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Main fields of research

- Chiral separation of helicenes
- Determination on $^nJ(^{13}\text{C}-^{13}\text{C})$ coupling constants
- Fluorous organocatalysis
- Carbosilane dendrimers in bioapplications
- Antitumor properties and mechanism of action of metallocenes modified by carbohydrate or heterocyclic substituents
- Synthesis of helicene derivatives and $[n]$ phenacene derivatives
- Sensors based on laser ablated graphene
- Silicon nanowires grown on metal substrates

Applied research

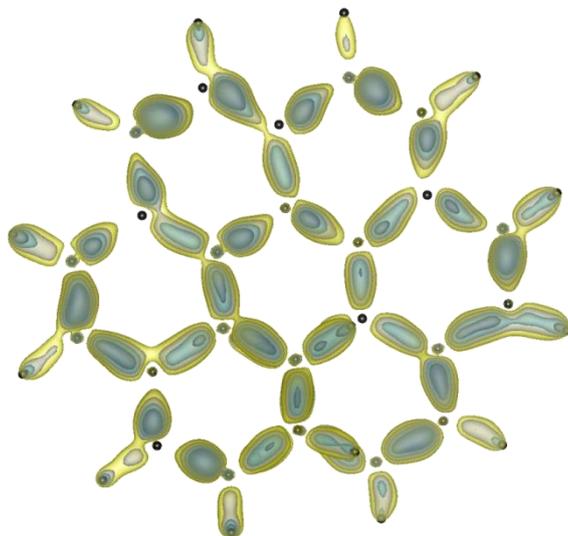
- Enzymatic optical sensor and optical fiber biosensor of glucose
- Printed optical chemical sensors
- Development of new analytical methods
- Analytical services to the research departments of ICPF
- Highly efficient catalyst and process for degradation of resistant antibiotics
- Macroporous titanium surfaces for enhancing bone osseointegration and adhesion to titanium implants

Research projects

Electron density distribution in polycyclic aromatic compounds through experiments; liquid state vs. solid state

(J. Sýkora, sykora@icpf.cas.cz; joint project with Charles University and Institute of Physics of the CAS; supported by GACR, project No. 15-12719S)

Project combines two experimental procedures to gain information about the bonding environment focusing on polycyclic aromatic compounds. The indirect spin-spin coupling constants ${}^nJ({}^{13}\text{C}-{}^{13}\text{C})$ will be measured by means of advanced NMR spectroscopy. The nJ values reflect the electron density distribution between two particular atoms. The NMR data will be correlated to the structural parameters (bonding distances and angles) provided by X-ray single crystal analysis. The found correlations will enable mutual prediction of these data. Both data types will also serve for the calibration of DFT calculations. Furthermore, suitable single crystals will be selected for experimental determination of charge density distribution. The results (critical points, partial charges etc.) will be also correlated to the original NMR data and also compared to DFT results. Final analysis of the liquid state (NMR) and vacuum (DFT) data when compared to XRD data will reveal the contribution of the intermolecular interactions in the packing to the final electron density distribution in a given molecule.



X-ray charge density analysis of [6]helicene

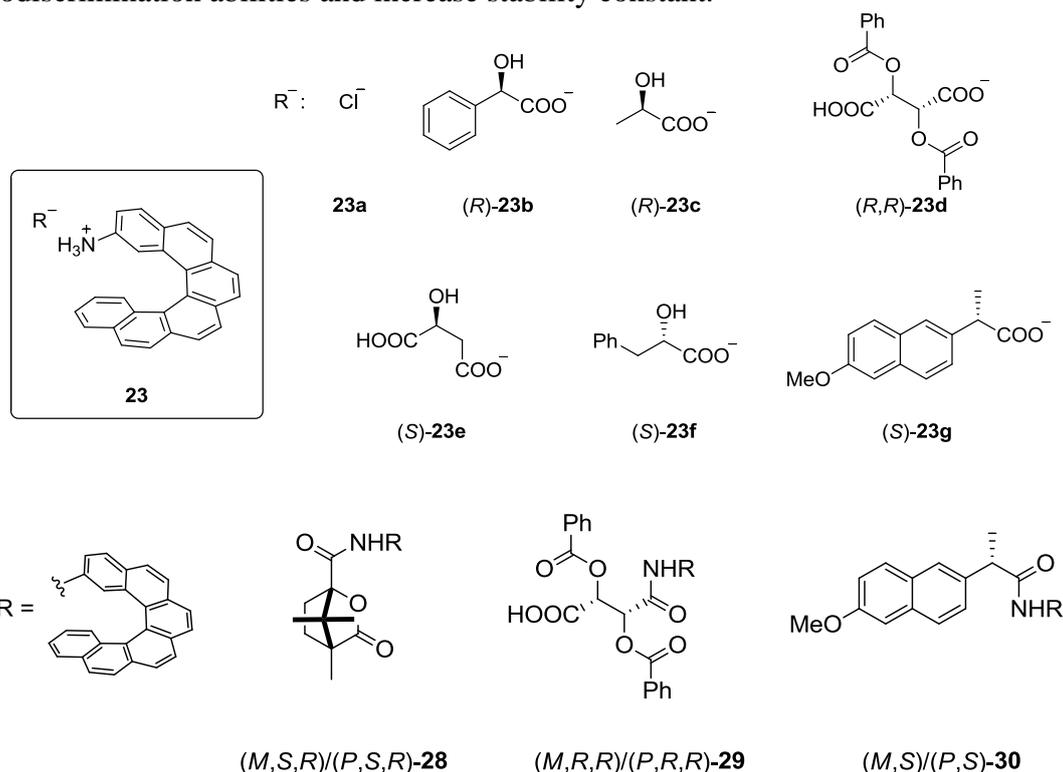
Chiral separation of helicenes

(J. Storch, storchj@icpf.cas.cz; joint project with Lach-ner, s.r.o.; supported by TACR, project No. TA04010082)

In the quest of our previous activities we have turned our attention to preparation of diastereomeric salts and amides derived from 2-amino[6]helicene as an important intermediate in further synthesis (Scheme below). It was found that diastereomeric salts are not sufficient for chiral separation since the stereocenter is too far from inherently chiral helicene backbone. Therefore we have focused on preparation of amides starting from chiral carboxylic acids. All chiral amides exhibited strong interactions in NMR spectra, however only aromatic ones can be separated on standard silicagel stationary phase with diastereomeric excess exceeding 90%. Optimization of the procedures is currently ongoing.

Electronically deficient cinchonidine and fluorenone CT-agents for complexation of unsubstituted helicenes prepared in previous year were thoroughly studied by means of NMR.

Results obtained will be further used for improvement of structures in order to improve enantiodiscrimination abilities and increase stability constant.



Diastereomeric salts and amides derived from 2-amino[6]helicene

Synthesis and Characterization of a Helicene-Based Imidazolium Salt and Its Application in Organic Molecular Electronics

(J. Storch, storchj@icpf.cas.cz; project supported by ICPF)

This work is focused on preparation of substituted imidazolium cation by [6]helicene backbone in order to improve helicene properties for the surface immobilization and enhancing the solubility in polar solvents. The deposited layer was used for the development of organic molecular semiconductor device and construction of novel type of fully reversible humidity sensor. [Ref. 28]

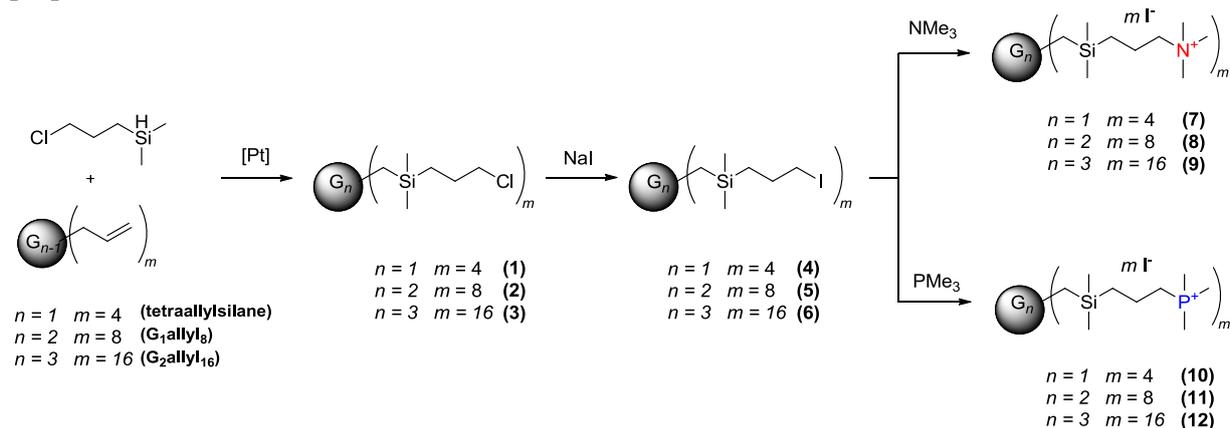


Helicene-Imidazolium salt spincoated on SiO₂ substrate with annealed gold contacts and the SEM micrographs of the amorphous (bottom) and the crystalline (upper) thin film after thermal treatment at 100 °C

Novel carbosilane dendrimers for biomedical applications - interactions with biomolecules and biomembranes

(T. Strašák, strasak@icpf.cas.cz; supported by GACR, project No. 15-05903S)

We designed and synthesized novel types of generation 1-3 carbosilane dendrimers (CS-DMMs) surface modified with various types of phosphonium and ammonium terminated CS-DDMs. In the first step carbosilane dendrimers with chloropropyl terminal were synthesized via hydrosilylation of allyls terminated starting compounds by (3-chloropropyl)dimethylsilane. Chloropropyl functionality was subsequently transformed to iodopropyl by Finkelstein reaction. Ammonium and phosphonium groups were connected by quarterisation of trimethyl amine or an appropriate phosphine by iodopropyl-terminated dendrimers. Publication is in preparation.

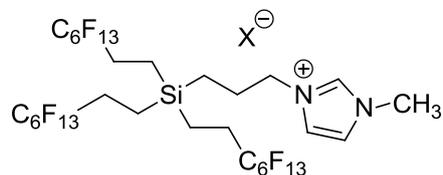


Synthetic routes leading to cationic carbosilane dendrimers

Highly fluorinated cyclopentadienes for applications in catalysis

(J. Čermák, cermak@icpf.cas.cz; supported by GACR, project No. P106/12/1372)

Fluorous ionic liquids based on 1-methylimidazolium substituted in position 3 with 3-[tris(3,3,4,4,5,5,6,6,7,7,8,8,8-tridecafluorooctyl)-silyl]propyl group were synthesized as precursors of fluorinated N-heterocyclic carbenes functioning as catalysts of redox esterification of α,β -unsaturated aldehydes. The reaction runs smoothly with primary and secondary alcohols and aldehydes that do not have a strong electron donating group. A model reaction was used to measure reaction kinetics with the salts and with a non-fluorous precatalyst [BMIM]⁺I⁻. The reaction was also tested with supercritical CO₂ as the solvent. [Refs. 29, 35]



- (5a)** X = I
(5b) X = BF₄⁻
(5c) X = PF₆⁻
(5d) X = Br⁻

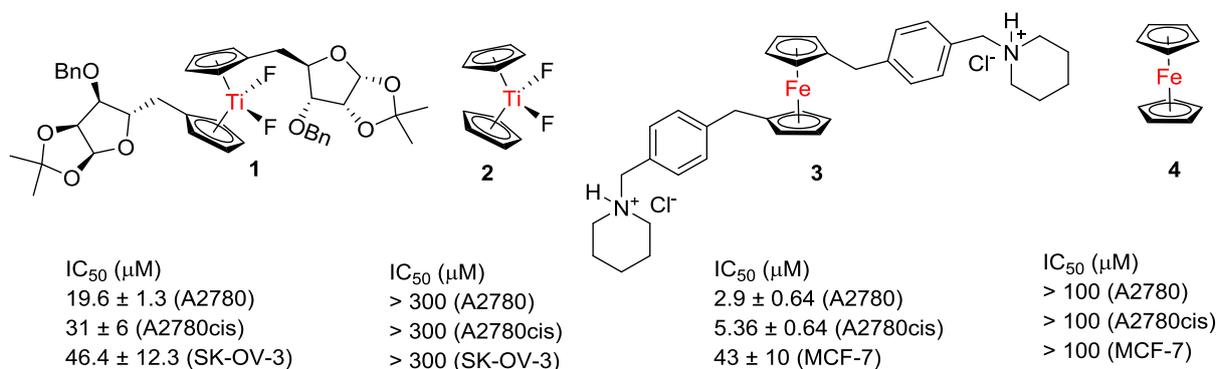
Fluorous ionic liquids

Antitumor properties and mechanism of action of metallocenes modified by carbohydrate or heterocyclic substituents

(J. Karban, karban@icpf.cas.cz; supported by ICPF)

The accidental discovery of cytotoxic properties of cisplatin [*cis*-Pt(NH₃)₂Cl₂] in 1965 initiated a continuous interest in anti-cancer properties of other transition metal complexes. Metallocenes and metallocene dihalides and pseudodihalides were soon identified as promising anti-cancer drug candidates. Titanocenes and ferrocenes were among the first metallocenes reported for anti-cancer properties. The cytotoxic activity, solubility, hydrolytic stability and other medicinally relevant properties of metallocenes are substantially influenced by substituents attached to the cyclopentadienyl ring. We initiated a synthetic and biomedical program focused on titanocenes and ferrocenes modified by a carbohydrate or heterocyclic substituents at the cyclopentadienyl ring. Titanocene difluoride substituted by *O*-protected α -D-ribofuranos-5-yl moiety **1** exhibited higher cytotoxicity than cisplatin in A2780cis (IC₅₀ 31 μ M) and SK-OV-3 (IC₅₀ 46.4 μ M) human ovarian cancer cell lines (values for cisplatin: IC₅₀ 50 μ M and 175 μ M, respectively) [Ref. 13]. Unmodified parent titanocene difluoride **2** was inactive. Further studies suggested that a stress of the endoplasmic reticulum followed by autophagy was responsible for the cytotoxicity of **1** [Ref. 13].

We have also prepared piperidinium-substituted ferrocene **3** [Ref. 1]. This compound showed cytotoxicity in a low micromolar range in human ovarian A2780 (IC₅₀ 2.9 μ M) and A2780cis (IC₅₀ 5.36 μ M) cancer cell lines. We have also developed a simple and inexpensive electrochemical method based on differential pulse voltammetry for the determination of the uptake of **3** by cancer cells [1]. Parent ferrocene **4** showed no cytotoxic properties in the tested cell lines. This correlates with the fact that only a negligible amount of ferrocene **4** penetrated into cells as showed by our electrochemical method [Ref. 1].

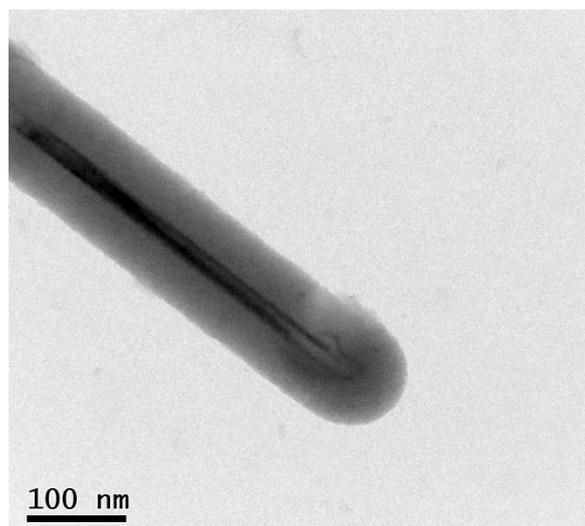


Comparison of the cytotoxic activity of modified titanocenes and ferrocenes with the parent complexes

Silicon Nanowires Grown on Metal Substrates via Self-Catalyst Mechanism

(V. Dřínek, drinek@icpf.cas.cz; supported by GACR, project No. 13-25747S)

Low Pressure Chemical Vapor Deposition (LPCVD) was applied to grow Silicon Nanowires (SiNWs) without any heteroatom catalyst or special pretreatment of substrates used. Silane (SiH₄) as a precursor was pyrolyzed at 500 °C in an oven at molybdenum or iron substrates. NWs were several microns long, about 100 nm thick and possessed core-jacket structure. The thin core is composed of crystalline silicon oriented in <110> direction whereas the jacket is amorphous silicon. Unlike other approaches this one makes possible to avoid contamination caused by metal heteroatom seeds and/or applying special procedures for substrate pretreatment initializing/supporting NW growth. [Ref. 5]

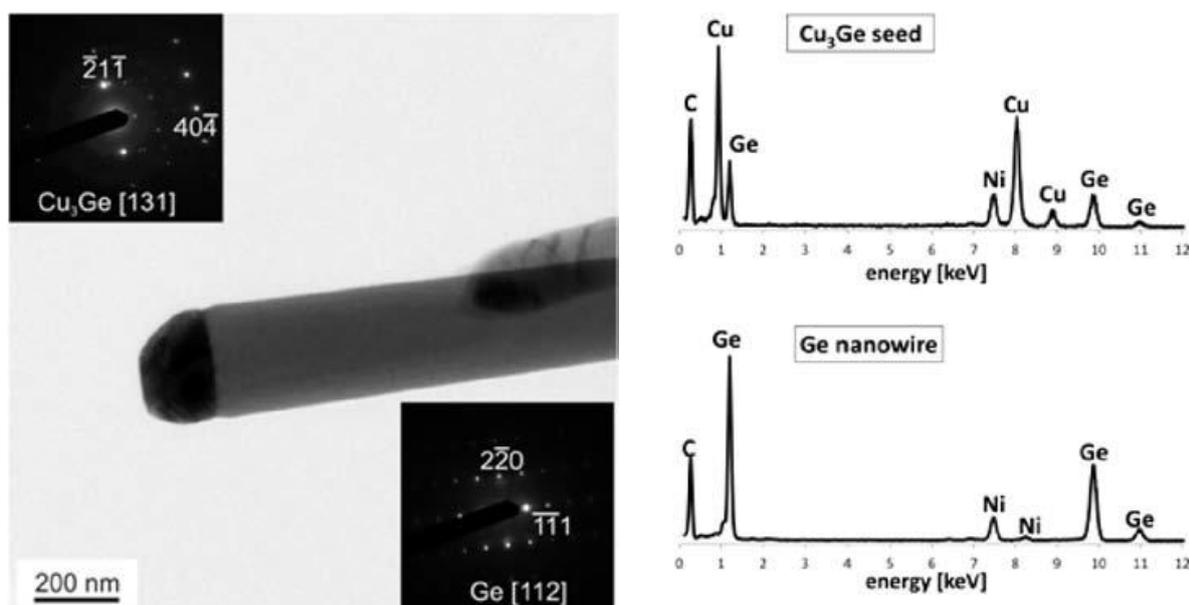


A silicon nanowire with distinguished crystalline core and amorphous jacket

Formation of $\text{Cu}_{1-x}\text{Ge}_x$ Nanoplatelets Using LPCVD of Ge_2Me_6 or $\text{Ge}_2\text{Me}_6/\text{Et}_4\text{Pb}$ Mixture

(V. Dřínek, drinek@icpf.cas.cz; supported by GACR, project No. 13-25747S)

Unlike synthesis of nanowires (1D nanoobjects), the synthesis of nanoplatelets (2D nanoobjects) has not been performed frequently. Here we prepared Cu-Ge based nanoplatelets with a high surface-to-volume ration using low pressure CVD of Ge_2Me_6 and a mixture of $\text{Ge}_2\text{Me}_6/\text{PbEt}_4$. Nanostructured deposits are composed of $\text{Cu}_{1-x}\text{Ge}_x$ nanoplatelets, Ge nanowires and Ge nanoparticles. The nanoplatelets, which have the lateral size up to several tens of micrometers and thickness of 100-400 nm, belong to the cubic α phase of $\text{Cu}_{91}\text{Ge}_9$ alloy (Ge admixture in cubic Cu) and hexagonal ζ phase of $\text{Cu}_{85}\text{Ge}_{15}$ alloy. Nanowires composed of cubic Ge have a diameter of about 30 nm and length of several tens of micrometers. [Ref. 6]



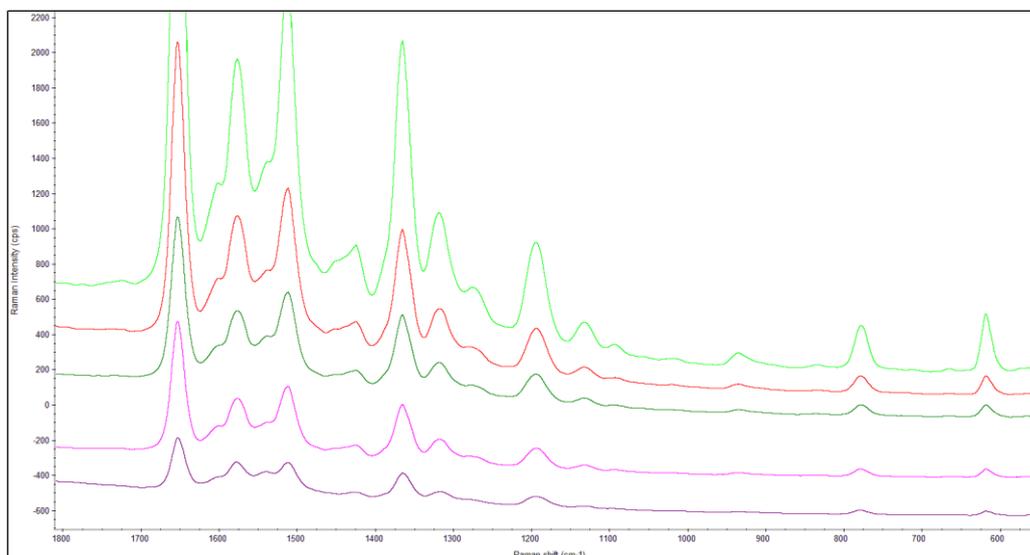
TEM observations of a deposit prepared from Ge_2Me_6 : (left) bright-field image of a Ge nanowire capped by a Cu_3Ge seed and (right) EDX spectra of nanowire and the seed

Novel sensors based on laser ablated graphene

(R. Fajgar, fajgar@icpf.cas.cz; supported by NATO, project No. 984399)

Research was focused on optimization of SERS properties of graphene based substrates and demonstration of their ability to enhance Raman scattering. To achieve lower detection limits of the technique, it is necessary to tune SERS substrates by tuning metal nanoparticle plasmons to be efficiently excited. Deposition of Au/Ag alloy nanoparticles was carried out under 10 Pa of helium used as a background gas for 6 min at 60 mJ/pulse and 10 Hz laser repetition frequency. Bulk Au/Ag alloys prepared by alloying of elemental metals were used as targets. Compositions 60Ag/40Au, 40Ag/60Au, pure Au and Ag were used for deposition of corresponding metal nanoparticles. Ablation conditions were chosen to avoid graphitization of deposited graphene/polymer composites. Prepared substrates were tested for their SERS activity towards Rhodamine 6G used as a model compound. Previous study revealed this compound as the most active model for our SERS system including Raman spectrometer with 473 nm excitation line.

Rh6G was prepared as a solution (effective concentrations 10^{-5} - 10^{-10} mol/l) in deionized water. Based on UV-VIS absorption spectrum, Rh6G electronic transition is centered at 530 nm, thus the SERS measurement should be regarded as non-resonant. Typical Raman spectra of the model compound obtained from Rh6G/Ag nanoparticles are shown below:



Rh6G/Ag SERS spectra (concentrations 10^{-6} , 10^{-7} , 10^{-8} , 10^{-9} , 10^{-10} mol/l from top to bottom)

Pure silver nanoparticles (and alloys 60Ag/40Au nanoparticles) show the best SERS properties. Increasing content of gold deteriorates the quality of spectra and gain factors of the metal nanoparticles. Our experiments demonstrate that highly non-equilibrium ablation conditions lead to homogeneous composition of Ag/Au nanoparticles and show extreme sensitivity of the SERS technique to metal surface composition.

Highly efficient catalyst and process for degradation of resistant antibiotics

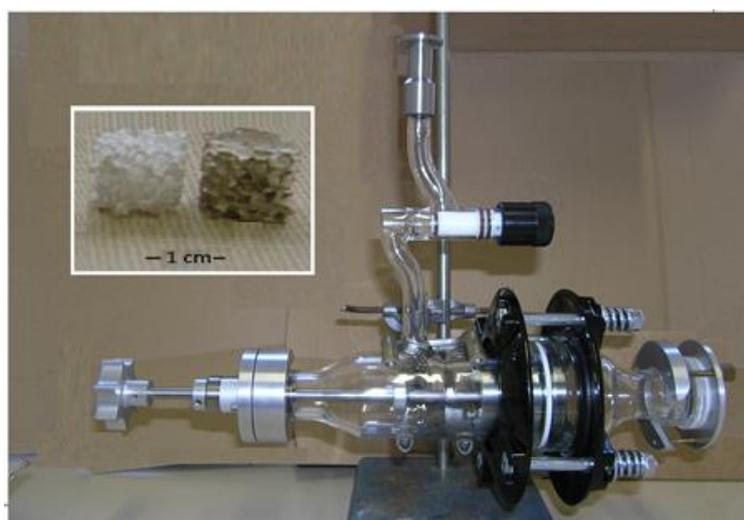
(J. Pola, pola@icpf.cas.cz; supported by TACR, project No. TA04020860)

The aim of the project is (a) the development and efficiency verification of a new-type nanocomposite heterogeneous catalysts for technological process of advanced oxidative degradation of refractory antibiotics occurring in waste waters, (b) the development and optimization of a model reactor for this process, (c) the development and optimization of a model technological process in combination with MBBR system for biological waste water

post-treatment. The novel catalysts based on grains of ferrous spinels will be laser-immobilized on high surface mesostructured walls of macroporous ceramic carriers and will have high priorities in efficient adsorption of pollutant molecules to high-surface mesoporous structure and in pollutants degradation taking place exclusively on the incorporated grains.

In 2015, ferrous spinels (magnetite, ulvospinel, chromite, hercynite), synthesized and available as raw materials served as bulk precursors for UV and IR laser ablative deposition of films on flat Ta and porous alumina surfaces. These films were examined by electron microscopy in order to determine stability of these spinels in the laser plume and their modification in the laser ablative process. Functional samples of porous catalysts to be used in Fenton degradation of antibiotics in waste water have been prepared by laser ablative deposition of magnetite, one of the studied ferrous spinels, on porous substrates from alumina and lupek by the use of pulsed radiation from TEA CO₂ and Nd:YAG lasers.

The samples were characterized by mercury porosimetry and by means of SEM and BET analyses. They possess sufficient mechanical stability suitable for the use in filtration beds of flow reactors.



Set-up for laser ablative deposition of spinels on porous alumina substrates

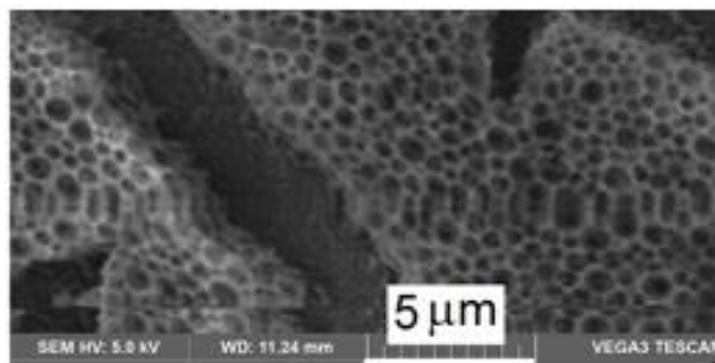
Porous and macroporous titanium surfaces with embedded submicrometer-sized Si-, SiO_x- and TiO_x- moieties for enhancing bone osseointegration and adhesion to titanium implants

(J. Pola, pola@icpf.cas.cz; supported by TACR, project No. TA04010169)

The main goals of the project are the development and optimization of a new-type of biocompatible surfaces of titanium implants for enhanced osseointegration and adhesion of bone tissue, a know-how for the production of the novel model implants, and finally the fabrication of model samples for commercial sphere. The partial goals are (a) fabrication of macroporous surface layers of bulk titanium by using laser radiation-induced structural modification of titanium, (b) penetration of sol-gel polymerizing titania and silica precursors into these layers to achieve macro-porous bicontinuous titanosilicate structures, (c) laser-induced incorporation of SiO_x and TiO_x nanoparticles and hydrated SiO_x and TiO_x nanoparticles into these structures catalyzing the growth of bone tissue, and (d) incorporation of hydroxyapatite grains into these structures, which will serve as seeds of growing bone tissue.

In 2015, an examination of the sol-gel polymerizing TiO₂ and SiO₂ progenitors for producing bicontinuous titanosilicate structures, as well as micron-sized poly/metacrylate-styrene/template copolymers-assisted polymerization of these precursors have been studied. These

polymerizations have been performed in sub-millimeter-sized porous Ti substrates and the porous silica/titania fillings have been examined by SEM analysis.



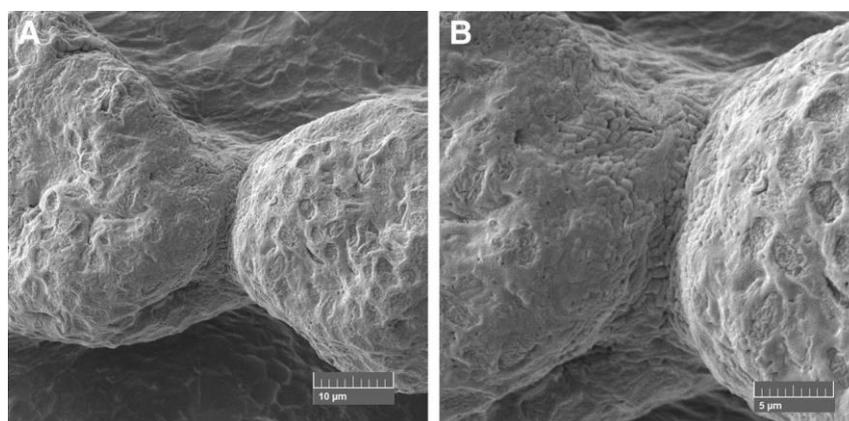
Micron-porous silica/titania filling

Printed Optical Chemical Sensors (POS)

(G. Kuncová, kuncova@icpf.cas.cz; joint project with Invos, s.r.o.; supported by TACR, project No. TA03010544)

Non-invasive detection of spoiled food inside a package enable the optical biosensor of biogenic amines with optical oxygen transducer. To estimate the behaviour of the sensor in parametric space, a mathematical model of the reaction–transport processes inside the sensing layer was developed. The model revealed the qualitative relations between the sensor analytical features, the characteristics of the sensitive layer and concentrations of substrates. The results of the mathematical modelling may serve as guidelines in the design of optodes for specific applications [Ref. 15].

The studies [Ref. 23, 24] demonstrate bioluminescent bioreporters *E. coli* ARL1 as a detector of contamination of bioavailable mercury in salty waters and tap water. The developed analytical assays overcome drawbacks of mercury detection with *E. coli* ARL1 via addition of tryptone into induction solution and preconcentration of mercury on the sorbent comprising yeast wall envelopes. The new detection assay with *E. coli* ARL1 made possible the detection of 0.57 μg/L of HgCl₂ in double-diluted artificial sea water. In tap water was detected semiquantitatively 0.025 μg/L by the induction of bioluminescence of *E. coli* ARL1 in medium with tryptone after preconcentration using a method of standard addition. The detected mercury concentrations were below limits allowed for drinking water 2 μg/L in USA, and 1 μg/L in EU.



***E. coli* ARL1 on the sorbent with adsorbed Hg²⁺ under condition of bioluminescence induction**

International co-operations

- Centre for Environmental Biotechnology, University of Tennessee, Knoxville, TN, USA:
Improved biomaterials for the encapsulation of living cells
- Environmental Sciences Division Oak Ridge National Laboratories, Oak Ridge, TN, USA:
Application of nanomaterials and novel organic-inorganic materials in optical sensors
- Graz University of Technology, Graz, Austria: ^{29}Si and ^{119}Sn NMR
- Institut de Chimie Moléculaire de Reims, CNRS 7312, France: Fluorous organocatalysis
- Lehrstuhl für Organische Chemie I, Friedrich-Alexander-Universität Erlangen-Nürnberg,
Germany: Chemistry of hetero[n]phenacenes
- Institut de Physique et Chimie des Matériaux de Strasbourg, France (CNRS): Complexation
study of chiral helicene derivatives with DNA
- Faculty of Technology and Metallurgy, University of St. Cyril & Methodius, Skopje,
Republic of Macedonia: Preparation of SERS active substrates based on graphene
- King Fahd University of Petroleum and Minerals, Dhahran, Saudi Arabia: Preparation of
Ag/C nanocomposites by laser-induced carbonization of n-hexane
- Southeast University, Department of Physics, Nanjing, China: Preparation of Ag/C
nanocomposite by laser-induced carbonization of n-hexane
- POLYMAT, Institute for Polymer Materials, San Sebastian, Spain: Laser ablation of
graphene-based composites

Publications

Original papers

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Patents

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