Dear Conference participants, Visitors and Colleagues,

We are proud to invite you to the VŠB- Technical University halls, which are hosting their first international conference **Nano Ostrava 2008**, a meeting of scientists, experts, students and companies from all around the world, working and researching in field of nanotechnology.

This conference is under kind auspice of university rector prof. Ing. Tomáš Čermák, CSc.

Our university VŠB – Technical University of Ostrava is one of the oldest Czech universities. Traditional faculties of Mining and Geology, Metallurgy and Materials Engineering and Mechanical Engineering are complemented with other technical faculties such as Electrical Engineering and Computer Science, Civil Engineering, Safety Engineering and Economics. Last year our university opened a new dimension of education in two new interdepartmental fields of study Nanotechnology and Mechatronics.

The interest in these advanced disciplines from the students, researchers and industrial partners was the first impulse for arranging this event.

The significance of the conference for city Ostrava points out the fact that Moravian-Silesian Region and hetman Ing. Evžen Tošenovský, Dr.h.c. are patrons of this conference.

We appreciate the sponsors and attendance of prestigious companies representing producers of analytical and technological equipments, testing and measurement instruments, and laboratory tool.

We are proud, that the Nano Ostrava 2008 conference is being attended by significant international scientists from fourth continents, as well as world known Czech scientists.

We believe that this gathering could be good place to start new friendships and cooperation, and our conference will be a good base for further years

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Wellcome from the Chairman of the Nano Ostrava 2008

During last few years, nanotechnology has become highly promising field of science with many applications in various branches of materials research and industry; from chemistry, biochemistry, pharmacology to new sensors and optoelectronic devices.

The conference programme **NANO Ostrava 2008** covers selected fields of nanomaterials research:

- Nanocatalysts and photocatalysts
- Polymeric, ceramic and metallic nanocomposites
- Nanostructured materials for magnetooptics, electronics and photonics
- Modeling of nanomaterials and structure analysis
- Particles and nanopowders
- Biotechnology for nanomaterials fabrication, health safety and environmental risk of nanomaterials

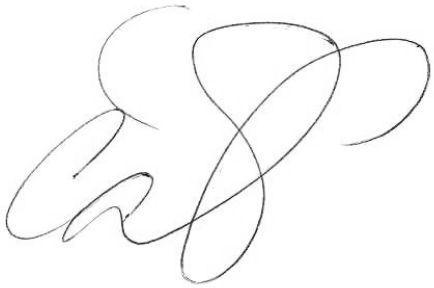
It must be emphasized that the conference programme is not meant to provide a full coverage of all latest developments. Anyway it offers an informative survey of the contemporary state of art and new trends in research fields mentioned above.

Dear colleagues,
I hope that you will take this opportunity to expand your professional contacts and beyond the professional aspects you will find some time for sight-seeing in Ostrava, which is educational, cultural and industrial centre of the North Moravian part of Czech Republic.

I would like to thank the rector of VŠB-Technical University Prof. Ing. Tomáš Čermák, CSc. and hetman of the Moravian-Silesian region Ing. Evžen Tošenovský, Dr.h.c. for their auspices.

Last but not least my acknowledgements and respect belongs to my co-workers from Nanotechnology centre who willingly cooperated in organization of this conference.

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



Pavla Čapková (Chairman)

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

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Photoactivity evolution of titanium dioxide nanoparticles in course of aging of their colloidal solutions

H. Měšťánková[1], M. Kolář[1], G. Grabner[2], J. Jirkovský[1*]

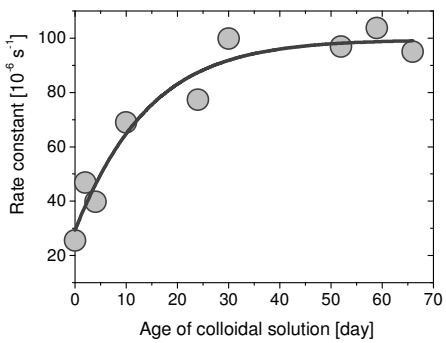
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Aqueous colloidal solutions of quantum sized particles of titanium dioxide (Q-TiO_2) undergo an ageing process since the moment of their preparation. In course of time, the mean size of nanoparticles is gradually increasing and some of their physico-chemical properties are changing as well. The decrease of blue spectral shift of semiconductor absorption threshold was measured to determine the corresponding changes of band gap energy of the Q-TiO_2 particles. In parallel, the decrease of specific surface area of these particles was followed through their complexation with salicylic acid and thiocyanate anions. The formation kinetics and thermodynamic equilibrium of these surface charge-transfer complexes were investigated in detail by means of UV/VIS absorption spectroscopy. Based on the obtained results (equilibrium constant and molar absorption coefficients), the concentration of complexation sites (surface Ti atoms) was derived that can be used to characterize the surface area of the colloidal particles.

In the TiO_2 / SCN^- system, the dependence of surface complex concentration on bulk SCN^- concentration was compared to quantum yield of the pulsed-laser-induced $(SCN)_2^-$ radical formation. The quantum yield generally increased with the increase of bulk SCN^- concentration and with the percentage of iron doping. The found non-linear shape of these dependences was less pronounced for smaller semiconductor particles that for larger ones. A quantitative kinetic model for description of dependences of the absorbance of primary radical products of interfacial charge-transfer on the laser pulse energy was derived. It is based on the assumption that photogenerated electrons and positive holes are repeatedly trapped and fast thermally excited into corresponding either conduction or valence band. Competitive processes of electron-hole recombination on the one hand and of charge transfers on the other hand are described as functions of irradiation intensity, numbers of available trapping sites, and numbers of reactants, i.e. electron-acceptors as well as electron-donors, adsorbed on the particle surface.

The photocatalytic activity, which represents the most important quality of titatium dioxide from the practical point of view, was repeatedly determined through photocatalytic degradation of 4-chlorophenol in the colloidal solution of Q-TiO_2 particles in the course of its ageing for several months. The corresponding reaction rate increased in the first weeks more than three times; afterwards it approached a limiting value. In the figure, a dependence of the first-order rate constants of photocatalytic degradation of 4-chlorophenol on the age of Q-TiO_2 colloidal solution.



Keywords: titanium dioxide, photoactivity, charge-transfer surface complex, colloidal solution, aging

Carbon based polymeric nanocomposites (processing and properties)

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Nanocomposite materials encompass a large variety of systems, made of distinctly dissimilar components and mixed at the nanometer scale. The properties of nanocomposite materials depend not only on the properties of their individual parents but also on their morphology and interfacial characteristics. The current work focuses on carbon nanocomposites (carbon nanofiber filled polymers) with an emphasis on purification, surface functionalization and heat treatment temperature of nanofibers. Two methods were used (heat treatment and Soxhlet extraction) for purification and removal of impurities of the pristine nanofibers. Cleaning of nanofibers by subsequent heat treatment and Soxhlet extraction resulted in chemical and physical property improvements due to the removal of the surface dielectric layer.

Prior heat treatment of carbon nanofibers lead to alignment of graphene layers within the nanofibers allow for a more efficient transfer of phonons and electrons resulting in significant decreases in electrical resistivity and increases in thermal conductivity of the neat epoxy resin. However, heat treatment beyond 1800°C lead to increased conversion to graphene layers and resulted in lower mechanical properties due to poor adhesion to the epoxy matrix.

Functionalization of nanofibers showed that simply the oxygen content of a functionalized sample does not correlate with improved matrix adhesion. Instead, the type of functional group is a factor in determining mechanical properties and curing rates. The phenol group improves adhesion with the epoxy matrix to a greater degree than carboxylic acid groups. Oxygen groups cause a negative impact on electrical properties of the nanofiber, but do not tend to effect thermal properties.

Based on the results of the testing of heat treated and surface functionalized nanofiber based polymer composites, the possibility exists for tailoring of nanocomposite physical properties.

Keywords: nanocomposite, nanofiber, purification, functionalization

Capability of X-ray diffraction and transmission electron microscopy for the microstructure analysis of nanomaterials

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Development of new materials is nowadays based on the knowledge of the relationship between the materials microstructure and the materials properties, as it is well known that the modification of the materials microstructure leads to the adjustment and to the tailoring of the materials properties. Currently, defect engineering is discussed as a possible tool for the development of materials with the desired properties. The most relevant instruments of the defect engineering are the kind, the density and the ordering of microstructure defects. The importance of the microstructure defects for the materials properties can excellently be illustrated on nanocrystalline materials and nanocomposites, in which the volume of the microstructure defects like the crystallite and grain boundaries or the interfaces between different phases approaches the volume of the "undistorted" material. In general, the presence of internal interfaces in nanocrystalline materials and in nanocomposites strongly affects the competition between the surface energy and the strain energy that is frequently responsible for the microstructure formation.

In order to be able to utilize the relationship between the materials microstructure and the materials properties and to tailor the materials microstructure in particular technological processes, thorough microstructure analysis is needed. In this contribution, the capability of the combination of the X-ray diffraction (XRD), the conventional transmission electron microscopy (TEM) and the transmission electron microscopy with high resolution (HRTEM) for the microstructure analysis on nanocrystalline materials and on nanocomposites will be illustrated on the examples of the hard Ti-Al-(Si-)N, Cr-Al-(Si-)N and Zr-Al-(Si-)N thin films nanocomposites, ultrahard BN-nanocomposites and hot-rolled perlitic steels. It will be shown that XRD, TEM and HRTEM are complementary experimental methods for microstructure analytics that allow the following microstructure features to be determined: the crystallinity, the phase composition and the homogeneity of individual phases in nanocomposites. For crystalline phases, it will be discussed, how the combination of these experimental methods can furthermore used to visualize and to determine the size, the shape and the global preferred orientation of nanocrystallites, the local disorientation of neighboring nanocrystallites, the atomic ordering at the nanocrystallites boundaries and the microstructure defects that are responsible both for the formation of the internal interfaces and for development of the internal stresses. Additionally, some microstructure-related phenomena will be discussed in the talk, which are related to the mechanical properties of the nanocomposites, like a strong anisotropy of the elastic constants and the partial coherence of adjacent nanocrystallites.

Keywords: nanocomposites, microstructure, X-ray diffraction, transmission electron microscopy

Advantages of gas phase prepared nanoparticles in understanding carbon nanotube growth

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The use of gas phase prepared nanoparticles as growth catalysts for 1D structures opens up new possibilities for ex-situ studies on the role of the catalyst. This is well exemplified when applied to carbon nanotube (CNT) nucleation and growth. Conventional techniques, on the whole, do not use predefined catalyst particles. This limits the information available on the correlation between the initial catalyst nanoparticle and the resultant CNT. Gas phase prepared nanoparticles provide a key advantage, viz. information on the nanoparticles a priori. This information combined with post synthesis studies provide a rich base with which to decipher key aspects of the growth process of CNT, which is highly relevant if CNT are to play a role in future molecular electronics and other devices.

Systematic spectroscopic and microscopy studies on carbon nanotubes synthesized via chemical vapor deposition (CVD) using nano-engineered particles on oxide supports were conducted [1]. Our findings show that both the diameter and number of walls of the resultant CNT increase proportionally to the size of the catalyst particle. This behavior is fully consistent with catalyst volume to surface area constraints as predicted by data using floating catalyst nanoparticles [2].

In addition, detailed HREM post-synthesis studies on the carbon nanotubes show capped tops and open-ended roots. The data points to the consecutive inward formation of concentric graphene caps during nucleation constricting and elongating the catalyst particle within the tube core. Studies of gas phase prepared nanoparticles on graphite corroborate this model. Further, the data suggest that after nucleation continued growth stems from the oxide support itself rather than from the catalyst particle [3]. Studies on typical oxide supports alone show that such oxides can graphitise carbon species probably via defect and/or step sites. This suggests CNT growth may occur through similar mechanisms as found for heterogeneous catalysis where the dissociation of molecules on a surface takes place preferentially at atomic steps and kinks.

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Keywords: nanoparticles, catalysis, TEM, carbon nanotubes

Magnetic nanoparticles and biotechnology

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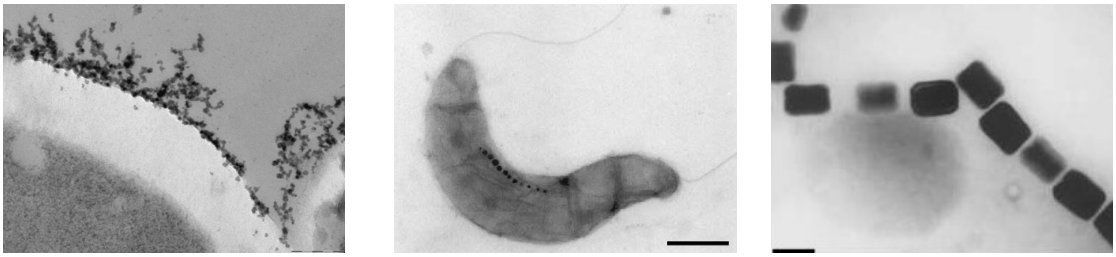
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Magnetic nano- and microparticles have already found many important applications in various areas of biosciences, medicine, biotechnology, environmental technology etc. Magnetic particles belong to the group of "smart materials" or "stimuli responsive materials" which exhibit response to external magnetic field. These particles can be efficiently separated from difficult-to-handle samples and targeted to the desired place, used as contrast agents for magnetic resonance imaging, to generate heat during exposition to alternating magnetic field, to modify biomolecules and biological structures etc.

Different areas of biotechnology have already benefited from the application of biologically active compounds (antibodies, enzymes, lectins, nucleic acids, oligonucleotides, specific affinity ligands) immobilized to magnetic nano- and microparticles (e.g., immunomagnetic assays of target compounds, affinity separation of cells, cell organelles or molecules, molecular biology applications, magnetofection, etc.). Magnetically responsive microbial cells or lignocellulose, useful for xenobiotics removal, were prepared by ferrofluid modification. Industrial waste water treatment process "Sirofloc" is based on the application of magnetite microparticles.

Biotechnology approach can be used to prepare specific types of biocompatible magnetic nanoparticles *in vivo* or *in vitro*. Several types of magnetotactic bacteria, forming intracellular magnetosomes composed of maghemite or greigite, have been cultivated successfully and the released magnetosomes used as carriers for immobilization of antibodies or enzymes. Extracellular magnetite nano- and microparticles are formed during bacterial reduction of ferric ions. *Fusarium oxysporum* extracellular proteins enabled *in vitro* formation of magnetite nanoparticles.

Both biotechnology and nanotechnology can have enormous benefit from close mutual cooperation.



Kluyveromyces fragilis cells modified with magnetic fluid (left), magnetotactic bacterium *Magnetospirillum gryphiswaldense* (center), and typical examples of magnetosomes (left)

Keywords: magnetic nanoparticles, magnetic biocomposites, magnetotactic bacteria, magnetic separation

Design of well-defined TiO_2 for photocatalysis

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Powder nanoporous TiO_2 with narrow monodisperse pore-size distribution in the range from 2 to 30 nm were prepared by self-assemble micelle system in nonpolar and polar solvents at laboratory temperature and pressure. Nonionic surfactants Triton X-102, 100, 114, 45, 15 were used for preparation of micelle systems. Various approaches were applied for removing of residual organic matter from prepared titania gels: a) calcination, b) improved extraction by supercritical (CO_2) and subcritical fluids (H_2O, CH_3OH) and c) the combination of both forenamed approaches.
The influence of different calcination temperature, time and temperature ramp as well as the extraction temperature and pressure on the purity and the change of TiO_2 PSD width and the shift of PSD maxima were observed.
Deeper textural and structural information was obtained via physical adsorption of nitrogen (ASAP2020, Micromeritics), helium pycnometry, high-pressure mercury porozimetry, TiO_2 XRD phase analysis, organic elemental analysis and TEM.

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Keywords: sol-gel, TiO_2 powder, supercritic fluid extraction, pressurized fluid extraction, texture, nanostructure, nitrogen physical adsorption, XRD

The correct determination of texture properties in microporous-mesoporous solids

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Evaluation of micropore volume and mesopore surface area in microporous-mesoporous samples is presented. A series of mechanical mixtures of microporous zeolite NaY, and mesoporous γ-alumina was used for verification of the applicability of the (three-parameter) modified BET equation. The nitrogen adsorption isotherms (77 K) were analyzed by the modified (three parameters) BET equation and compared with the independent textural information obtained a) from t-plots (with the standard isotherm of nonporous α-alumina and the standard isotherms of Lecloux-Pirard) and b) by subtracting the isotherm of the parent NaY from mixed sample isotherms. Similarly, nitrogen adsorption isotherms of rutile samples with micropores blocked by n-nonane, from the literature were tested. For illustration, the failure of classic (two-parameter) BET equation for microporous-mesoporous samples is demonstrated.

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Keywords: BET method, porous materials, t-plot, texture parameter

Titanium dioxide growing on the kaolinite particle surface and its practical utilization in the building industry

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Titanium dioxide (TiO_2) growing on the silicate substrate is one of the possibilities how to improve its photocatalytical activity. Other significant advantage results from the fact, that the TiO_2 nanoparticles are fixed on the silicate matrix and in this way doesn't mean serious problem for living environment in comparison to the often discussed pure photoactive TiO_2 nanoparticles.

There are several successfully utilized methods for the highly photoactive TiO_2 nanoparticles preparation, whereas sol-gel method is the most often used. The most often used precursors for the sol-gel method include titanium alkoxides ($Ti(OR)_n$), titanyl sulphate ($TiOSO_4$) and titanium tetrachloride ($TiCl_4$). The hydrolysis of these compounds under suitable conditions (temperature, solution used for hydrolysis …) leads to TiO_2 nanoparticles formation. After the hydrolysis the photoactivity of the prepared nanoparticles is often improved using proper thermal treatment.

Among well known applications of the photoactive TiO_2 nanoparticles belongs building industry. The patent [1] proposed by Italcementy company describe the TXactive® technology, where photoactive TiO_2 is used in combination with Portland clinker to produce the concrete with self-cleaning properties as well as with positive impact to the living environment.

Kaolinite belongs to 1:1 phyllosilicate family and represents important raw material utilized in building industry. After its heat treatment above approx. 480 °C the dehydroxylation occur and former well crystalline kaolinite transfer to almost amorphous metakaoline what is approved with disappearing of the 001 basal diffraction on its powder difractogram. Metakaoline is latently hydraulic material and after its addition to the concrete mixture improve mechanical properties of the final hardened concrete significantly. Other possible activation of metakaoline includes its reaction with alkaline hydroxides or water glass what leads to formation of hardened geopolymers.

In our work we focused on the kaoline/TiO_2 composite preparation with the aim to obtain composite with the latently hydraulic properties and high photocatalytic activity.

The financial support of the Czech Ministry of Industry and Trade research project FT-TA4/025 is gratefully acknowledged.

Reference:

[1] Patent no. WO 9805601 (1998).

Keywords: TiO_2, kaoline, photoactivity, building materials

Self-supporting pellets from (Al-)SBA-15 molecular sieve

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Thanks to high surface areas and large pores with narrow pore size distribution, the mesoporous molecular sieves attracted much attention as sorption materials, catalysts and catalyst supports [1]. Because the powder form cannot be used directly for industrial applications [2], the development of methods for the preparation of extrudates or pellets from mesoporous molecular sieves is desirable. One of the most interesting materials is SBA-15 due to its hydrothermal stability, pore wall thickness and tunable pore size [3].

Usually, the extrudated catalyst is prepared with the aid of binders and plasticizers, typically bentonite and methylcellulose, respectively [2,4]. To the best of our knowledge, only one paper reporting the preparation of Al-SBA-15 pellets without the use of additives was reported so far [5]. However, the detailed characterization of pelleted Al-SBA-15 lacks in this paper. Moreover, it can be expected that high pressure (~ 120 MPa) used for the formation of the pellets caused the collapse of mesoporous structure of powder Al-SBA-15. The aim of this preliminary study was to investigate texture and catalytic properties of self-supporting (Al-)SBA-15 pellets prepared from powder catalyst without the addition of binders or plasticizers.

In order to describe the relationship between the pressure used for pellets forming and their texture properties, samples of powder SBA-15 were pressed into self-supporting pellets using four various pressures. When the forming pressure was 0.7 MPa, only small decrease in the surface area of mesopores (8 %) was revealed, while the pore size and micropore volume remained unaffected. With increasing pressure the surface area was decreasing and when the pressure was 2.7 MPa and higher, partial structural collapse of SBA-15 was observed.

Al-SBA-15 catalyst in the form of powder and self-supporting pellets was employed in the alkylation of toluene with benzyl alcohol under moderate conditions (110°C). The reaction was carried out in the batch reactor stirred with inert gas under reflux conditions. Even the lowest forming pressure (0.7 MPa) was sufficient to form a pellet that was enough mechanically stable to maintain the structural integrity during the reaction. However, the catalytic activity of Al-SBA-15 pellets was about four times lower than that of powder catalyst due to diffusion limitations.

The financial support of Grant Agency of the Czech Academy of Sciences (A 4072404) and AS CR Program Nanotechnology for Society (KAN 400720701) is gratefully acknowledged.

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Keywords: mesoporous molecular sieves, Al-SBA-15, pellets, Friedel-Crafts alkylation

Anti-wear coatings on cutting tools for wood machining

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Currently, machine tools used in wood industry are characterized by high performance. Development of this industry has resulted in the full automation of the production process. Howewer, cutting tools applied in this area do not fulfil all the requirements for full exploitation of the machines potential. In this case, the main problem is related to differences in physical and chemical structures between wood and metals. Wood is characterized by good machinability allowing high cutting and feed speed. However wood-like materials (such as plywood and laminates) are characterized by nonuniform internal structure and a very hard sub-surface layer, thus making them exceptionally difficult to machine.

Currently, the cutting tools for wood machining are produce from carbides, Polycristalline Diamond (PCD), high speed steels and hardened steels. Among these materials, steel is the cheapest. However, steel cutting tools are characterized by weak wear resistance. For this reason, modification of these tools is required. In this case great hope is directed to carbon coatings, particularly Diamond Like Carbon (DLC)coatings..

DLC coatings have very attractive anti-wear properties such as low friction, high hardness, chemical inertness and high surface quality. These beneficial properties of DLC are due to the mixture of tetrahedral (sp^3) and trigonal (sp^2) bonding constituents of carbon in the coating. For example, the level of hardness of carbon coating depends on the sp^3 concentration in the deposited coating. High concentration of sp^3 bonds is correlated with high hardness of the coating.

In this paper, we investigate the cutting behavior of carbon coatings for cutting operations on an wood and wood-like materials. We have deposited carbon coatings on steel cutting knife. Carbon coatings have been produced by Radio Frequency Plasma Assisted Chemical Deposition (RF PACVD) method. For the synthesis of carbon films pure methane has been used. Coatings were produced under different deposition conditions; flow rate of gas, negative self-bias voltage, and time of deposition were changed..

Carbon coatings obtained in this way was characterized by the following studies. The roughness was measured by optical profilometry. The scratch test has been used to assess thin coating adhesion to the substrate. Scanning electron microscopy (SEM) was used to examine the coatings as fractured cross-sections with the aim of characterizing their microstructure. In order to get information on the chemical composition of the coatings glow discharge optical spectroscopy (GDOS), **and X-ray Photoelectron (XPS)** were taken out. Thickness of the coatings was determined by GDOS.

Keywords: carbon coatings, wear resistance, RF PACVD, wood machining

Surface metal/polymer 2D nanocomposites: fabrication and application in catalysis

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Recently, we systematically studied the effect of glass transition temperature reduction in polymer surface layer by the novel nanoscale surface probing technique. This technique is based on the control (using atomic force microscope) of the embedding of colloidal metal nanoparticles, preliminary adsorbed on the polymer surface, under the system stepwise annealing in the temperature range between the room temperature and the polymer glass transition temperature (T_g). The information obtained allowed us to formulate the algorithm of preparation of new type of polymer/metal composites, i.e., surface 2D nanocomposites with tunable properties. Its main steps are as follows.

1. Synthesis of metal sols with the given nanoparticle size, shape and surface chemistry.
2. Deposition of the monolayer metal nanoparticle ensembles (with controlled packaging density) on the polymer surface by adsorption from colloidal solution.
3. Adsorbed metal nanoparticle ensemble embedding in polymer surface layer for a given depth by annealing at the temperature between surface glass transition temperature (T_g') and T_g values.

Experimental results concerned with the practical realization of this algorithm will be reported in presentation.

In particular, **t**he adsorption kinetics of gold nanoparticles from their colloidal solutions onto the polystyrene and poly(2-vinylpyridine) surfaces was studied using atomic force microscopy and quartz crystal microbalance. The comparison of the results obtained by these two independent techniques showed their good accordance. The adsorption kinetics is limited by nanoparticles diffusion from hydrosol to both polymers studied and is virtually irreversible up to rather high surface coverages.

To solve the problem of "two-dimensional" nanoparticle ensembles incorporation in polymer surface (for a depth less then the particle diameter) we proposed several modification methods of polymer surface layer which lead to its T_g' depression. These methods are as follows: UV irradiation, addition of small amounts of oligomeric homologue or other polymer with the lower T_g value to the "matrix" polymer.

To obtain the dense nanoparticle monolayers, which are needed for some practical applications, we developed the procedure of enlargement of metal nanoparticles incorporated in polymer surface layer using an aqueous solution of chloroauric acid ($HAuCl_4$) and hydroxylamine mixture as a growth solution.

Also an example of the surface 2D-nanocomposite application as a catalytic system will be presented. It will be shown that polystyrene/Pt-nanoparticles 2D-nanocomposites reveal high catalytic activity in the reaction of methyl viologene reduction by molecular hydrogen in basic medium.

Keywords: metal nanoparticles, polymer surface layer, nanocomposites

Metal organophosphonates and their intercalation compounds as inorganic-organic hybrid materials and nanomaterials

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Metal organophosphonates belong to a group of inorganic-organic hybrid compounds which have received much attention with the aim to obtain novel materials with interesting properties. Layered structures of phosphonates contain an ordered network of active sites, where active molecules anchored to the host layers can be well organized. In addition, new compounds can be prepared, in which a functional group is attached to the organic part of the compound, thus enlarging the intercalation potential of these phosphonates. From this point, organophosphonates containing carboxylic, sulfonic, or amino groups seem to be very interesting.

We prepared several new phenylphosphonates and 4-carboxyphenylphosphonates of alkaline earth metals and copper and characterized them by powder X-ray diffraction, thermogravimetric analysis, energy-dispersive X-ray analysis, elemental analysis and infrared spectroscopy. For four of these compounds, their structure was determined from their powder X-ray diffraction patterns using an ab initio FOX program. The prepared alkaline-earth phenylphosphonates with general formula $MeC_6H_5PO_3 \cdot yH_2O$ (Me = Ca, Sr, Ba) were found to be suitable hosts for intercalation reactions.

These metal phenylphosphonates are able to intercalate amines and alcohols providing compounds which contain one molecule of the guest per formula unit of the host. We presume that the guests are anchored to the layers of the host by coordinating to the metal atom via nitrogen or oxygen atoms of their functional groups. By intercalation the distance between the host layers, the interlayer distance, increases. It was found that for the intercalates of $BaC_6H_5PO_3 \cdot yH_2O$ the interlayer distance d depends on the length of the alkyl chain n_C both for the intercalated amines and alkanols with roughly the same slopes of d vs. n_C dependencies. The values of these slopes indicate that the guest molecules are arranged in the interlayer space so that the alkyl chains are roughly perpendicular to the planes of the host layers.

The ability of these phenylphosphonates to intercalate amines and alcohols indicates their possible applications in anchoring commercially interesting species in the interlayer space of the hosts. By intercalation of polymers (or monomers and subsequent polymerization) new two-dimensional composite nanomaterials can be prepared.

Keywords: phosphonate, alkaline-earth metals, intercalation, alkylamines, alkanols

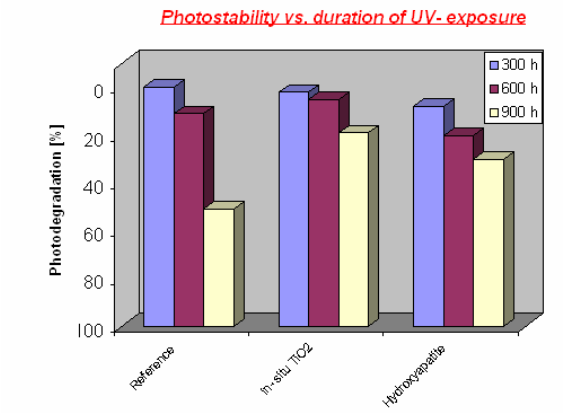
Inorganic nanoparticles for the protection of transparent polymer composites against UV-Light-Exposure

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In order to improve the outdoor durability of polymer materials, new types of UV-stabilization agents are necessary. Commonly applied UV-protection agents based on organic compounds are not intrinsically long-term-resistant against exposure to ultraviolet radiation. This suggests to test out different types of robust inorganic nanoparticles as additives for the photostabilization of materials such as powder coating polymers. New problems, however, connected to some of these additives may arise due to the rather high surface energy of such inorganic particles, which tends to cause significant aggregation phenomena. Furthermore, in the case of semiconductor-based additives such as the frequently applied compounds TiO_2 or ZnO, the photocatalytic activity of these particles may lead to undesirable aging and degradation effects of polymer materials such as polyester.
In this context, the role of various inorganic additives for the photostabilization of commercially important powder coating materials has been investigated. The beneficial effects of functional nanoparticle additives have clearly been documented (among other experiments) by a series of industrial-scale UV-exposure tests as shown schematically in the figure below:



An important issue for many practical applications of the polyester-based coatings studied in our work is to guarantee sufficient long-term transparency of the polymer under outdoor ambient conditions. Therefore, it is very important to avoid light-scattering, which occurs whenever the embedded inorganic additives are too large or dispersed inhomogeneously. In order to achieve and maintain the desired transparency, two possible strategies have been followed in our work. On the one hand, the particle-size and aggregate formation has been controlled, and on the other hand, improved and photochemically inert additives with a refraction index closely matching that of the polymer matrix have been developed. As will be shown, our search for innovative additive strategies succeeded especially by using hydroxyapatite-nanoparticles, and by producing titanium-dioxide-particles *in-situ* during the polyester synthesis.

Keywords: nanoparticles, photostabilization, polymer materials, transparent coatings

Protonization of methylene blue and its intercalation into montmorillonite

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The protonization of methylene blue (MB) in acid aqueous solutions was studied and protonization constant evaluated. The percentages of protonized methylene blue (MBH^{2+}) cations were calculated for various acid aqueous solutions of different ionic strengths using calculated protonization constant.

The cations of MB (MB^{+}, MB_n^{n+} and MBH^{2+}) present in acid aqueous solutions were adsorbed on montmorillonite SAz and adsorption curves were measured. For the photometric determination of total methylene blue analytical concentration in aqueous solution the wavelengths of absorption maxima 660 nm (relating to MB^{+}) and 745 nm (relating to MBH^{2+}) were used. By this way two independent results of methylene blue concentration were obtained. Adsorption curves determined in acid aqueous solutions differ from that one determined in neutral aqueous solution by more complicated shape. Adsorption curves determined in acids aqueous solutions consist of two waves which are similar to the sixth type of physical adsorption isotherm for adsorption that run in several separate steps. It was found that the first wave relates to ion exchange of MB forms from aqueous solution with cations present in SAz interlayer; the second wave relates to the adsorption of MB forms on outer SAz surface. This relatively high amount of MB adsorbed on outer surface is attributed to acid outer surface of montmorillonite and to presence of MBH^{2+} with double charge.

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Keywords: protonization of methylene blue, montmorillonite, adsorption

The PP/clay nanocomposites: using of commercial fillers

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This study has studied the effect of commercial filler on the properties of prepared nanocomposites and the influence of condition of preparation was studied too. The nanocomposites were comprised by polymer matrix, maleated polypropylene and filler. The polymer matrix was comprised by polypropylene. Maleated polypropylene was used for better compatibility between polymeric and filler. The concentration was 5 wt. %. We have used two types of commercial nanofillers – Dellite 72T and Dellite 67G which are suitable for polyolefinic matrix. The concentrations of fillers were different: 2, 4, 6, and 10 wt. %. The nanocomposites were prepared on mixing device Brabender Plasti-Corder. The conditions of preparation were different too. The level of intercalation and the changes of mechanical properties were obtained. The results of X-ray diffraction and transmission electron microscopy showed that the level of intercalation of filler did not better, because the polymeric matrix contained some agglomerate of filler. However, all nanocomposites had higher mechanical properties than the virgin polypropylene. The compare of type's fillers showed that the better intercalation and higher mechanical properties had nanocomposites contained filler named Dellite 72T. All measured dates are showed in tables and graphs.

Keywords: nanocomposites, commercial filler, intercalation, mechanical properties

Preparation of organovermiculites and testing of naphthalene sorption

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Organophilic smectites are commonly used for sorption of hydrophobic organic compounds from aqueous media. In this study, vermiculite as clay mineral with higher CEC value than CEC values for smectites was used for preparing of material with higher sorptive properties.
Organic cations of HDTMA (hexadecyltrimethylammonium) and HDP (hexadecylpyridinium) were used for preparing of organovermiculites with 50%, 100% and 170 % of exchanged vermiculite CEC. The prepared organovermiculites were characterized with XRD analyses, TOC analyses and the experimental values were compared with theoretically calculated values. Lower cation exchanges and TOC content were reached comparing theoretical and measured values in all prepared organovermiculites.
The prepared organovermiculites were exposed to: 1) naphthalene vapors in closed container and 2) water spiked with naphthalene solution in methanol. In the first case we observed changes in basal diffractions d_{001} and TOC content after naphthalene vapor sorption. Molecular modeling was used for describing of naphthalene interactions with chains of organic cations in interlayer gallery. In the second case the naphthalene sorption isotherms for organovermiculites containing organic cations in 50% of vermiculite's CEC were created using purge and trap and thermal desorption method and gas chromatography with mass spectrometry for naphthalene analysis in solution after sorption process.
In organovermiculites with higher cation exchange capacity (100 and 170%) partial delamination of organovermiculites and creation of very small vermiculite particles containing chains of organic cations was observed. It occurred as foaming of solution after sorption process that was purged by nitrogen to prepare naphthalene analysis. The presence of the vermiculite particles was confirmed with XRD analyses.
Both 50% organovermiculites were used for sorption of organic nonpolar compounds from real waste water. The sorption effectiveness was in range 74-95% for different organic compounds whereas HDP vermiculite seemed to be more efficient. Connection between organic compound affinity and coefficients K_{ow} and K_{oc} was unambiguously demonstrated.

The present work was solved in the frame of the research project of GAČR 205/08/0869 and Ministry of Education, Youth and Sport of Czech Republic MSM 619 891 0016.

Keywords: organovermiculite, HDTMA, HDP, naphthalene

Polymer/Clay nanocomposite – a brief description and a practical using

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Recently nanocomposites based on polymer matrix and clay mineral nanofiller have attracted a great amount of research. It is caused by specific properties of these types of materials. In comparison with conventionally produced composites in the case of nanocomposites, the much lower amount of nanofiller can bring the same of even better material properties, such as thermal stability, barrier or mechanical properties. This ability of nanofillers brings their high aspect ratio, because clay mineral nanofillers could be taken as big platelets with the thickness in the size in nanometers.

Nowadays montmorillonite (MMT) is the most used clay mineral. It is a layered silicate, composed of very small platelets. MMT has the structure 2:1, which means that it consists of two fused silica tetrahedral sheets sandwiching an edge- shared octahedral sheet aluminium hydroxide. The MMT interlayer is capable to be intercalated by several types of organic or inorganic molecules. The choice of a suitable compound is done by the purpose of the using.

This presentation brings brief information about the polymer nanocomposite preparation and mainly about the examples of their practical using.

Electromagnetic scattering from imperfectly periodic structures

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When a plane wave is incident on a perfectly periodic structure, the Floquet theorem claims that the scattered fields are pseudo pseudo-periodic. This means that each field component is a product of a periodic function and an exponential phase factor, and allows us to reduce the analysis region to the single periodicity cell. This fact also means that the fields have discrete spectra in the wavenumber space. Recently, structures in which the periodicity is locally broken have also received much interest because they yield distinct properties. One of the most promising properties is the field localization induced by defects in the photonic crystals, which is thought to allow realizing essential optical elements of wavelength size. For example, a lump of defects may behave as a microcavity resonator and a line defect may behave as a waveguide. In such structures, the Floquet theorem is no longer applicable, and the computation has been mainly performed with the finite differences time-domain method, the finite element method, the time-domain beam propagation method, the method of fictitious sources, etc. The analysis region of these approaches have to cover all the scatterers under consideration and the approaches for scatterer with infinite extent has been limited to perfectly periodic structure with plane wave incidence.

This study shows an approach for scattering problems of imperfectly periodic structures with non-plane wave incidence with the use of the pseudo-periodic Fourier transform (PPFT). Let $f(x)$ be a function of x and d be a positive real constant. Then PPFT and the inverse transform are formally defined as followings:

$$\bar{f}(x;\xi) = \sum_{m=-\infty}^{\infty} f(x-md)\, e^{imd\xi}, \qquad f(x) = \frac{d}{2\pi}\int_{-\pi/d}^{\pi/d} \bar{f}(x;\xi)\, d\xi, \quad (1)$$

where ξ denotes the transform parameter. The transformed function $\bar{f}(x;\xi)$ has a pseudo-periodic property with the pseudo-period d in terms of x: $\bar{f}(x-md;\xi) = \bar{f}(x;\xi)e^{-imd\xi}$ for any integer m. Also $\bar{f}(x;\xi)$ has a periodic property with the period $2\pi/d$ in terms of ξ: $\bar{f}(x;\xi-2\pi m/d) = \bar{f}(x;\xi)$, and the inverse transform is given by integrating on ξ over the period as shown in Eq. (1). PPFT converts the electromagnetic fields to pseudo-periodic functions of x, and the transformed fields are expressed in the generalized Fourier series expansions. Then, an artificial discretization on ξ makes us possible to apply the conventional grating theory based on the Floquet theorem to the scattering problems of imperfectly periodic structures. The present formulation is applied to fundamental structures in which periodicity are locally collapsed and numerical examples validate the effectiveness of the proposed method. This numerical experiments also show that we should take into account Wood's anomalies to discretize ξ adequately for practical computation.

Keywords: electromagnetic scattering, imperfectly periodic structure, pseudo-periodic Fourier transform

Fitting of iron nano-film properties in planar optical structure

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The study of the response from optical nano-structure containing a ferromagnetic component offers wide applications among other in magneto-optics, optical sensing or by designing of nano-systems with required properties. Since experimental investigation is very time and cost consuming, the important attention appertains the theoretical modeling and/or simulation of optical nano systems.

We present some results obtained by fitting of iron nano-layer properties in planar optical system created in basic version as the Fe thin film with gold protecting cover deposed on bulk Si substrate. The thicknesses of layers have been fitted from spectral-ellipsometric data measured in laboratory of Dept. of Physics at VŠB-TUO. The computational scheme based on the matrix model of multilayer has been realized by the use of own algorithms and implemented in Matlab codes.

The results obtained by analysis of three samples with different geometry have been used to theoretical predicting of surface plasmon resonance (SPR) response from described system supplied by coupling prism with fixed Au layer. This one enables the plasmon-waves excitation in e. g. Kretchmann-Raether confi-guration.

Keywords: planar optical nanostructures, fitting, iron nano-layer, surface plasmons

Surface plasmon resonance arrangement

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The method of surface Plasmon excitation (Surface Plasmon Resonance - SPR) belong to modern optical method that during last ten years found apply in detection and analysis of solid and liquid materials. The extreme sensitivity of SPR to small changes in refractive index (up to 10^{-8}) is used primarily in sensors, especially as very sensitive detectors of various substances in biology and chemistry, determining thicknesses of layers adsorbed on metal surface or to study of chemical reactions kinetics. This method can be also used to study layered or periodical nanostructures which occur frequently in microelectronics.

In Department of Physics at VŠB-TU it was decided to extend experimental devices for SPR. Final implementation has been tested on several basic measurements and obtained experimental data were compared with theoretical model. We shall disposal two experimental arrangements for SPR measurement. First of them was developed at VŠB- TU of Ostrava. It is adaptation of Gaertner L119 ellipsometr. The measurements are realized at five different wavelengths. The second used Multiscope arrangement for SPR experiments has been developed by Optrel. At the present it works at wavelength of 632.8 nm.

Keywords: surface plasmon resonance, SPR, thin layers

Mathematical simulation of severe plastic deformation

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Project of new geometry of extrusion (forming) tool was based on results of the experiment aimed at simulation of uniformity of deformation distribution of the sample in its volume during forming by the ECAP technology, which was made at the Faculty of Mechanical Engineering (FME) of the VŠB-TUO. Engineering design of the new tool for the given technology was based on results obtained with use of classical geometry. It concerns in particular change of the route of deformation at the first pass through the ECAP channel. It results in increased amount of deformation leading to higher grain refinement and therefore to overall increase in efficiency of the process of severe plastic deformation. Design modifications concerned horizontal channel, which is deflected by 10° and 20° in respect to the horizontal axis.

Comparison of the obtained magnitude of deformation intensity for the channel geometry without deflection and with deflection of 10 and 20° and angles $\phi = 90°$ and $\psi = 90°$ in the software QForm has unequivocally confirmed that deformation intensity at the first pass through the channel of the ECAP tool has been significantly increased (see Fig. 1). This design modification is experimentally verified in the laboratory of the Department of mechanical technologies of the FME VŠB-TUO.

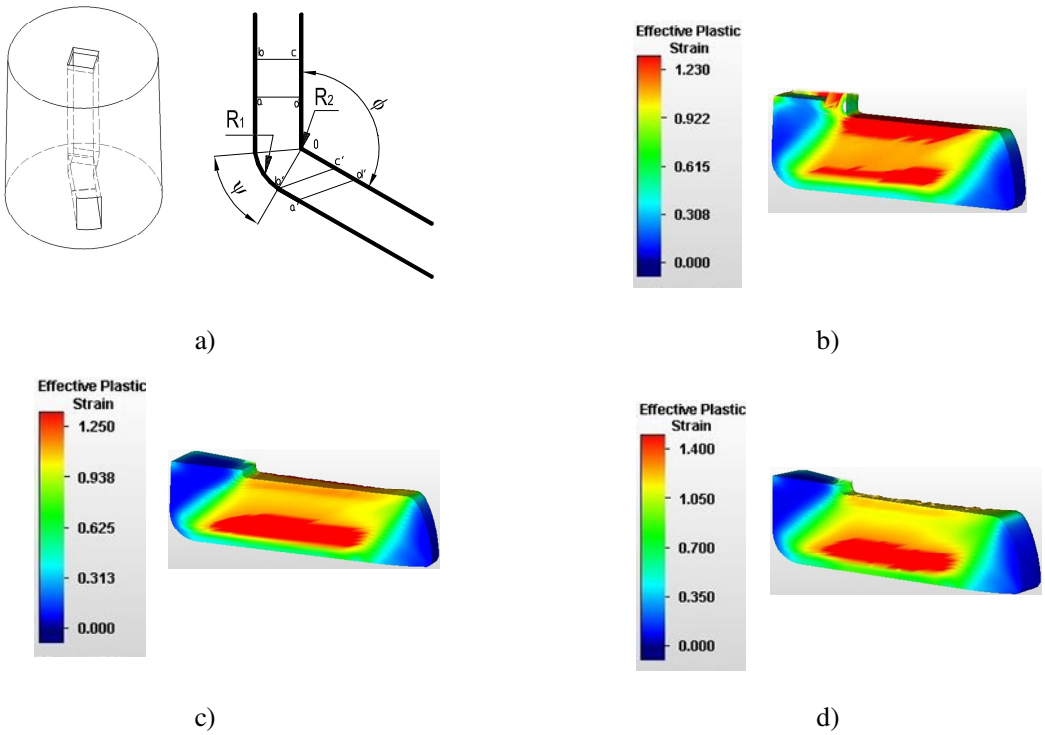


Fig. 1. Development of deformation intensity

a) geometry of the channel ECAP, b) channel without deflection c) channel deflection by 10°, d) channel deflection by 20°

Deflection of the outgoing part of the channel [°]	Effective strain [-]
0	1.0
10	1.15
20	1.3

Keywords: ECAP process, Al alloys, effective strain, die geometry

Bending buckling analysis of carbon nanotubes and graphene sheets by molecular structural mechanics approach

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In this article, bending buckling of two kinds of single wall carbon nanotubes (SWCNT) and single layer graphene sheets(armchair and zigzag) are studied. An exact relation for nanotubes' diameter is introduced. Also, all of internal loads in each element are considered. Buckling of CNTs is very important in micro electromechanical systems (MEMS) and nano electromechanical systems (NEMS). In this article, two types of zigzag CNTs, three types of armchair CNTs and two kinds of graphene sheets with free-fix end condition are studied.
According to nanotubes' geometry, two formulas are suggested for nanotube's diameters in present work:

$$d = \frac{3a}{\sin(\frac{180}{n})} \qquad (1) \qquad\qquad d = a\frac{\cos 30}{\sin(\frac{90}{n})} \qquad (2)$$

Equations (1) and (2) reperesnt armchair(n,n) and zigzag(n,0) nanotubes' diameter, respectively. In these equations, parameter a reperesents each element length (carbon-carbon bond length). In almost all articles the following formula is considered as general(n,m) nanotube's diameter.

$$d = \frac{a_0\sqrt{(m^2+mn+n^2)}}{\pi} \qquad (3)$$

Where a_0 is the length of unit vector in the direction of chiral vector. By application of formulas (1) and (2) the length of all elements comes to be the same. This is the benefit of our suggested formulas. Critical buckling force is determined by means of molecular structural mechanics approach. In this approach, CNTs and graphene sheets are considered as space frames. By constructing equality between strain energies of each element in structural mechanics and potential energies of each bond(which are according to all kinds of bond deformations), equivalent space frames with CNTs and graphene sheets can be achived. Finite element method is applied to buckling eigenvalue problem of equivalent space frame.
Aspect ratio is defined as the length to diameter ratio for nanotubes and the length to width ratio for graphene sheets. Results are shown as diagrams stating critical buckling forces with respect to aspect ratios for several kinds of nanotubes and graphene sheets. This study shows that bending buckling force decreases by increasing nanotube's length and decreasing its diameter. It can be observed that, critical buckling force in CNTs is larger than that of graphene sheets at the same aspect ratios.

Keywords: molecular structural mechanics approach, nanotube, graphene sheet, bending buckling

Refractive index designing of liquids using SPR

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The surface plasmon resonance (SPR) technique belongs to optical methods for designing of the refractive index of very thin layers. In presented paper we summarize some results obtained by testing of recently prepared mathematical model for inverse optical scattering problem. The computational scheme is based on the matrix model of multilayer [1] and implemented in Matlab-code which uses the built-in Nelder-Mead algorithm for non-linear regression.
The reflectometric measurements have been realized in laboratories of Dept. of Physics at VŠB-TU. The most common geometrical setup of SPR (Kretschmann configuration) has been used from coupling prism (60°, SF10 glass) with 44 nm thin gold film that allow excitation of surface plasmon wave. The prism has been connected with the cell containing at first the NaCl-water solution of variable concentration or with the magnetic liquid.
The refraction index of solution has been fitted by different degrees of salinity for wavelengths 604 nm, 612 nm and 633 nm. The computed results obtained at 633 nm show the good agreement with the data published in [2].

References:

[1] J. Pištora, O. Vlašín, M. Lesňák, J. Vlček, A. Talik: SPR application for materials with induced anisotropy. *11th International symposium on microwave and optical technology* (ISMOT – 2007), Monte Porzio Catone, Rome, Italy, December 17-21 (2007), ISBN 978-88-548-1476-9, pp. 143 – 146

[2] www.topac.com/salinity-brix.html

Keywords: SPR, planar optical system, fitting of refractive index

Molecular modeling in nanomaterials design

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Molecular modeling using empirical force field (molecular mechanics and classical molecular dynamics) represents very efficient tool in molecular nanotechnology designing molecular systems with various functionalities allowing the fast structure and property prediction necessary for the development of materials with desirable properties. Empirical force field approach offers two main advantages in materials research: (1) enables to analyze structures of large supra-molecular systems, which cannot be treated using quantum chemical calculations and (2) allows the characterization of disordered crystal structures where the conventional diffraction analysis fails. It is evident that the description of the total system energy by an empirical force field is an approximation which requires extremely careful strategy of modeling, where the experiment must be an integral part of the structure analysis. In the mosaic of experimental methods used in the structure-properties analysis the X-ray diffraction and IR spectroscopy play the key roles.

The main problem in molecular nanotechnology based on the host-guest interaction is the host-guest complementarity, which is characterized by a series of factors describing the presence, ordering and distance of active sites on the host structure, the size of guest molecules, the charge distribution on the host matrix and guest molecules and mutual relation between the host-guest and guest-guest interaction energy. The final supramolecular structure is a result of mutual cooperation of all these factors, which can lead to an ordered or partially ordered crystal structure and/or to completely disordered structures [1]. Molecular mechanics and molecular dynamics combined with experiment have been used for the variety of problems in nano-materials design and characterization:

- Intercalated layered structures (graphite, layer silicates, vanadyl and zirconium phosphate, tantalum sulfate)
- Phase transitions in molecular crystals (liquid crystals and fat crystals)
- Liquid crystalline polymers
- Break up of energetic molecules
- Adhesion of metal, metal oxide and metal sulfide nanoparticles on the crystalline substrates

Molecular modeling explored the host-guest complementarity [1], revealed the mechanism of the phase transitions [2,3] and helped in prediction of structure and properties of intercalated structures [3-5].

References:

[1] P. Čapková, H. Schenk; *J. Incl. Phenom. and Macrocyclic Chemistry*, 47 (2003) 1.
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[4] P. Čapková, M. Pospíšil, Z. Weiss; *J. Mol. Model*, 9 (2003) 195.
[5] P. Čapková, J. Votinský; *Molecular Crystals and Liquid Crystals,* 341 (2000) 301.

Keywords: molecular modeling, nanomaterials, intercalates, nanoparticles

Material grinding by water jets – feasibility and limits

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Disintegration of brittle material particles by water jet takes place during generation of the injection abrasive water jet just in the mixing process inside the mixing chamber and focussing tube. The physical limits of these processes are the topic of the theoretical investigation the results of which are presented in this contribution. The mean particle size resulting from the mixing process can be evaluated from the mean particle size of the input material particles and basic water jet parameters using the theoretical equation (1).

$$a_n = \frac{24\rho_o E_P c_o^2 a_o^3}{24\rho_o E_P c_o^2 a_o^2 + C_D \pi a_o d_o^2 \mu^2 p_o^2 \gamma_R^2} \qquad (1)$$

a_o - input mean particle size, a_n - mean particle size after mixing process, c_o - velocity of sound in liquid, C_D - drag coefficient of material particle in liquid jet, d_o - water nozzle diameter, E_P - specific surface energy of material, $\gamma_R = (1-\gamma p_o)$ - γ is water compressibility, μ - water nozzle discharge coefficient, p_o - water pressure in front of the water nozzle, ρ_o - water density, ρ_a - ground material density.

Equation (1) is carefully analyzed and the results of this analysis are used for explanation of the basic problems of particle grinding by water jet. Special observance is focused on specific surface energy of material. There are discussed both the theoretical and experimental results prepared in last years. Subsequently, some propositions are made for improvement of the water jet grinding process. The primary disintegration process, taking place in the mixing chamber and focusing tube, can be followed up by subsequent processes magnifying material pulverization – interaction with rigid target or collision with opposite jet of the same type. These processes are also investigated and the basic theoretical postulations of their efficiency are formulated.
Using of chemical additives increasing water jet coherence, modulation or pulsation of water flow utilization and inducing of cavitation phenomena inside flow and their utilization during mixing process are the self-sustaining problems the benefits of which are discussed, especially their contribution to growth of effectiveness of brittle material particle grinding.
The theoretical studies are supported by results of experiments aimed at the disintegration processes inside mixing chamber and focusing tube and opposite jet collision. Some results of jet – rigid target interaction are also presented and discussed. All theoretical and experimental conclusions are also analyzed from the economical point of view and the most important conditions for laboratory and even industrial use of this type of mineral grinding are submitted.

Keywords: water jet, mixing process, grinding, brittle material

Particle characterization of nanoparticle materials in Water Jet Mill device

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Particle characterization of nanoparticles prepared by the water jet mill technology in a nano scale has been presented in the paper.
During the investigation, a size distribution change and a particle shape of nanomaterials prepared of the WJM technology as well as optical material properties have been developed.
Silicon nanoparticles created by the WJM technology by which direct mechanical destruction of particles has been taken with their impact to a solid butt made of wolfram carbide after outputting of cavity tube of the WJM.

The project the particle characterization as well as the submitted paper have been supported by the Grant No. MSM 619 891 0016 under Ministry of Education, Youth and Sports of the Czech Republic.

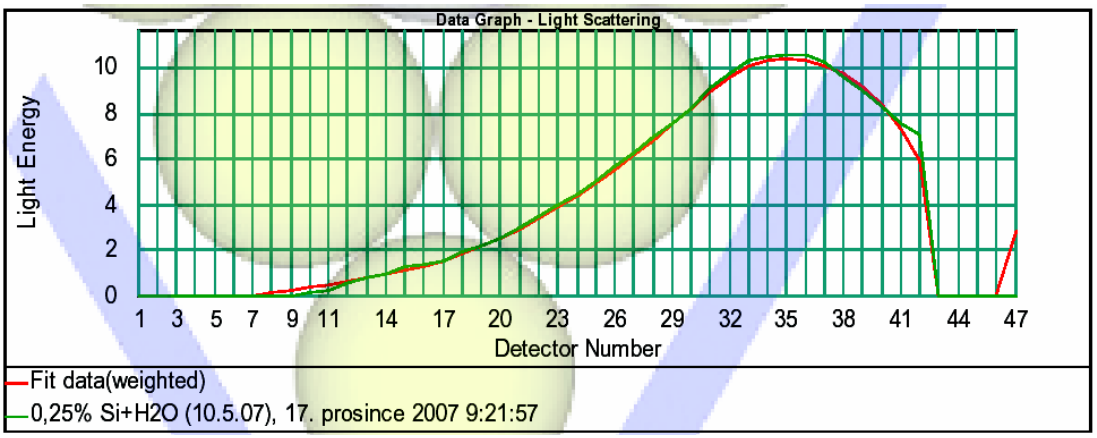


Fig. 1. Data graph-Light scattering : Light energy Vs. Detector Number of the investigated nanoparticle material

Keywords: particle characterization, water jet mill

Identification of 3D inner conception of a particle material rising of its internal friction using the LBM-Nano-Machine for 3D

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Model conception described in the paper is developed by a long-time observation of a friction parameters behavior of particle material groups in the Laboratory of Bulk Materials (LBM) located by VSB-Technical University of Ostrava. A general model of the LBM-Nano-Machine has been designed for a 3D bulk material identification using an internal friction curve or it is possible to design of a individual structure layer to has a expected-conducted/stabilized value of internal friction in a general external behavior.

LBM-Nano-Machine for 3D is developed in the area of a contact friction $\varphi \in$ <30°,70°) in the regime of a layer and particle arrangement which proceed to a shear plane.

The paper has been supported by the Grant No. MSM 619 891 0016 under Ministry of Education, Youth and Sports of the Czech Republic.

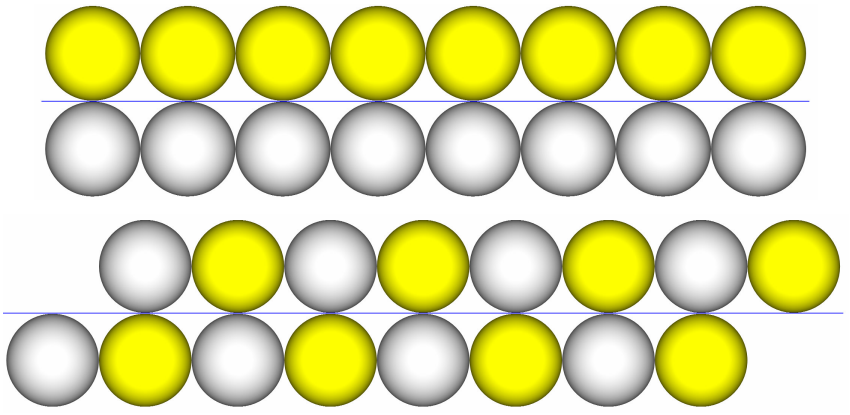


Fig. 1. A schematic figuration of a particles rearrangement principle in LBM-Nano-Machine for 3D

Keywords: internal friction, particle conception, internal friction model

Fluorescent resonant energy transfer in hybrid systems based on rhodamines and synthetic saponite

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Fluorescent resonant energy transfer (FRET) is the interaction between chromophores via a long-range dipole-dipole coupling and occurs in close proximity between interacting molecules (to 10 nm). It is based on the energy transfer between different chromophores; one representing an energy donor and the other an energy acceptor. FRET is the phenomenon taking place in the dimensions of nanometers. We studied energy transfer between different rhodamine dyes by using fluorescence spectroscopy. FRET was observed as the quenching of the emission of energy donor in favor of the emission of energy acceptor, although the energy acceptor had not directly been excited. First series of the experiments were performed to characterize the energy transfer in colloidal dispersions. Relatively efficient energy transfer took place at bulk dyes concentration 10^{-5} M to be over-concentrated at the surface of silicate colloidal particles at loading 5 10^{-5} mol/g. Efficiency of the energy transfer was tested by varying the amount ratio of energy donor and acceptor components. Different dyes loadings and inclusion of the alkylammonium surfactant to the system were tried to suppress molecular aggregation thus preventing the quenching of the fluorescence. The effect of ionisable groups in rhodamine cations may influence the dye properties and adsorption on the silicate surface and, in this way, the energy transfer process. More efficient energy transfer was observed in the systems based on hybrid thin films. Although much larger amount of the energy donor was present in the films, small amounts of the energy acceptor were efficient to quench the energy donor emission.

This work was supported by the Slovak Research and Development Agency (No. APVV-51-027405) and VEGA agency (2/6180/27).

Keywords: organic dyes, energy transfer, molecular aggregation, fluorescence

Preparation of Nanoparticle Materials in New Device WJM (Water Jet Mill)

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Nanoparticle preparation (Fig. 2) in a device of new design called "Water Jet Mill"(Fig. 1) -WJM has been presented in the paper.
Disintegration of solid particles has been developed with a rapid mechanical stress in an extreme speed gradient and cavity implosion.
These disintegration areas have been created by cavity bubble implosions cumulated with an impacted wave. The impacted wave proceeds to parallel with a different intensity in the all parts of desintegration volume of the WJM device. Direct mechanical destruction of particles has been taken with their impact to a solid butt made of wolfram carbide after outputting of cavity tube of the WJM.

The paper has been supported by Czech Grant Agency (GAČR) during the project No. GAČR 106/08/1092.



Fig. 1. Water Jet Mill (WJM)

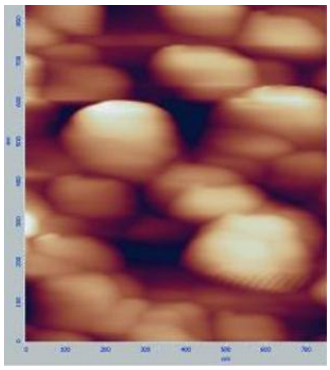


Fig. 2. Silicon nanoparticles created by the WJM technology

Keywords: nanoparticle, disintegration, cavitation, milling

Electrophoretic size determination of gold clusters from Au magic numbered clusters to Au nanoparticles

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The physicochemical properties of nano-sized materials are highly sensitive to their size and shape so that isolation of the particles as a function of size and the determination of the size as well as shape is the first step of characterization of these substances of current interest. Polyacrylamide Gel Electrophoresis (PAGE) is a simple and powerful tool both for isolation and for the determination of the molecular weight of protein and of DNA, and the methodology is well established in biochemistry. However, no trial is reported so far to determine the size of nano-particles solely from a wet chemical method.

In this paper, we present how the PAGE technique is applied to water soluble gold thiolate cluster and nano-particles and show usefulness of this method to determine the size as well as shape of metal nanoparticles without the use of mass spectrometric technique or transmission electron microscopy, both of which need sophisticated instrumentation or a special technique of vacuum. As a reference material, we accepted glutathione reduced form (GTR) and made more than twenty Au-GTR species in isolated form. After about 5 hr PAGE-operation, the gel pattern shows well isolate series of bands as shown in Figure 1. The composition of each band was analyzed by ESI mass spectrometry and correlated with the Rf-value from PAGE. It was found that there is a good relationship between logarithm of the number of gold atoms determined by ESI method and by TEM observations and Rf-values. In addition, close examination of the plot revealed that much better correlation was attained by plotting log m vs. Rf, in which m is the number of GTR molecules adsorbed on a cluster. The inclination of curve A is much larger than that of B, suggesting that the conformation or composition of GTR changes at around Au40.

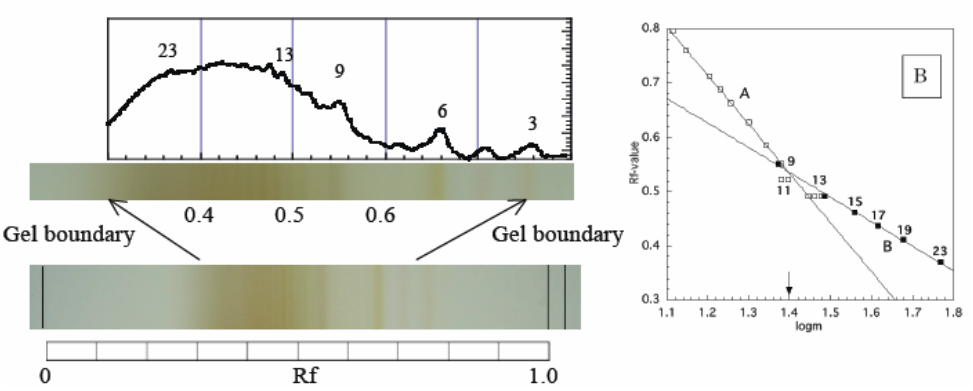


Fig. 1. PAGE pattern and the numbering of the fraction bands. Lower panel shows a whole pattern from top of resolving gel of 16 % T to the bottom gel boundary of 26 % T. Middle panel is expansion of panel A. Upper panel shows the digitized data of the concentration of the band in which numbers stand for the precise position of each band.

Fig. 2. Relationship between relative mobility Rf and logarithm of m.

Keywords: PAGE, gold cluster, nanoparticles, GTR

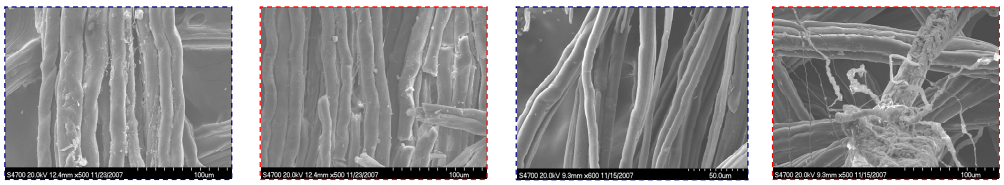
Preparation and characterization of micro- and nano-fibrils from natural fibers I. jute

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Jute fibers are bundles of individual strands held together by binders. In order to prepare micro-fibrils and nano-fibrils from jute, the binders have to be cleaned. A new technique including chemical (room temperature alkaline, special acid, and 80°C alkaline) and physical (high pressure) treatments of jute fibers was developed. The effects of chemical and physical treatments on the morphological development of jute fibers from the micro- to the nano-scale were observed using scan electric microscopy (SEM), as shown in Fig. 1. This novel natural fiber treatment technology has two advantages compared with others. One is the long strands of jute fibers can be kept their length by special acid treatment (Fig. 1a), but the traditional acid treatment shorten the length of jute fibers (Fig. 1b). Another one is the high pressure treatment made the jute fibers nano-fibrils (Fig. 1d). Before the high pressure treatment the micro-fibrils can be obtained (Fig. 1c). The thermal property of before and after treated fibrils was determined using thermogravimetric analysis (TGA), as shown in Fig. 2. It indicates that after treatments, the thermal stability was enhanced.



(a) (b) (c) (d)

Fig. 1. Chemical and physical treatments of jute fibers (a) after special acid, (b) after traditional acid, (c) after 80°C alkaline, and (d) after high pressure.

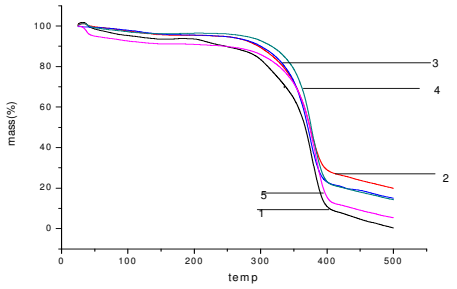


Fig. 2. TGA curves of jute fibers (1) original, (2) after room temperature alkaline, (3) after special acid, (4) after 80°C alkaline, and (5) after high pressure.

Keywords: jute fibers, nano-scaled fibers, natural fibers, chemical treatments

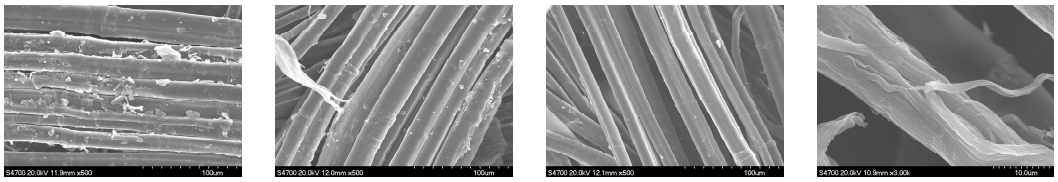
Preparation and characterization of micro- and nano-fibrils from natural fibers II. flax

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Flax fibers are a type of bundled natural cellulose fibers. The effects of chemical (room temperature alkaline solution and special acid) and physical (high pressure) treatments on the formation of micro- and nano-fibrils were characterized using scanning electric microscopy (SEM). On the surface of flax fibers there are a lot of binders conglutinating tiny fibrils (Fig. 1a). The binders can be removed by alkaline treatment mostly (Fig. 1b) and further removed by special acid treatment. Then the micro-fibrils were obtained (Fig. 1c). The high pressure treatment made fibrils nano-sized (Fig. 1d). The chemical compositions of the binders cleaned off by alkaline and special acid treatments after carefully collection and drying were analyzed using fourier transform infrared spectrometer (FTIR), as shown in Fig. 2. They are mainly lignin after alkaline treatment and mainly hemicelluloses after special acid treatment.



(a) (b) (c) (d)

Fig. 1. Chemical and physical treatments of flax fibers (a) original, (b) after alkaline solution, (c) after acid steam, (d) after high pressure steam.

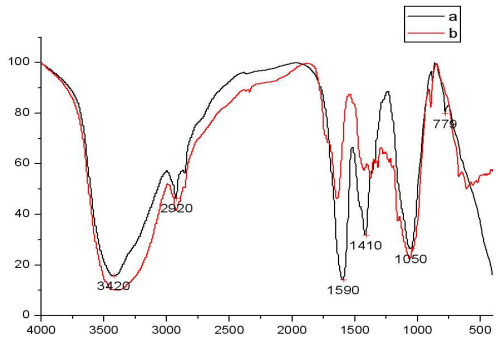


Fig. 2. TFIR spectra of binders cleaned off by chemical treatments (a) after alkaline and (b) after special acid.

Keywords: flax fibers, nano-scaled fibers, cellulose, chemical treatment

Preparation and characterization of micro- and nano-fibrils from natural fibers III. hemp

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Hemp fibers are a kind of natural cellulose fibers. The nano-fibrils can be prepared from hemp fibers after chemical (room temperature alkaline solution and special acid) and physical (high pressure) treatments. Nano-fibrils were obtained after special acid treatment and the morphology of the nano-fibrils was characterized using scanning electric microscopy (SEM), as shown in Fig. 1. It was proved that the special acid treatment is a novel technique compared with the conventional acid treatment. The thermal stability of the nano-fibrils analyzed using thermogravimetric analysis (TGA) was enhanced compared with the original hemp fibers, as shown in Fig. 2. The structure of treated and untreated hemp fibers was analyzed using fourier transform infrared spectrometer (FTIR), as shown in Fig. 3. It indicates that after treatments the amount of the binders, lignin and hemicelluloses, were increased.

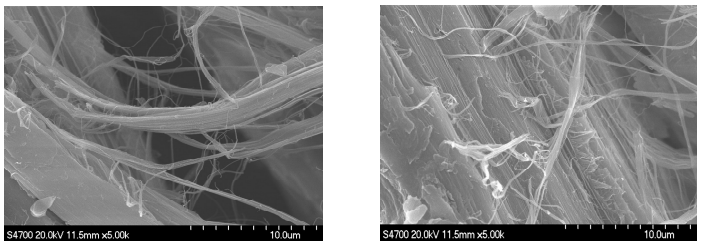


Fig. 1. Nano-fibrils prepared after special acid treatment.

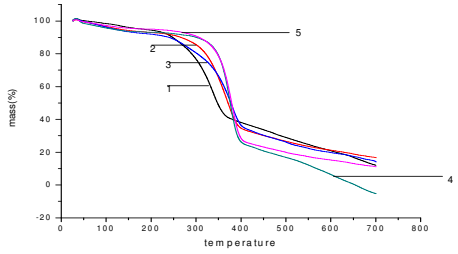


Fig. 2. TGA curves of hemp fibers (1) original, (2) after room temperature alkaline, (3) after special acid, (4) after 80°C alkaline, and (5) after high pressure.

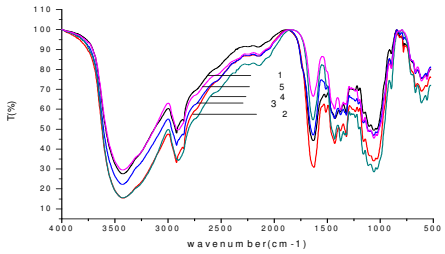


Fig. 3. FTIR spectra of hemp fibers (1) original, (2) after room temperature alkaline, (3) after special acid, (4) after 80 °C alkaline, and (5) after high pressure.

Keywords: flax fibers, nano-scaled fibers, cellulose, chemical treatment

Delamination of vermiculite particles using ultrasonic treatment

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The present paper deals with the thermal ultrasonic treatment on vermiculite. Expanded Mg-vermiculite purchased from fy. Aldrich (dry sieved the particles size fraction < 100 μm) was used for investigation of exfoliation effect after hydrogen peroxide and/or ultrasonic treatment at 70°C. After 60 or 180 minutes treatment of powdered Mg-vermiculite sample in water solution or in 10 %, 30% and 50 % hydrogen peroxide solution, the reduction of the peaks intensity of the 001 diffraction were observed. Particle size distribution (PSD) of fine, ultra-fine and nanostructured samples is taken on dispersed particles in available medium-water using the low-angle laser method of PSD (Analysette C22). To describe mechanism of exfoliation, the content of Na, Ca, Mg, K, Si and Al in water solution after treatments were determined by the atomic emission spectrometry with inductive couplet plasma (JY-24). In comparison with the results in paper [1] more effective was the ultrasonic treatment in 50 % hydrogen peroxide solution.

The financial support from the projects MSM 6198910016 and GAČR 205/08/0869 is gratefully acknowledged.

Reference:

[1] Z. Weiss, M. Valášková, J. Seidlerová, M. Šupová-Křístková, O. Šustai, V. Matějka, and P. Čapková, *Journal of Nanoscience and Nanotechnology* 6 (2006) 726.

Keywords: exfoliation, ultrasonic treatment, Mg-vermiculite, hydrogen peroxide, ICP, XRD

Nanocrystalline silicon and its interaction with bio-environment

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The presented work is aimed at the development of nontoxic nanocrystalline silicon fluorescence marks, that are biodegradable in living body. This new fluorescence marks could by a very good substitute for commercially used quantum dots (e.g. cadmium compounds quantum dots) which are toxic according to the latest results. We prepare porous nanocrystalline silicon (por-Si) by the electrochemical etching of Si wafers in HF solutions, with possibility to tune the luminescence peak through the visible spectrum.

The behavior of por-Si (Si nanocrystals with diameter about 3 – 5 nm), pulverized silicon (crystallites of mean diameter of about 1μm) in biological environment (L929 mouse fibroblast and HeLa cells) is studied by optical transmission microscopy, time-lapse microphotography of cell culture evolution, fluorescent microscopy, fluorescent microspectroscopy, and scanning electron microscopy (SEM).

Por-Si with different fluorescence spectra are not having significantly negative effect on evolution of the cell culture. At high concentration of pulverized silicon we see meronecrosis, which is caused by strong mechanical stress of the cell outer membrane derivable from incorporated pulverized silicon. Por-Si has had also mildly negative effect on the evolution of the cell culture at very high concentrations, but cells are viable and cell division proceeds normally – the next cell generation shows normal behavior. Fluorescence microscopy applied on orange emitting por-Si shows that por-Si is concentrated probably on cell membranes and its presence in internal cell environment is also proved.

Keywords: silicon nanocrystals, fluorescence, microspectroscopy, biocompatibility

Electrochemical migration of electronic components at sea environments – theoretical study

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Seawater is associated with two main effects, high humidity and chloride ions, that have direct influence on electrochemical migration occurrence in electronic components which might eventually lead to short circuit failure. This phenomenon occurs when metal ions migrate from anode electrode to cathode, via electrolyte, under bias voltages. Other factors that contribute to cause this phenomenon are electrolyte composition (halide ions), pH, and temperature; in addition to the chemical composition of the substrate, such as printed circuit board. Moreover, polarization effects, and the distances between the electrodes is another parameter.

In order to reduce the possibility of electrochemical migration, this study has tried to collect information and data from other literatures, to analyze and investigate the interactions between the factors that affect this phenomenon. The main concept in this study is characterization the interactions and interferences between the chemical composition of printed circuit boards, i.e. resin : glass ratio, and ability for absorption, which changes their dielectric properties, and capacitance, and showing how they affect electrochemical migration of electronic devices at seawater.

The survey showed that acidic solution causes electrochemical migration more than basic solutions, also chloride ions, contaminants, and bias voltage will accelerate the phenomenon. Moreover, by interpreting the effects of each parameter individually and discussing the interferences between all the factors together, this study could show that the chemical composition of printed circuit boards has an influence on the absorption rate, and changing the dielectric constant of the insulator material between conductors, which act to increase the capacitance of the substrate, and subsequently encouraging ion migration.

Keywords: electrochemical migration, printed circuit board, absorption rate, dielectric constant

New nanohybride materials for magnetic fluid hyperthermia

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Among various methods of the oncologic therapeutic methods there is a continuous increase of an interest about the magnetic fluid hyperthermia (MFH).
The method is based on an intratumoral deposition of stable and nontoxic suspensions of the magnetic nanoparticles followed by a short-time exposure to a high frequency electromagnetic field. The dissipation of energy connected with magnetic losses results in a local heating by the active particles and consequently to the destruction of the cancer cells.
Two kinds of magnetic heating and consequently of the materials should be distinguished. Superparamagnetic particles, where heating is effectuated by rotation of the particles magnetic moments and ferro- or ferrimagnetic particles, where heating is due to hysteresis losses. Successful use of the latter type requires maintenance of the magnitude of magnetization for small particles as high as possible and adjustment of the coercivity to a reasonable value assuring sufficient heating efficiency but not too high for practical medical heating application.
Simultaneously the temperature coefficient of magnetization providing self-controlled heating mechanism in the range of ~ 40–70° C is desirable. It is obvious that these conditions can be hardly fulfilled by currently used simple iron oxides, magnetite and maghemite. A possible approach to solve this task is the use of complex magnetic oxides, where the properties of nanoparticles cores can be properly tailored by various ways, like:

a) modification of the intrinsic properties depending on the composition and structure,
b) modification of the extrinsic properties like particle size depending on the synthesis procedure and alternatively employing composite multiphase materials.

In fact these approaches are usually combined and we have investigated the outlined possibilities on the following magnetic oxides exhibiting typical distinct features, $CoFe_2O_4$ cobalt spinel, Sr-hexaferrite/maghemite composites and $La_{1-x}Sr_xMnO_3$ manganese perovskites.
The syntheses of the nanoparticles via wet chemistry methods and by subsequent thermal treatment and relations between magnetic properties and the heating efficiency will be presented.
Particular attention was paid to $La_{0.75}Sr_{0.25}MnO_3$ (LSMO) manganese perovskite where a reproducible method of the encapsulation by silica in order to ensure water colloidal stability of the cores and to suppress the toxicity caused by the presence of manganese ions was developed. The viability tests carried out on the culture medium of rat mesenchymal stem cells and loading of the cells by LSMO@SiO_2 will be shown.

The support by projects ASCR KAN20020061, KAN201110651, 1QS100100553, MSMT1M0538, GACR309/06/1594 is gratefully acknowledged.

Keywords: magnetic nanoparticles 1, complex oxides 2, encapsulation 3, hyperthermia 4

Study of Pd/InP interfaces for hydrogen detection

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For preparation good performance sensor it is necessary to prepare high-quality Pd/InP interface with high Schottky barrier. In this sense it is not convenient to prepare this interface by high energetic means of metal deposition because it leads to Schottky barrier decrease induced by Fermi level pinning explained by disorder-induced gap states (DIGS). Our effort is to develop reproducible method for preparation such interfaces with the highest Schottky barrier possible.

For the deposition we used electrophoretic deposition method which put Pd nanoparticles from colloid solution on the InP wafer. This process occurs in a presence of electromagnetic field, which accelerate Pd nanoparticles in the direction to the wafer.

We prepared several interfaces by this method. These structures were analyzed by current-voltage measurements, capacitance-voltage measurements, impedance measurements, AFM, and secondary mass spectroscopy. This measurements show the good rectifying character of this structure and the Schottky barrier high calculated from I-V curves was about 1eV. We also tried to improve properties of this interface by annealing which had positive impact on Schottky barrier high.

This talk is to refer about the progress in the project of central goal to find reproducible method for fabrication Pd/InP interfaces with high-quality properties.

Keywords: Pd nanoparticles, Schottky barrier, gas sensor, electrophoretic deposition

N_2O catalytic decomposition - effect of pelleting pressure on activity of the Co-Mn-Al mixed oxide catalyst

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The effect of pelleting pressure during preparation of the Co-Mn-Al mixed oxide catalyst on its activity for N_2O catalytic decomposition was examined in this study. Although increase of pelleting pressure caused an increase in bulk density and a decrease of pore size and pore volume of the prepared catalyst (resulting in lower values of N_2O effective diffusion coefficient), no direct correlation between pelleting pressure used and catalyst activity has been found. The temperature dependences of N_2O conversions are shown in Fig. 1. Volume of micropores of about 20 mm^3 g^{-1} was evaluated using modified BET equation. This value practically did not change with increasing pelletization pressure excepting the sample formed under pressure of 10 MPa. Estimation of effectiveness factor for spherical particles showed that effectiveness factor of about 0.07 and 0.2 can be obtained for spheres with radius of 1.5 and 0.5 mm, respectively. An appropriate pelleting pressure of about 6 MPa should be used for the catalyst preparation.

This work was supported by the Czech Ministry of Education, Youth and Sports (MSM 6198910016 and 6046137302) and by Czech Science Foundation (104/07/1400).

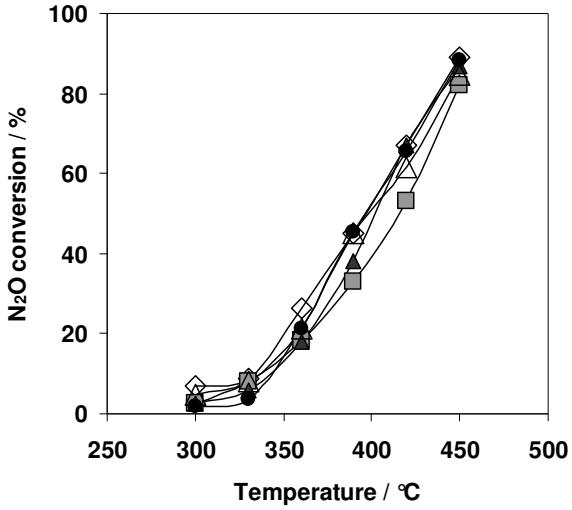


Fig. 1. Temperature dependence of N_2O conversion over Co-Mn-Al spinel catalyst formed to tablets at different pressure: 0 MPa (◇), 4 MPa (■), 6 MPa (▲), 8 MPa (△), 10 MPa (●). Conditions: 1000 ppm N_2O balanced by He, space velocity 60 l g^{-1} h^{-1}

Keywords: pelleting pressure, nitrous oxide, catalytic decomposition, mixed oxide

Size dependency of nanocrystalline TiO_2 on photocatalytic reactivity exemplified by CO_2 photoreduction

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The reduction of CO_2 by photocatalysts is one of the most promising methods since CO_2 can be reduced to useful compounds by irradiating it with UV light at room temperature and ambient pressure. The aim of this work is to assess the effect of particle size on photocatalytic reactivity of TiO_2 exemplified by the photoreduction of CO_2 by water. As the particle size decreased higher methane yields over the TiO_2 nanoparticles under the illumination of the light were obtained. Results were compared with yields of methane over Degussa P25, which is non-porous. We demonstrated that photoreactivity of Degussa P25 was lower than all tested TiO_2 nanoparticles.

The financial support of the Czech Ministry of Education, Youth and Sports, research project MSM 6198910019 and AS CR Program Nanotechnology for Society (KAN 400720701) is gratefully acknowledged.

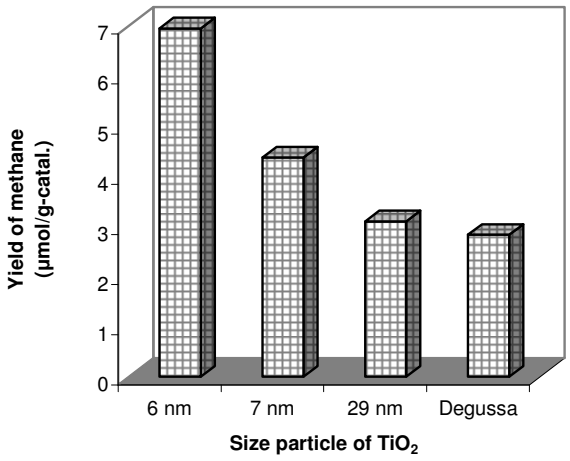


Fig. 4. Comparison of methane and methanol yields (24 h) over different particle size TiO_2 and Degussa P-25.

Keywords: Carbon dioxide, Photocatalysis, Reduction, Titanium dioxide, Size of Particle

The effect of reaction conditions for sol-gel synthesis of TiO_2 with photocatalytic properties

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The existing and promising applications of TiO_2 nanomaterials include paint, toothpaste, UV protection, photocatalysis, photovoltaics, sensing, and electrochromics as well as photochromics. TiO_2 is regarded as the most efficient and environmentally benign photocatalyst, and it has been most widely used for photodegradation of various pollutants[1,2]. Doping of the TiO_2 with various metal elements (Cu, Ag, Fe, …), with nitrogen or carbon or their combination, as well as preparation of binary mixtures of TiO_2-MeO_x or TiO_2-MeS_x significantly influence the photocatalytic activity in comparison with pure titanium dioxide[3].

Sol-gel is successful technique for preparing of nano-sized metallic oxide with high photocatalytic activity[4]. For preparation of TiO_2 (pure and Cu doped) the hydrolysis of titanyl sulphate ($TiOSO_4$) precursor was used. The effects of reaction conditions (temperature, amount of water for hydrolysis) were investigated by hydrolysis of $TiOSO_4$ (industrial grade). The photocatalytic activity of prepared samples against methylene blue (MB) was determined after UV irradiation (365 nm, 2 mW/cm^2). The prepared samples were characterized using X-ray powder diffraction method.

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Keywords: titanium dioxide, titanyl sulphate, thotocatalyst, Cu-doping

Acid orange and methylene blue as model substances for evaluation of photodegradation ability of composites clay/TiO_2

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Titanium dioxide (TiO_2) belonging to the family of transition metal oxides is commonly used as a white pigment (approx. 51% of total production), in plastic industry (19 %), paper industry (17 %), other minor end-use sectors are textiles, foods, pharmaceuticals … . TiO_2 exists in three stable modifications: rutile, anatase and brookite. From these modifications anatase or mixture of anatase/rutile are reported as the most efficient photocatalysts for photodegradation of pollutants.
The natural clay minerals (e.g. kaolinite, montmorillonite, vermiculite) represent suitable matrix for growing of nanosized highly photoactive TiO_2. This particles are anchored in the interlayer space of montmorillonite, vermiculite or grafted on the surface of these clays (kaolinite) and can be used as material with photo-functional properties

In this work we focused on the comparison of photodegradation ability of the kaoline/TiO_2 composite against selected model pollutants. Photocatalytic activity of prepared composites was evaluated by the degradation of methylene blue (MB) and acid orange 7 (AO7). Basically the water suspension of tested composite and selected pigment is prepared. After the 60 min long adsorption in the dark tested suspension are exposed to UV irradiation (365nm). The discoloration of the suspensions due to the photocatalytic degradation is determined using UV-VIS spectrophotometer.

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Keywords: titanium dioxide, acid orange 7, kaoline, methylene blue

Fluorescent resonance energy transfer in the systems based on layered inorganics and organic dyes

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We have studied the systems based on layered inorganic templates with adsorbed (intercalated) organic dyes. Our objective was to prepare such materials which would exhibit energy transfer process. This is not a simple task, since adsorption of the organic molecules on solid templates leads to the formation of molecular aggregates and often to complete depletion of luminescent properties. Fluorescence spectroscopy indicated the intermolecular interactions based on non-radiant energy transfer taking place between isolated dye cations and supramolecules. Various systems were investigated based on the energy transfer between two rhodamine dyes, between rhodamine and oxazine dye, porphyrin and phthalocyanine dye, etc. Basic system presents the energy transfer from rhodamine to oxazine. Molecules of rhodamine play a role of molecular antennas absorbing green light (λ=500-520 nm). Light absorption was accompanied with a non-radiant energy transfer to the molecules of the second dye, oxazine. The transfer was detected as a quenching of the light emission from the rhodamine (580 nm) in favour of the red light luminescence from oxazine (near 635 nm). Oxazine had not been directly excited by green light and did not emit in the absence of rhodamine. The phenomenon indicates the possibility of application of inorganic layered materials to mediate the transfer of electromagnetic energy between adsorbed dye cations. The results of our research could contribute to the development of novel types of hybrid materials potentially useful for laser or optical technologies.

This work was supported by the Slovak Research and Development Agency (No. APVV-51-027405) and VEGA agency (2/6180/27).

Keywords: energy transfer, layered silicates, laser dyes

Molecular assemblies of cationic porphyrin dyes in colloidal dispersions of layered silicates

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Supramolecular assemblies of organic dyes have been extensively investigated. Molecular assemblies are important for the processes such as light harvesting and energy transfer, intermolecular interactions requiring limited degree of freedom, etc. The objective of this work was to compare molecular aggregation of cationic porphyrin dyes in colloidal dispersions of layered silicates. Three silicates specimens were tested, two synthetic hectorite samples (high and low charge densities) and montmorillonite. For specific experiments related to the influence of the layer charge, series of reduced change montmorillonites was synthesized. The interaction with two porphyrin dyes based on tetracationic (*meso*-tetrakis(N-methylpyridiniumyl)porphyrin) and dicationic molecules *meso*-bis(*N*-methylpyridiniumyl)porphyrin were studied by means of UV/VIS absorption and fluorescence spectroscopies. Tetracationic porphyrin did not extensively form molecular assemblies in the colloids at low loadings and concentrations. Only small amounts of the molecular aggregates were detected in the colloids of the highest charge silicate, fluorohectorite. This phenomenon can be interpreted in terms of the repulsion electrostatic forces preventing the self assembly of the porphyrin tetracations. Porphyrin dication with trans-configuration of the cationic groups in the molecule exhibited higher tendency to form molecular assemblies. This was confirmed by the changes in absorption spectra in both Soret and Q-bands regions. The spectral changes exhibited direct relation to the layer charge of the silicates. Fluorescence did not exhibit qualitative changes, but the emission yields seemed to be related to the silicate properties. Fluorescence measurements indicated dicationic porphyrin formed mainly J-type assemblies with a head-to-tail association.

This work was supported by the Slovak Research and Development Agency (No. APVV-51-027405) and VEGA agency (2/6180/27).

Keywords: aggregates, porphyrin, layer charge, fluorescence

Synthesis and characterization of novel organoclay prepared from vermiculite and Chlorhexidine Acetate

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It is well known that a number of clay minerals have a layer structure which can accept organic and/or inorganic cations within their interlayer space by an exchange reaction. The study of organoclay-based nanocomposites has made great progress, in which organoclays were synthesized by modifying clay minerals with surfactants. These nanocomposites often exhibit remarkable improvement in materials properties when compared with virgin organic compound.

Chlorehexidine Acetate (abbreviated as CA hereafter) is a kind of bivalent cationic surfactant with efficiency as an antibacterial ion. CA has been widely used for diminishing inflammation, disinfecting, washing surface of a wound.
There are only a limited number of reports about CA/layered silicate nanocomposites based on montmorillonite but no based on vermiculite.

A series of novel organoclays were synthesized using Na-vermiculite and Chlorehexidine Acetate (CA) by ion-exchange. The structure of resultant organoclays were characterized by X-ray powder diffraction (XRD) and Fourier transform infrared spectroscopy (FTIR).

This work was supported by the Czech Grant Agency (project GAČR No. 205/08/0869), and by the MSMT of the Czech Republic (MSM 6198910016).

Keywords: chlorhexidine acetate, vermiculite, organoclay

Changes in visible and luminescence spectra of RCM complexes with methylene blue

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The montmorillonites (M) with reduced charge (RCM) were obtained by heating of Li saturated M at temperatures 40, 130 and 210°C. The cation exchange capacity (CEC) and X-ray diffraction pattern (d_{001}) decrease with increasing temperature of RCM preparation, i.e. with decreasing charge of RCM. These samples were used to study visible and luminescence spectra of methylene blue complexes (MB/RCM). Their fluorescence intensity increases with decrease of RCM charge, dye loading and with decrease of methylene blue concentration in equilibrium aqueous solution. The fluorescence intensity of fully saturated MB/RCM complexes is very poor while that one for unsaturated MB/RCM complexes is high. In the visible spectra of MB/RCM suspensions three absorption bands (monomer, higher aggregates and protonated cations) of MB dye were observed. Usually with increasing time after MB/RCM formation the changes among concentrations of dye higher aggregates – monomer and protonated dye cation (and/or J-aggregates) were observed. The visible spectra of MB/RCM suspensions also depend on charge of RCM. In MB/RCM suspensions prepared from RCM with higher charge (i.e. prepared at lower temperature) higher agglomerates or dimers of MB prevail. Contrary in suspensions prepared from RCM with less charge (i.e. prepared at higher temperature) methylene blue prevails in monomeric form.

The authors are grateful for support from project of Ministry of Education of the Czech Republic MSM 6198910016.

Keywords: reduced charge montmorillonite, methylene blue, visible and fluorescence spectra

Controlling the nano- and microstructure in POSS containing epoxy networks

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In this contribution, we present organic-inorganic materials. The epoxy-amine network based on diglycidyl ether of Bisphenol A and poly(oxypropylene)diamine (Jeffamine D2000) was modified with several types of well-defined nanobuilding blocks – polyhedral oligomeric silsesquioxanes (POSS). The nanosized POSS unit (diameter ~ 1.5 nm) serves as a nanofiller in the organic matrix. The epoxy- or amino- functionalized POSS monomers were covalently attached to the epoxy network as pendant blocks or as junctions of the network.

The hybrid structure and morphology were controlled by synthesis procedure (one-step or two-step), varying type of POSS organic substituents (isooctyl-, isobutyl and phenyl) and number of functional groups in POSS monomers (mono-, poly- and nonfunctional POSS). The two-step synthesis consisting in preparation of the adduct DGEBA-POSS in the first stage provides more homogeneous organic-inorganic network. The POSS with phenyl substituents shows a very strong POSS-POSS interaction and a tendency to aggregation of crystallites within the matrix. On the contrary, POSS with flexible isooctyl substituents forms only small amorphous aggregates.

The structure of the modified networks was determined by using chemorheology experiments, by SAXS and WAXS. Thermomechanical properties were followed by dynamic mechanical analysis (DMA).

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Intercalation of clay materials with inorganic cations

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This work is focused on basic research of intercalated clay materials characteristics - synthesis, structure and properties of the nanomaterials based on intercalated fylosilicates. Intercalation of inorganic and organic compounds into the texture of clay materials is studied intensively because the texture and quality of these intercalated materials are of a great significance in the field of chemical industry and also in the environmental pollution, since the quantity of waste and toxic compounds in the environment is still increasing. The research focused on intercalated materials can notably help to capture these dangerous materials by means of the sorption of e.g. trace elements (Cu, Hg, Pb, Cd, Zn etc., including radioactive Cs and Sr tracers) into the texture of montmorillonites. Currently the particular attention is paid to the study of the possibility of intercalation of some big cations, such as Keggin cation, into the clay material structure. In this contribution the attention is paid to Keggin $[Al_{13}O_4(OH)_{24}(H_2O)_{12}]^{7+}$or another polycations intercalation into structure of the clays in order to describe the modified nanomaterials. This cation was prepared by titration of $AlCl_3$ with NaOH solution with different OH/Al ratio (1.0; 1.6; 2.0; 2.3 and 2.5) and consequently it has been removed by sulphate sodium. In aqueous intercalating solutions there are various hydroxyl- and oxy-hydroxyl Al(III) forms. The existence of this cation and another forms has been verified at different pH and its influence on intercalates characteristics has been studied by NMR, IR-spectroscopy, X-ray diffraction, thermal analysis, etc. Intercalation of Keggin cation has been performed into the Na-clay (SWy-2) with CEC 0.7002 mmol/g and sorption curves at five different pH (1.5; 2.0; 2.5; 3.0 and 3.8) were obtained. Maximal sorption capacity obtained at all these pH has reached 2.5 mmol/g.

The authors thank to Grant Agency of Czech Republic (project 104/08/P274) and Ministry of Education (project MSM 619 8910016) for financial support, which permitted to conduct this study.

Keywords: Keggin cation, intercalation, NMR analysis, IR spectroscopy

Vermiculite filler for polymeric nanocomposites

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Layered fillers for polymeric nanocomposites represent still area with high interest from business and therefore the scientific sphere as well. The demand of nanocomposites for filler properties which is expected utilization. Natural minerals are keeping one of the prior positions in that significance. The thermal properties and strength of the requested properties expected to be improved for polymeric materials. Modification of that layered materials is still necessary to assure the compatibility of the filler with polymer with non-polar character.

The organically modified montmorillonite is still the most frequently studied mineral for the application as the filler. Comparing montmorillonite and vermiculite we can note the difference in layer charge of silicate layer. Thus the vermiculite with higher charge could be more stable. Modification of jet milled vermiculite with amine and phosphonium salts using thermal treatment was performed using 10% of organic amount. The test of PS compatibility was done for vapor sorption in closed container for several days. The sample after sorption was polymerized and the changes of interlayer space were studied using X-ray diffraction analysis.

Keywords: nanocomposite, nanofiller, vermiculite

Study $CuZn_{39}Pb_2$ brass deformation

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During fastening of Brass Bodies containing temperature sensor to the engine the cracks are developed. Position and feature of cracks depend on both manufacture process and composition of Pb in the alloy. X-ray diffraction analysis in combination with SEM+EDS elemental analysis revealed the mixture of ductile α-grains and brittle β-grains. Spherical and platelet Pb precipitates are observed for 2 wt.% and 3 wt.% respectively. Raw material entering metallurgical process meets one of two standards: CuZn39Pb2 F44 according to DIN 17 672 and CuZn39Pb3 R430 according to DIN EN 12164. Besides that a prior orientation of grains after mechanical stress beyond elasticity limit is observed. Influence of ambient temperature processing on rearrangement of grains is discussed.

Keywords: brass, heat treatment, grains orientation, stress

Clay mineral mixtures for porous cordierite ceramics

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The cordierite ceramics due to its low thermal expansion coefficient, high chemical durability, low dielectric constant and high resistance to thermal shock are promising candidates in many applications. The conventional methods for their synthesis include the solid-state sintering of individual magnesium, aluminum and silicon oxides corresponding to the chemical composition of cordierite, $2MgO.2Al_2O_3.5SiO_2$, crystallizing the glasses or the sintering of the natural raw materials. The monophase cordierite ceramic is difficult to prepare and in the $MgO–Al_2O_3–SiO_2$ system and very often occurs with spinel, cristobalite, mullite, forsterite, periclase, and corundum. Commercial cordierite can be sintered at temperatures in excess of 1300 °C with the help of alkali oxides, typically K_2O, that promote liquid phase sintering. The properties of porous ceramics greatly depend on pore morphology, size and distribution.

Cordierite was synthesized from four different mixtures, prepared based on the clay minerals kaolinite $2SiO_2.Al_2O_3.\ 2H_2O$ and talc $4\ SiO_2.\ 3MgO.H_2O$, and from those mixtures with vermiculite $4\ (Si,Al)O_{2,3}\ 3\ MgO.H_2O$. Firing the specimens at tube furnace in Ar atmosphere at 1300 °C for 1 h yielded cordierite ceramics with open pores. Enstatite $MgSiO_3$ and/or spinel $MgAl_2O_4$ and forsterite Mg_2SiO_4 formed dependently on deviation from equilibrium $MgO–Al_2O_3–SiO_2$ system.

The research was supported by projects: Ministry of Education, Youth and Sport of Czech Republic (ME08040), Ministry of Education of the Czech Republic (project MSM 6198910016) and the Czech Grant Agency (projects GA ČR 205/08/0869).

Keywords: cordierite, vermiculite, ceramics, X-ray powder diffraction

Nanocrystalline Fe thin films on Si(100) substrate

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The different effects of the substrate are believed to be due to interfacial being due to lattice mismatch between films and substrates. Among the interesting properties of Fe thin films, magnetic anisotropy has attracted a lot of attention, mainly the existence of a positive (perpendicular) anisotropy. This anisotropy is explained either by a reduced Fe magnetization due to interdiffusion which lowers the shape anisotropy or to a contribution of stress. Moreover, the relation between the structural and magnetic properties of thin films is still an interesting topic in thin film magnetism, even in polycrystalline and relatively thick films.

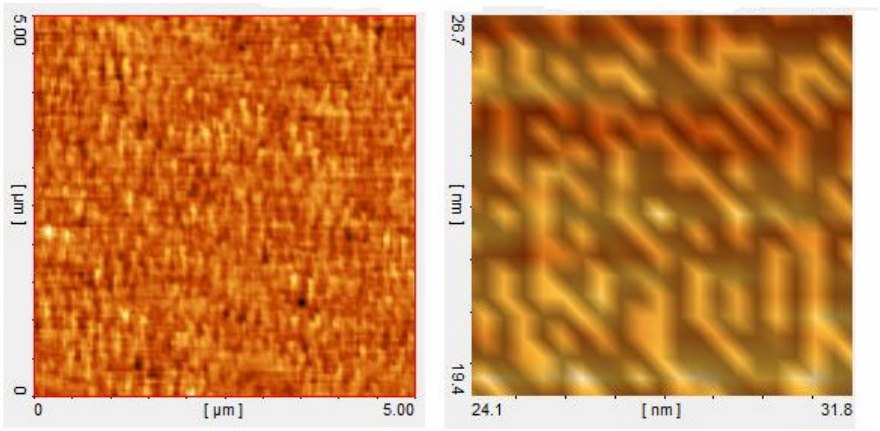


Fig. 1. The AFM morphology of surface (left) and MFM magnetic domain (right) images of 100nm, Fe/Si(100) thin film.

In this paper, the nanocrystalline, structural, magnetic properties and domain structures of evaporated Fe thin films on Si(100) substrate are reported. AFM technique has been used to study the roughness of the surface of these Fe films. An example of AFM image is shown in Fig. 1(Left), for the 100 nm thick Fe/Si(100) sample. Root Mean Square value for this sample is found 1.04nm.
It is well known that the extrinsic magnetization behaviors of a ferromagnetic material depend strongly on its domain structure. MFM is one of the most used techniques for imaging magnetic domains. Fig. 1(Right) shows an MFM image taken from 100 nm thick Fe/Si(100) sample in the permanent state. One can see, in this image, bright and dark areas that can be regarded as corresponding to the position of inner down and up domains. The domain wall of the wide basic domain reflects in the MFM images as a transition region of the dense stripe domain. The inhomogeneous stress relaxation during the annealing treatment may account for the appearance of the metastable domain structure, which turns to a stable single-domain state after magnetization.

Keywords: nanostructures, thin films, root mean square, magnetic anisotropy

Silver nanoparticles in titanium dioxide matrix exhibiting multicolour photochromic effect

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Silver and titanium dioxide nanoparticles are widely studied because of their indispenseble contribution to photocatalysis, photovoltaics and antibacterial materials. In 2001 unique inorganic composite Ag-TiO_2 material exhibiting multicolor photochromic behaviour was invented [1]. The material consists of silver nanoparticles embedded in titanium dioxide matrix. The extinction spectrum of the samples is inhomogeneously broadened over the whole visible spectral region due to different shapes and sizes of the silver nanoparticles. The photochromic response is caused by an optical deactivation of the selected silver nanoparticles under monochromatic irradiation. After irradiation the originally brownish grey samples seem to take the color of the incident light.

We implemented the method of the preparation of the samples by the UV photocatalytic reduction of silver ions from the aqueous solution of $AgNO_3$ into porous titanium dioxide matrices prepared on glass substrates by spin-coating of ethanol solution of the commercial titanium dioxide nanopowder (Degussa Aerosil P90). We optimized the preparation procedure in order to increase the photochromic response.

The samples were studied under various conditions of irradiation (cw, pulse laser irradiation with different fluence intensities). The results help to reveal the microscopic background of the observed extinction changes.

The foreseen applications of this nanomaterial are e.g. multiwavelength optical memories or large displays.

Reference:

[1] Y. Ohko, et al., *Nat. Mater.* 2 (2003) 29.

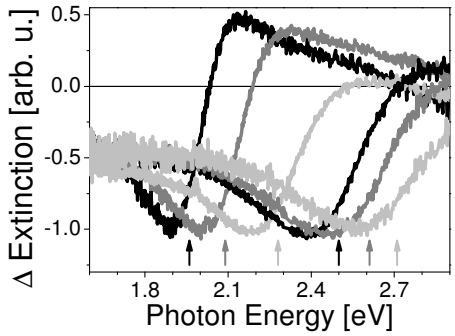


Fig.1. Illumination induced changes in optical extinction spectra of the Ag-TiO_2 photochromic film caused by six different lasers. The spectral positions of the lasers are indicated by the arrows.

Keywords: silver nanoparticles, titanium dioxide, photochromism, optical spectroscopy

Domain walls in ferromagnetic metallic-semiconductors nanostructures

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Experimental results of domain walls for evaporated Fe on Si(111) and Si(100) has been reported. The results are shown in figure below.

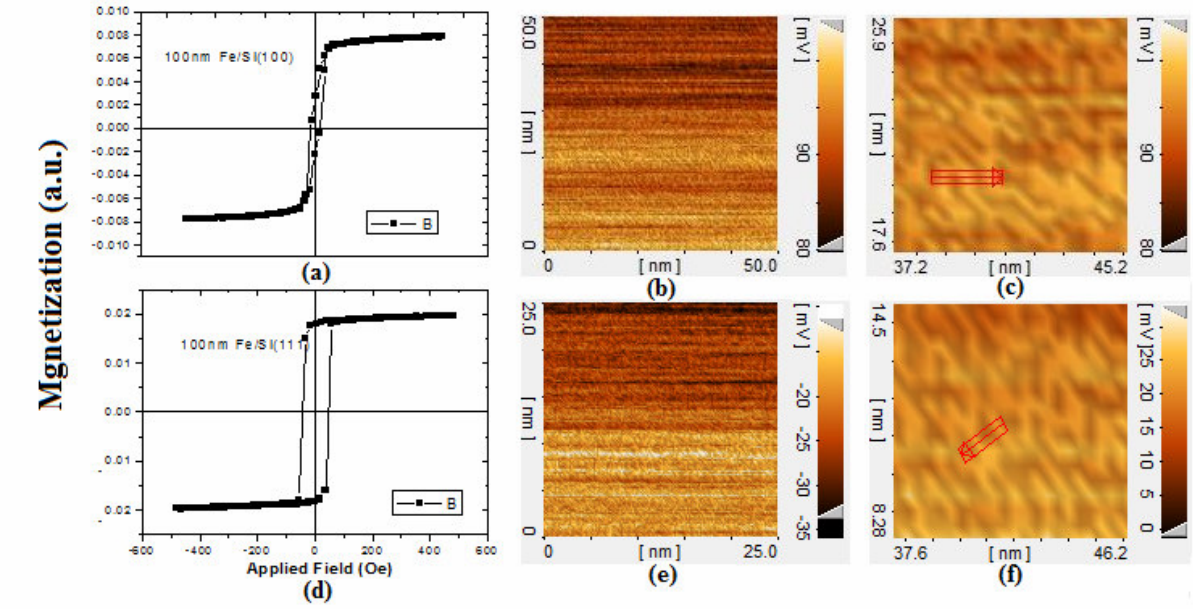


Fig. 1. Magnetization and MFM surface view of as deposited Fe/Si(100) and Fe/(111)

The magnetic domain patterns clearly visible in the MFM picture have been taken at the magnetic remanence. In Fig.1 (b, e), MFM results for 100 nm of Fe/Si (111) and Fe/Si (100) substrates are presented. In (c,f) parts of fig.1, zoom to part of this images are shown. Domain walls of specimen in 7×7 nm. Magnetization loops were measured in directions of perpendicular and parallel to the film plane by alternating gradient magnetometry (AGFM). Hysteresis diagrams show a good ferromagnetic characteristics of these samples where is shown in Fig.1(a) and (d). The domain wall's width in samples is found 0.5-1.5 nm.

Keywords: magnetic nanostructures, Fe thin films, magnetic properties, magnetic anisotropy

Surface characterization of metal-semiconductor nanostructures thin films

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We present experimental results on the surface characterization of nanostructures metal-semiconductor thin films. Four series Cu and Fe thin films have been deposited by thermal evaporation onto Si substrates from a 99.99% purified powder. The Fig.1 shows results for these series of thin films consist of samples with different thicknesses.

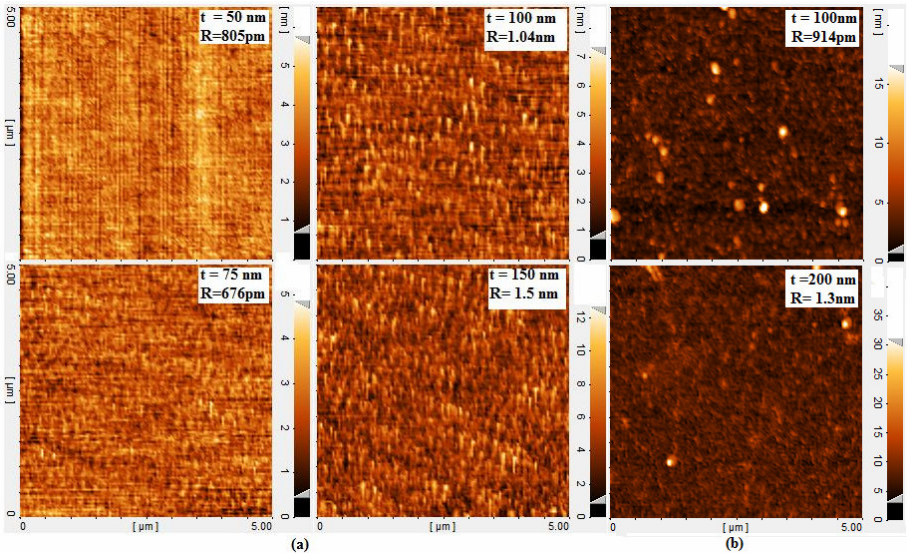


Fig.1. AFM Surface topography images of samples with different thicknesses.

AFM technique has been used to study the roughness of the surface of these films with 50-150nm for Fe and 20-200nm for Cu thin films thickness. Iron on Silicon substrate AFM images are shown in Fig. 1(a) and copper on Silicon substrate AFM images are shown in Fig. 1(b). The scanned area is 5×5 μm^2. The root mean square deviation (R) was estimated that are reported in figure with 45^0 texture direction. The Root Mean Square slope is estimated between 0.02-0.06 for Fe films and 0.01-0.03 for Cu films.

Keywords: nanostructures, thin films, root mean square, AFM

Defined zinc oxide particles preparation employing milling technique

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The particles and nanoparticles are extensively studied for many years. Nowadays the interest is focused on their morphology, shape and particle size. Both size and shape of nanoparticles influence their properties (such as chemical, electrical, optical, bio-properties etc.) and depend on their preparation methods.

Zinc oxide (ZnO) is in the interest of scientist and producers in nanotechnology field for several decades. Zinc oxide has a wide range of applications including functional devices, catalysts and pigments, optical materials, cosmetics, UV-absorbers and additives for many industrial products. Especially, ZnO nanoparticles with narrow particle size distribution have great potentiality for being used in solar cell, gas sensors, chemical absorbent, varistors, nanostructured semiconductors etc. Therefore, many investigations on the synthesis and modification of ZnO nanoparticles have been in progress to control particle size, shape and particle size distribution.

Numbers of methods for the synthesis of ZnO nanoparticles have been developed. ZnO nanoparticles have been prepared by various physical and chemical methods including chemical vapor deposition, electrochemical deposition, homogeneous precipitation, hydrothermal synthesis, molecular beam epitaxy, sol-gel process, and solid-state reaction method. Generally, milling process has been applied as a process of solid-state reaction method contributing to the synthesis of a wide range of nanoparticulate materials. Important role in ZnO nanostructure preparation plays Zn-precursors. For zinc oxide particles, various shapes including nanorods, whiskers and nanowires have been successfully prepared.

ZnO nanoparticles have been synthesis via mechanochemical method with two type of precursor: zinc chloride and zinc acetate dihydrate. ZnO nanoparticles have been defined using the particle size distribution (PSD) analysis. Character and shape of ZnO nanoparticles have been studied using scanning electron microscopy (SEM). Structural and phase analysis has been performed using X-ray diffraction (XRD) method.

The present work was solved in the frame of the research project of GAČR 205/08/0869 and Ministry of Education, Youth and Sport of Czech Republic MSM 619 891 0016.

Keywords: zinc oxide, milling technique, size effect, particle shape.

Synthesis and plasmonic properties of silver nanoshells on cores of different nature

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Composite nanoparticles with dielectric cores and gold or silver shells, possessing unique optical properties, are of growing interest (in particular, from the point of view of their potential use in photovoltaics and medicine). Such nanostructures are characterized by the peak of surface plasmon resonance which position can be "tuned" in a wide spectral range, varying the size (and/or the form) of a core and the shell thickness.

According to theoretical estimations, the optical characteristics of composite nanoparticles with silver shells essentially surpass ones of similar structures with Au shells. However, it is rather difficult to synthesize particles with Ag shells of high quality (uniformity). At the same time the problem of the synthesis of composite nanoparticles with gold shells of the predetermined thickness is practically solved [1]. Developed algorithm of the core/Au-shell structures' fabrication includes the following basic steps: 1) synthesis of dielectric cores of the desired size and form; 2) modification of their surfaces with aminosilane adsorbed layer; 3) synthesis of Au or Ag seeding particles 1–5 nm in diameter and their adsorption onto core surface; 4) growth of seeding nanoparticles in a solution containing ions of corresponding metal and a mild reductant up to formation of continuous metal shell of the required thickness.

The aim of our work – colloidochemical synthesis of composite (dielectric core/silver shell) nanoparticles with predetermined size and form and, as a result, position of surface plasmon resonance.

The following main results have been obtained.

1) The methods of synthesis of SiO_2 spherical particles and anisotropic particles ("nanorice") of FeOOH with the desired size are developed. In some cases the particles of aminated polystyrene latex (Diapharm, St.-Petersburg) have been used as the cores also.

2) Procedures of surface modification of SiO_2 and FeOOH particles with aminosilane adsorption layers are optimized. They provide the subsequent formation of monolayers of metal seeding particles on such cores. The densities of bound aminosilane molecules on the core surfaces are quantitatively estimated.

3) New route for the attachment of metal seeding nanoparticles (1–5 nm in diameter) on the core surfaces is proposed and realized. It consists in reduction of Au or Ag ions by sodium borohydride directly in the presence of core particles.

4) For the first time, the possibility of enlargement of gold or silver seeding nanoparticles adsorbed on a core surface (up to the formation of a continuous silver shell) in a solution containing silver nitrate and ascorbic acid is demonstrated.

5) For such composite structures, it is possible to fine tune the position of plasmon absorption maximum in a 600–1200 nm range varying the core diameter-to-shell thickness ratio.

Reference:

[1] C. Loo, A. Lin, L. Hirsch, *Technol. Cancer Res. & Treatment.* 3 (2004) 33.

Keywords: core/shell nanoparticles, plasmon resonance, nanostructures, composite nanoparticles

Dynamics Phenomena of Cavitation Disintegration of Nanoparticles in a Liquid Suspension using Water Jet Mill (WJM)

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Physical analysis of cavitation breakage (Fig. 2) of solid particles focused with a practical application during fine particle disintegration in a liquid suspension has been developed in the paper.
Cavitation source in the dynamics phenomena is an extreme velocity gradient developed by high-energy liquid jet mixing together with a slow suspension of milled particles.
Preparation of a nanoparticle silicon using WJM (Fig. 1) has been demonstrated as a sample of method applicability in the range of a hundred of nanometers.

The paper has been supported by Czech Grant Agency (GAČR) during the project No. GAČR 106/08/1092.



Fig. 1. Water Jet Mill (WJM)

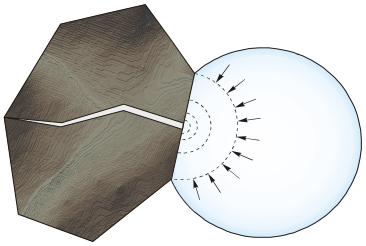


Fig. 2. Mechanism of cavitation breakage

Keywords: nanoparticle, cavitation, implosion, particle breakage

Internal damping in epoxy resin – silicone dioxide systems

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The control of vibration has become more important at present. Materials for vibration damping are mainly polymers, because of their viscoelastic character. But viscoelasticity is not the only mechanism for damping. The microstructures can greatly affect the damping of the materials and various interfaces in composite systems contribute to damping due to their slippage one another. The utilization of nanoparticles seems to be very interesting.

This paper presents the experimental study of composite systems epoxy resin – silicone dioxide filler damping behavior. Particle shape, size and volume fraction on internal damping of the composite systems was evaluated. Fumed silica (diameter of primary particles 40 nm, length of aggregates hundreds of nanometers), quartz sand (diameter of particles hundreds of micrometers) and silicon dioxide flakes created from submicron silicone dioxide fibres (diameter of fibres hundreds of nanometers) were used as filler without surface modification. Free vibration (frequency about 50 Hz and temperature approximately 22^{o}C) were performed on the composite samples (single cantilever bending) to determine the above mentioned effect. The photoelectric equipment was used to measure the vibration damping of the samples, the loss coefficient tan δ was calculated by common method.

The application of above mentioned fillers tends to change of nanocomposite systems damping capacity. A scanning electron microscopy was used to observe fracture surfaces of the samples and to evaluate the character of interfacial surface in the composites and homogeneity of the systems.

This work was financial supported by MSM 4674788501.

Keywords: internal damping, composite systems, silicone dioxide, particle fillers

Chiral functionalization of monolayer-protected silver nanoclusters by chiral ligand-exchange reactions

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Passivation of metal nanoclusters with chiral molecules results in unique electronic and chiroptical responses that are unlike in those of the component parts. For example, we have reported large chiroptical responses of size-selected gold nanoclusters protected by a pair of enantiomers of chiral penicillamine. We have also succeeded in preparing chiral penicillamine-protected silver nanoclusters with core sizes ranged in 1-3 nm. Unlike the case of the gold nanoclusters, the silver nanoclusters covered with penicillamine racemate interestingly exhibited different absorption profiles from those covered with the enantiopure species in the same core size. On the other hand, reactivities of monolayer-protected metal nanoclusters or nanoparticles respond to both the metal core and the nature of the monolayer ligand shell. An important example is the ligand-exchange reaction. Surface ligands on a nanoparticle surface can be exchanged by stronger ligands, and this ligand-exchange reaction provides an important means for the chemical functionalization of nanoparticles or nanoclusters. If such ligand-exchange reactions can be utilized for introducing chirality or optical activity onto the achiral monolayer-protected metal nanoclusters, they will offer a useful synthetic route for fabricating chiral functionalized nanoclusters. We report here the ligand-exchange reaction between the optically inactive racemic penicillamine monolayer on a silver nanocluster surface and enantiopure D- or L-penicillamine dissolved in solution. As a consequence, chiral functionalization of the achiral silver nanoclusters is successful.

Enantiopure (D-/L-) or racemic penicillamine was used as a monolayer protecting agent. Briefly, a freshly prepared aqueous $NaBH_4$ solution was added into the mixture of $AgNO_3$ and penicillamine (D-/L-Pen, or rac-Pen) in methanol under vigorous stirring, followed by the addition of ethanol into the solution yielding a dark-brown crude precipitate. After completely washing the precipitate, the sample power was obtained by a vacuum-drying procedure. The silver nanocluster sample prepared by using D-Pen, L-Pen, or rac-Pen is termed as Ag-D-Pen, Ag-L-Pen, or Ag-rac-Pen, respectively. To separate or purify the silver nanoclusters that differ in size and charge, we applied polyacrylamide gel electrophoresis (PAGE) using a slab gel with the thickness of 2mm. On the basis of electrophoretic mobility of the silver nanocluster compounds, the separated compounds are referred to as 1, 2, … (consecutive numbers), with the order of mobility 1 > 2 > …. When distinguishing these compounds in regard to the stereochemistry of the surface ligands, a suffix L, D or rac is added at the end the compound number; for example, 1_D, 2_D, … for the Ag-D-pen nanocluster sample. In chiral ligand-exchange reactions, we used an optically inactive silver nanocluster sample as the starting material. The reaction was conducted by stirring an aqueous solution (1.0 mL) of the as-synthesized rac-Pen-protected silver nanocluster powder (10.0 mg) and enantiopure D- or L-Pen (5.0 mg) overnight at room temperature under an inert (N_2-saturated) atmosphere. Fortunately, emergence of the identical band positions in the gel electrophoresis separation assures the presence of size-invariant silver nanoclusters (1.05 nm for compound 1 and 1.30 nm for compound 2 in core diameter) during the ligand-exchange reaction and allows us to further examine the optical/chiroptical properties of these nanoclusters.

Figure 1 shows the pattern of the anisotropy factor (or g-factor), which is defined as the ratio of the molar dichroic absorption to the extinction coefficient, of compounds $1_D/1_L/1_{rac}$. The separated

compounds $1_D/1_L$ exhibited large Cotton effects in metal-based electronic transition regions, whereas 1_{rac} was optically inactive. Note that, in enantiopure D- and L-Pen-protected nanocluster compounds ($1_D/1_L$), the absorption spectral shapes are almost identical within the same compound numbers, supporting that each numbered compound has similar core size, geometry, and chemical compositions. In silver nanocluster compounds covered with rac-Pen or penicillamine in a low ee (enantiomeric excess) value, on the other hand, the spectral shapes involving peak or shoulder positions were slightly different from those of $1_D/1_L$. Hence we can conclude that the ee value of the chiral surface ligand influences the optical properties of the silver nanoclusters in the same core size. The ligand enantiomeric purity also had an influence on the Cotton effects of the silver nanoclusters. Importantly, we found that the maximum anisotropy factor (g_{max}-factor) was approximately proportional to the ligand ee value: In compound 1, for example, the g_{max}-factor was 1.5×10^{-3} for $1_D/1_L$, whereas it decreased to be 7-8 x 10^{-4}, scaled by a factor of ~0.5, for the clusters covered with 50% ee penicillamine. In ligand-exchange reactions using achiral nanoclusters, a noticeable modification was observed in lanes in the PAGE separation, but the positions 1 and 2 were invariant as the magic-numbered silver nanocluster compounds. The ligand-exchanged samples are referred to as $1_{eD}/1_{eL}$. Figure 2 shows the pattern of g-factor of $1_{eD}/1_{eL}$. A key feature is the appearance of intense Cotton effects (chiral functionalization) from the metal clusters modified through the chiral ligand-exchange reaction. The pattern of the g-factor as well as its intensity of $1_{eD}/1_{eL}$ are similar to those of the normally synthesized silver nanoclusters with 50% ee penicillamine, respectively, suggesting that the surface ligands would be partially (probably ~50% ee) displaced by penicillamine with specific handedness through the exchange reaction, and as a consequence, the chiral ligand-exchange reactions yielded enantioenriched optically active silver nanoclusters.

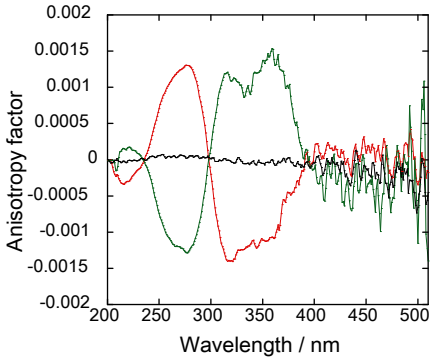


Fig. 1. Anisotropy factor of $\mathbf{1}_D/\mathbf{1}_L/\mathbf{1}_{rac}$

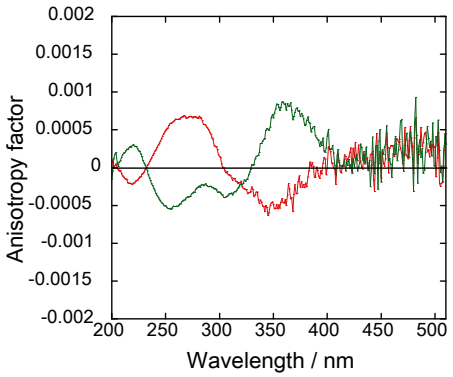


Fig. 2. Anisotropy factor of $\mathbf{1}_{eD}/\mathbf{1}_{eL}$

Keywords: chiral functionalization, silver nanoclusters, ligand-exchange reaction, dissymmetric field model

Formation of nanostructured powders obtained by dry grinding of layer silicates: a bidimensional particle size model

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Physico-chemical changes during the grinding of a material produces mechanochemical effects. They are more frequently observed when grinding is carried out in equipment using impact and friction forces among particles, such as vibratory, oscillating and planetary mills. Wet or dry grinding are commonly used as procedures for the preparation of fine and reactive powders and for further processing steps. Deagglomeration and elimination of soft aggregates are produced by wet grinding. In contrast, dry grinding is most intensive when acting on solids. It originates mechanical stress which are quite diverse, producing important changes such as lattice distortions, amorphization, decrease in particle size (both crystallite size and grain) and an increase in surface area, besides the formation of new phases by mechanical alloying. Thus, the reactivity of ground nanostructured solids is enhanced and, therefore, it leads to mechanochemical reactions. From this, there is a general interest in the study of the physical and chemical induced effects by grinding in solids.

The effects of dry grinding on layer silicates and clay minerals have been extensively studied in past years due to their relevant importance in some applications, for instance kaolinite and talc as ceramic raw materials. In the present research on grinding effects on layer silicates, several industrial samples of kaolinite (1:1 layer silicate) and talc (2:1) have been selected. Dry grinding experiments by planetary milling have been performed in order to examine the effects of different conditions in the resultant powders. Several techniques have been used to follow the evolution of the layer silicates, mainly XRD, surface area (nitrogen adsorption), SEM, TEM, and particle size analysis by Laser scattering.

Modifications of surface area, particle size and shape of short and prolonged grinding on the crystal structure of kaolinite and talc have been evaluated. The observed changes have been related to a progressive delamination and structural breakdown during grinding, with final formation of a turbostratic-type structure characterized by a partial or complete absence of three-dimensional order of the finest particles, mainly along [00*l*]. Short grinding times resulted in the breakdown and drastic size reduction of relatively thin particles. However, the rate of size reduction decreased with time and a limit is reached. The particle size distribution analysis for the original and ground samples agree with these findings. Increasing grinding produced more rounded particles from the layer silicates and aggregates, with a decrease of surface area. These changes also affected the pore size distribution from nitrogen adsorption measurements. It can be also observed an increasing in amorphization as a consequence of the loss of long-range order. TEM and SEM observations have used to follow the progressive particle size reduction of the original layer silicates and associated morphological changes, showing the formation of nanometer size ground powders and further particle agglomeration. The particles gradually lost their original layered shape with grinding, producing round-edged particles with a rugged surface, as observed by SEM.

Taking into account all these results, it has been developed a bidimensional model of particle size evolution and changes of particle shape on grinding the 1:1 and 2:1 layer silicates kaolinite and talc, respectively. Grinding produces a loss of periodicity perpendicular to the layer silicate plane, and the dimensions of the crystallites along the c-direction resulted too small to produce coherent X-ray scattering. On the other hand, it has been found an inverse relationship between coherent X-ray domain and microstrains in the ground powders.

The reduction of particle size and domain thickness by grinding have been also found directly related to the state of increasing structural disorder. In this model, the aspect ratio, domain thickness and average particle size have been compared in both layer silicates and considering the different samples and experimental grinding conditions. Short grinding times induces some changes in the regular stacking mode of the silicate layers into a more random one. A more isometric particle shape is produced than in either the original talc and kaolinite samples or in those ground for short grinding times. The calculated shape factors demonstrated also its dependence on particles orientation, which influence the X-ray diffraction results.

In summary, on the basis of the present results it is proposed a bidimensional particle size model, which is interesting to analyze the formation of nanostructured powders when grinding layer silicates.

The financial support of Regional Government (Junta de Andalucía) to the Research Group TEP 204 is acknowledged. Thanks are extended to the Research Project CTQ2005-998 cofinancied with Feder funds.

Keywords: grinding, layer silicates, nanostructures, model

Montmorillonite as a heavy metal sorbent

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The sorption removal of some heavy metal cations such as Zn(II), Cd(II), Pb(II), Hg(II), As(VI), Be(II) and Tl(I) from aqueous solution onto montmorillonite has been studied using the batch method with initial metal ion concentrations within the range 1-10 mg/l. The natural clay and clay treated with hydrochloric and sulphuric acid were used. The percentage effect of the sorption and equilibrium concentrations using atomic absorption flame (Zn, Cd, Pb) Spectrometry and atomic emission spectrometry with inductive couplet plasma (JY-24) were determined. The montmorillonite surface after sorption treatment was studied by scanning electron microscope XL 30, with X-ray micro-analyser EDAX (PHILIPS). In detail the study was focused on equilibrium of Tl sorption. The influence of pH solution was study too. The results show that natural montmorillonite has a considerable potential for the removal of heavy metal cationic species from aqueous solution and wastewater.

The financial support from the projects MSM 6198910016 and GAČR 205/08/0869 is gratefully acknowledged.

Keywords: sorption, heavy metal, montmorillonite

Analytical investigation of solidification characteristics of PCM with nanoparticles

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The cold thermal energy storage (CTES) is also used to adjust a time lag between demand and supply of cold thermal energy (CTE). A peak load of electricity due to an air conditioning during a summer afternoon is shifted to an off-peak period, by producing cold or ice through a night and using the CTES. For cold thermal energy storage application phase changes of materials are a perspective ways of thermal energy storage. Such materials are called phase change material (PCM). During the phase change in a phase change material (PCM) storage system, the solid-liquid interface moves away from the heat transfer surface and the surface heat flux decreases due to the increasing thermal resistance of the molten or solidified medium.

By the dispersion of ultra fine metallic (Cu) nanoparticle within the encapsulation of PCM(Calcium chloride hexahydrate) resulting as nanofluid, which tremendously enhances the heat transfer characteristics of the original fluid. The main objective of this paper is to develop a simplified analytical model which predicts the solid-liquid interface location and temperature distribution of PCM with nanoparticles at various time positions in the solidification process with a constant end-wall temperature. The effect of suspended ultra fine metallic (copper) nanoparticle within the enclosure is analyzed and effective thermal conductivity maps are developed for various controlling parameters. Comparison with conventional PCM are performed and found to excellent agreement.

Keywords: cold thermal energy storage, phase change material, nanoparticle, heat transfer, thermal conductivity

Thermal conductivity measurement of nanofluids by transient hot wire method

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Heat transfer fluids with suspended ultra fine particles of nanometer size are named as nanofluids, which have opened a new dimension in heat transfer processes. The recent investigations confirm the potential of nanofluids in enhancing heat transfer characteristics. This paper presents the experimental investigation of thermal conductivity of silver/water and Al_2O_3/water nanofluid. The nanoparticles are prepared by the chemical route and the TEM photographs are presented and analysed. The thermal conductivity of nanofluids are measured by transient hot wire method, the effect of volume fraction ranging from (1% to 5%) and temperature on thermal conductivity are studied. Thermal conductivity of nanofluid increased as the temperature and volume fraction is increased. Particle random motion and the thermo-physical properties of nanofluid are found to be the reasons for the enhancement. It is also observed that the silver/water nanofluid shows more enhancement of thermal conductivity than Al_2O_3/water nanofluid. Possible mechanisms for the enhancement are presented in the paper and discussed.

Keywords: nanofluids, thermal conductivity, transient hot wire method

Gold doped poly(vinyl alcohol) optical films: synthesis and characterization

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A simple one-step process is developed to synthesize optical films of Au-doped polymer of poly(vinyl alcohol) (PVA) with selective Au-contents from 0.1 to 5.0 wt%. The synthetic procedure involves a caloric $Au^{3+} \rightarrow Au$ reaction in aqueous PVA at 60-90°C temperatures followed by casting the sample {Au- *nanoparticles* (NPs) embedding in the PVA polymer molecules}as films or other shapes. The NPs are thin platelets of triangular, square, rectangular, or hexagonal shapes. There are very few pentagonal platelets and nanorods. As analyzed with microstructure, the NPs formation results in a structural anisotropy by the preferential adsorption of PVA on selective facets. An Au-content dependent emission occurs in the 400-650 nm regions, useful for possible optical data storage, nonlinear optical devices, and color pigments.

Keywords: gold nanoparticles; poly(vinyl alcohol); microstructure; optical films

Emplacement of particulate materials phase between fluids and solid materials: synergetic interpretation

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Comparison a synergetic and *a classic* approach to a particle material arrangement description in dependence on its angle of internal friction φ from 0 deg to 90d deg has been presented in the paper.
The Fig. 1 describes a transformation of a mass state via an angle of internal friction φ = 0 deg-Fluids through particulate materials to Solids φ = 90 deg.
The particulate material area and studying of the particulate materials are important for description, identification and nanoparticle systems research with regarding to particle properties and identification of particle aggregates as well as their property forecasts.

The paper has been supported by the Grant No. MSM 619 891 0016 under Ministry of Education, Youth and Sports of the Czech Republic.

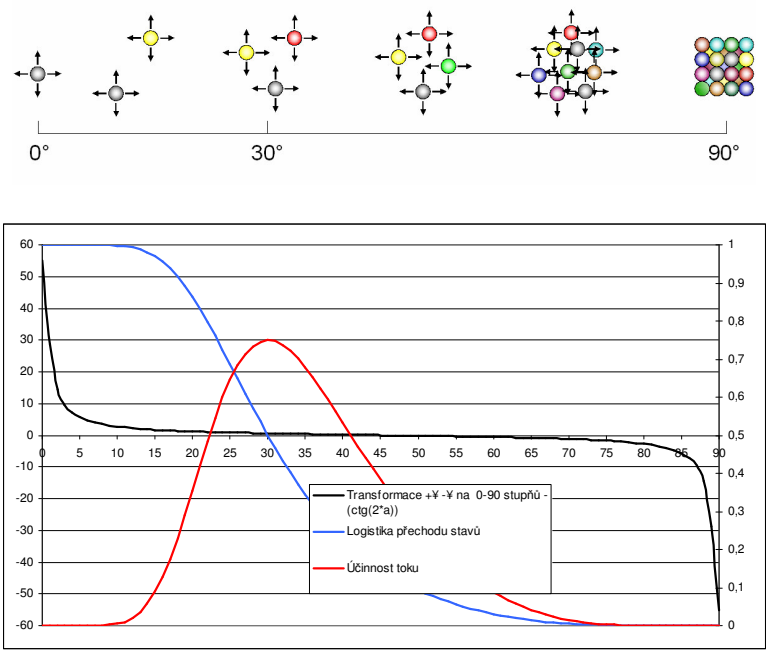


Fig. 1. Emplacement phase Vs. angle of internal friction of a particulate material.

Keywords: nanoparticle, particle aggregates, synergetic

Identification of 2D design of opencast structures of micro and nano layers from an internal friction curve - LBM-Nano-Machine for 2D

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In the paper, a model identification of overcast layers structure which is in a direct contact with a moving particle has been developed. The model is developed by a referent particle movement observation. A model of a local contact friction is draft, and using a mathematical analysis of values on a local friction, a general model of friction and a contact structural layer are designed.

The paper has been supported by the Grant No. MSM 619 891 0016 under Ministry of Education, Youth and Sports of the Czech Republic.

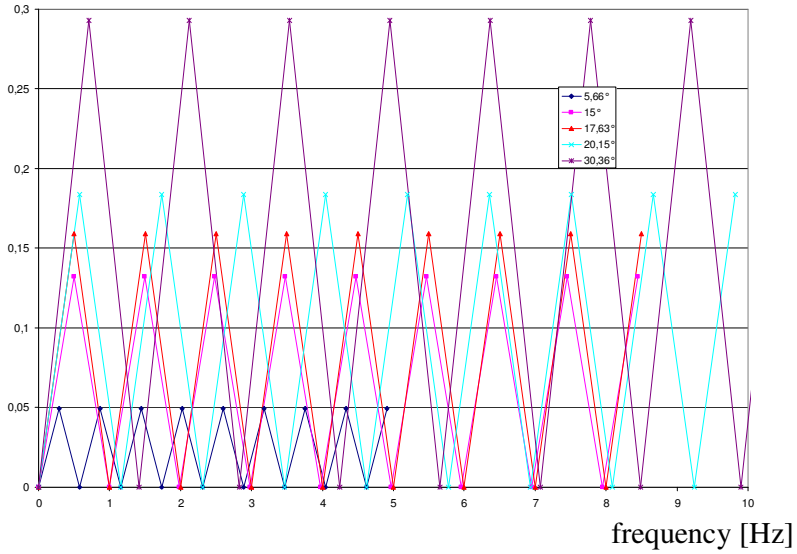


Fig. 1. A ration of an amplitude height and a frequency characterization of initial opencast layers for the value of internal friction: 5,56° , 15° , 17,63° , 20,15° a 30,36° .

Keywords: internal friction, overcast geometry, internal friction model

Nanoparticles of $La_{1-x}Sr_xMnO_3$ perovskite phase for magnetic fluid hyperthermia and MR imaging

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Magnetic nanoparticles assigned for medical applications like Magnetic Fluid Hyperthermia (MFH) and Magnetic Resonance Imaging (MRI) have to fulfil special demands regarding their physical properties and in addition they have to be biocompatible. The core-shell architecture often divides these tasks. Magnetic properties are foremost determined by the core, whereas the shell ensures biocompatibility and stability. The perovskite phase $La_{1-x}Sr_xMnO_3$ (LSMO) represents a promising and unique core material with tunable Curie temperature and sufficient power losses in terms of MFH. The former is a way how to rule out the risk of overheating and the later enables sufficient damage of cancer cells during hypertermia treatment of tumor.

LSMO is prepared via sol-gel procedure, followed by thermal and mechanical treatment involving both rolling and milling. This material is coated with silica which provides colloidal stability in water and should resolve the toxicity of the core. The particles stabilized in water by citric acid were encapsulated using tetraethoxysilane (TEOS) and finally certain fraction was separated via centrifugation. TEM evidenced very good morphology (see Fig. 1), silica character of the shell was confirmed in IR spectrum and other characterizations like magnetic measurements and zeta-potential measurement were carried out as well. Another approach was used in order to enable further functionalization of the particles. Amine-derivatized particles with complex shell were prepared using TEOS and 3-aminopropyltriethoxysilane together encapsulating LSMO after its stabilization by poly(vinylpyrrolidone). The density of accessible amine groups was determined in photometric assay based on coupling of amine groups with *p*-nitrobenzaldehyde.

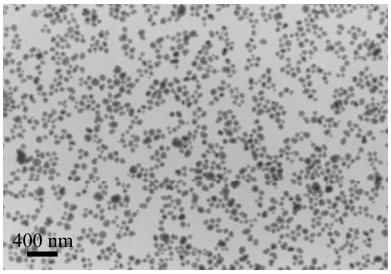


Fig. 1. LSMO@SiO_2

Cell labelling and viability of cells in the presence of LSMO@SiO_2 at concentration around 0.1 mM_{Mn} (determined via AAS) were studied on rat mesenchymal stem cells with incubation period of 48 hours. Cell viability varied between 50 % and 70 % in comparison to 97 % for unlabelled cells but it was less than 5 % for "poorly coated" particles therefore it is a considerable improvement. According to the relaxivity measurements the amount of manganese inside the cells is low

(0.24 pg Mn/cell) compared to the content of Fe in the case of Feridex® loaded cells (14.6 pg Fe/cell). In fact the LSMO@SiO_2 labelled cells exhibit even a better contrast than Feridex® labelled ones due to high T_2 relaxivity of LSMO and probably due to its low aggregation inside cells. T_2 relaxation rate for LSMO@SiO_2 and Feridex® labelled cells was 18.1 $s^{-1}/(10^6$ cells) and 17.3 $s^{-1}/(10^6$ cells) respectively.

The support by projects ASCR KAN20020061, KAN201110651, 1QS100100553, MSMT1M0538, GACR309/06/1594 is gratefully acknowledged.

Keywords: Magnetic Nanoparticles, Manganites, Magnetic Fluid Hyperthermia, MRI

Functionalized nanomaghemite from iron(II) acetate as a potential contrast agent in MRI

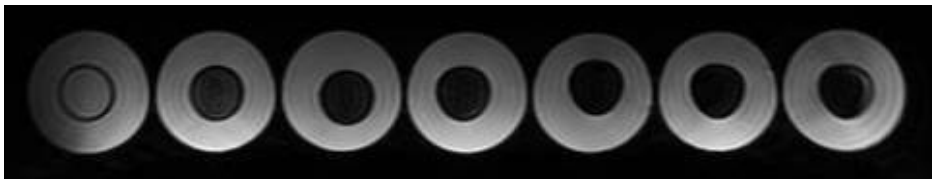
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Nowadays Magnetic Resonance Imaging (MRI) is one of the most powerful techniques in diagnostics, clinical medicine and biomedical research. MRI is a noninvasive method which gives information on physical and biochemical properties of desired organs or soft tissues with the use of a highly uniform static and alternating magnetic fields. Superparamagnetic iron oxides (SPIO) contrast agents, consisting of iron oxides magnetite (Fe_3O_4) or maghemite (γ-Fe_2O_3), are extremely strong enhancers of the proton relaxation and thus enhance the contrast of desired tissues causing predominantly negative signals in T2-weighted images.
We have prepared crystalline maghemite nanopowder by thermal decomposition of iron(II) acetate in air at a temperature of 400 °C for 1 hour. The average size of the final nanoparticles was found to be approximately 25 nm. Relatively highly monodisperse nanoparticles of maghemite with the specific surface area of 150 m^2/g exhibited the saturation magnetization of 53 emu/g at 2 K. These magnetic, size and surface properties of the as-prepared maghemite nanoparticles thus make them very promising in the field of biomedical applications.
In our work, we have focused on the preparation of the superparamagnetic maghemite contrast agent for the medical diagnostics of the gastrointestinal tract via MRI. To improve biocompatibility and uniform dispersion of maghemite nanoparticles in a suitable solution, maghemite particles were incorporated into bentonite matrix. By optimization of the synthesis condition, we have confirmed an excellent contrast effect of this suspension in *in vitro* phantoms by MRI. Currently *in vivo* tests of the prepared contrast agents for gastrointestinal tract imaging are performed.

In general, maghemite nanoparticles are considered to be a nontoxic material. In order to confirm nontoxic character of the prepared maghemite samples, standard toxicological tests are currently performed.



MRI contrast effect of maghemite particles modified with bentonite (concentrations of maghemite are increasing from left (pure water) to right (30mg maghemite/50ml water)) in T2-weighted images.

Keywords: maghemite, magnetic resonance imaging, thermal decomposition, gastrointestinal tract

Characterization of nanoparticles at the environment: a case study at the Canary Islands (Spain) by airborne dust accumulation contribution

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Investigations on nanoparticles and nanopowders is of widely interest, not only by health safety. The global climate change studies involve the role of dust in a number of important processes. For instance, desert dust could affect climate through direct effect by absorbing and reflecting solar radiation and indirectly by modifying cloud properties. Both effects could contribute to the variation of the earth´s radiation budget. The degree of these effects is determined by chemical, physical and mineralogical properties of dust particles. On the other hand, soil particles from damaged lands enter suspension and become part of atmospheric dust load. Dust obscures visibility and pollutes the air, causes accidents, fouls machinery and affect animal and human health. Aeolian sediments also pollute water bodies and cause associated problems.

Airborne dust form desert, in particular Sahara desert, has a significant effect on adjacent and also at global scale. Calima (haze) is one local Canarian name for the metereological condition by which Saharan dust is transported by wind. It was previously demonstrated that Saharan dust is an usual particulate input in Canary Islands soils and has an important weight on the annual mean dust deposition. The dust sources in the island of Gran Canaria can be autochtonous, i.e. particles of diverse origin generated and/or recycled within the insular area, and allochthonous from the neighboring African continent and transported during haze conditions. The Saharan Desert generates a wide variety of particles and nanoparticles owing to the range of different origin and ages present in Norwest Africa. The source areas of the dust components were established by data (Total Ozone Mapping Spectrometer) to be in two major areas: the Bodelé depression (Southern of Chad) and the Eastern Mauritania-Western Mali-Southern Algeria area. Quantification of airborne dust can be measured either volumetrically or through accumulation rates.

In the present research it has been carried out a study on the presence of nanoparticles at the environment with a case study of airborne dust accumulation at a Canary Island. The island of Gran Canaria located approximately in the center of the Canary island Archipelago. The aim of this study has been to characterize the identified nanoparticles. Dust sampling design (Pyrex glass trays on top of a glass surface covered with a PVC mesh) and sample collection were representative of the island from both a metereological and altitudinal point of view during a period of a year (2002-2003). Dust samples were collected once each week. Deionized water was used to collect the samples of atmospheric dust in dry and immediately cleaned the tray. Four soil profiles close to each one of the selected sampling plots were described and sampled for comparative purposes (one sample per horizon). Grain size analyses were made using a Laser analyzer. The crystalline phases and chemical analysis of the samples were performed by X-ray powder diffraction and X-Ray Fluorescence, respectively. Transmision and Scanning Electron Microscopies (TEM and SEM) and Energy Dispersive X-Ray Spectroscopy (EDX) were used to characterize the particles. Possible organic matter was also investigated by elemental analysis (CHN) and IR spectroscopy.

Mean values of dust accumulation rates (in wt %) decrease with increasing altitude due to the gravitational settling. Components of collected dust samples were mostly in the coarse silt grain fraction with low percentages of clay size lower than 2 μm according to particle size analysis with detection of nanoparticles in the polymodal distribution plots.

The phase analysis by XRD allowed to identify a complex association of quartz, salts (mainly halite) and carbonates in major proportion, with feldspars, gypsum, diopside (Ca-Mg silicate) and magnetite in medium proportion. K and Mg salts, such as sylvite and epsomite, and clay minerals were found in low proportion. The main clay minerals were the layer silicates montmorillonite, illite, chlorite-kaolinite and halloysite. Quartz was considered allochthonous because it is not present in the volcanic substrate of the island, although additional experimental evidences suggested an external and recycled input of this mineral. The clay minerals of the airborne dust can derive from insular soils as well as from the Saharan region.

Chemical data by XRF allowed to quantify the main components such as SiO_2 (range 76.11 to 86.43 wt %) and Na_2O (14.57 to 23.59 wt %), and the presence of CaO and MgO. The XRF results confirmed Cl and S, associated to the presence of chloride and sulphate salts (i.e., halite and gypsum). Minor elements (in ppm) were also investigated. It should be noted the presence of Ni, Pb, Zr, and Zn. Elemental analysis of C and N (in wt %) range from 6.2 to 0.56 and 3.1 to 0.2 wt %, respectively, suggesting a very low proportion of organic matter.
TEM and SEM-EDX observations confirmed the nanometer size of components of collected dust, in particular layer silicates and iron oxides as identified by EDX. In the island of Gran Canaria, silica saturated rocks are present in the form of rhyolitic rocks and residual amounts of microcrystalline silica (agate-chalcedony-moganite). In addition, quartz particles have been detected in sediments collected in the Canary Islands, with this mineral assumed to be of Saharan origin. The volcanic substrate of the sampling plots is often composed of olivine, clinopyroxenes, amphibole, feldespatoids and zeolites, among others. However, the studied aeolian samples did not contain these minerals or they are present in minimal amounts. Taking into account the detection limit of the analytical techniques employed, this is associated to the comparatively large rock grain sizes and low fragmentation or the transformation through weathering in other minerals.
Additional observation by SEM of prismatic carbonate (dolomite) crystals displaying smoothly rounded edges in some samples could be associated to aeolian abrasion due to transport. On the other hand, the halite crystals were considered of marine origin. In summary, the presence of nanoparticles in airborne dust is evidenced. However, it is a first step on these investigations and further work must be conducted to evaluate their environmental impact, in particular in climate change studies and additionally the sinergy effect of nanoparticles production by the increasing activity of industries.

The partial financial support by the University of Las Palmas de Gran Canaria (Project UNI2001/17) and Research Group TEP 204 are acknowledged. The collaboration to the present work of some institutions is also acknowledged: Secondary Education Institute of the town of San Mateo, the Agricultural Research Centre of the Island Council of Gran Canaria, the Gando Air Force Base and the National Institute of Metereology of Spain.

Keywords: nanoparticles, airborne dust accumulation, environment, mineral phase

Magnetically responsive *Saccharomyces cerevisiae* cells for hydrogen peroxide removal

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Hydrogen peroxide (HP) is a very effective microbicidal agent. A major reason for the growth in popularity of HP in the food, cosmetic, and medical fields is its low toxicity at use concentrations as well as its safe decomposition products (water and oxygen). HP is effective against a wide spectrum of microorganisms, including bacteria, yeast, moulds, viruses and spore-forming organisms. The use of HP in food industry has been approved in several countries, for example in the production of Swiss cheese and for milk cold pasteurization.

The excess of hydrogen peroxide is usually destroyed by adding catalase (hydrogen-peroxide:hydrogen-peroxide oxidoreductase, EC 1.11.1.6). Commercial catalase preparations are rather expensive. In selected cases crude microbial extracts from appropriate microbial producers can be used. Microbial cells can serve as inexpensive substitutes of purified enzymes. In the case of catalase, readily available *Saccharomyces cerevisiae* cells (bakers' yeast) are of special interest, due to their broad application in food industry; they are classified as "Generally recognized as safe (GRAS)". The cells can be used both in the native state and in the immobilized form.

In order to simplify manipulation with the biocatalyst, we have prepared magnetically responsive alginate beads containing entrapped *Saccharomyces cerevisiae* cells and magnetite microparticles. Larger beads (2-3 mm in diameter) were prepared by dropping the mixture into calcium chloride solution while microbeads (the diameter of majority of particles ranged between 50 and 100 μm) were prepared using the water in oil emulsification process. In general, microbeads enabled more efficient HP decomposition. The prepared microparticulate biocatalyst caused efficient decomposition of HP in water solutions up to the concentration of 2 %, leaving very low residual HP concentration after treatment (below 0.1 %). The biocatalyst was quite stable; the same catalytic activity was observed after one month storage at 4 ºC and the microbeads could be used at least five times. Due to the presence of variety of enzymes in the entrapped yeast cells, this inexpensive biocatalyst can also be used for other biotechnology processes.



Magnetically responsive alginate microbead containing entrapped *Saccharomyces cerevisiae* cells and magnetite microparticles. The scale bar corresponds to 50 μm.

Keywords: Saccharomyces cerevisiae cells, alginate, hydrogen peroxide, magnetic separation

Magnetic adsorbents for extraction of alkylphenols and nonylphenol mono and diethoxylates from water

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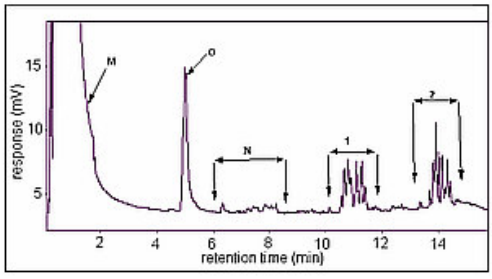
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Alkylphenols are basic materials for the production of e.g. paints and pesticides. 4- Nonylphenol ethoxylates (NPnEO) synthesized from a technical nonylphenol (NP; a very complex mixture of 22 isomers) are often used as nonionic surfactants in pharmaceutical, agrochemical, cosmetic, textile, petroleum and chemical industrial production. Residues of NPnEO and their biodegradation products can occur everywhere in the environment, especially in surface water, where they are partially converted to more persistent and toxic metabolites.

Concentrations of NP, NP1EO and NP2EO in surface water, and in water after biodegradation tests of NPnEO are low, therefore it is necessary to accomplish extraction, clean-up and extract concentration before chromatographic separation and determination. The aim of our work was to extract NP, NP1EO and NP2EO from different water samples using magnetic solid phase extraction and compare these results with liquid liquid extraction (LLE).
Different types of adsorbents, such as Chromosorb, Tenax, Porapak and Chezacarb were magnetically modified and extraction parameters were optimized (sorption 2 min; elution by 1 ml methanol, 1 min). The target analytes were isolated from spiked distilled and tap water, real samples from river Labe and the reservoir Rozkos and from water phase after biodegradation tests of oxyethylated nonylphenols. Extracts of pollutants were analysed by capillary gas chromatography.

Magnetic solid phase extraction showed to be a suitable method for fast extraction of alkylphenols with middle length alkyls and oxyethylated nonylphenols with one or two oxyethylated groups from water samples. Magnetically modified Chezacarb B is appropriate for extraction NP1EO and NP2EO to achieve recovery 98%. For extraction of NP technical mixture, magnetic Chromosorb 103 was used with the recovery 96%. The results are similar to LLE with the recovery of NP1EO and NP2EO 93-95% and NP technical mixture 88%. The concentration of nonylphenols in real surface water samples was under the detection limit, but NP1EO and NP2EO were detected (1.5 - 2.2 μg/l) by both methods. It is obvious, that LLE can be superseded by MSPE, besides, MSPE is more economical, faster and less demanding than LLE and it is especially suitable for emulsion forming samples.



Chromatogram of technical mixture of 4-nonylphenols and 4-nonylphenol mono- and diethoxylates. M = Methanol; O = 4-tert-octylphenol; N = technical NP; 1 = NP1EO; 2 = NP2EO

Keywords: Magnetic solid phase extraction, magnetic adsorbents, alkylphenols, water

Nanoparticles of zinc-substituted cobalt ferrite as prospective material for biomedical applications

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In pursuance of appropriate material for biomedical application as hyperthermia we explored the production and properties of nanoparticles of zinc-substituted cobalt ferrite $Co_{1-x}Zn_xFe_2O_4$. The material was prepared by co-precipitation from water solution of nitrates of cobalt, zinc and iron with solution of ammonium hydroxide. The samples where then annealed at 400, 600 and 800 °C. The resulting particles were characterized by methods of XRD, TEM, Mössbauer spectroscopy, and dc and ac magnetometry. The heating experiments in ac magnetic field of various amplitudes were performed with diluted systems of particles suspended in agarose gel and the results were interpreted on the basis of the ac magnetic losses measured at various temperatures.

The XRD analysis combined with chemical analysis revealed single-phase compositions of the samples and the presence of the cubic spinel phase. Annealing of the sample with x = 0 at temperatures 400, 600 and 800 °C of the material prepared by coprecipitation resulted in the increase of the mean sizes of nanoparticles of the single-phase spinel in the range from 18 to 41 nm, measured by x-ray diffraction and shown qualitatively by TEM.

The Mössbauer spectra of the above mentioned sample indicated that the originally random distribution of rather high concentrations of Co ions over the tetrahedral sublattice is slightly changed by annealing at 600 °C. The thermal treatment at 800 °C resulted, however, in a change of Co distribution towards the preferred inverse spinel structure with the concentration of Co in octahedral sites (~0.84).

The dc magnetic measurements demonstrated little change with particle size in the magnitude of magnetization at room temperature in intermediate magnetic fields, while both coercivity and remanence were much more affected. The hysteresis losses measured at constant amplitude of the ac magnetic field (of 35 mT) displayed rather diverse dependences with temperature reflecting the changing ratio of this amplitude to coercivity. The experiment with heating efficiency of the particles was performed with the amplitude of the ac field changing from 34 to 80 mT. With the small amplitudes the heating curves displayed the usually observed character with decreasing slope and a tendency to saturation, but the highest used amplitudes already led to changed convex shapes of these curves with progressively increasing slopes. In spite of a different character of the experimental conditions, the specific absorption rates increasing with temperature are in close agreement with the measurements of the ac losses. The increase of magnetic losses and consequently of the heating efficiency with increasing temperature is explained by the strong dependence of the constant of magnetocrystalline anisotropy of cobalt ferrite on temperature.

The support by projects GAAV KJB100100701, AS CR KAN20020061 and AS CR 1QS100100553 is gratefully acknowledged.

Keywords: magnetic nanoparticles, cobalt zinc ferrite, specific absorption rate, mössbauer spectra

Possible utilization of Sr-hexaferrite/maghemite composite nanoparticles for magnetic fluid hyperthermia

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Among various methods of the oncologic therapeutic methods there is a continuous increase of an interest about the magnetic fluid hyperthermia. The method is based on an intratumoral deposition of the magnetic nanoparticles followed by a short-time exposure to a high frequency electromagnetic field. The dissipation of energy, most efficiently due to hysteresis losses, results in a local heating by the active particles and consequently to the destruction of the cancer cells. Successful use requires maintenance of the magnitude of magnetization for small particles as high as possible and adjustment of the coercivity to a reasonable value assuring sufficient heating efficiency but not too high for practical medical heating application. Simultaneously the temperature coefficient of magnetization providing self-controlled heating mechanism in the range of ~ 40–70° C is desirable. It is obvious that these conditions can be hardly fulfilled by currently used simple iron oxides, magnetite and maghemite. One of possible approaches to solve this task is the use of Sr-hexaferrite/maghemite ferrimagnetic composites, consisting of the phases possessing markedly different magnetocrystalline anisotropies. Their properties thus can be suitably adjusted by the ratio of the content of the respective magnetic phases. The studies were carried out on a set of nanoparticles with size below 50 nm prepared via sol-gel method completed by thermal heating. The d.c. magnetic studies showed the shapes of the hysteresis loops, expected from a model calculation, exhibiting an increase of the coercivity and remanent magnetization with increasing content of the Sr-hexaferrite phase. The calorimetric measurements carried out on the agarose gel suspensions under a.c. magnetic field evidenced that a combined contribution of the magnetic properties of a selected ratio of Sr-hexaferrite and maghemite spinel phases allows to adjust the coercivity and remanence of the composites appropriately for the magnetic fluid hyperthermia application. Therefore in this way the values of HC = 5.2 kA/m and 22.3 kA/m measured for composites of [SrFexO19/(1+x)]/ [γ-Fe2O3/2] molar ratio equal to 0.28 and 0.5 made possible to reach a relatively high heating efficiency of 7 – 25 W/g(Feferromagnetic) for the applied Hmax in the range of 30 – 45 kA/m and frequency ν = 108 kHz which can be substantially increased by an adjustment of the alternating field parameters.

The support by projects GAAV KJB100100701, AS CR KAN20020061 and AS CR 1QS100100553 is gratefully acknowledged.

Keywords: specific absorption rate, magnetic nanoparticles, strontium hexaferrite, maghemite

Advances in scanning probe microscopy techniques

G. Kada

Agilent Technologies, Austria

The unique combination of characterization and modification of molecules on solid surfaces with high resolution (nanometer and sub-nanometer atomic scale) in all environments (air, gas atmosphere or solution) makes Atomic Force Microscopy (AFM) and Scanning Tunneling Microscopy (STM) the techniques of choice for Nanotechnology.

Examples will demonstrate IMAGING of various substrates ranging from molecular layers to live cells, PROBING local properties such as elasticity, charge and conductivity, and MANIPULATION structures such as pulling or moving molecules or atoms.

1) **Atomic resolution imaging under controlled conditions:** operation in air and solution; both controlled temperature, controlled environment, electrochemical control and low force imaging

2) **Nanolithography**: reproducible movement on the sample surface for accurate positioning, structuring and lithography applications.

3) **Triple lock-in operation**: higher harmonics imaging, Q control and Kelvin Probe Force Microscopy for surface potential measurements.

4) **Topography and recognition:** advances in the unique AFM technology called **TREC**-simultaneous Topography and RECognition in a single scan.

5) **AFM in combination with inverted light microscope** (BioScan) for simultaneously gathering light, fluorescence and probe data of living cells.

6) **Advanced Force spectroscopy**: from intermolecular forces of ligand-receptor pairs to intramolecular forces involved in protein unfolding. New data analysis enables the correlation between kinetic and thermodynamic (affinity) data. In addition, measurements of stiffness and elasticity on various substrates will be presented.

Nanoparticle characterization using Light Scattering Techniques

Malvern Instruments Ltd., Grovewood Road, Enigma Business Park, Malvern, Worcestershire, WR14 1XZ. UK

Particle size and zeta potential are important parameters for the characterization of nanoparticles. The technique of dynamic light scattering is well suited to the measurement of the size of nanoparticle dispersions. Traditionally, measurement of very small and/or poorly scattering particles or samples that are very dilute were difficult unless high-powered lasers were used. These problems have been addressed in a light scattering instrument incorporating novel non-invasive backscatter optics (NIBS®). This novel optic arrangement maximizes the detection of scattered light while maintaining signal quality.

Laser Doppler electrophoresis is an accepted method for the measurement of particle mobility and zeta potential of dispersions of colloidal size materials. A novel scheme using a combination of Phase Analysis Light Scattering (PALS) and a patented method of measurement called M3 to improve the accuracy and ease of use of the technique will be discussed.

These technologies have been incorporated in the Zetasizer Nano Series of instruments from Malvern Instruments Ltd (Malvern, Worcestershire, UK). Examples of the application of these novel light scattering techniques to a wide range of nanoparticle samples will be presented which will illustrate the capabilities of the technology.

Nanosurf on Mars and Earth

Björn Pietzak

Nanosurf AG, Switzerland

For the first time ever, nanostructures have been measured on another planet. On July 9, the NASA "Phoenix" Mars Probe recorded images with nanometer resolution using its onboard Swiss-made atomic force microscope, and successfully transmitted these images back to Earth. The first images of the microscope — which was developed by the universities of Neuchâtel and Basel, and by the Swiss company Nanosurf — show the surface of a test grid with unprecedented detail. The successful imaging of this test grid serves as a calibration for the nanomicroscope and documents full functionality of the instrument, which is an absolute prerequisite for the scheduled microscopy measurements on Mars.
Nanosurf offers a variety of instruments and the talk will give an overview of the different product lines and its applications.

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