technique.

The present work was solved in the frame of the research project of GAČR 205/09/0352 and Ministry of Education, Youth and Sport of Czech Republic MSM 619 891 0016.

Keywords: vermiculite, ultrasonic treatment, particle morphology, structural change

Doping effect of Fe^{3+} and Ni^{2+} cations on synthesis of spinel $LiM_xMn_{2-x}O_4$ cathode active nanoparticles for rechargeable lithium ion batteries

E. Bulut[1], A. Örnek[2], M. Özacar[1]

[1]Department of Chemistry, Sakarya University, Turkiye

[2]Institute of Science, Sakarya University, Turkiye

ebulut@sakarya.edu.tr

Spinel $LiMn_2O_4$ is an attractive cathode active material for Li-ion batteries due to its non-toxic nature, ease of preparation when compared with other layered oxides such as lithium cobalt oxide ($LiCoO_2$) and lithium nickel oxide ($LiNiO_2$) and low cost.Although, the main disadvantage of $LiMn_2O_4$ spinel as cathode material is the decrease of the specific capacity during cycling of the lithium-ion batteries.The
capacity fading mechanisms on spinel $LiMn_2O_4$ are complex and not fully understood, several reasons are considered to explain its poor cycling performance, including structural instability, Jahn–Teller distortion and Mn dissolution into electrolyte, etc. As a result, partial substitution of manganese ions with other transition metal ions such as Fe^{3+} and Ni^{2+} enhances the cyclability of the $LiMn_2O_4$ spinel.

Several low-temperature preparation methods such as sol–gel method, chemical precipitation, pechini process and hydrothermal method, have been used to obtain spinel $LiMn_2O_4$and doped $LiM_xMn_{2-x}O_4$cathode activematerials for use in rechargeable lithium-ion batteries.

In this study, we investigateddopping effect of Fe^{3+} and Ni^{2+} cations on the lattice framework of spinel $LiMn_2O_4$ by calculating the cell parameters.The structure of undoped $LiMn_2O_4$ and doped $LiM_xMn_{2-x}O_4$ were characterized by X-ray diffraction (XRD) (Fig. 1d). Size of the nanoparticles were determined by scanning electron microscopy (SEM) (Fig. 1a and c) and calculated with Debye-Scherrer equation. Dopping ratios of substituted cations were proved by electron diffraction spectroscopy (EDS) (Fig. 1b).

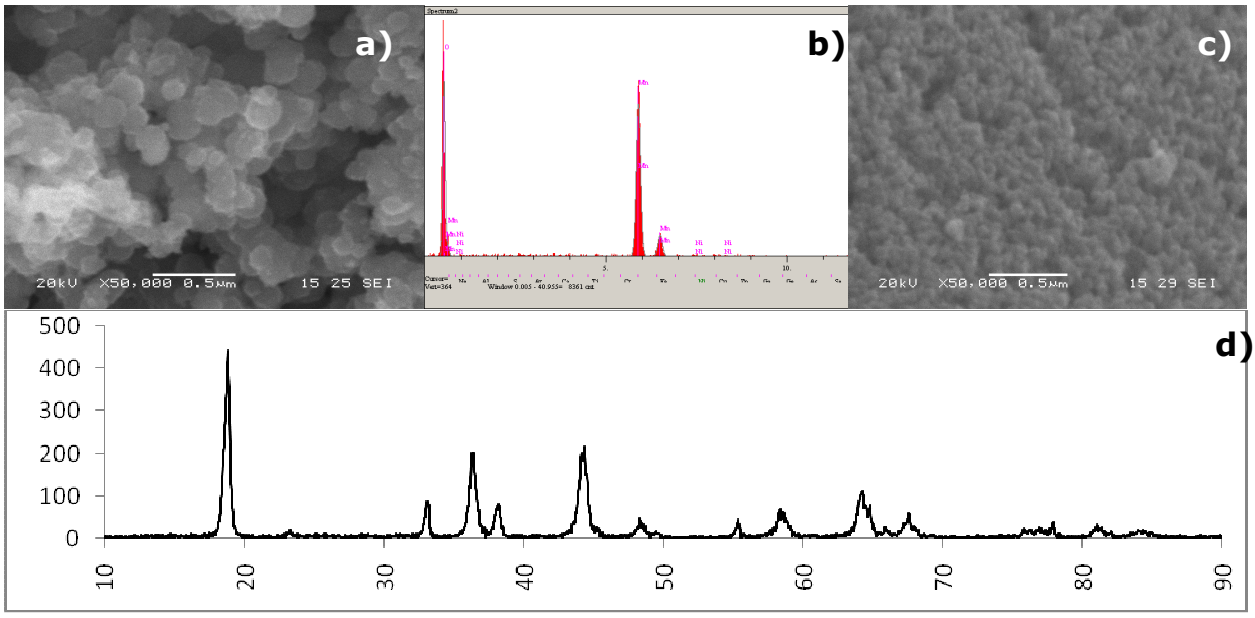


Figure 1. SEM images (a and c), EDS (b) and XRD patterns (d)of $LiMn_2O_4$and doped $LiM_xMn_{2-x}O_4$cathode active nanoparticles

Keywords: Spinel $LiMn_2O_4$, Cation dopping, Li-ion batteries, Cathode active nanoparticles

Effect of calcination temperature on synthesis of $LiMn_2O_4$ cathode active nanoparticles for rechargeable lithium ion batteries

E. Bulut[1], A. Örnek[2], M. Özacar[1]

[1]Department of Chemistry, Sakarya University, Turkiye

[2]Institute of Science, Sakarya University, Turkiye

ebulut@sakarya.edu.tr

Rechargeable lithium ion batteries have a significant interest for a few decades due to their high energy density, high capacity, light weight and long cycle lifein the areas of portable electronics, such as notebook computers, cell phones, and electrical vehicles.

Spinel $LiMn_2O_4$ with a three dimensional cubic structure is the most attractive cathode active material for Li-ion batteries due to its low cost, high abundance and environmentally benign nature. Theoretical charge storage capacity of $LiMn_2O_4$ is 148 mAh/g. However, $LiMn_2O_4$cathode has capacity fading during cycling. Reducing particle size of $LiMn_2O_4$ to the nano size has been shown to enhance power performance due to the large surface to volume ratio that allows for a large electrode-electrolyte interface area.

Here, we synthesized $LiMn_2O_4$ cathode active nanoparticles which have grain size of 100 nm and less. Effect of calcination temperature of the cathode active materials on the grain size, morphology and structure were investigated. Structural characterizations of as synthesized $LiMn_2O_4$ nanoparticles were performed by X-ray diffraction spectroscopy (XRD) (Fig. 1c) and morphological characterizations by scanning electron microscopySEM (Fig. 1a and b). In addition, cell parameters were calculated from XRD data and interpreted.

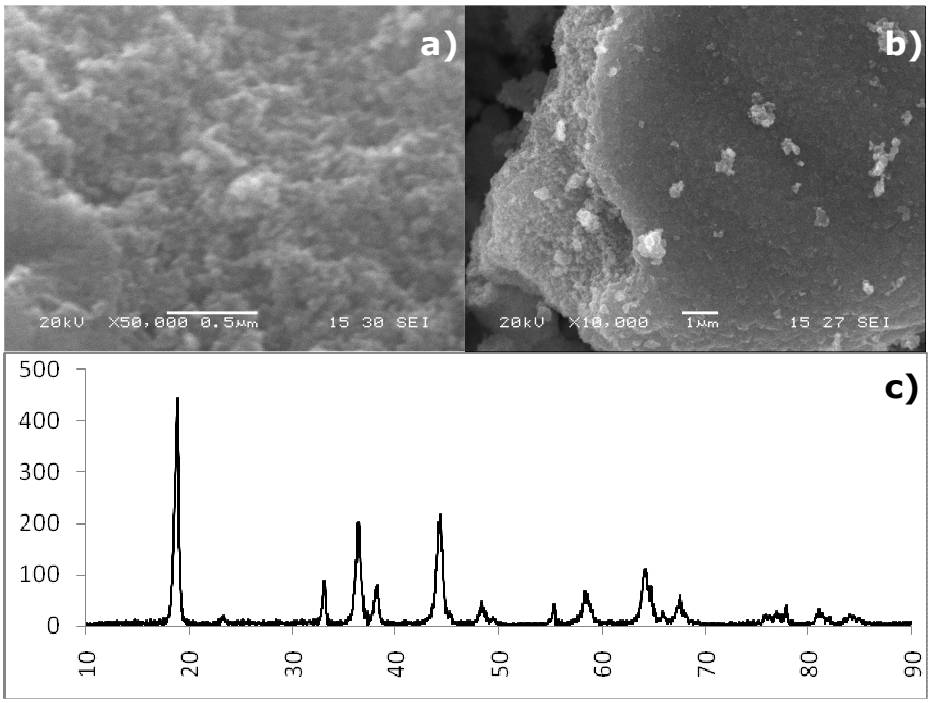


Figure 1. SEM images (a and b) and XRD patternof $LiMn_2O_4$ cathode active nanoparticles

Keywords: Li-ion batteries, $LiMn_2O_4$, Cathode active materials, Nanoparticles

Plasma treatment of polyamide fibers and nanofibers

B. Cigánová[1], J. Wiener[1]

[1]Technical University of Liberec, Department of Textile Chemistry, Liberec, Czech Republic

barbora.ciganova@tul.cz

Polyamide is used for clothes, carpers and technical textiles. Polyamide is one of the most common fibers in the world.

Polyamide is typical by the hydrophilic properties in comparison with other synthetic fibers, but cotton and many other natural fibers are more hydrophilic. For many industrial and clothing applications is interesting to change the wetting properties of polyamide to obtain extreme hydrophilic or extreme hydrophobic properties.

New and very interesting method is change of polyamide surface properties by plasma treatment. Nowadays technology of atmospheric plasma is enough intensive to change to surface properties of polymer in duration of few seconds. In this study is used special source of plasma "barrier discharge BD" – this method is typical by high energy plasma in air or in other gas with atmospheric pressure. Samples were treated by different times.

Polyamide used for experiments was in fiber form. Interesting results we obtained by compare of polyamide fibers in form of classical fibers and nanofibers. Classical fibers are in bonded at plasma treatment in fabric; nanofibers are used as a non-woven. Nanofibers are created by electro-spinning technology in semi-industrial scale.

A surface and morphological change of polyamide (in form of fibers and nanofibers) was tested by electron microcopy, infrared spectroscopy, wetting/wicking and sorption properties.
Plasma treated samples were more hydrophilic after plasma treatment in air. The effect of hydrophobization depends on duration of plasma treatment and on the fiber diameter.

This work was supported by KAN101630651.

Keywords: nanofibers, fibers, plasma treatment

Study of preparation of composite materials containing silver nanoparticles

M. Karlíková[1], L. Kvítek[1], A. Panáček[1], R. Prucek[1], M. Kolář[2]

[1]Regional Centre of Advanced Technologies and Materials, Department of Physical Chemistry, Palacky University, Olomouc; Czech Republic

[2]Institute of Microbiology, Faculty of medicine, Palacky University, Olomouc

mar.karlikova@centrum.cz

The increase of application potential of metal nanoparticles (NPs) brings their anchoring on a solid substrate. Such composite materials can be characterized by their unique mechanical, optical and electrical properties. They are often used as catalysts, antibacterial materials, materials for solar cells or sensors construction etc. Moreover, the anchoring of nanoparticles on suitable substrate can minimize the ecological risk linked with the use of nanoparticles [1].

The main attention is engaged in preparation of composite materials containing silver NPs, mainly for their antimicrobial properties and for almost no resistance which bacteries create against these silver NPs [2, 3]. The several kinds of silver-composites are known as silver-zeolite [4], silver-polymers nanocomposites (e.g. silver-polypropylene fibers) [5] or silver-metal oxides including an antibacterial Ag-SiO_2 nanocomposite [6]. The silver NPs and their composites can be used for antibacterial modification of textile materials or they can be embedded on an active carbon surface and engaged as an antibacterial medium for water purification [7].

Our attention was focused on study of preparation of composite materials containing silver NPs and on the verification of the influence of sorbent on properties of silver NPs.

The aqueous dispersion of silver NPs was prepared by modified Tollens process, by reduction of diammine silver(I) complex by disaccharide maltose. The average size of thus prepared particles was about 30 nm with narrow size distribution, determined by dynamic light scattering (DLS). The composites were prepared by adsorption of these silver NPs on inert adsorbent, which was active carbon with high surface area and two types of clay minerals – bentonite and montmorillonite. All of these adsorbents had been dried for 2 hours at 105 °C. Thereafter the amount of 0.5 g of clay or active carbon sample had been shaken in Erlenmayer flask with 150 ml of silver NPs dispersion with increasing concentration of silver (0.03–1.00 mmol L^{-1}). Three hours later, after the equlibrium was established, this system was filtered and the prepared silver-composite materials had been air-dried for 24 hours.

The structure of silver-composite materials was characterized by means of transmission electron microscopy (TEM – Figure 1a) and by powder X-ray difraction (XRD). The amount of anchored silver NPs was interpreted by UV/Visible spectrophotometry from decrease of absorbance of solution after the adsorption and these results were confirmed by thermal analysis of prepared nanocomposites.

The adsorption isotherms were obtained from these experimental results for all kinds of adsorbent. They revealed that the adsorption process is described by the model of Langmuir adsorption isotherm (Figure 1b). It was also found out that the type of adsorbent significantly influenced the amount of anchored silver NPs from aqueous dispersion. In case of active carbon (with specific surface area of 1270 m^2/g), the maximum of anchored silver NPs was equal 11.21 mg per 1 g of active carbon. In the case of inorganic substrates amount of adsorbed silver NPs decreased to value 4.36 mg per 1 g of montmorillonite.

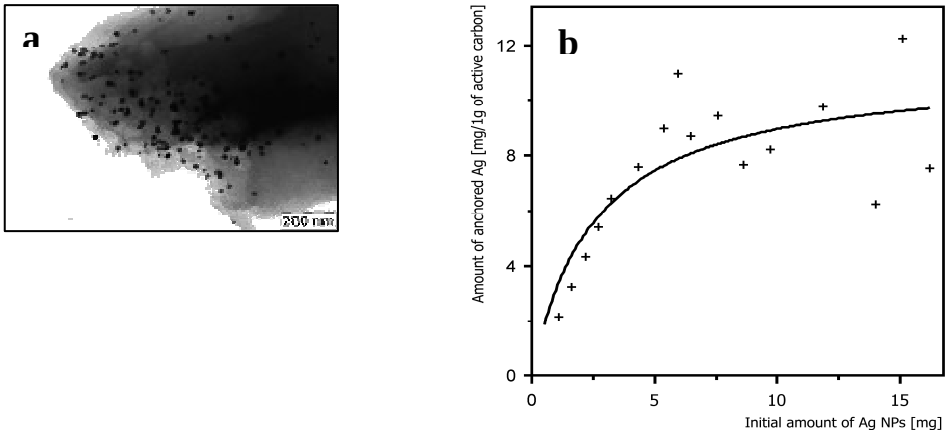


Figure 1. a) TEM image of anchored silver NPs on the surface of active carbon (silver-composite material). b) Adsorption isotherm: The function of adsorbed amount of silver NPs anchored in 1g of active carbon depending on initial amount of Ag NPs in dispersion.

Desorption of silver from composite materials was studied too. The 1% (w/w) aqueous dispersion of this composite was prepared and this system has been shaken for two hours. Then it was filtered and the amount of silver NPs in filtrate was determined using atomic absorption spectroscopy (AAS). Less than 1% of silver was removed from composite in comparison with the initial amount of adsorbed silver NPs.

Additionally the influence of the inert adsorbent on the antimicrobial and antifungal activity of silver NPs was studied. Antibacterial effect against Gram-positive (*Staphylococcus aurea*) and also Gram-negative bacteria (*Enterococcus faecium*, *Klebsiella pneumoniae* or *Escherichia coli*) was studied. Very low concentrations of several μg/ml of these silver composite materials, cause the significant inhibition of the studied bacteria growth.

This work has been supported by the Operational Program Research and Development for Innovations - European Social Fund (CZ.1.05/2.1.00/03.0058) and by the Ministry of Education of the Czech Republic (1M6198959201) and also by internal grant of Palacký University in Olomouc (PrF_2011_020).

Keywords: composite materials, silver nanoparticles, adsorption, active carbon, clay mineral

J. Tokarský, P. Čapková, D. Rafaja, V. Klemm, M. Valášková, J. Kukutschová, V. Tomášek, Appl. Surf. Sci 256 (2010) 2841–2848.
A. Panáček, L. Kvítek, R. Prucek, M. Kolář, R. Večeřová, N. Pizúrová, K. Sharma, T. Nevěčná, R. Zbořil, J. Phys. Chem. B 110 (2006) 16248–16253.
M. Rai, A. Yadav, A. Gade, Biotechnol. Adv. 27 (2009) 76–83.
J. Talebi, R. Halladj, S. Askari, J. Mater. Sci. 45 (2010) 3318–3324.
S. Y. Yeo, S. H. Jeong, Polym. Int. 52 (2003) 1053–1057.
Y. H. Kim, D.K. Lee, H.G. Cha, Ch. W. Kim, Y.S. Kang, J. Phys. Chem. C 111 (2007) 3629–3635.
R. Bandyopadhyaya, M. V. Sivaiah, P. A. Shankar, J. Chem. Technol. Biotechnol. 83 (2008) 1177–1180.

Coating Ni-Co-MWCNT Metal Matrix Nanocomposites by Electrodeposition

R. Karslıoğlu, M. Uysal, M. Sancar, H. Akbulut

Sakarya University Engineering Faculty Department of Metallurgical and Materials Engineering Sakarya 54187, Turkey

rkarslioglu@sakarya.edu.tr

Ni-based multi-walled carbon nanotube (MWCNT) composite coatingsat three different applied current densities were produced by electrodeposition. Ni-Co alloys and MWCNT were deposited on low carbon steel substrates. At the frist step of the depaoition of MWCNT Ni-Co matrix composğtes the MWCNT were suspended in the electrolyte by activating nanotrubes and introducing proper surfactants. Zeta potential of the susspended electolytes were measured to optinmize the amount of MWCNT depending on the activating and surfactant agents. Ni-Co metal matrix composite (MMC) coatings were prepared from a modified Watt's type electrolyte containing MWCNT particles by direct current (DC) plating method to increase the surface hardness of the electrodeposited Ni-Co. For these purposes, effect current density on the microstructural and mechan,ical proeprties were investigated. The characterization of the coatings was investigated by scanning electron microscopy (SEM). The efefct of MWCNT addition on the hardness of the resultant coatings was also measured and found a remerjkable incdfease.

Keywords: Electro co-deposition 1, nano MMC 2,MW CNT 3, Ni-Co 4

Fabrication of nano-sized SnO_2 powder for anode of Li-ion battery by sol-gel method

H. Köse[1], A. O. Aydın[1], H. Akbulut[1]

[1]Sakarya University, Turkey

hkose@sakarya.edu.tr

SnO_2 have been extensively studied because of its application as anodes for lithium ion batteries, transparent electrodes for solar cells, liquid crystal displays; antistatic coatings and gas sensors; transistors, catalyst supports; nano and ultra filtration membranes and anticorrosion coatings. Fine grain structured tin oxide is especially targeted to produce as anode active materials for lithium-ion batteries. The finer the grain sizes for the anode electrode the higher the capacity.

Many techniques have been used to prepare the tin oxide powders. Among these techniques, sol–gel process is considered as a suitable method due to its simplicity, inexpensive, high efficiency, and capability to control of stoichiometry.

In this work, sol-gel method was applied to synthesize SnO_2 nanopowders. The SnO_2 precursor solution was prepared by dissolving $SnCl_2 \cdot 2H_2O$ in absolute ethanol. The solution was stirred for 1 h, and then deionized water was added to clear solution for hydrolysis. After that, a few milliliters of diluted NH_4OH solution was gradually dropped to sol as catalyst. A yellow gel slowly formed via stirring the solution at 50 ^oC. The gel was oven-dried at 100 ^oC to remove the solvent. After drying, the powders were calcinated at 550 ^oC with a heating rate of 2^oC/min.

SEM, EDS and XRD analysis were performed to characterize nanopowders. The results indicated that the average size of SnO_2 powders is 50 nm. Consequently, these nanostructured SnO_2 powders are assessed as applicable for Li-ion batteries.

Keywords: Tin oxide, Nanopowder, Sol-gel, Li-ion battery

Prediction of change of energy embodied in materials in ECAP technology

M. Kušnerová[1], J. Valíček[1], M. Zeleňák[1], M. Harničárová[2], P. Hlaváček[1], S. Hloch[3], M. Řepka[4], V. Kuběna[1]

[1]Institute of Physics, Faculty of Mining and Geology, VSB-Technical University of Ostrava, Czech Republic

[2]Department of Manufacturing Technologies, Faculty of Manufacturing Technologies in Prešov, Slovak Republic

[3]Faculty of Manufacturing Technologies of Technical University of Košice with seat in Prešov, Slovak Republic

[4]Institute of Economics and Control Systems, VŠB-Technical University of Ostrava, Czech Republic

milena.kusnerova@vsb.cz

This paper deals with the ECAP technology in order to determine a deformation work of created samples. As a number of extrusion increases, the energy rate of extruded material reduces. With a higher number of extrusion there is a risk, that deformation pressure applied to so much hardened and homogenized material can cause equipment failure. Therefore it is necessary to predict a maximum number of extrusion for the sample of concerned material. The paper presents a new way for evaluating the results of mechanical work (embodied energy in materials) depending on the number of extrusion. According to a course of this regression function it is possible to determine the maximum number of extrusion without material fracture.

Keywords: Equal channel angular dressing 1, Intense plastic deformation 2, Mechanical work 3, Prediction of change 4

Preparation of multilayer TiO_2 films

J. Lang[1], V. Matějka[1], J. Vontorová[1]

[1]Vysoká škola báňská - Technical university of Ostrava, Czech Republic

vlastimil.matejka@vsb.cz

The utilization of the photocatalytic materials fall to the different areas of industry: energetic (converting of solar energy, production of batteries), ecology (water and air purification), medicine (antibacterial coatings) in building industry (self cleaning windows) etc.. In principle the nanosizes of photocatalytic materials can be achieved by two basic approaches, the first one includes preparation of nanopowders, the second one is based on the preparation of thin layers, which thickness falls to the sizes in the order of the tenth nanometers. The preparation of photocatalysts in the form of thin films brings number of advantages like better manipulation and diminishing of the environmental problem arising with nanosize of particles. Titanium dioxide represents the most studied photocatalyst in the form of nanopowder as well as in the form of thin layer.

In the present work we focused on the preparation of TiO_2 thin layer. TiO_2 sol was prepared by the sol-gel method using titanium tetraisopropoxide, the prepared sol was then deposited on the soda-lime glass sheets using dip-coating method. The obtained samples were heat treated at 400 and 500 °C with the aim to study the resistance of the layers against forming of the cracks invoked by the thermal treatment. The chemical composition of prepared layers was studied using optical emission glow discharge spectroscopy (OE GDS), the morphology of the layers' surface was studied using atomic force microscopy (AFM).

Keywords: Titanium dioxide, sol-gel, dip coating, OE GDS

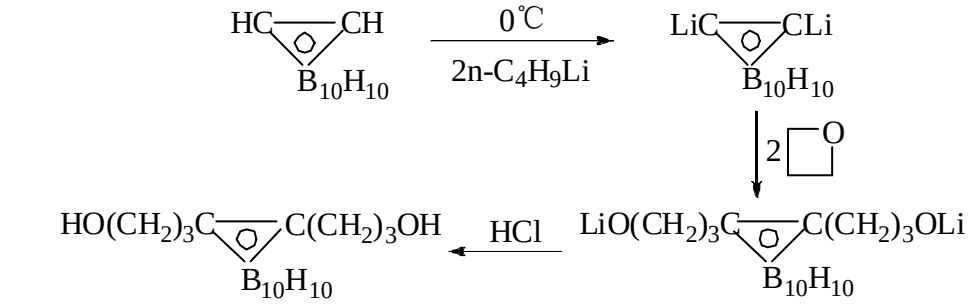
Synthesis and characterization of carborane polyesters

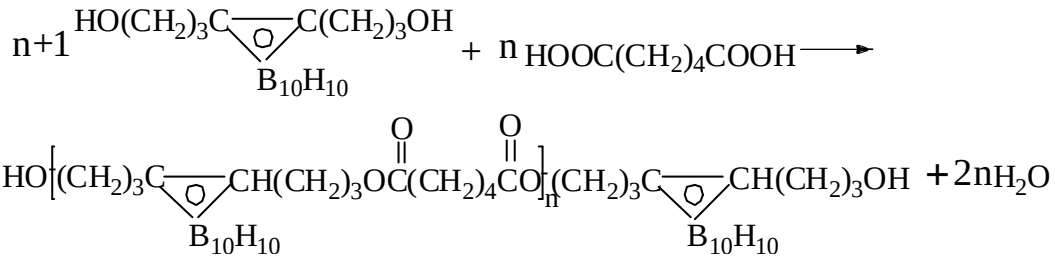
Xiaoning Zhao, Shicheng Qi, Yafei Lu

Key Laboratory of carbon fiber and functional polymers, Ministry of Education, Beijing University of Chemical Technology, Beijing 100029, China

ylu623@gmail.com

Carborane polymers are a kind of organic-inorganic hybrid nanocomposites that the nano-scaled carborane was linked to polymer main chains with superior thermal stability and mechanical properties. In this research, the 1,2-carborane (or 1,7-carborane) based polyesters were synthesized by two steps: (1) preparation of 1,2-bis(hydroxypropyl)-carborane (or 1,7-bis(hydroxypropyl)-carborane) and (2) reactions of 1,2-bis(hydroxypropyl)-carborane (or 1,7-bis(hydroxypropyl)-carborane) with adipic acid. The structure of both 1,2-carborane and 1,7-carborane based polyesters was characterized using ^{1}H-NMR, FTIR and GPC. The synthetic route of the carborane polyesters is shown as follow.





Keywords: Carborane polyesters, Synthesis, Characterization

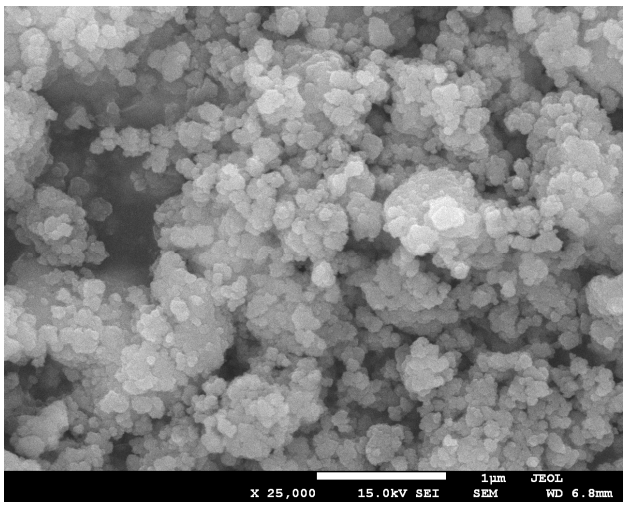
Simple methods of synthesis ZnS nanoparticles

K. Luberda-Durnaś[1], V. Matějka[2], K. Mamulová-Kutláková[2], W. Łasocha[1, 3]

[1]Insitute of Catalysis & Surface Chemistry PAS, Poland, [2] Nanotechnology Centre, Technical University of Ostrava, Czech Republic, [3]Faculty of Chemistry, Jagiellonian University, Poland

ldkasia@gmail.com

The main goal of our work was to develop a simple, cheap and fast method for the synthesis of ZnS nanoparticles. The way of the synthesis proposed by us is based on the use of microwave irradiation, $ZnSO_4$ as a source of zinc and i) thioacetamide, or ii) thiourea as possible sources of sulfur. Based on the experimental results we observed that five minutes microwave treatment is sufficient to create ZnS nanoparticles in both cases.
Obtained samples where characterized using X-ray diffraction (XRD), UV-vis spectroscopy and scanning electron microscopy (SEM). The samples exhibit fotocatalytic activity as revealed by photodegradation of acid orange 7. Base on X-ray diffraction the estimated grain size of ZnS nanoparticles is about 20-30 nm. That confirms also SEM measurement.



Keywords: ZnS nanoparticles, X-ray diffractions, UV-vis spectroscopy, SEM

Review: Enhancement of the Interfacial Strength between Carbon Fiber and Matrix in Composites by Growing Carbon Nanotubes on Carbon Fiber

M. Najari[1], M. Kasiri[1], H. Tiznobaik[2], S. Niakan[2]

[1]Islamic Azad University, Najafabad Branch, Iran; [2]Iran University of Science and Technology, Iran

saeed_n1361@yahoo.com

The mechanical behavior of composites depends, not only, on the properties of the constituent materials, but on the characteristics of the interface between the constituents as well. In continuous fiber-reinforced composites, the tensile load is transferred from the matrix to the fiber through shear stress at the interface, causing the fiber to elongate. Good interfacial adhesion provides composites with structural integrity and efficient load transfer from fiber to matrix.

Previously, the interfacial strength can be improved through various methods, the most common being through improving the chemical adhesion of the fiber with the matrix, removing the weak outer layer of the fiber produced during fiber fabrication, or by producing an interphase region through the use of a thin polymer sizing [1,2]. However, these methods can also introduce mechanical defects onto the fiber and create bulky or impenetrable additional phases around the fiber.
In contrast, directly growing nanomaterials such as carbon nanotubes (CNTs), having high aspect ratio and desirable thermal, electrical, and mechanical properties, onto the reinforcing fiber can modify the interfacial properties. Generally, CNTs have been grown by many methods including electric arc discharge [3], laser ablation [4], and chemical vapor deposition (CVD) [5, 6]. Of these, only CVD process provides direct control of location, alignment, morphology, and packing density of CNTs while providing strong bonds with a substrate [7].

In an effort to investigate the effects of growing nanotubes on carbon fibers, multi–walled carbon nanotubes (MWCNT) have been grown on T650 carbon fibers both in radialy (Fig. 1a) and randomly (Fig. 1b) aligned morphology and the products have been compared with sized and unsized T650 carbon fibers from two mechanical property aspects (interfacial shear strength and tensile strength).

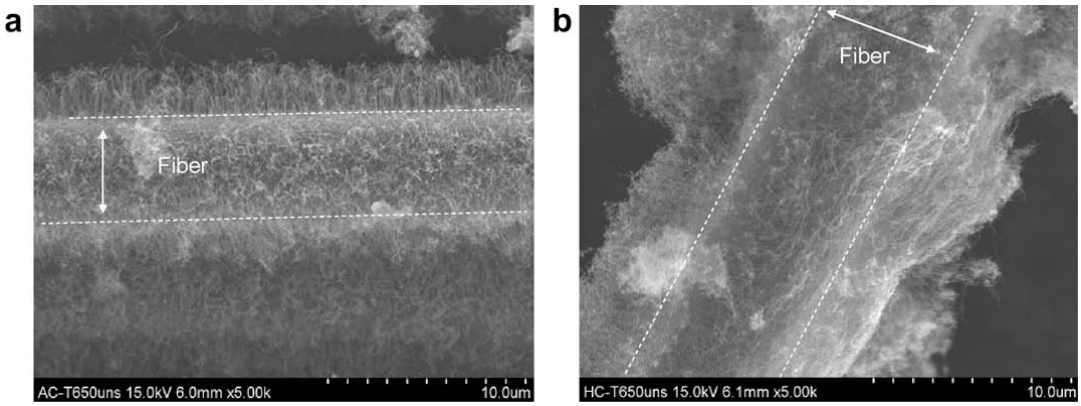


Figure 1. High-resolution SEM images of (a) carbon fiber with radially aligned MWCNTs and (b) carbon fiber with randomly oriented MWCNTs [8].

Single-fiber fragmentation tests have indicated that commercially sized fibers have the highest interfacial shear strength, while unsized fibers have the lowest. Randomly oriented MWCNT and aligned MWCNT coated fibers have demonstrated a 71% and 11% increase in interfacial shear strength over untreated, unsized fibers. This increase can be attributed to an increase in, both, the adhesion of the matrix to the fiber and the interphase shear yield strength due to the presence of the nanotubes. The increase in interphase shear strength observed for the randomly oriented MWCNT case is most likely due to the alignment of MWCNTs with the principal tensile stress direction in the interphase [8].
Tensile tests have revealed that CVD processing significantly reduces the ultimate tensile strength of the fiber by an average of 37% in the case of the radially aligned MWCNTs and by 30% in the case of randomly oriented MWCNTs. Reductions in mechanical properties of the fiber due to processing can be attributed to the addition of surface flaws on the fiber through thermal degradation and surface oxidation [8]. Decrease in processing temperatures as well as elimination of oxygen within the processing chamber have been shown to decrease this degradation in mechanical properties [9]. To reduce the degradation of tensile strength caused through CVD process, a desired growth condition, which depends on growth temperature, flow rate of the carbon/catalyst mixture and growth time, has been found [10].

To conclude, despite the fact that a slight degradation in tensile strength of the fiber caused through CVD process has been observed, the strength role of carbon fiber in composite has been increased due to significant enhancement in shear strength.

Keywords: Carbon Nanotube, Carbon Fiber, Shear Strength, Tensile Strength

References:

[1] Drzal LT, Rich MT, Lloyd PF. Adhesion of graphite fibers to epoxy matrices: I. The role of fiber surface treatment. J Adhes 1982;16:1–30.
[2] Drzal LT, Rich MT, Koenig MF, Lloyd PF. Adhesion of graphite fibers to epoxy matrices: II. The effect of fiber finish. J Adhes 1983;16:133–52.
[3] Ebbesen TW, Ajayan PM. Large-scale synthesis of carbon nanotubes. Nature 1992;358(6383):220–2.
[4] Thess A, Lee R, Nikolaev P, Dai HJ, Petit P, Robert J, et al. Crystalline ropes of metallic carbon nanotubes. Science 1996;273(5274):483–7.
[5] Joseyacaman M, Mikiyoshida M, Rendon L, Santiesteban JG. Catalytic growth of carbon microtubules with fullerene structure. Appl Phys Lett 1993;62(6):657–9.
[6] Endo M, Takeuchi K, Kobori K, Takahashi K, Kroto HW, Sarkar A, et al. Pyrolytic carbon nanotubes from vapor-grown carbon-fibers. Carbon 1995;33(7): 873–81.
[7] Dai L, Patil A, Gong X, Guo Z, Liu L, Liu Y, et al. Aligned nanotubes. Chem Phys Chem 2003;4:1150–69.
[8] Sager R, Klein P, Lagoudas D, Zhang Q, Liu J, Dai L, Baur J. Effect of carbon nanotubes on the interfacial shear strength of T650 carbon fiber in an epoxy matrix. Compos Sci Technol 2009;69(7-8):898-04.
[9] Zhang Q, Liu J, Sager R, Dai L, Baur J. Carbon nanotubes on carbon fibers: influence of growth conditions on fiber tensile properties. In: SAMPE fall technical conference and exhibition, SAMPE, October 29–November 1; 2007.
[10] Zhang Q, Liu J, Sager R, Dai L, Baur J. Hierarchical composites of carbon nanotubes on carbon fiber: Influence of growth condition on fiber tensile properties. Compos Sci Technol 2009;69:594–601.

Effect of different carbon sources on a particle size of $LiFePO_4$/C nanocomposite cathode active material for lithium-ion batteries

A. Örnek[1], E. Bulut[2], M. Özacar[2]

[1]Institute of Sciences and Technology, Sakarya University, 54187 Sakarya, Turkiye

[2]Department of Chemistry, Art and Science Faculty, Sakarya University, 54187 Sakarya, Turkiye

mozacar@sakarya.edu.tr

Lithium iron phosphate ($LiFePO_4$) belonging to the family of phosphate olivines is a promising cathode material alternative for lithium-ion batteries. It has become of great interest because of its low price, high specific energy density, and excellent safety, especially thermal stability at rather high temperatures providing safety security to high power or large capacity batteries. The main obstacle for this material is poor rate capability because of low electronic conductivity and low lithium-ion diffusivity through $LiFePO_4$–$FePO_4$ interfaces.

Carbon coating is the common and cheaply way to overcome the limited rate capacity because the dispersed carbon provides pathways for electron transferring, resulting in improvement of the conductivity and electrochemical properties. Various carbon sources have been reported for the $LiFePO_4$/C composite, e.g., hydroxyethyl cellulose, sugar, resorcinol-formaldehyde gel, polypropylene, and carbon black. Based on these results, it can be contended that the carbon resource and synthesis conditions have a very obvious influence on the particle size of $LiFePO_4$ nanocomposite. In this study, investigation of particle size $LiFePO_4$/C using three different carboxylic acids (tartaric, citric and stearic acid) as carbon sources for lithium-ion batteries. $LiFePO_4$/C was synthesized by sol-gel method and characterized XRD and SEM. Characterized nanoparticles are presented in Figs. 1 and 2.

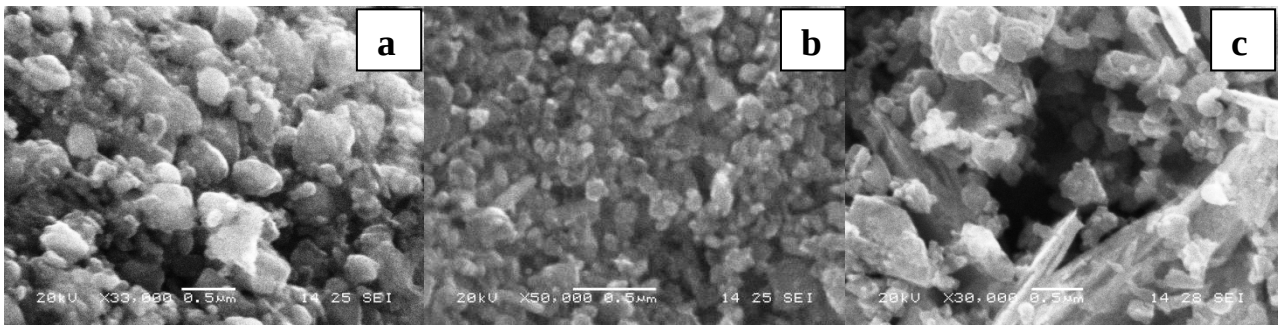


Figure 1. SEM images of the $LiFePO_4$ samples coated with various carboxylic acid (a. tartaric acid, b. stearic acid and c. sitric acid) as carbon source

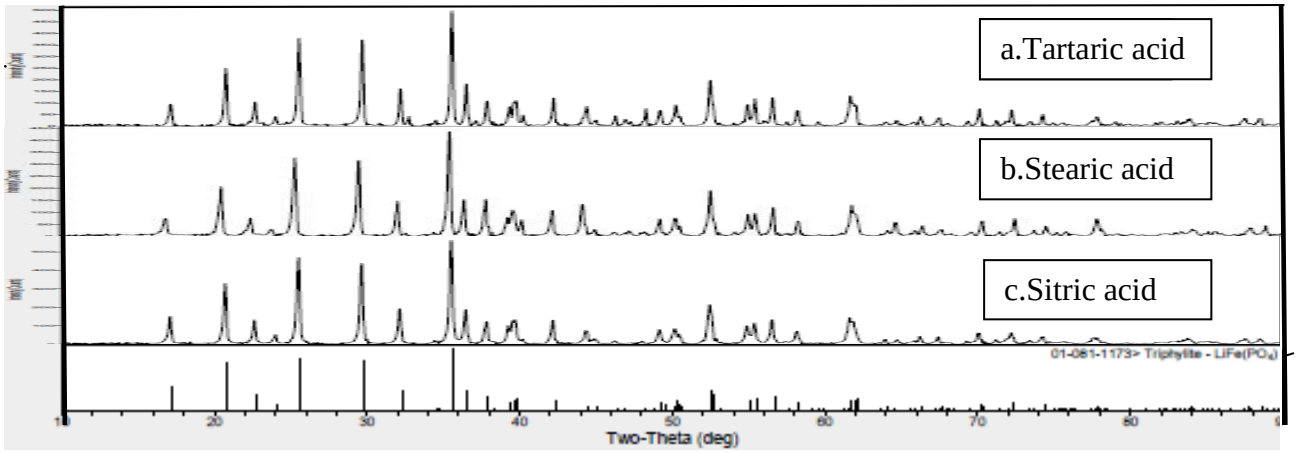


Figure 2. XRD patterns of the $LiFePO_4$ samples coated with various carboxylic acid as a carbon source

Keywords: Lithium iron phosphate, Carbon coating, Nanoparticles, Lithium-ion batteries

Effect of temperature on synthesis nano $LiFePO_4$/C cathode active material for lithium-ion batteries

A. Örnek[1], E. Bulut[2], M. Özacar[2]

[1]Institute of Sciences and Technology, Sakarya University, 54187 Sakarya, Turkiye

[2]Department of Chemistry, Art and Science Faculty, Sakarya University, 54187 Sakarya, Turkiye

mozacar@sakarya.edu.tr

The lithium iron phosphate ($LiFePO_4$) has attracted much attention as a potential cathode material for rechargeable lithium batteries. $LiFePO_4$ has offered several unique benefits when compared to conventional cathode materials such as $LiCoO_2$, $LiNiO_2$ and $LiMn_2O_4$. $LiFePO_4$ exhibits a flat discharge profile, good thermal stability, high theoretical capacity (172 mAh/g) and environmentally friendly properties. However, a significant problem with the use of this material lies in its conductivity. This is due to the one-dimensional diffusion of Li^+ ions along the b axis formed by edge-shared LiO_6 octahedra, compounded by poor electronic and ionic conductivity from the $LiFePO_4$–$FePO_4$ interface. Two methods have been employed to circumvent the poor conductivity issue. The first is to reduce the grain size of the cathode particles, which would shorten the diffusion path length for both electrons and Li^+ ions. The second is the use of surface-modified $LiFePO_4$ with a conductive matrix made of carbon, copper and silver, or doping with some guest species.

In this study, in order to effect of calcination temperature on $LiFePO_4$/C grain size, three samples were synthesized. The samples of $LiFePO_4$/C were synthesized with 600°C, 650°C and 700°C temperatures by sol-gel method. All samples were characterized by XRD and SEM as shown in Figs. 1 and 2, respectively.

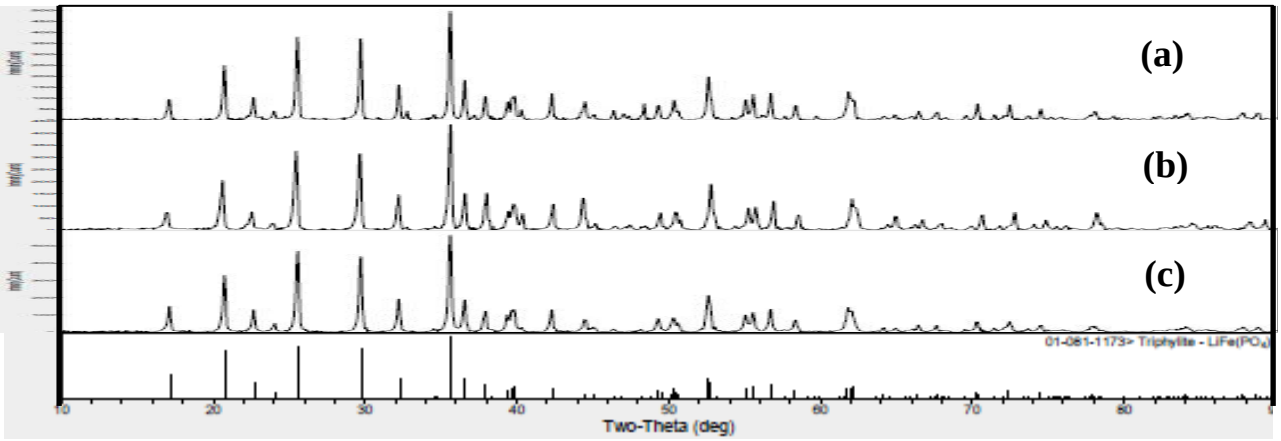


Fig. 1. XRD patterns of the $LiFePO_4$ samples with different synthesis temperature (a. 600°C, b. 650°C and c. 700°C)

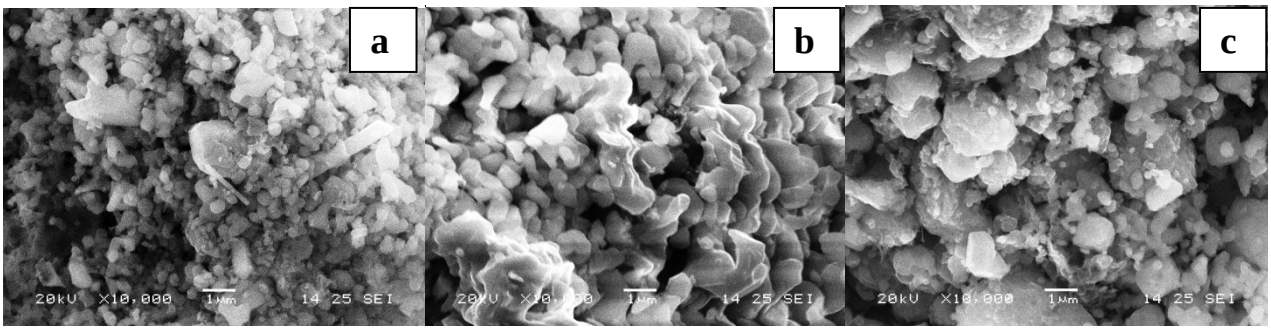


Fig. 2. SEM images of the $LiFePO_4$/C samples with different synthesis temperature (a. 600^0C, b. 650^0C and c. 700^oC)

Keywords: Lithium iron phosphate, Sol-gel method, Particle size, Lithium-ion batteries

Effect of the Temperature on SnO_2 Thin Films Deposited by APCVD

F. Dogan[1], M. Özacar[2], H. Akbulut[3], M. O. Guler[3]

[1]Institute of Sciences and Technology, Sakarya University, 54187 Sakarya, Turkiye

[2]Department of Chemistry, Art and Science Faculty, Sakarya University, 54187 Sakarya, Turkiye

[3]Department of Metallurgy and Materials, Engineering Faculty, Sakarya University, 54187, Sakarya Turkiye

mozacar@sakarya.edu.tr

Li-ion batteries are now commercially available products for many applications. Some metals, alloys and metal oxides have been recently proposed as possible negative electrodes in Li-ion batteries. Metal oxides and also their composites have been investigated by various researchers. Amongst these oxides, thin oxide (SnO_2) is known to be chemical most stable in ambient atmosphere. Tin oxide is a wide band gap (~4eV) and indirect band gap (of about 2.6eV) nonstoichiometric semiconductor. It has a low n-type resistivity (10^{-3} Ωcm) and high transparency (%90) in the visible region.

At low deposition temperatures the resistivity of the grown layers is high, but it decrease with increasing deposition temperature to a minimum at about 400-500 ^oC and then increase again. The resistivity decreases with increasing deposition temperature because the grain size and the crystallinity of the layer increase. After a minimum value at about 450 ^oC the resistivity of the layer increases as a result of decreasing carrier concentration. This is caused by the elimination of oxygen vacancies or by a lower concentration of active chlorine ions at high temperatures.

In this study, it was investigated effect of the temperature between 400-550 ^oC on SnO_2 thin films deposited by APCVD. As deposition temperature was increased, grain size was increased. At higher temperature, it was showed in larger grains and better crystallinity resulting in a higher carrier concentration, a higher mobility, and thus a higher conductivity. Below 450 ^oC the layers showed a preferential orientation of (101) planes, while above 450 ^oC the (200) planes had the highest XRD intensity. The exact type of texture is not only dependent on the thickness, but also on the temperature of the substrate. The effect of temperature on the SnO_2 thin films is shown in Figure 1.

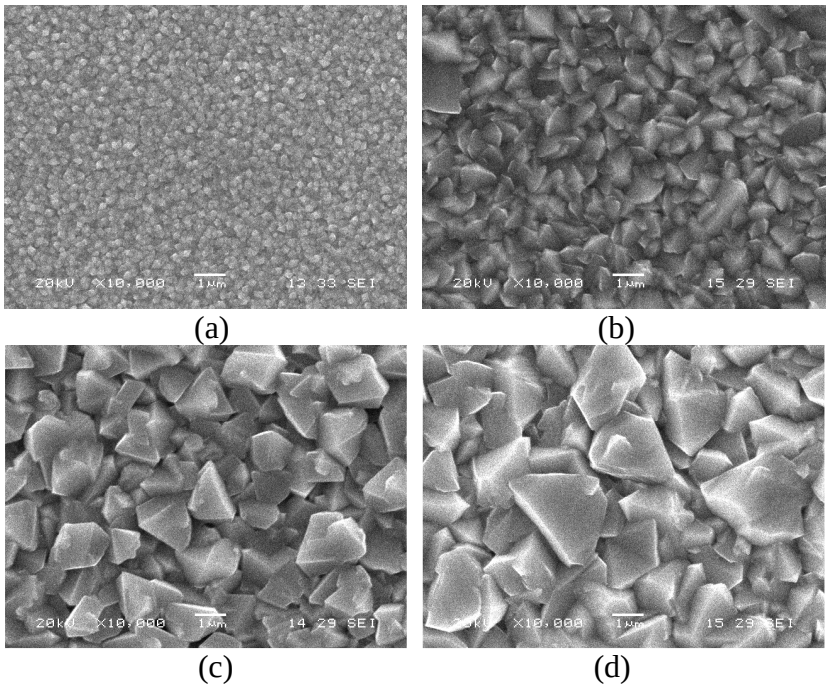


Figure 1. SEM characterization of SnO_2 thin films at (a) 400, (b) 450, (c) 500, (d) 550 ^{o}C

Keywords: Tin oxide, Li-ion battery, SEM, CVD

Production of Nanoparticles of Polypyrrole by Ball Milling

A. M. Rehan Abbasi[1], V. K. Baheti[1], Jiri Militky[1]

[1]Dept. of Textile Materials, Technical University of Liberec, Czech Republic

rehan_abbaci@hotmail.com

For the first time nanoparticles of polypyrrole (PPy) were produced by high energy ball milling. Polypyrrole was chemically synthesized by polymerization of pyrrole and p-toluene sulfonic acid (PTSA) as dopant in the presence of oxidant FeCl3 and Polyethylene glycol (PEG 8000) at -5°C for 48 hours. High energy milling was carried out at 850 rpm in the completely dry atmosphere with the balls of 10mm in diameter. Nanoparticles were collected on to nano-filter and then characterized by *SEM* (see Fig. 1) and Malvern Instruments' *Zetasizer* (Nano series); it was found that the size of the particles of PPy is a decreasing function of time of milling (see Fig. 2).

Electrical conductivity was measured immediately after dispersing PPy nanoparticles into distilled water and it was observed that PPy nanoparticles are much more conductive than nanoparticles of PPy which was polymerized by keeping all the conditions same but without PEG 8000. This method of measuring conductivity was chosen just for comparison purpose.

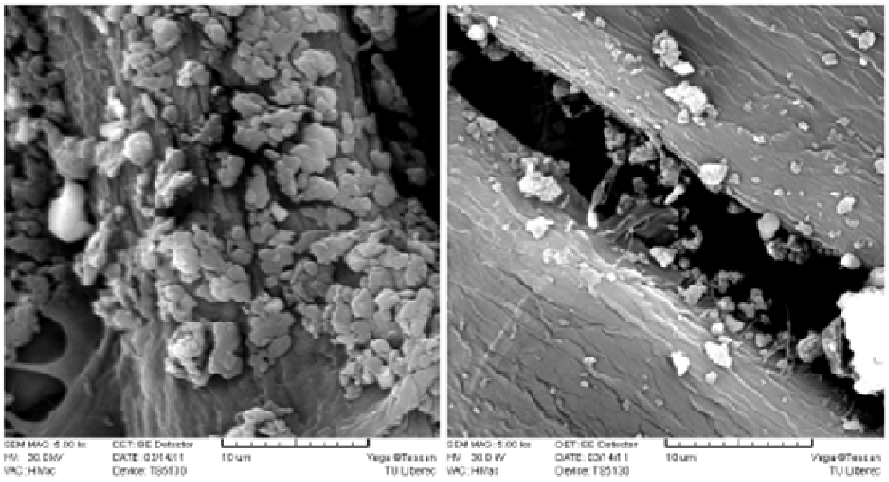
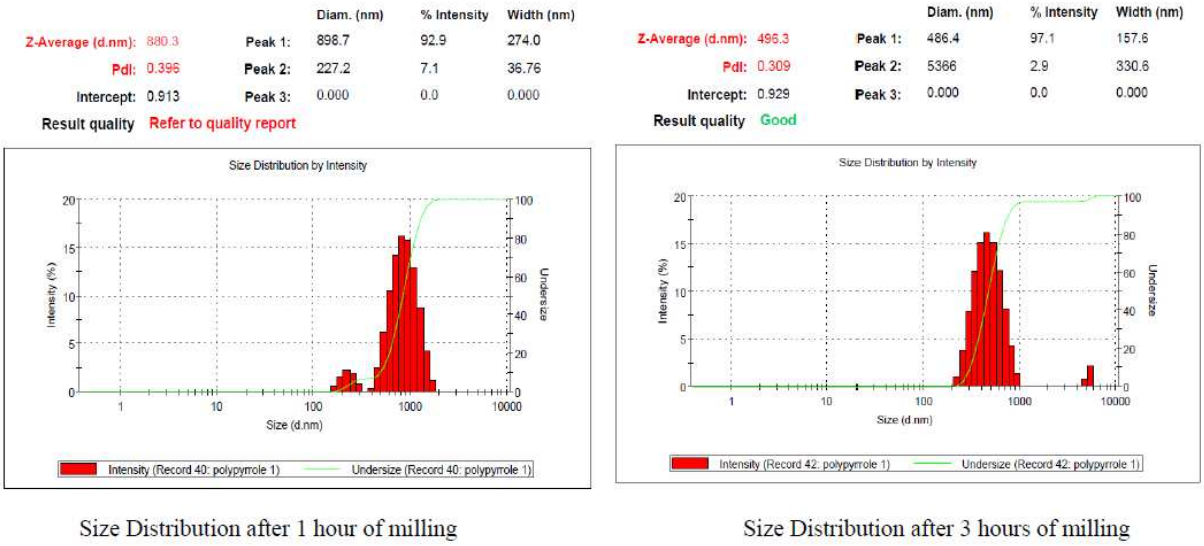


Fig 1. Changes of PPy particles shape and form after milling

PPy nanoparticles were deposited on Polyurethane (PU) films to prepare nanocomposite electrical conductive films. Influence of presence of PEG during polymerization on size and electrical conductivity of PPy nanoparticles were also investigated. Simple synthesis of polypyrrole and developing nanoparticles via high energy ball milling offer a potential in using as conducting fillers in making conducting polymer nanocomposites. These nanocomposites can be used as a sensors (composed of thin conductive layers) and EMI shields on textile substrate.



Size Distribution after 1 hour of milling

Size Distribution after 3 hours of milling

Fig. 2 Changes of PPy particles size distribution after milling

Keywords: Polypyrrole Nanoparticles, High Energy ball milling, Flexible conductive, Nanocomposites

Preparation of organoclays with antibacterial properties

M. Samlíková, S. Holešová

Nanotechnology centre, VŠB – Technical University of Ostrava, Czech Republic

magda.samlikova.st@vsb.cz

The development of clay materials with the ability to inhibit bacterial growth has been of great interest in recent years. The most commonly used are the planar hydrous phyllosilicates clay minerals montmorillonite and vermiculite. Their layered structure shows cation exchange reaction ability occuring mainly within the interlayer space. The exchangeable interlayer hydrated cations Na^{+}, K^{+}, Mg^{2+}, Ca^{2+} of original montmorillonite and vermiculite can be replaced by antibacterial anorganic and/or organics ions and give rise to resulting antibacterial material.

The aim of this work was to prepare organoclay filling agent with antibacterial properties for its potential use into nanocomposite materials.

The organoclays antibacterial compounds were synthesized from montmorillonite, vermiculite and their Zn^{2+}, Cu^{2+} and Ag^{+} forms using chlorhexidine acetate.

Chlorhexidine acetate is a bisbiguanide antiseptic and disinfectant which is bactericidal or bacteriostatic against a wide range of Gram-positive and Gram-negative bacteria.

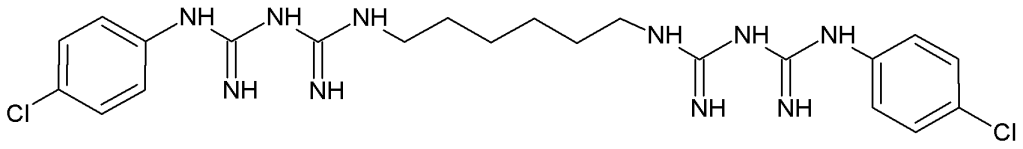


Figure 1. Structure of chlorhexidine

In a first step montmorillonite and vermiculite were converted into Zn^{2+}, Cu^{2+} and Ag^{+} form using cation exchange reaction with 1.0 M aqueous solution. Next step was intercalation chlorhexidine into the structure of prepared monoionic form of clays.

The structures of resulting samples were characterized by X-ray diffraction and infrared spectroscopy. The antibacterial activity of prepared organoclays against *Entrerococus faecalis*, *Escherichia coli* and *Pseudomonas aeruginosa* were evaluated by finding minimum inhibitory concentration.
Antibacterial tests showed that the prepared organovermiculites have a better ability to inhibit bacterial growth than organomontmorillonite. It was concluded that discrepancy arised probably due to different means of bounding of molecule chlorhexidine at montmorillonite and vermiculite.

Keywords: organoclays, chlorhexidine acetate, intercalation, antibacterial activity

Study of Silver Nanoparticles Stabilization Performed by Gelatin

M. Sivera, L. Kvitek, R. Prucek, A. Panacek

Regional Centre of Advanced Technologies and Materials, Department of Physical Chemistry, Palacky University, Olomouc, Czech Republic

MartinSivera@seznam.cz

Nanomaterials are more and more becoming part of the human life. This is particularly true for silver nanoparticles, which are for antibacterial properties applied in a number of textile, cosmetics and medical products [1]. The high antibacterial activity of silver nanoparticles due to growing bacterial resistance to penicillin antibiotics accelerates research of their application in specific areas such as medical treatment for burns, antibacterial development of surgical instruments, dental materials and prosthetic devices [2]. Silver nanoparticles are also applied in the development of highly sensitive sensors based on their optical properties, typically in the surface enhanced Raman spectroscopy (SERS), which provides highly sensitive analytical technique [3,4].

The aim of this paper was study the stabilization of silver nanoparticles prepared via modified Tollens process and stabilized by gelatin. The prepared nanoparticles were characterized by measuring their size by dynamic light scattering (DLS), UV–Vis spectrometry and transmission electron microscopy (TEM). For experiments were used silver nanoparticles of size range from 25 to 30 nm. Stabilization of the dispersion of the particles was performed with gelatin (concentration in the dispersion was 0.01% w/w). TEM image (see Figure 1) demonstrates morphology and polydisperzity of stabilized silver nanoparticles.

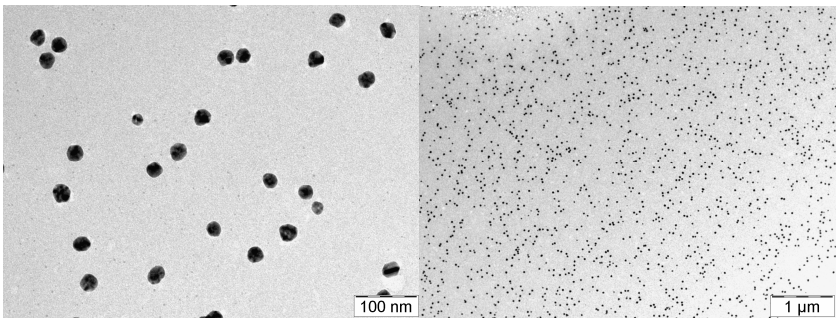


Figure 1: TEM image of the silver NPs prepared via the modified Tollens process and modified by gelatin.

The study of aggregation stability silver nanoparticles was performed by determining the critical coagulation concentration (CCC) for different types of electrolytes – $NaNO_3$, $Ca(NO_3)_2$ and poly(diallyldimethylammonium) chloride (PDDA). By comparing the observed values for the CCC for unmodified and modified silver nanoparticles was demonstrated robust stabilizing activity of gelatin. In all cases there was a significant rise of the values of the CCC. In case of PDDA value of CCC increased from $2.47 \cdot 10^{-5}$ % w/w for unmodified silver nanoparticles to value of $3.47 \cdot 10^{-4}$ % w/w for the modified nanoparticles (see Figure 2). For $NaNO_3$ CCC increases from a value from 0.08 $mol \cdot L^{-1}$ to 3.31 $mol \cdot L^{-1}$ and in case of $Ca(NO_3)_2$ value CCC increases from 0.50 $mmol \cdot L^{-1}$ to 11.30 $mmol \cdot L^{-1}$.

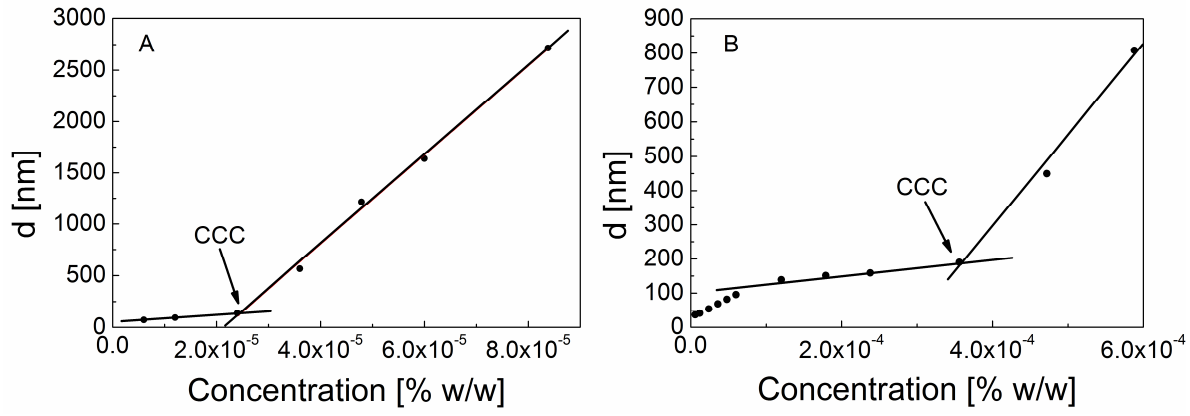


Figure 2: Evaluation of critical coagulation concentration from the dependence of the size of silver nanoparticles on the PDDA concentration (unmodified nanoparticles part A and modified nanoparticles part B)

This presented work demonstrates that gelatin is for silver nanoparticles an excellent modifier. Such modified nanoparticles may be due to the nature of the gelatin used in medical and cosmetic products.

This work has been supported by the Operational Program Research and Development for Innovations - European Social Fund (CZ.1.05/2.1.00/03.0058), by the Ministry of Education of the Czech Republic (1M6198959201) and by Internal Grant of Palacky University Olomouc (PrF_2011_020).

[1] Kvítek, L.; Panáček, A.; et al. *Journal of Physical Chemistry C* **2008**, *112*, 5825–5834.
[2] Chaloupka, K.; Malam, Y.; Seifalian, M. A. *Trends in Biotechnology* **2010**, *28*, 580–588.
[3] Kvítek, L.; Prucek, R.; et al. *Journal of Material Chemistry* **2005**, *15*, 1099–1105.
[4] Anker, J. N.; Lyandres, O.; Shah, N. C.; et al. Nature Materials **2008**, *7*, 442–453.

Keywords: silver nanoparticles, aggregation, stabilization, gelatin

Modification of hydrogel using collagen and nanostructured clay

S. Šustek[1], L. Svoboda[1], J. Zelenka[2], K. Zetková[2], J. Kára[1]

[1]University of Pardubice, Czech Republic; [2]Synpo a.s., Czech Republic

stanislav.sustek@student.upce.cz

Recently various eco-friendly products and technologies are developed in the world to replace environment threatening ones. In the field of superabsorbent materials the efforts are directed to substitute synthetic polymers for natural materials or a combination of these two groups of substances. Such products are used for example in agriculture, horticulture and forestry to improve soil water retention and nutrient utilization by plants. The presented work is focused on modification of the commercially available polyacrylate hydrogel Agrisorb (Stockhausen GmbH & Co.KG, Germany) with a mixture of collagen and nanostructured clay with the aim of conservation or increasing swelling capacity of the product.

The clay was used in three forms: as a powder, aqueous suspension and in jelly-consistence. Encapsulation was carried out as follows. Collagen was added to the water under stirring and heating (60°C), subsequently was added the clay and finally, after perfect homogenization, was added 1g of swollen hydrogel. Then the beaker was let to stand 10 minutes and solid phase filtered on a Buchner funnel. The filter cake was transferred into a dish and dried. Prepared samples were examined under an optical microscope and compared with non-encapsulate gel. The samples were also tested for swelling in saturated solution of $(NH_4)_2SO_4$ and KNO_3. Coated gel was placed into the tea-bag which was then immersed in selected solution of salt. The bags were in given time periods removed from the solution and left to drain freely, then were weighed and immersed again. Their weight was recorded in dependence on the swelling time and compared to the values for uncoated gel. The best swelling ratio had a value 24,69 grams of $(NH_4)_2SO_4$ solution per one g of the original gel, in the case of KNO_3 it was reached the value 35,86g/g. Both these values are higher than for non-encapsulated gel (17,57g/g for $(NH_4)_2SO_4$), 28,02g/g for KNO_3). After swelling samples were dried in an oven at 100°C. Rate of ammonium sulfate or potassium nitrate release from modified hydrogel was measured using conductivity test during immersion samples in distilled water. It was proved that modified hydrogel saturated with $(NH_4)_2SO_4$ solution released ions slower than non-encapsulated gel. In the case of KNO_3 solution significant differences were not observed.

In this work compatibility of collagen with nanostructure clay was tested and the possibility of using this system to modification the polyacrylate hydrogel was assessed. Some of the prepared hydrogels reached higher values of swelling than the original unmodified material. It makes possible to decrease content of polyacrylate component in the composite hydrogel and to reduce content of non-biodegradable residues in soil after using hydrogel as water and nutrient regulator in agricultural applications.

Keywords: nanostructure clay, collagen, hydrogel

Impact of mechanical and chemical treatments on talc structure

J. Zdrálková, M. Valášková

Nanotechnology Centre, VŠB-Technical University of Ostrava, 17. listopadu 15, 708 33 Ostrava-Poruba, Czech Republic

jana.zdralkova.st@vsb.cz

Talc ($Mg_3Si_4O_{10}(OH)_2$) is a common clay mineral belonging to the planar hydrous phyllosilicates. The layered structure with type of 2:1 layers is electrically neutral. Pulverized talc has wide industrial applications as high-quality filler in rubber, textile, paints, plastic, composites; as a loading agent for paper of all kinds; in the agriculture as a carrier of insecticidal and pesticidal dust. Among its many uses, talc is also an important raw material for magnesium ceramics (steatites, cordierite, enstatite and forsterite products).

The purpose of the present study was modification of natural talc for its further use into nanocomposites. Therefore combination of chemical acid activation and mechanical planetary ball milling was utilized to increase the specific surface area (SSA) and required reactivity of natural talc. Talc is very resistant to acid attack. Disorder of the layered structure caused by grinding may promote its leaching behavior.

The effects of dry grinding and hydrochloric acid treatment on the structural characteristic of talc from Egypt were studied using the X-ray powder diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), Scanning electron microscopy (SEM), thermogravimetric analysis (TGA) and the SSA measurement.

The powder XRD pattern of natural talc showed the admixture (~ 5 wt.%) of other clay minerals kaolinite (K) and dolomite (D). Grinding and following acid treatment caused increasing of the surface area from 10,1 cm^2/g to the 11,8 m^2/g. When the opposite procedure was employed (the former acid activation and the later grinding in ball mill) the highest value of the SSA of 26,2 m^2/g was measured.

Prepared samples were further tested for intercalation ability. For this purpose, the urea as intercalation guest species was chosen.

Keywords: talc, hydrochloric acid, intercalation, structural change

Quartz/TiO_2 composite and its acute aquatic toxicity

K. Gröplová, S. Rebilasová, P. Peikertová, V. Matějka, J. Kukutschová

Nanotechnology Centre, VŠB – TU Ostrava, Czech Republic

kamila.groplova@vsb.cz

Based on the literature review it can be found that bare nanoparticles of titanium dioxide (TiO_2) can have a negative effect on the living environment. If the TiO_2 nanoparticles are deposited onto the surface of the suitable type of carrier the new composite material is obtained, and the environmental hazard caused by the nanosize of TiO_2 is diminished. So obtained nanocomposites show even higher photodegradation activity against selected compounds in comparison to bare TiO_2 nanoparticles.

The present work deals with determination of photocatalytic activity and aquatic toxicity of SiO_2/TiO_2 composites. The composites with different content of TiO_2 (10 wt% and 50 wt%) were prepared and thermally treated at selected conditions (dried at 105 °C or calcined at 600 °C). The composites were studied using X-ray fluorescence spectroscopy (XRFS), X-ray powder diffraction (XRD), scanning electron microscopy (SEM), Diffuse reflectance spectroscopy in UV-VIS, Fourier transform infrared spectroscopy (FTIR), Raman microscopy.

The method based on the discoloration of aqueous solution containing Acid orange 7 was used for the testing of photodegradation activity of prepared SiO_2/TiO_2 composites. Acute aquatic toxicity testing of SiO_2/TiO_2 composite was performed according to the Czech Standard EN ISO 8692: Water quality – Test for inhibition of growth of freshwater green algae organism *Desmodesmus subspicatus*.

The study was supported by projects SP/201186, MSM 6198910016.

Keywords: nanocomposite, toxicity, Quartz/TiO_2, green algae

Chemical and physical properties of salivary gland stones

E. Gryčová[1], J. Seidlerová[1], P. Štrympl[2]

[1]VŠB – TU, Ostrava, [2]FN s poliklinikou, Ostrava

eva.grycova@vsb.cz

Salivary stones are consequence of illness, called Sialolithiasis and affects a large salivary glands – sialolith. The stones inside the salivary gland or salivary duct are formed during the Sialolithiasis and block the saliva drain. The exact cause of the formation of sialoliths is unknown. Patients suffering from this disease feel a lot of pain. The pain can be sudden and intense just after starting a meal. Commonly, salivary stones measure from 1 mm to less than 1 cm and rarely they measure more than 1,5 cm. Treatment is often performed by surgical removing. Thanks to collaboration with the Department Otorhinolaryngology of Teaching hospital in Ostrava – Poruba were gained 30 pieces of sialoliths until today.

Several instrumental methods were used for detailed description of the salivary stones. Their choice was influenced by a sample size and a sample preparation method. With regard to previous statement and with effort to obtain as much information as it was possible, we used the following methods: infrared spectroscopy, electron microscopy, X-ray diffraction and thermal analysis.

The Infrared spectroscopy showed that nucleus of all the studied sialoliths consists of carbonate apatite. This fact was also confirmed by a X-ray powder diffraction. Furthermore, X-ray diffraction analysis clarified the presence of compounds such as Calcium Hydrogen Phosphate Hydroxide, Hydroxylapatite, Calcium Sodium Magnesium Phosphate (Fig. 1). Thermal analysis of select samples determined, that particular samples differed in content ratio of Calcium Carbonate and Magnesium Carbonate.

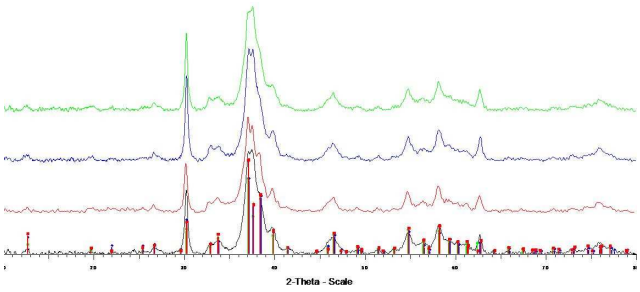


Fig. 1.: Diffractogram of sialolithes

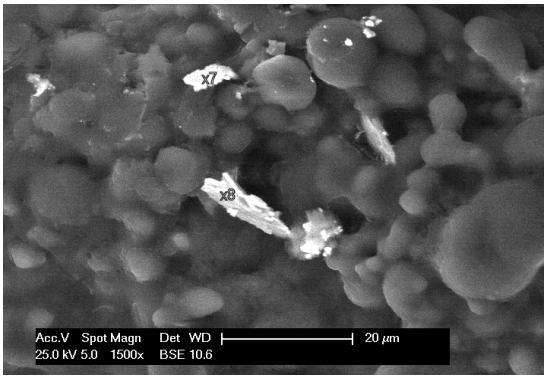


Fig. 2.: Surface of sialolith with particles of Cu

Electron microscopy allowed a research of samples surface and it also enabled a point and surface analysis. Photographs showed the diversity of surface, which is mostly smooth and divided by numerous of cracks. But there are also presented various shapes of micro to nano size (Fig. 2). Point analysis proved the presence of elements involving in construction of a human body (C, O, Ca, P, S, N). There were found particles containing metals such as Al, Fe, Cu, Ni, Pb, Ti or Zn in 67% of the sialolith´s samples. In most cases the micrometer size particles of metals were spread on the surface

in the form of unidentified compounds. In rare cases pure metal particles there were found too, as it is shown on Figure 2 containing significantly lighter particles of Cu. The relationship between the presence of metals in sialoliths, especially Cu, Pb, Zn and Ni, and the environment, eventually the patient's lifestyle, is the main goal of further study.

The financial support from project MSM 6198910016 is gratefully acknowledged.

Keywords: salivary stones, Sialolithiasis, chemical characterization of salivary stones

Nanoparticles emissions from molten metals

P. Danihelka[1],V. Mička[2], K. Lach[2], V. Hase[1], D. Kupka[1]

[1]VŠB – Technical University of Ostrava; [2]Institute of Public Health Ostrava

pavel.danihelka@vsb.cz

In last decade, the production of engineered nanoparticles (NPs) has become an important sector of nanotechnology and both number of types and quantity of production increased significantly. The study of various nanoparticles has shown that they can pass biological barriers including cell membranes, many of them exhibit high mobility in environment and living organisms and some have negative health effects. Consequently, the investigation of possible health effects of nanoparticles started and nowadays it is a concern of several research projects including international ones. Nevertheless, the study of health effects is complicated and still in early stages and this is why the precautionary principle should be applied in occupational health and safety issues and environmental health consideration related to nanoparticles. The behaviour of engineered nanoparticles has attracted attention also to other type of nanoparticles in working and living environment, i.e. to non-intentionally produced nanoparticles. In the literature, several studies dealing with non-intentional nanoparticles production are present and examples of them are carbon particles from diesel engines, particles from friction processes or nanoparticles produced by tarnishing of paints and other materials. Among various types of engineered nanomaterials, large group is formed by metallic or metal-containing nanoparticles, e.g. nanomaterials based on silver, zinc oxide or titanium oxide. Many other metals are able to form nanoparticles either by dissipation processes or by thermal processes, what implies the idea, that nanoparticles are formed during common metallurgical processes as melting, founding or other processes with liquid metals. Nanomaterials produced by these operations may be an important source of contamination of working environment and environment generally by metal-containing nanoparticles.

To examine the possibility of nanoparticles creation from molten metals in contact with gaseous phase, following pilot tests were realized: In the air-tightly closed ceramic cup, industrially used metals with low melting points (Al, Pb, Sn, Zn and alloy Pb50-Sn50) were melted and purged by nitrogen. Influence of metal temperature and volume of nitrogen bubbled throw molten metal was investigated. Temperatures 500, 700 and 900 deg C respectively (only above melting point of metal) were applied and particular nanoparticles aerosol fractions concentration in the range 5 to 100 nm measured by FMPS spectrometer. Samples of all tested metals and alloy aerosols were collected during the last test phase at the highest testing temperature by NanoID WRAS sampler for subsequent SEM and ICP-MS analyses.
The experiments show that high temperature (900 deg C) leads to significant increase of fine NPs (5 – 12 nm) of all tested metals. The relationship between metal vapour partial pressure and the quantity of distributed NPs cannot be clearly identified. Further, the relationship between purged nitrogen amount and quantity of distributed NPs is rather ambiguous. The experimental results suggest that there is changing of fractioning. Changing the dynamics of purged nitrogen changed distribution of NPs, what can be caused by different mechanisms of particles generation. The series of concurrent processes (evaporation followed by condensation, oxidation, splashing) are proceeding during the metal melting and differently sized particles are generated.

Keywords: nanoparticles, industrially used metals, working environment, metal temperature

Nanoparticles in the Ostrava urban atmosphere

P. Danihelka[1], K. Lach[2], V. Mička[2], V. Hase[1]

[1]VŠB – Technical University of Ostrava; [2]Institute of Public Health Ostrava

pavel.danihelka@vsb.cz

The atmosphere of the Ostrava region has been rightfully considered unsatisfactory in the long term. Combination of the geographical, climatic and meteorological conditions on the one hand and the accumulation of industrial plants, small local heating sources and transportation on the other hand, results in the basis to regularly repetitious situations – inversion accompanied with considerable increase of PM_{10} (and $PM_{2.5}$) concentration. The number of days during which the immission limit of PM_{10} daily concentration can be exceeded is 35 days/year. The real situation is about 6 times worse. The immission limit of $PM_{2.5}$ and PM_1 has not been introduced yet and the value of 0.8 of PM_{10} and $PM_{2.5}$ concentration ratio of Ostrava airborne aerosol shows relatively higher apportionment of finer fractions in Ostrava atmosphere, compared to other Czech or European towns. Moreover there are not available systematic data of ultrafine aerosol fractions in the Ostrava urban atmosphere so far.

With respect to present-day findings in aerosol science and toxicology, the risk potential of airborne aerosol particles narrowly coheres with particle size, within the range of particles smaller than 1 µm the health-related number concentration and surface area besides, or instead of the mass concentration, are considered to be more relevant in a health effect assessment. Also, the particle size distribution and the particle surface area characterization could help the better understanding of a daily and seasonal variation of benzo(a)pyrene concentration and possibly also help with the PAH's emission source identification.

In this work we present two campaigns results of a nanoparticles monitoring conducted under different meteorological conditions at four locations in Ostrava: Ostrava-Poruba, which is considered to be a suburb with a relatively low pollution level, Ostrava-Přívoz, close to the city centre and the coking plant, Ostrava-Mariánské hory and Ostrava-Bartovice considered to be the most polluted locality in the region, often mentioned as the most polluted village of the central Europe. The nanoparticles concentration in the Ostrava urban atmosphere was measured in the range 5 to 100 nm by FMPS spectrometer.

The total particle number and particle size distribution shows remarkable variations in all localities, primarily characterised by size distributions modality. Given the fact there is much greater danger of ultrafine fractions of airborne dust, it will be paid more attention to this issue in the future. The nearest monitoring campaign will continue in the particle number and size distribution measurement, but also shall be focused on the sampling of aerosol samples in range of approx. 1 nm – 20 µm by various sampling equipment for further physicochemical properties like shape and surface observation by SEM, TEM and chemical compositions of samples obtained from various localities and various seasonings.

Keywords: nanoparticles, airborne aerosol particles, urban atmosphere, immission limits

Delamination of phylosilicates

P. Majorová[1], J. Seidlerová[1], G. Simha Martynková[1]

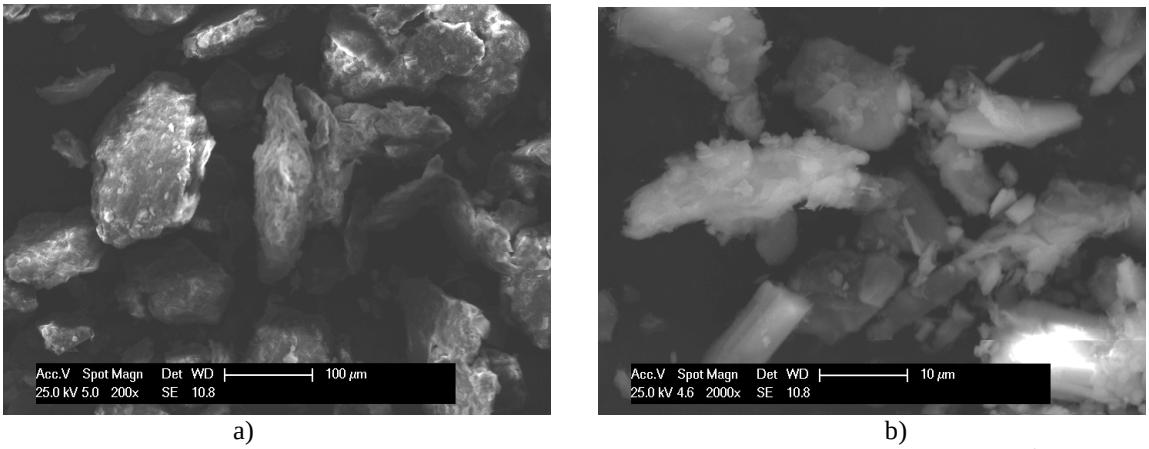
[1]VŠB – TU, Ostrava

jana.seidlerova@vsb.cz

Phylosilicates are very interesting materials for different industrial applications (cosmetic and pharmaceutical products, sorbents, filler for nanocomposites etc. One of significant is vermiculite. The crucial factor for nanocomposites preparation is the particle size. There are several methods of the phyllosilicate delamination/exfoliation described in literature. The first method of delamination is the intercalation of some organic molecules (aliphatic amines) into the interlayer space of the phylosilicate structure.

The present paper deals with influence of oxalic acid on vermiculite structure. Natural powdered Mg-vermiculite from Brazil, with following crystallochemical formula $(Si_{6,32}Al_{1,58}Ti_{0,1})(Mg_{4,75}Ca_{0,34}Fe_{0,91})O_{20}(OH)_4Ca_{0,04}K_{0,38}$ was used. Before the treatment the vermiculite was modified by grinding and the particles size less than 0.2 mm was selected. After 120 minutes treatment of powdered Mg-vermiculite sample in water solution with oxalic acid at five different temperatures in interval 25 ^oC - 95 ^oC the vermiculite was separated by filtration. The changes of vermiculite structure were study using rtg. diffraction analyses (BRUKER D8 ADVANCE), particle size distribution (PSD) using the low-angle laser method of PSD (Analysette C22) and scanning electron microscope with EDS detector (PHILIPS XL-30). To describe mechanism of exfoliation the content Na, Ca, Mg, K, Si and Al in water solution after treatments were determined by the atomic emission spectrometry with inductive couplet plasma (instrument JY-24). The results were compared with vermiculite treatment in deionised water at the same condition.

The most expressive reduction of the peaks intensity of the 001 diffraction was observed after vermiculite treatment at temperature 95^oC. The changes of particles size are obvious from Fig. 1, which compares the original vermiculite and after its treatments with oxalic acid at 95^oC. The results confirmed that vermiculite delamination with oxalic acid is possible.



a) b)

Fig. 1: Vermiculite particles a) original b) after treatment with oxalic acid solution at 95 ^oC.

The financial support from project MSM 6198910016 is gratefully acknowledged.

Keywords: exfoliation, Mg vermiculite, phylosilicates

Clay supported 2-phenylimidazole derivatives for catalysis of Henry reaction

S. Holešová[1], P. Pařík[2], M. Ludwig[2]

[1]Nanotechnology Centre, VSB- Technical University Ostrava, Czech Republic

[2]Faculty of Chemical Technology, University of Pardubice, Czech Republic

Eight derivatives (**1-8**) of 2-phenyl-1*H*-imidazole were tested as catalysts of Henry nitroaldol reaction. Two types of catalysis, homogeneous and heterogeneous, were examined and compared. Clay minerals Ca-montmorillonite (Ca-MMT) and Cu-montmorillonite (Cu-MMT) were used as solid supports for heterogeneous catalysis.

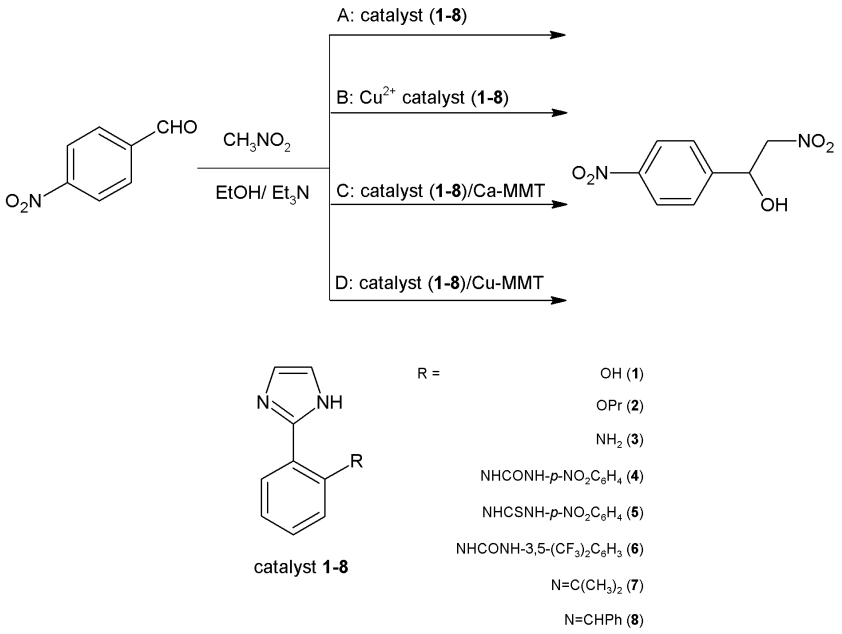


Figure 1. Catalysis of Henry reaction

Catalysis of Henry reaction was assessed from point of view of yield and reaction time. Primarily, reaction was accomplished without application of catalyst for further comparison; in this case reaction time was 24 hours and yield 58%. Secondly, reaction was accomplished in presence of solid supports Ca-MMT and Cu-MMT. In both cases, reaction times are comparable with reaction time of reaction without catalyst, but yields are evidently higher, so from the point of view of yield, chosen solid supports are catalytically active.

The best results were obtained using compound *N*-benzyliden-2-(1*H*-imidazol-2-yl)aniline (**8**) under conditions of heterogeneous method D from the point of view of yield and reaction time.

This work was supported by the Czech Grant Agency (project GACR No. 205/08/0869), and the Ministry of Education, Youth and Sports of the Czech Republic (projects MSM 6198910016 and MSM 0021627501).

Keywords: Henry reaction, imidazole, montmorillonite, catalysis

In-situ synthesis of oriented zeolite MFI layers

P. Hrabánek[1], A. Zikánová[1], J. Drahokoupil[2], M. Matějková[1], I. Jirka[1], L. Brabec[1], V. Fíla[3], B. Bernauer[3], M. Kočiřík[1]

[1]J. Heyrovský Institute of Physical Chemistry of the ASCR, v.v.i., Czech Republic

[2]Institute of Physics of the ASCR, v.v.i., Czech Republic

[3]Institute of Chemical Technology Prague, Czech Republic

pavel.hrabanek@jh-inst.cas.cz

The optimization of synthesis procedures to prepare preferentially oriented zeolite MFI (ZSM-5 or silicalite-1) layers is a challenging topic attractive for applications in the field of membranes, microreactors, sensors and optoelectronic devices. Due to the simplicity and possibility to easily coat the surface of complex geometrical objects such as grains, grids or monoliths, the one step direct *in-situ* hydrothermal synthesis of silicalite-1 layers was the primary focus of this work. The preferentially *b*-oriented silicalite-1 layers were synthesized on mercury surface, silicon wafer, non-porous and porous stainless steel (TRUMEM^{TM}) supports. The synthesis procedures were based on the preparation of *b*-oriented zeolite MFI monolayer films and optimization was made by the addition of the second colloidal silica source (TOSIL/LUDOX) to promote preferred orientation and crystal intergrowth. The main factors that were observed after the synthesis of silicalite-1 layers included: (i) preferred crystal orientation, (ii) crystal intergrowth and (iii) layer compactness that were evaluated from Scanning Electron Microscopy (SEM, JEOL JSM 5500LV) and X-ray diffraction (XRD, PANalytical X`Pert diffractometer in Brag-Brentano geometry with Co radiation). Crystallographic Preferred Orientation (CPO) indexes were used to quantify the degree of preferred orientation of silicalite-1 crystals in the layers. Fig. 1 shows two examples of preferentially *b*-oriented silicalite-1 layer prepared on *P*-doped silica wafer (Fig. 1a) and fine-polished porous stainless steel support (Fig. 1b).

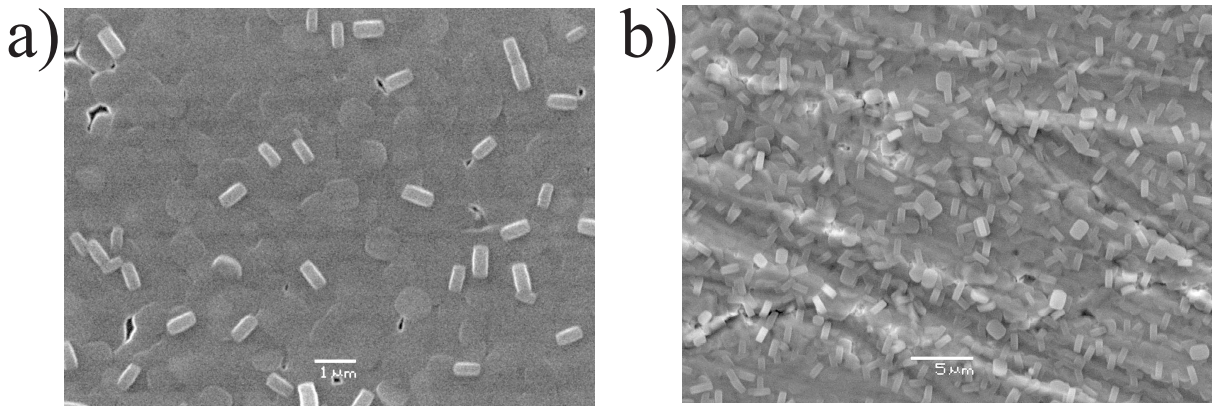


Figure 1. Preferentially *b*-oriented silicalite-1 layers synthesized on *a) P*-doped silica wafer, *b)* fine-polished porous stainless steel support.

The preferentially *b*-oriented silicalite-1 layers were prepared on mercury, silicon wafer, non-porous and fine-polished porous stainless steel supports. The synthesis solutions of two silica sources accelerated crystallization and promoted crystal intergrowth based on the observation of self-supported silicalite-1 layers.

The financial support by The Grant Agency of the AS CR via grants KAN 400720701 and IAA 400400909 is gratefully acknowledged.

Keywords: zeolite MFI, support, orientation, CPO index

Nanocomposit ZnS-MMT for photocatalytic reduction of CO_2

M. Reli, O. Kozák, P. Praus, K. Kočí, L. Obalová

Technical University of Ostrava, Faculty Metallurgy and Material Engineering

kamila.koci@vsb.cz

Greenhouse gases such as CO_2, CH_4 and CFCs are the primary causes of global warming. The concentration of carbon dioxide in the atmosphere has increased as a result of human activities, further accelerating the greenhouse effect. The efficient photocatalytic reduction of CO_2 by H_2O is one of the most desirable and challenging goals in the research of environmentally friendly technologies. Zinc sulphide is a semiconductor with wide direct band gap energy about 3.7 eV. It appears to be a very promising material for photocatalysis due to a rapid generation of electron–hole pairs by the photoexcitation and highly negative reduction potentials of excited electrons. The goal of this work was to determine the optimal concentration of ZnS-MMT catalyst for the photocatalytic reduction of CO_2. ZnS nanoparticles for the photocatalytic reduction of CO_2 were deposited on montmorillonite (MMT) in the presence of cetytrimethylammonium (CTA). UV spectrometry and transmission electron microscopy proved the formation of nanoparticles with diameters ranging from 3 nm to 5 nm. The band gap energy of nanosize ZnS was estimated at 3.89 eV. The photocatalytic reduction of carbon dioxide was carried out in a stirred batch annular reactor with a catalyst suspended in 0.2 M NaOH and illuminated by UV 8 W Hg lamps (254 nm). GC/FID/TCD was used for the analysis of reaction products. The catalyst concentrations were 0.5; 1.0; 1.5 and 2.0 g dm^{-3}. Fig. 1 shows the yields of all reaction products as functions of the concentration of ZnS-MMT catalyst. The optimal concentration of ZnS-MMT photocatalyst was determined at 1 g dm^{-3}.

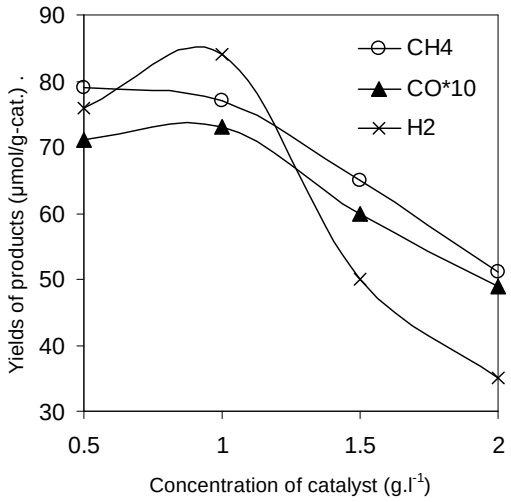


Fig. 1 Dependence of the product yields (after 24 hours of irradiation) on concentration of ZnS-MMT catalyst

The financial support of the Grant Agency of the Czech Republic (P107/11/1918) and EU project CZ.1.05/2.1.00/03.0069 is gratefully acknowledged.

Keywords: Photocatalytic reduction, CO_2, ZnS-MMT, Catalyst concentration

The alkali metals as the promoters in Co-Mn-Al mixed oxide

K. Karásková[1], T. Kulová[1], P. Kustrowski[2], L. Obalová[1], K. Jirátová[3], F. Kovanda[4]

[1]Faculty of Metallurgy and Materials Engineering, Technical University of Ostrava, Czech Republic

[2]Faculty of Chemistry, Jagiellonian University, Krakow, Poland

[3]Institute of Chemical Process Fundamentals CAS v.v.i., Prague, Czech Republic

[4]Institute of Chemical Technology, Prague, Czech Republic

lucie.obalova@ vsb.cz

The alkali promoted Co-Mn-Al mixed oxides were prepared by impregnation of the calcined Co-Mn-Al layered double hydroxide (Co/Mn/Al = 4/1/1) by nitrate solutions of alkali metals (Li, Na, K, Rb and Cs). The prepared spinel catalysts were characterized by AAS, AES, SEM, XRD, TPR, TPD and XPS. The elemental mapping of alkali metals indicated homogeneous promoter distribution on the catalysts' surface (Fig. 1). Substantial differences between surface (XPS) and bulk chemical composition (AAS, AES) and changes of acido-basic surface properties (TPD-CO_2, TPD-NH_3) were observed.

The effect of alkali promoters on N_2O decomposition over prepared catalysts was investigated in inert gas. In Fig. 2, the temperature dependences of N_2O conversion are shown. Li-promoted catalyst was less active for N_2O decomposition in comparison with the non-modified one, while the same catalytic activity on Na-containing catalyst was observed. On the other hand, potassium, rubidium and cesium caused the increase of N_2O conversion. Their beneficial influence on the catalyst activity increases in the order: K < Rb < Cs and is probably of electronic nature.

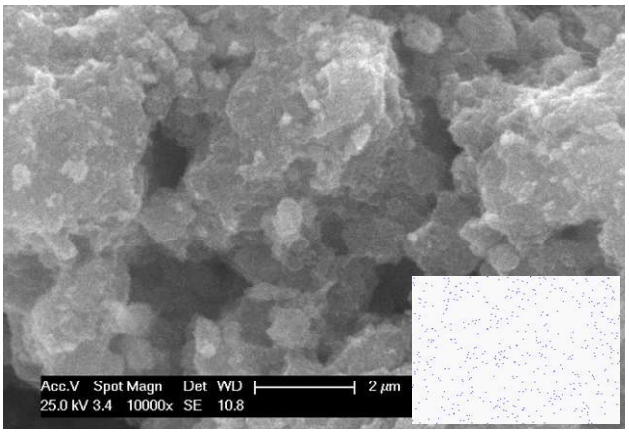


Fig. 1 Characteristic SEM image of Cs-promoted Co-Mn-Al mixed oxide catalyst.
The dots in the inserted picture represent cesium surface distribution

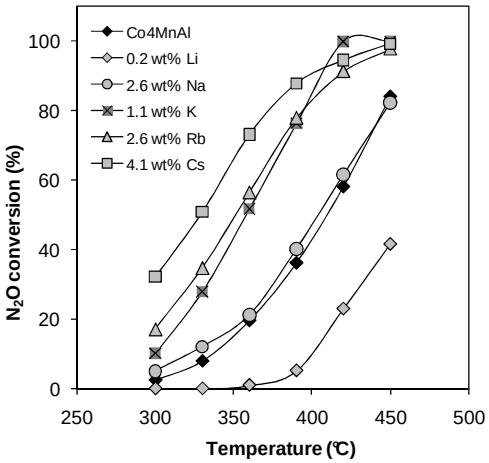


Fig. 2 Temperature dependence of N_2O conversion on alkali promoted Co-Mn-Al mixed oxide.
Conditions: 0.1 mol% N_2O He, space velocity 60 l g^{-1} h^{-1}, alkali molar content 0,0003 mol/g of catalyst

This work was supported by the Czech Science Foundation (project No. 106/09/1664).

Keywords: Mixed Oxide, Layered Double Hydroxide, Alkali Promoters, N_2O decomposition

APPENDIX

A

Akbulut, H. **OP-18**,1,2,3,8,20 22,23,28 PP-21,28,29,37,38
Alp, A. PP-21, OP-18,20,28
Andrýsek, T. OP-9
Aydin, A. O. OP-23
Aydin, G. OP-28
Aydin, Y. **OP-1**

B

Baciu, A: **OP-14**
Bačovská, R. PP-16
Baheti, V. K. **PP-22**,38
Bachmatiuk, A. **IL-1**
Barabaszová, K. **PP-23**
Berger, J. **PP-1**
Bernauer, B. PP-49
Brabec, L. PP-49
Bretšnajdrová, E. **PP-2**
Büchner, B. IL-1
Bulut, E. **PP-24,25**,35,36
Bureš, Z. **PP-3**
Burtica, G. OP-14,16

C

Cada, M. **IL-2**
Cevreh, O. **OP-2**,1,8,11
Cigánová, B. **PP-26**
Ciprián, D. OP-10
Czochara, R. **PP-4**

Č

Čapek, L. PP-6
Čapková, P. OP-9, PP-8
Černošek, Z. OP-29

D

Danihelka, P. PP-45,46
Dědková, K. **OP-30**
Dogan, F. PP-37
Drahokoupil, J. **OP-34**, PP-49
Dvorský, R. **OP-19**, PP-11
Dvořáčková, J. OP-32
Dzik, P. OP-6

F

Fajgar, R. OP-5, PP-1
Fečko, P. OP-31
Fernández, V. A. OP-30
Fíla, V. PP-49
Filip, P. OP-32
Foldyna, J. OP-26

G

Grégr, J. OP-24
Gröplová, K. **PP-43**
Gryčová, E. **PP-44**
Gül, H. **OP-20**, 18,21
Guler, M. O. **OP-3**,1,2,8,11 PP-37,38
Gultekin, D. OP-1,2,8,11

H

Hrach, R. OP-7
Harničárová, M. PP-30
Hase, V. **PP-45,46**
Hlaváček, P. PP-30
Hloch, S. PP-30
Holešová, S. **PP-48**,39
Holišová, V. OP-17
Holubová, J. OP-29
Horská, K. IL-4
Hovorková, I. PP-16
Hrabánek, P. **PP-49**, OP-34

CH

Charvát, R. OP-25
Chládová, A. PP-19
Chotěbor, M. OP-24
Chvojka, J. **OP-21**

I

Ibrahim, I. IL-1

J

Jirátová, K. PP-51
Jirka, I. PP-49

K

Káfuňková, E. PP-10
Kaj, F. PP-15
Kalivoda, P. PP-23
Kalus, R. **OP-4**
Kára, J. PP-41
Karásková, K. PP-51
Karlíková, M. **PP-27**
Karslioğlu, L. **OP-22,**28, **PP-28**
Kasiri, M. PP-34
Klich, J. OP-26
Klouda, K. OP-19
Klusoň, P. OP-6
Kočí, K. **PP-50**,6, OP-35
Kočiřík, M. OP-34, PP-49
Kolář, M. PP-27
Köse, H. **OP-23, PP-29**
Košťáková, E. **OP-24**

Koval, L. **OP-31**
Kovalová, L. OP-31
Kovanda, F. PP-10,51
Kovář, P. OP-35, PP-10
Kozák, O. OP-36, PP-11,50
Kratošová, G. **PP-5**, OP-17
Krejčíková, S. **PP-6**
Krogul, A. PP-7
Kroteň, M. A. **PP-7**,9
Kuběna, V. PP-30
Kučerová, L. OP-31
Kučerová, R. OP-31
Kukutschová, J. **OP-32**, PP-43
Kulová, T. PP-51
Kupka, D. PP-45
Kustrowski, P. PP-51
Kušnerová, M. **PP-30**
Kvítek, L. **OP-33**, PP-40

L
Lach, K. PP-45,46
Lang, J. **PP-31**
Lang, K. PP-10
Łasocha, W. PP-33
Lesňák, M. OP-10,12
Litwinienko, G. PP-4,7
Lu, Y. **OP-32**
Luberda-Durnaś, K. **PP-33**
Ludwig, M PP-48
Lukáš, D. OP-21,25
Luňáček, J. OP-19
Lyčková, B. OP-31

M
Macháčková, M. **PP-8**
Majorová, P. PP-47
Malachová, K. OP-32
Maléterová, Y. OP-5
Mamulová Kutl., K. OP-35, PP-33
Manea, F. OP-14,16
Mansori, M. E. **IL-3**
Martinec, P. OP-26
Matěj, Z. OP-5
Matějka, V. **OP-35**,32 PP-31,33,43
Matějková, M. PP-49
Matějová, L. **OP-5**
Matlochová, A. **OP-15**
Matoušek, J. **PP-16**
Mendes, R. G. IL-1
Mészáros, L. OP-24
Mička, P. PP-45,46
Mikeš, P. **OP-25**
Militký, J. PP-22,38
Morozová, M. **OP-6**
Moslemi, M. PP-15
Motoc, S. **OP-16**

N
Najari, M. **PP-34**
Nakatake, Y. OP-13
Natšinová, M. OP-17
Navrátilová, Z. PP-17
Niakan, S. PP-34
Novák, S. **OP-7**
Nováková, D. OP-26
Novotný, F. PP-1,14
Ntaka, S. PP-20

O
Obalová, L. **PP-51**,6,50
Örnek, A. **PP-35**,24,25,36
Özacar, M. **PP-36,37**,24,25,35
Özkar, S. PP-18

P
Pálka, K. OP-29
Panáček, A. OP-27,30,33 PP-27,40
Pařík, P. PP-48
Peikertová, P. PP-43
Piksová, K. OP-19
Piotrowski, P. **PP-9**
Pištora, J. OP-10,12
Plachá, D. OP-15
Plášek, V. PP-5
Plevová, E. **PP-17**
Pode, R. OP-16
Pokorný, P. OP-24,25
Pop, A. OP-14,16
Pospíšil, M. **PP-10**
Praus, P. **OP-36, PP-11**,50
Procházka, M. PP-14
Pokrop, R. PP-4
Proška, J. PP-1
Prucek, R. PP-27,40

Q
Qi, S. PP-32

R
Rakap, M. **PP-18**
Ranjbaran, S. PP-15
Rebilasová, S. PP-43
Rehan Abbasi, A. M. **PP-38**,22
Reli, M. PP-50
Remes, A. OP-14
Richter, I. PP-14
Rosická, D. PP-13
Rümelli, M. H. IL-1

Ř
Řepka, M. PP-30

S
Sadeghi, H. PP-15
Samlíková, M. **PP-39**
Sancar, M. PP-28
Seidlerová, J. **PP-47**,44
Schröfel, A. **OP-17**
Schroeter, S. OP-29
Simha Martynková, G. IL-1, PP-17,47
Sitek, L. **OP-26**
Sivera, M. **PP-40**
Skupińska, J. PP-7
Soukupová, J. OP-33
Staněk, F. **OP-10**
Suchomel, P. **OP-27**
Svoboda, L. PP-2,41

Š
Šafařík, I. **IL-4**, OP-17
Šafaříková, M. IL-4, OP-17
Šembera, J. **PP-13**
Šolcová, O. OP-5,6, PP-6
Štolcová, L. **PP-14**
Štrympl, P. PP-44
Šupová, M. OP-35
Šustek, S. **PP-41**
Švec, M. OP-7

T
Tiznobaik, K. PP-34
Tocoglu, U. **OP-8,11**
Tokarský, J. **OP-9**, PP-8
Trojková, J. OP-19

U
Uysal, M. **OP-28**,22, PP-28

V
Vaculíková, L. PP-17
Valášková, M. **PP-12**,23,42
Valíček, J. PP-30
Valovičová, V. PP-17
Vávra, I. **IL-5**
Vejsadová PP-19
Veselý, M. OP-6
Višňovský, Š. **IL-6**
Vítek, A. OP-4
Vlček, J. **OP-12**
Vlček, M. **OP-29**, PP-3
Vontorová, J. PP-31

W
Watanabe, K. **OP-13**
Wiener, J. **PP-19,20**,26

Z
Zdrálková, J. PP-42
Zeleňák, M. PP-30
Zelenka, J. PP-2,41
Zetková, K. PP-41
Zhao, X. PP-32
Zikánová, A. OP-34, PP-49
Zolanvari, A. PP-15