

INSTITUTE OF CATALYSIS AND SURFACE CHEMISTRY POLISH ACADEMY OF SCIENCES

ANNUAL RESEARCH REPORT

2009



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INSTITUTE OF CATALYSIS AND SURFACE CHEMISTRY

Polish Academy of Sciences

From theory to experiment and application

Institute of Catalysis and Surface Chemistry (ICSC) of the Polish Academy of Sciences in Krakow is the only scientific institution in Poland and one of very few in the world devoted entirely to research in catalysis and chemistry of interfaces. The Institute pursues interdisciplinary studies of the phenomena occurring at gas/solid, gas/liquid and liquid/solid interfaces, with emphasis on their significance in catalysis, materials engineering, adsorption, coating technologies and biomedical applications of dispersed systems. ICSC integrates fundamental theoretical and experimental studies, and combines them with applied research so that the results obtained can be used directly to improve technological processes.

The number of employees is about 100, of which half are research staff. Over 20 PhD students are also involved in the research. ICSC is equipped with state-of-the-art research instrumentation, which serves not only Institute's employees but is also available to the whole scientific community.

Theory

An important aspect of the research carried out at ICSC is to provide the theoretical background for experimental studies. The methodologies employed involve quantum chemistry and solid state physics modelling methods, as well as molecular mechanics and Monte-Carlo simulation techniques. The field of expertise ranges from elucidating catalytic reaction mechanisms and modelling the structures of transition metal oxides, composite nanomaterials and enzyme active centres, to describing surfactant adsorption at liquid/gas and liquid/liquid interfaces, and simulating polymer particle adsorption on solid homogenous surfaces.

Experiment

The research activities of the Institute are very broad and versatile. The bulk of research is focused on the practical implementation of scientific results and encompasses design, synthesis, testing and practical verification of new, advanced materials and processes. In the area of heterogeneous catalysis, lots of effort is put into developing new, "intelligent" materials, with welldefined structure-property relations, tuneable to the requirements of a particular catalytic reaction. The materials under investigation include new types of oxidic structures, supported metal catalysts, organometallic complexes and novel micro- and mesoporous inorganic solids. New magnetic materials and interactions between metallic nanoparticles deposited on single crystalline oxide surfaces are also investigated. The field of application encompasses selective oxidation, oxidative dehydrogenation, selective hydrogenation and isomerization of hydrocarbons. End-of-pipe environmental catalysis and green chemistry reactions are among the major points of interest. The work also involves the description of kinetic and mechanistic aspects of the studied processes. Research into homogeneous and enzymatic catalysis concentrates on bioactive and biomimicking systems. In the field of surface chemistry of dispersed systems, investigations are focused on synthesis of nano- and colloid particles and their interactions, formation and properties of thin composite films, microencapsulation and biocompatible materials as well as on description of adsorption phenomena and understanding the mechanism of formation and stability of foams. Research on application of combined photocatalytic-membrane technology for water treatment is an example of bridge between two main fields of ICSC activity. An important aspect of research is carried out by the culture heritage research group, whose work concentrates on the structure and properties of historic building and decorative materials, the deterioration mechanisms, as well as on the conservation and protection measures.

Application

Research carried out at ICSC is planned in such a way that the investigation outcomes can be turned into applications. In 1995, a spin-off company Katalizator Ltd. was created in order to promote the industrial implementation of catalytic materials designed at ICSC. This company, in which ICSC holds a control share packet, produces and sells catalysts synthesized according to ICSC-patented procedures to industrial partners in Europe and overseas. Currently, the Institute coordinates several large-scale projects aimed at implementation of results of research in practice.

Education

ICSC is a parent institution for the International Postgraduate School, organised in cooperation with the Faculty of Engineering and Chemical Technology of the Krakow University of Technology, and the Faculty of Chemistry of the Rzeszów University of Technology. The School is attended by ca. 50 PhD students. Institute also participates in the International Postgraduate Program (MPD) as well as in Interdisciplinary PhD Studies (ISD) financed by European Social Fund and operated by the Foundation for Polish Science.

National cooperation

ICSC has a long-standing tradition of animating and coordinating research in the field of catalysis and surface science in Poland. For over forty years the Institute has been organising the annual National Catalytic Colloquium, an extremely popular conference at which Polish scientists report their recent findings. The Institute is the coordinator of the National Network "SURUZ Surfactant and Dispersed Systems in Theory and Practice", and a member of the National Scientific Networks (currently financed): "PV-TECH Development of New Technologies and Research Techniques in Area of Photovoltaic Cells", "BIONAN Molecular Mechanisms of Interactions in Biological Nanosystems and in Biologically Active Systems modified with Nanoparticles" and "ARTMAG Magnetic Nanostructures for Spintronic Applications". ICSC is also the member of the National Scientific Network "New Materials–Nanomaterials for Technical and Medical Applications", the Polish Technology Platform of Sustainable Chemistry, the Regional Network of Centres of Excellence, the Regional Cluster of Information Technologies, the South Poland Regional Cluster of Clean Energy, the Consortium "Polish Synchrotron", the Consortium NANOTECH, the Consortium PAN-AKCENT, the Cluster Life Science, and the Polish Supramolecular Chemistry Network. In order to facilitate interdisciplinary research, a Joint Laboratory of Biotechnology and Enzymatic Catalysis (in cooperation with the Institute of Plant Physiology PAS), the Centre for Surface and Nanostructures Research (in cooperation with the AGH-University of Science and Technology and the Krakow University of Technology) and Joint Laboratory of Electrochemistry and Surface Chemistry (in cooperation with Institute of Physical Chemistry PAS) have been created. The Institute is also a co-founder of National Laboratory for Surface Studies.

International cooperation

ICSC is involved in a wide international cooperation. The activities include over 40 bilateral international collaboration schemes and participation in numerous EU scientific programmes. The bilateral collaboration with the Ukrainian Academy of Sciences led, in 1993, to the establishment of

a Ukrainian-Polish Laboratory of Catalysis. In the years 2002-2006 the ICSC was granted the status of an EC Centre of Excellence "CATCOLL – the Krakow Research Centre of Molecular Catalysis and Soft Matter Chemistry". CATCOLL activities contributed to the integration of the European scientific community through organising lectures by internationally renowned scientists, arranging study visits for researchers from different countries, and offering to young PhD students a unique opportunity to visit the leading European research laboratories. At present, ICSC is involved in activities of the Network of Excellence (NoE) IDECAT (Integrated Design of Catalytic Naomaterials for a Sustainable Production), which was launched in 2005 within 6th EU Framework Programme for Research and Technical Development. The Network is coordinated by Consorzio Interuniversitario per la Scienza e Tecnologia dei Materiali, Italy, and integrates 37 laboratories from 17 institutions in 11 countries (Italy, Belgium, Czech Republic, Finland, France, Poland, Spain, Sweden, Switzerland, The Netherlands, and The United Kingdom). The aim of IDECAT is to achieve a lasting integration between the main European Institutions in the area of heterogeneous, homogeneous and bio-catalysis via creating a coherent framework of research, know-how, training, promotion of science, spreading of excellence and technology transfer between the various catalysis communities.

Currently ICSC (2009) is involved in 20 European projects of which 5 are of 6th Framework Programme (ACENET ERA-NET, IDECAT, NANOMEDPART [in frames of MATERA ERA-NET] – coordination, PARFUN [in frames of MNT ERA-NET] and NANOREP II [in frames of MNT ERA-NET]), 6 are of 7th Framework Programme (InGAS, MUST, SMooHs, NEXT-GTL, ROCARE and F³ Factory), 2 are financed by EEA Financial Mechanism (NOM Remove and Wood). Institute is involved in 4 COST Actions: D36, D43, IE0601 and P21. Additionally 3 projects of Sectorial Operational Programmes (MPD, ISD and SPINLAB) are carried out. Several next 7th FP projects are being evaluated.

Cultural Heritage and Environmental Protection

One of the focal points in the research carried out at ICSC is the preservation of the natural environment and historical sites. In a collaborative effort with the Norwegian Institute for Cultural Heritage Research in Oslo, the cultural heritage research group of the Institute has animated a programme on establishing standards for the preservation polychrome wood, in particular of historic wooden churches, a unique heritage of Poland and Norway on the world scale. As a result of group activity, Roman cements, key materials for decorating facades of buildings during the nineteenth and early twentieth century, were re-established and used in renovation of several historic buildings in Europe, ensuring full compatibility between the historic substance and the repair technology. Recently, determination of the degradation mechanism of the early-Christian paintings in the St. Paulus Cave in the archaeological area of Epheus in Turkey and development of an action plan to mitigate the problems by controlling microclimate in the cave were successfully solved thanks to the research and preservation programme coordinated by the Institute's experts.

Moreover, the Institute has notable achievements in developing technologies for protecting the environment. One of them - SWINGTHERM - is a catalytic, low-energy-consumption process based on the Regenerative Catalytic Oxidation, targeted at the purification of waste gases from organic pollutants. It has already found its application in industry in Poland, Europe, Canada and Japan. In 2006 a new laboratory "Centre of Environmental Pollutants Analysis", active in the field of environmental protection control, was established thanks to the Sectorial Operational Programme grant.

Popularisation

It is our firm belief that making scientific research easily understandable to ordinary people is of immense importance for gaining public support in this area of human activities. Therefore, every year, ICSC organizes Open Door Days, during which visitors may listen to popular lectures, participate in spectacular laboratory presentations and attend poster sessions depicting Institute's research. The Open Door Days are aimed especially at young people from junior and senior high schools. They are very popular among students interested in science – every year ICSC receives ca. 1000 visitors. Institute participates also in the annual "Science in Krakow" Festival.

SCIENTIFIC TEAMS OF THE INSTITUTE 2008

Research Groups

Adsorption

Professor Władysław Rudziński, DSc, PhD Tomasz Pańczyk, PhD, Wojciech Płaziński, PhD

Catalysis I

Professor Jerzy Haber, DSc, PhD, Dhc, PAN, PAU, AE, NANU member Jan Połtowicz, PhD, Katarzyna Pamin, PhDEng, Erwin Lalik, PhDEng, Robert Karcz, MScEng, PhD student

Colloids I

Professor Zbigniew Adamczyk, DSc, PhDEng, PAU member Jakub Barbasz, PhDEng, Barbara Jachimska, PhDEng, Małgorzata Nattich, PhDEng, Lilianna Szyk-Warszyńska, PhDEng., Aneta Michna, PhD, Maja Morga, MSc, PhD student, Magdalena Oćwieja, MSc, PhD student, Monika Wasilewska, MSc, PhD student, Maria Zaucha, MSc, PhD student, Katarzyna Kusak, Eng, Elżbieta Porębska, technician

Colloids II - Colloid and Surfactant Systems

Professor Piotr Warszyński, DSc, PhDEng Grażyna Para, PhD, Ewelina Jarek, PhD, Marzena Noworyta, Eng, Anna Trybała, MSc, PhD student, Magdalena Elżbiecik, MSc, PhD student, Dorota Dronka-Góra, MSc, PhD student, Krzysztof Szczepanowicz, MSc, PhD student, Joanna Piekoszowska, MSc, PhD student, Małgorzata Adamczak, MSc, PhD student, Anna Pajor, MSc, PhD student, Justyna Dziedzic, MSc, PhD student

Colloids III - Colloid and Self-Assembling Systems

Associate Professor Paweł Weroński, DSc, PhD Małgorzata Nosek, MSc, PhD student

Cultural Heritage Research

Associate Professor Roman Kozłowski, DSc, PhD Łukasz Bratasz, PhD, Michał Łukomski, PhD, Sławomir Jakieła, PhD, Łukasz Lasyk, MSc, PhD student, Dariusz Wilk, MSc, PhD student, Bartosz Rachwał, MSc, PhD student

Dispersed Systems

Professor Kazimierz Małysa, DSc, PhD Marcel Krzan, PhD, Jan Zawała, PhD, Marta Barańska, Eng, Anna Niecikowska, MScEng, PhD student, Agnieszka Olszewska MSc, PhD student, Dominik Kosior MScEng, PhD student

Electrochemistry of Mineral Systems

Associate Professor Paweł Nowak, DSc, PhDEng Aleksandra Pacuła, PhDEng, Michał Mosiałek, PhD, Grzegorz Mordarski, MScEng, Dawid Wodka, MSc, PhD student, Małgorzata Krzak, MScEng, PhD student

Environmental Control

Tadeusz Machej, PhD Wojciech Rojek, MSc, Leszek Matachowski, MSc

Heterogenous Reaction Kinetics

Associate Professor Ryszard Grabowski, DSc, PhD Małgorzata Ruggiero-Mikołajczyk, PhD, Michał Śliwa, MSc, PhD student, Jan Mizera, MScEng, PhD student

Layered Minerals, Mesoporous Oxides, Nanostructures

Professor Ewa Serwicka-Bahranowska, DSc, PhD Małgorzata Zimowska, PhD, Roman Dula, PhDEng, Alicja Michalik-Zym, PhDEng, Dorota Duraczyńska, PhD, Elżbieta Bielańska, PhD, Daria Napruszewska, MScEng, Justyna Plona, MSc, PhD student

Polymer Based Composites as Heterogeneous Catalysts

Associate Professor Alicja Drelinkiewicz, DSc, PhD Robert Kosydar, PhD, Anna Knapik, MSc, PhD student, Adam Zięba, MSc, PhD student, Katarzyna Samson, PhDEng

Polyoxometalates

Professor Adam Bielański, DSc, PhD, Dhc, PAN, PAU member Anna Micek-Ilnicka, PhDEng, Urszula Filek, MScEng, PhD student

Quantum Chemistry I

Professor Małgorzata Witko, DSc, PhD, PAN member Renata Tokarz-Sobieraj, PhD, Robert Gryboś, PhD, Dorota Rutkowska-Żbik, PhD, Maciej Szaleniec, PhD, Jakub Goclon, PhD, Aneta Lewkowicz, MSc, PhD student

Quantum Chemistry II

Professor Ewa Brocławik, DSc, PhD Tomasz Borowski, DSc, PhD, Artur Góra, PhD, Paweł Rejmak, PhD

Surfaces, Thin films, Nanostructures

Professor Józef Korecki, DSc, PhD, Nika Spiridis, PhD, Jacek Gurgul, PhD, Robert P. Socha, PhD, Dorota Wilgocka-Ślęzak, PhDEng, Kinga Freindl, MScEng, Ewa Zackiewicz, MScEng, PhD student, Barbara Figarska, MSc, PhD student, Ewa Młyńczak, MScEng, PhD student

Zeolite Chemistry I

Professor Bogdan Sulikowski, DSc, PhDEng Ewa Włoch, PhD, Łukasz Mokrzycki, PhD, Urszula Filek, MScEng, PhD student

Zeolite Chemistry II

Associate Professor Mirosław Derewiński, DSc, PhDEng Jerzy Podobiński, MSc, Katarzyna Onik, MSc, PhD student, Adam Węgrzynowicz, MSc, PhD student

Laboratories

AFM Laboratory

Jakub Barbasz, PhDEng, Małgorzata Nattich, PhDEng

ESCA Laboratory

Jacek Gurgul, PhD, Robert P. Socha, PhD

X-Ray Diffraction and Thermoanalysis Laboratory

Professor Wiesław Łasocha, DSc, PhD Maciej Grzywa, PhD, Dariusz Mucha, PhD, Anna Dobija, MSc, Katarzyna Luberda-Durnaś, MSc, PhD student

Microcalorimetry Laboratory

Erwin Lalik, PhDEng

Scanning Electron Microscopy Laboratory

Elżbieta Bielańska, DSc, PhD, Małgorzata Zimowska, PhD

CEPA Centre for Environmental Pollution Analyses

Joanna Kryściak-Czerwenka, PhD, Janusz Janas, MSc, Joanna Opalińska-Piskorz, MSc

RESEARCH SUBJECTS AND PROJECTS IN 2009

Statutory Research

Substrate Interactions

Nanotechnology as the Basis of New Catalytic Materials

• Preparation of Colloidal, Monodispersed Suspensions of Zeolitic Nanoparticles and their Application in Preparation of New Catalytic Systems	16
 Structural Properties and Dynamics in Epitaxial Oxide and Metal Nanostructures Studied with Microscopic and Spectroscopic Techniques Radiation 	17
• New Peroxo-compounds of Mo(VI), W(VI) and V(V) – Synthesis Crystal Structure and Physico-chemical Investigations	18
Nanomaterials Derived from Layered Minerals	19
Catalytic Processes for Sustainable Development	
• Methanolysis of Triacetin as a Model Reaction System for the Transesterification of Natural Oils. Preparation and Characterization of Brønsted and Lewis Type Catalysts	21
• Synthesis of Transition Metal Salen Complexes and their Catalytic Behaviour in Processes of Removal of Allochtonic Compounds	22
 Reduced Copper and Palladium Salts of Heteropolyacids as Bifunctional Catalysts Pd and Au-Supported Catalysts, Preparation, Physicochemical Characterization and Catalytic Activity in Hydrodehalogenation of Organic Reagents 	23 24
• Investigation of CO ₂ Hydrogenation over Cu-ZnO-Al ₂ O ₃ (ZrO ₂) Catalysts Obtained from Hydrotalcite Precursors	25
Oxide-Silver Catalyst for Total Oxidation of Organic Compounds	26
• Alloys and Composites Consisting of Nickel, Cobalt, Iron, Rhenium and other Transition Metals as the Replacements of the Precious Metals in the Catalysts Used in Fuel Cells	27
 Some Reactions of the Monoterpene Hydrocarbons over Micro- and Mesoporous Materials 	28
Adsorption and Catalytic Processes Modeling	
• Structural and Electronic Factors in Catalytic Activity of Metal Sites in Enzymes and Nanoporous Materials: Quantum Chemical Investigations	30
• Sorption Processes Applied in the Environmental Protection: Theoretical Description of the Sorption Kinetics	31
• Electronic Structure of Model Heteropolyacids - Cluster and Periodic Investigation	32
Dynamics of Nanoparticle and Colloid Systems	
• Determination of Conformations of Globular Proteins in Electrolyte Solution and the Topology of their Layers at Heterogeneous Surfaces	34
• Analysis of Influence of the Air Presence at Hydrophobic Solid Surface on Kinetic of the Three-phase Formation	35
Self-Organized Structures and Functional Surface Layers	
 Thin Polymer Layers with Embedded Nanoparticles Modeling of Thin Multilayers Structures of Controlled Architecture. Theoretical Description of Spherical Particle Systems Including Particle-particle and Particle- 	37 38

Physical Chemistry in the Protection of Cultural Heritage

•	Monitoring of Preservation State of Surfaces of Historic Objects Using Optical Methods	40
Re	esearch projects of the Ministry of Science and Higher Education	
•	1 T09A 094 30 [2006-2009] Research of Effectiveness of Hydrogen Activation of the	42
•	Supported Catalysts Based on the Au Nanoparticles 1 T09A 123 30 [2006-2009] The Mechanism of Formation of Polyelectrolyte Multilayer	43
	Films with Controlled Microarchitecture on Metallic Surfaces	
•	1 T09A 122 30 [2006-2009] The Effect of Cadmium and Selenium onto Surface Properties of Model Lipid Layers Formed on the Basis of Material Obtained from Natural Cell Membranes	44
•	N 204 166 31/3734 [2006-2009] Structure, Interfacial Properties and Permeability of	45
-	Multilayer Polyelectrolyte Films for Surface Modification and Encapsulation of Chemicals	
•	N 204 164 31/3689 [2006-2009] Heteropolyacids (HPA) as he Catalysts in Gas Phase Ethyl- <i>tert</i> -butyl Ether (ETBE) Synthesis	46
•	N 205 022 31/1112 [2006-2009] New Method of Producing Nano- and Microcapsular	47
_	Systems Characterized by Targeted Functionality for a Selective Drug Delivery	40
•	Theoretical Studies on Surface Heterogeneity of Catalysts Based upon Vanadium Pentoxide	48
•	N 204 089 32/2633 [2007-2010] Mechanism of Catalytic Activity of "Nano-Au/Oxide"	49
	in Oxidation of CO	50
•	N 204 166 32/4313 [2007-2009] Synthesis of the Mesoporous Structures of the SBA-15 Type with Hierarchical Pore Distribution with the Use of Carbon or Latex Nanoparticles	50
	as a Hard Templates	
•	N 204 122 32/3142 [2007-2010] Employment of Macrocyclic Metallocomplexes of	51
•	Transition Metal Ions in Self-Assembling Systems for Catalytic Oxidation of Hydrocarbon N 204 1494 33 [2007-2010] The Influence of Kind of Central (P, Si) and Addenda Atoms	
-	(W, Mo) in the Silver Salts of Heteropolyacid with Keggin Structure on Direct	
	Decomposition of Nitrogen Oxide NO _x	
•	N N204 248634 [2008-2011] Multiple Cationic Surfactant Complexes with Polyelectrolytes as Elements of Nanostructures	53
•	N N204 249034 [2008-2011] The Study on the Application of Heterogenized Chiral	54
	Ruthenium Complexes in Enantioselective Hydrogenation of Ketones	
•	N N204 217734 [2008-2009] Photocatalytic Water Dissociation outside Earth Atmosphere. Example of Catalysis Usage in Missions of Solar System Exploration	. 55
•	N N202 248535 [2008-2011] Exchange-bias Effect in the Epitaxial Systems Obtained by	56
	Deposition of the 3d and 4f Metals on Polar and Non-polar Transition Metal Oxide	
•	Substrates N N301 093036 2009-2011] Molecular Mechanisms Determining Product Specificity	57
•	of 2-Oxoglutarate Dependent Oxygenases	57
•	N N204 028536 [2009-2012] Developing of the Methods of Formation Biologically	58
	Active Multilayers Contained Polyelectrolytes - Proteins - Dendrimers	-
•	N N507 269936 [2009-2012] New Nanostructured Carbon Materials, Oxide Materials and their Composites as Components of Capacitors	59
•	N N105 278536 [2009-2012] Acoustic Emission for Monitoring Museum Objects as a	60
	Universal Method of the Preventive Conservation	50
•	N N523 451336 [2009-2012] Controlled Adsorption as a Method of Protection Against	61
	the Environment Contamination Caused by the Oxidation of Metal Sulfides	

•	N N204 347737 [2009-2012] Theoretical and Experimental Analysis of Multilayered	62
	Spherical Colloidal Particles Porosity	

PhD research projects of the Ministry of Science and Higher Education

•	N 205 048 31/2120 [2006-2008] Synthesis and Characterization of Composite Multimodal	64
	Materials with Enhanced Acidic Properties Prepared by the Recrystallization Method	
•	N 204 150 32/3822 [2007-2009] Determination of the Influence of Adsorbed	65
	Polyelectrolytes on the Kinetics of Deposition and Topology of Particle Monolayers	
•	N 204 2145 33 [2007-2009] Deposition of Model Microcapsules on Metallic Surfaces	66
	Modified by Adsorption of Polyelectrolyte Mulitayers	
•	N 204 1238 33 [2007-2009] Transformations of Terpene Hydrocarbons over Zeolite	67
	Catalysts	
•	N N205 0189 34 [2008-2010] Influence of Modification of Electron Density of	68
	Catalysts Active Centre on Its Activity in Hydrocarbons Oxidation	
•	N N204 2559 34 [2008-2010] Encapsulation of Active Compounds in Emulsions Cores	69
•	N N204 166336 [2009-2011] Conformations of Selected Globular Proteins in Aqueous	70
	Solutions and on Interfaces of Controlled Heterogeneity	
•	N N204 279937 [2009-2011] The Physico-chemical Properties of Bifunctional Catalysts	71
	and their Catalytic Activity in Direct Syngas-to-Dimethyleter Process	
•	N N204 137537 [2009-2011] Mechanisms of Irreversible Adsorption of Nanoparticles	72
	and Selected Proteins on Interfaces Determined by Electrokientic Methods and the AFM	
-		
Po	ol-PostDoc projects of the Ministry of Science and Higher Education	
•	Epitaxial Oxide Nanostructures on Metallic Single Crystalline Substrates [2006-2009]	74

•	Establishing Standards for Allowable Microclimatic Variations for Polychrome Wood	75
	[2006-2009]	

•	Synthesis and Immobilization of New Chiral Ru(II) Complexes onto Mesoporous	76
	Supports. Application in Asymmetric Catalysis [2006-2009]	

Scientific networks of the Ministry of Science and Higher Education

	SURUZ Surfactants and Dispersed Systems in Theory and Practice (COORDINATION) PV-TECH Development of New Technologies and Research Techniques in Area of	78 79
	Photovoltaic Cells	
•	BIONAN Molecular Mechanisms of Interactions in Biological Nanosystems and in Biologically Active Systems modified with Nanoparticles	80
•	ARTMAG Magnetic Nanostructures for Spintronic Applications	81

Commissioned research projects of the Ministry of Science and Higher Education

- PBZ-MEiN-2/2/2006 [2006-2010] Chemistry of Perspective Carbon Transformation **83**
- PBZ-MNiSW-01/I/2007 [2008-2010] Investigations of the Physicochemical Properties of the Passive Layers on Aluminum, Magnesium and Titanium Alloys and Zn-Ni, Zn-Co, Sn-Zn, Co-P i Ni-P Alloy Coatings on Steel

Project of ''Technological Initiative I'' programme

 Development of a New, Wasteless Technology of Cyclohexanol/Cyclohexanone Synthesis 86 (Intermediates in Nylon 6 and Nylon 66 Synthesis) from Benzene via Cyclohexene [2008-2010]

EU 6th Framework Programme projects

- EC 6th FP NMP2-011730-2 IDECAT [2005-2010] Integrated Design of Catalytic **88** Nanomaterials for a Sustainable Production (NoE)
- EC 6th FP ERA-NET MATERA Project NANOMEDPART [2007-2010] Multifunctional **89** Particulate System for Nanomedicine (SPR-COORDINATION)
- EC 6th FP ERA-NET MNT Project PARFUN [2008-2011] Nanoscale Surface Treatments **90** to Functionalise Polymer Particles for Electronic Application (SPR)
- EC 6th FP ERA-NET MNT Project NANOREP II [2008-2011]Multifunctional Surface **91** Coatings and Highly Scratch Resistant Plactic Parts (SPR)

EU 7th Framework Programme projects

- EC 7th FP SCP7-GA-2008-218447 InGAS [2008-2011] Integrated Gas Powertrain Low 93 Emission, CO₂ Optimised and Efficient CNG Engines for Passenger Cars and Light Duty Vehicles (CP)
- EC 7th FP CP-IP 229183-2 NEXT-GTL Innovative Catalytic Technologies & Materials for **94** Next Gas to Liquid Processes (IP)
- EC 7th FP MUST [2008-2011] Multi-level Protection of Materials for Vehicles by "Smart" Nanocontainers (CP) 95
- EC 7th FP 212939 SMOOHS [2008-2011]Smart Monitoring of Historic Structures (FRP) 96
- EC 7th FP 226898 ROCARE [2008-2011] ROman Cements for Architectural Restoration 97 to New High Standards
- EC 7th FP CP-IP 228867-2 F3 [2009-2013] F3 Factory Flexible, Fast and Future Factory 98
 European Chemistry Consortium Begins the Journey into the Future of Production

EU COST Actions projects

•	EC COST D36 [2006-2011] Molecular Structure-Performance Relationships at the	100
	Surface of Functional Materials	
•	EC COST D43 [2006-2011] Colloid and Interface Chemistry for Nanotechnology	101
•	EC COST IE0601 [2006-2011] Wood Science for Conservation of Cultural Heritage	102

EC COST P21 [2006-2010] Physic of Droplets
 103

EEA Financial Mechanism projects

- EEA PL0084 NOMRemove [2007-2010] Effective Photocatalytic-Membrane Methods **106** of Removal of Organic Contaminants for Water Treatment
- EEA PL0086 [2007-2010] Establishing Standards for Allowable Microclimatic **107** Variations for Polychrome Wood

Projects of EU Sectorial Operational programmes

• PO IG 1.1.2 [2008-2012] MPD Krakow Interdisciplinary PhD Projects in Nanoscience **109** and Advanced Nanostructures

- PO KL 4.1.1 [2008-2012] ISD Advanced Materials for the New Technologies and Energy **110** of the Future
- PO IG 2.2. [2009-2011] SPINLAB National Centre of Magnetic Nanostructures for **111** Applications in Spin Electronic **111**

Other international scientific projects

- GRDE France [2007-2010] Enzymes as Nanotools Development of a New, Enzyme-Based Technology for Engineering Selectivity Permeable, Nano-Structured Membranes
- ECO-NET France [2007-2008] IUP Casein Polyelectrolyte Multilayer Films 114
- GDRI France [2007-2010] Catalysis for Environment Depollutation, Renewable Energy and Clean Fuels
- Intergovernmental Polish-Spanish Project [2007-2009] Nanostructural Micro/Mesoporous **115** Materials as New Catalysts for the Production of Environmentally Friendly Diesel Fuel
- Intergovernmental Polish-Russian Project [2008-2009] Electrostatic Interaction of Colloidal Particles Nearby to Charged Surface
- Intergovernmental Polish-Austrian Project [2009-2010] Adsorbates on Fe Monolayer on 116 W(110)

STATUTORY RESEARCH

Nanotechnology as the Basis of New Catalytic Materials

Preparation of Colloidal, Monodispersed Suspensions of Zeolitic Nanoparticles and their Application in Preparation of New Catalytic Systems

(Assoc. Professor Mirosław Derewiński, Veronika Pashkova, PhD. Jerzy Podobiński, MSc, Adam Węgrzynowicz, MSc)

The aim of the study was to prepare and characterize a highly porous systems obtained by assembling protozeolitic nanoclusters into the mesostructured material of wormhole- layered arrangement. The applied approach was chosen amongst methods of generation of acidic active sites in the mesoporous materials, due to its effectiveness in obtaining highly thermally stable and active catalytic systems.

The synthesis procedure applied consisted of two steps: i) preparation of suspension of a few nanometer protozeolitic seeds of BEA and MFI zeolite type structure and, ii) subsequent assembling into the meostructure using hexadecylamine as a template.

Although the XRD investigations did not show reflexes characteristic for the desired zeolites, FTIR spectra of prepared materials revealed much similarities with the reference MFI and BEA zeolite samples. The SEM images of the as-made material showed folded-sheet morphology of the preparations. The TEM analysis confirmed the layered structure and the presence of not-ordered wormhole-type mesopores in thin sheet-like layers. The obtained structures (after removal of organics) exhibited high specific surface area (above 900 m^2/g) and high pore volume ().

The pyridine sorption data showed presence of strong acid sites. Presence of such sites is one of the conditions required for high catalytic activity of the obtained materials. Preliminary results of catalytic tests (liquid phase isomerization of α -pinene) confirmed remarkable catalytic ability of the obtained new porous system.

Structural Properties and Dynamics in Epitaxial Oxide and Metal Nanostructures Studied with Microscopic and Spectroscopic Techniques Radiation

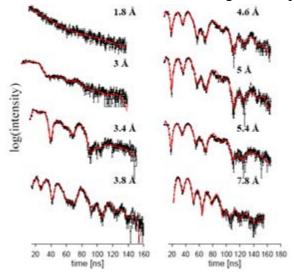
(Professor Józef Korecki, Nika Spiridis, PhD, Jacek Gurgul, PhD, Robert Soch, a PhD, Dorota Wilgocka-Ślęzak, PhDEng, Kinga Freindl, MSc, Ewa Zackiewicz, MSc [in cooperation with Academy of Mining and Metallurgy])

Hyperfine fields at the Fe(001)/MgO(001) interfaces studied by the conversion electron Mössbauer spectroscopy (CEMS).

CEMS combined the concept of isotopic ⁵⁷Fe probe monolayer in epitaxial films consisting otherwise of ⁵⁶Fe gives precise information on chemical, structural, and electronic properties at surfaces and interfaces. The Fe-MgO interface is important from the point of view of the Fe/MgO/Fe trilayers, which find spintronic application as TMR devices. Here we report on CEMS analysis of the hyperfine fields at the Fe(001)/Mg(001) interfaces in the both configurations: (i) with an MgO epitaxial film as a buffer layer and (ii) as a capping layer. The ⁵⁷Fe probe layers was placed alternatively at one of the interfaces in the epitaxial structure 5nm-MgO(001)/20nm-⁵⁶Fe(001)/5nm-MgO(001) grown on a cleaved MgO single crystal by MBE. The samples were characterized *in situ* by LEED, AES, STM and CEMS. The analysis of the CEMS spectra revealed that the studied interfaces are different both from chemical and electronic point of view. The differences were explained by different growth modes of Fe on MgO and MgO on Fe, as documented by LEED and STM measurements. Formation of an Fe oxide on a sub-monolayer scale was also evidenced by the CEMS analysis.

Growth and spin structure of ultrathin Fe films on W(110)

We investigated structure and magnetism of uncovered ultrathin epitaxial Fe films grown on W(110). In addition to standard surface sensitive techniques we applied in-situ Grazing Incidence Nuclear Resonant Scattering of X-rays (GI-NRS). The experiment was performed in a multichamber ultra high vacuum (UHV) system (base pressure $1*10^{-10}$ mbar) at ID 18 at ESRF Grenoble. ⁵⁷Fe was evaporated in a series of room temperature deposition steps, each one corresponding to evaporation of 0.4 Å, till 10 Å (~ 5ML) of the total Fe thickness was reached. After each deposition step, the NRS time-spectrum was collected at room temperature. The whole process of took only 1.5 h, and therefore the residual gas adsorption effects were negligible. The GI-NRS data were



fitted using the program CONUSS, as shown in the figure for selected Fe thickness. The spectrum of the 1.1 psML (1.8 Å) film shows no quantum beat pattern and could be fitted assuming a single paramagnetic site. The site is characterized by a distribution of the quadruple splitting due to the electric field gradient with the principal axis perpendicular to the surface. A quantum beat structure appeared in the time spectra when the nominal Fe thickness reached 3 Å, which corresponds to 1.8 psML. For coverage between 3 and 5.8 Å, analysis of the time spectra indicated a gradual transition from nearly perpendicular magnetization for 3Å film to the in plane collinear magnetic order above 5.8 Å. For intermediate coverage a complex magnetic structure was derived from the numerical analysis. The magnetic structure

is related to the film morphology characterized by a deviation from a layer-by-layer growth mode beyond the first monolayer. Competition of out-of-plane and in plane magnetic anisotropy for double layer Fe patches and for thicker Fe areas, respectively, leads to coexistence of non-collinear spin structures at buried layers, which could not be solved using traditional methods.

New Peroxo-compounds of Mo(VI), W(VI) and V(V) – Synthesis Crystal Structure and Physico-chemical Investigations

(Professor Wiesław Łasocha, Maciej Grzywa, PhD, Dariusz Mucha, PhD, Anna Dobija, MSc)

Recently we have obtained a few polynuclear peroxo-compounds of molybdenum and tungsten. Polynuclear peroxo-compounds are more stable in room temperature than mononuclear tetraperoxo- compounds, thus they can be useful in oxidation processes and photo-dynamical therapy. Our goal was to obtain quite stable, non-toxic, prone to external stimulus (e.g. light) peroxo-compounds. Since stability of peroxo-compounds depends on cation and the structure of anion, many synthesis routes, based on crystallization techniques used in protein crystallography, were tested. Among trials we have carried out, we can list:

1) saturation of H₂O-H₂O₂-(NH₄)₆Mo₇O₂₄ solutions by vapors of selected methylpyridines

2) saturation of H_2O -(NH₄)₆Mo₇O₂₄-methylpyridine solutions by H_2O_2 vapour

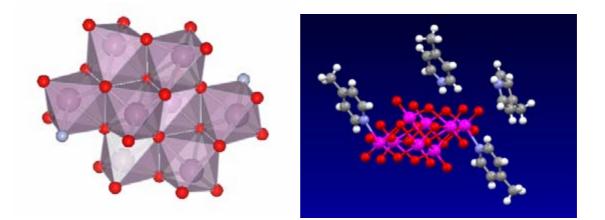
3) crystallization from series of H_2O -(NH₄)₆Mo₇O₂₄-methylpyridine-H₂O₂ solutions

4) crystallisation from series of H₂O-H₂O₂-H₂MoO₄-methylpyridine solutions

5) crystallisation from series of H₂O-H₂O₂-(NH₄)₆Mo₇O₂₄-nicotinic acid solutions

Since diffusion and crystallisation processes (in applied conditions) are rather slow, some precipitates are still a subject of observation and investigation. Till now many crystalline phases were obtained and they are the subject of structural investigations.

However, due to slowness of diffusion, high reactivity of reagents and their instability the results of synthesis are not always as one can expect. Among few new compounds we have obtained and investigated thoroughly, the most interesting seems to be γ -octamolybdate of 4-methylpyridine. In this compound some N atoms of 4-methylpyridie molecules are directly bonded to Mo atoms in octamolybdate moiety. Such compounds seem to be very interesting for medical and bio-catalytic applications.

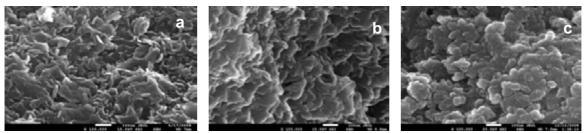


Formula - C₃₆H₄₆Mo₈N₆O₂₆, FW - 1746.31, T - 293(2) K SG - P2₁/c, a,b,c [Å]11.586(2), 15.526(2), 16.247(2), β [°]118.7530(10), V [Å³] 2562.23(6)

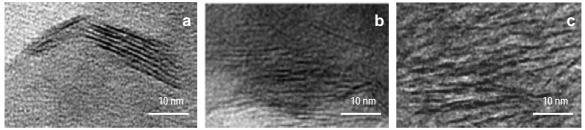
Nanomaterials Derived from Layered Minerals

(Professor Ewa Serwicka-Bahranowska, Małgorzata Zimowska, PhD, Alicja Michalik-Zym, PhDEng, Dorota Duraczyńska, PhD, Roman Dula, PhD, Elżbieta Bielańska, PhD, Daria Napruszewska, MScEng)

The study aimed at design, synthesis and physico-chemical characterization of porous clay heterostructures (PCH) derived from Laponite clay. Laponite is a synthetic layered silicate widely used in industry for manufacturing surface coatings, household cleaners and personal care products. Its easy commercial availability coupled with well defined composition makes it a particularly attractive substrate for materials design. In order to understand transformations leading to PCH formation, careful physico-chemical characterization (HRTEM, SEM, EDS, XRD, nitrogen adsorpion/desorption at -196°C, ²⁹Si MAS NMR, and thermal analysis) was carried out for solids obtained at every step of the synthesis procedure. HRTEM study showed that upon formation of PCH structure the Laponite tactoids exfoliate into primary basic units and become more randomly oriented. As a result, in contrast to materials obtained from other clays, the Laponite-derived PCHs appear amorphous to X-rays. SEM analysis shows that the molecular interactions leading to selfassembly in the Laponite/HDTMA/DDA/TEOS system induce a break up of larger plate-like Laponite agglomerates. The PCH products prepared in this work are free of sodium, which was exchanged for HDTMA⁺ cations at the stage of organoclay synthesis, and contain 3-4 times more silicon than the starting Laponite. ²⁹Si MAS NMR analysis showed that the silica network developed upon hydrolysis of TEOS and subsequent calcination contains both bulk Q⁴ and surface Q^3 sites, the former in larger quantity. Formation of PCH structures leads to an increase of specific surface area, total pore volume and average pore diameter. The change in the isotherm character from IVb for Laponite to IIb for PCHs is due to the formation of new type of linkage developed by the porous silica between the Laponite particles. The use of double amount of TEOS in the synthesis results in a material of significantly larger specific surface area (728 m²/g vs. 463 m²/g) but lesser resistance to sintering.



SEM images of a) starting Na-form of Laponite, b) HDTMA-exchanged Laponite, c) porous clay heterostructure (Laponite-mesoporous silica composite)



HRTEM images of a) starting Na-form of Laponite, b) HDTMA-exchanged Laponite, c) porous clay heterostructure (Laponite-mesoporous silica composite).

Catalytic Processes for Sustainable Development

Methanolysis of Triacetin as a Model Reaction System for the Transesterification of Natural Oils. Preparation and Characterization of Brønsted and Lewis Type Catalysts

(Assoc. Professor Alicja Drelinkiewicz, Adam Zięba, MSc, Leszek Matachowski, MScEng)

Silver salts of 12-tungstophosphoric acid namely $M_xH_{3-x}PW_{12}O_{40}$ (HPW) of silver content within the range from x = 0.5 up to 2.5 (abbreviated as Ag-0.5, Ag-2.5) were studied in the transesterification of triglycerides with methanol to give bio-ester in Biodiesel production. Catalytic testes were performed at mild reaction conditions (atmospheric pressure, temperature of 50 - 60⁰C). The course of reaction was followed by HPLC (triglyceride) and GC (methyl esters) techniques. Reaction was studied for a model triglyceride, triacetin (a shortest triglyceride molecule) and for vegetable oil, castor oil composed of triglycerides of ricinoleic acid (84 wt %) and traces of typical natural triglycerides. The results obtained over Ag-salts were compared with the data obtained in homogeneously catalysed reaction by well soluble $H_3PW_{12}O_{40}$.

Ag-salts of HPW were precipitated using appropriate amounts of aqueous $AgNO_3$ and HPW solutions. After precipitation, the obtained white colloidal suspension were slowly evaporated to dryness at temperature of 40 $^{\circ}$ C. Various techniques (BET, FTIR, XRD, XPS, laser diffraction, electron microscopy, SEM, EDS) were used to characterize as-received Ag-salts and selected samples separated after the catalytic tests. The results obtained by XRD and EDS methods evidenced the presence of crystalline phase of parent HPW at low silver content x=0.5–1. At higher content of silver, i.e. Ag-1.5 – Ag-3 no crystalline HPW was observed. Microscopic (SEM) results showed that all Ag-salts form well shaped crystalline particles of sizes differing depending on the Ag-content. The results of EDS analysis (the Ag/W atomic ratio) showed no homogeneous contribution of Ag-element in the salts, thus suggesting a presence of a mixture composed of Ag-3 and free HPW. This inhomogeneous composition was especially significant at Ag-loading below 1.5.

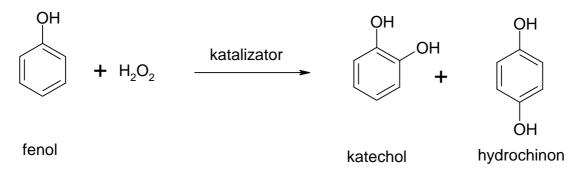
Because of polar solvent (methanol is the reactant) silver salts formed colloidal dispersion during the catalytic reaction evidenced by laser diffraction technique. Under such conditions, all silver salts prove to be active for transesterification of triglycerides. The conversion of both triglycerides, triacetin and castor oil gradually decreases as the protons in heteropolyacid are replaced by Ag⁺ cations. Partial glycerides, intermediates such as diacetin and monoacetin were formed. Methanolysis of triacetin in the presence of Ag-salts of Ag<1 was catalysed by homogeneously acting H₃PW₁₂O₄₀, owing to the leaching of acid in contact with methanol, i.e. reactant. This is not surprise owing to high solubility of HPW in methanol and highly inhomogeneous composition of these salts. Thus, owing to leaching effect of parent $H_3PW_{12}O_{40}$ by dissolving in methanol the contribution of homogeneous catalysis was high especially in the presence of Ag-salts of low silver content, such as Ag_{0.5}H_{2.5}PW₁₂O₄₀. The morphology of all the salts was remarkably changed after the catalytic tests. The form (size) of crystalline particles and the Ag/W atomic ratio were changed. This was ascribed to partial dissolution of Ag-salts in methanol, especially with Ag loading <1.5. Catalytic performance of Ag-salts for methanolysis of triacetin, a short-chain triglyceride differed remarkably from that for castor oil comprising natural, long-chain triglycerides. After transesterification of castor oil, initially crystalline particles of Ag-salts partially rearranged to "gel-type" material which makes difficult their further processing. On the other hand, although no "gel-type" particles were formed after reaction of triacetin, the formation of "nanowires" due to partial reduction of some of Ag⁺ ions was observed.

Synthesis of Transition Metal Salen Complexes and their Catalytic Behaviour in Processes of Removal of Allochtonic Compounds

(Professor Jerzy Haber, Jan Połtowicz, PhD, Katarzyna Pamin, PhDEng)

The development of civilization and industry results in pollution of environment with allochtonic compounds which do not occur in the natural environment. Phenols are pollutants most frequently present in natural environment. The removal of phenols can be accomplished by means of/on the way of catalytic oxidation. Metalloporphyrins, metallophthalocyanines and metallosalens are often used catalysts in oxidation reactions.

The aim of our study was the synthesis of cationic porphyrin and salen complexes with different transition metals and their physicochemical characterization. In the second stage of our work the synthesized catalysts were tested in oxidation of phenol in the presence of H_2O_2 as oxygen donor. Metallocomplexes were characterized by means of UV-Vis and FTIR spectroscopy.



Oxidation of phenol using hydrogen peroxide as oxygen donor in the presence of synthesized catalysts leads to the formation of catechol and hydroquinone. In standard experiment 0.05 mol of phenol was dissolved in acetonitril and combined with 0.05 mol of hydrogen peroxide (30%). Catalyst (7.8 x 10^{-6} mol) was added to the reaction mixture heated up to 80° C.

All the tested metalloporphyrins and metallosalens were active in tested reaction. For metalloporphyrins the following order of activity was established:

Moreover, catalytic activity of metallosalens was changing in the following way:

 $FeSalen - [CH_2 - P - (C_6H_5)_3]_2 Cl_2 > MnSalen - [CH_2 - P - (C_6H_5)_3]_2 Cl_2 > CoSalen - [CH_2 - P - (C_6H_5)_3]_2 Cl_2.$

FeTMPyP metallloporphyrin turned out to be the most active of all tested catalysts. FeSalen- $[CH_2-P-(C_6H_5)_3]_2Cl_2$ was the most active among all metallosalen complexes.

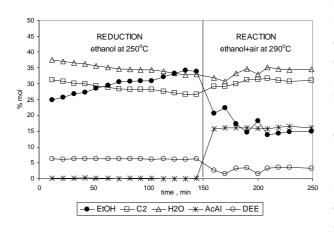
The type of central atom influences the catalytic activity of studied catalysts. The most active were iron complexes. On the contrary, cobalt catalysts were the least active. Moreover, it was proven that also the ligand structure influences the catalytic activity of tested catalysts. Metallocomplexes with positive charge on nitrogen atoms demonstrated higher catalytic activity than metallocomplexes with positive charge on phosphorous atoms.

Reduced Copper and Palladium Salts of Heteropolyacids as Bifunctional Catalysts

(Professor Adam Bielański, Anna-Micek-Ilnicka, PhDEng, Urszula Filek, MScEng))

As it was show previously by our study of $Ag_6P_2W_{18}O_{62}$ the salts of heteropolyacids containing easily reducible cations are interesting bifunctional catalysts, active both in acid-base and redox type reactions. In 2009 an analogous study of copper salt $Cu_3P_2W_{18}O_{62}$ has been carried out by us. The crystalline $Cu_3P_2W_{18}O_{62}$ ·c.a. $30H_2O$ salt has been obtained by the precipitation from aqueous solution of $H_6P_2W_{18}O_{62}$ with stoichiometric amount of copper acetate. Conversion of ethanol in gas phase has been chosen as the catalytic test reaction of the reduced salt.

Each catalytic run comprised two stages: - reduction of copper salt with ethanol (230-290°C) or with methanol vapors (200-290°C) - the proper catalytic test in which ethanol vapour conversion was registered (230-290°C). Helium or air was used in the second stage of experiment as the carrier gas for the saturated ethanol



vapour. Fig. 1 shows a typical course of the whole run. The observed products of the catalytic reaction were ethylene (C2) and diethyl ether (DEE) forming on the catalytic acid centers and also acetaldehyde (AcAl) forming on redox type centers. In the first stage of experiment (reduction) the catalyst exhibited already certain activity. After the stabilization of which the second stage (catalytic reaction at a chosen temperature) was started. At the absence of air conversion of ethanol was about 10%, and the main products of catalytic reaction were C2 and DEE. On the

other hand if air was used as the carrier gas selectivity to AcAl increased at 250°C nearly to 65.5% while selectivity to C2 and DEE although reduced was still appreciable. thus showing the distinct bifunctionality of the catalyst. The exact data concerning its activity are given in the Table.

reduced by ethalor at 250 C, in ethalor conversion								
	Reaction.	Conversion	Selectivity to (%)			Yield (%)		
Reaction	temperature	(%)						
	(°C)		DEE	C2	AcAl	DEE	C2	AcAl
	230	6.1	15.5	82.3	2.2	1.0	5.0	0.1
in helium	250	5.6	2.4	89.3	8.3	0.1	5.0	0.5
	290	8.2	8.5	78.3	13.2	0.7	6.4	1.1
	230	19.5	25.1	37.9	37.0	5.2	7.5	6.7
in air	250	39.6	9.3	25.2	65.5	4.2	11.2	24.1
	290	75.8	11.1	58.3	30.6	8.5	44.2	23.1

Table. The average conversion, selectivity and yield for CuD, reduced by ethanol at 250°C, in ethanol conversion

The X-ray microanalysis of non reduced copper salt carried out in a scanning electron microscope has shown very uniform distribution of both copper and tungsten at the surface of catalyst. However, in the case of salt reduced with ethanol at 250° C distinct aggregations of copper needles of the size of about 1µm were observed.

Pd and Au-Supported Catalysts, Preparation, Physicochemical Characterization and Catalytic Activity in Hydrodehalogenation of Organic Reagents

(Assoc. Professor Alicja Drelinkiewicz, Wojciech Stanuch, MSc)

Palladium and gold catalysts (1 - 2wt. % metal) supported by polyaniline (PANI), an electroactive polymer were prepared and characterized by BET, XRD and electron microscopic techniques (SEM, TEM). They were prepared by impregnation method using aqueous solutions of AuCl₃ and PdCl₂ of appropriate acidity, adjusted by HCl. Due to redox properties of PANI, during preparation stage, palladium was partially reduced to Pd-metal which appeared in the form of crystallites of ca. 20 nm in size uniformly distributed throughout the polymer aggregated particles. The reduction of Au-ions was complete and randomly distributed particles of Au-metal of size within wide range from 5 nm up to 200 nm were formed. They were composed of a number of nano-particles being highly aggregated particles. This shows that in contrast to palladium, polyaniline matrix did not stabilize well dispersed nano-particles of Au. Therefore, palladiumsupported by PANI were selected for catalytic experiments. The activity of Pd/PANI composites was evaluated in hydrodehalogenation of aryl ketone, namely p-chloroacetophenone. Reaction was studied at atmospheric pressure of hydrogen, temperature of 20-40 ^oC using multiphase solvents system composed of isooctane-water (1 : 1 v/v). In view of literature data two-phase solvents system is preferred because HCl – a poison for palladium formed by dehalogenation of the substrate is under such conditions removed from isooctane phase of reactants, i.e. the phase were reaction is accomplished. Literature data demonstrated that to facilitate the process of HCl removing from isooctane phase, various phase transfer agents were commonly used, like Aliquat 336 (tricaprylmethylammonium chloride). Moreover, in reaction catalysed by Pd/C, Pd/SiO₂ catalysts, the presence of Aliquat 336 was found to facilitate chemoselective hydrodehalogenation of p-chloroacetophenone to phenylethanol, without transformation of the latter to ethylbenzene or hydrogenation of aromatic ring. Without Aliquat 336, a mixture of various products was commonly obtained. In the present experiments, apart from Pd/PANI catalysts of various Pd content (1-2 wt % Pd) also 2%Pd/SiO₂ catalyst was tested. This typical catalyst was used in order to find the role of phase transfer agent, Aliquit 336. Hydrodehalogenation of p-chloroacetophenone in the presence of 2%Pd/SiO₂ was studied without and with Aliquat 336. By changing the loading of catalyst and the content of Aliquat 336 relative to the catalyst, the conditions giving chemoselective reduction of p-chloroacetophenone to phenylethanol were established. Catalytic experiments in the presence of Pd/PANI catalysts were performed using various loading of Pd/PANI catalysts and various initial concentration of p-chloroacetophenone. Furthermore, NaOH was also added to water phase in order to neutralize HCl formed in the course of hydrodehalogenation reaction. It was observed that in contrast to reaction catalysed by 2%Pd/SiO₂, in the presence of polyanilinesupported Pd no phase transfer agent was necessary to have chemoselective formation of phenylethanol (selectivity ca. 95 %). This shows specific role of polyaniline support. Polyaniline exhibits alkaline character (due to the presence of N-groups) and HCl formed in the reaction reacted with PANI forming polyaniline-salts. This effect was evidenced by FT-IR spectra. However, the presence of PANI-salt, which may be potential acid catalyst, did not facilitate the formation of ethylbenzene via hydrogenolysis of phenylethanol. Therefore, chemoselective hydrodehalogenation of p-chloroacetophenone to phenylethanol in the presence of Pd/PANI catalysts was ascribed to an influence of polymer matrix in the nearest environmental of Pd-particles on geometry of substrate adsorption, facilitating its adsorption via aromatic ring.

Investigation of CO₂ Hydrogenation over Cu-ZnO-Al₂O₃(ZrO₂) Catalysts Obtained from Hydrotalcite Precursors

(Assoc. Professor Ryszard Grabowski, Professor Ewa Serwicka-Bahranowska, Michał Śliwa, MSc, .Justyna Plona,MSc, Jan Mizera, MSc, Małgorzata Ruggiero-Mikołajczyk, PhD)

Activity of the CuZnAl and CuZnAl-ZrO₂ catalysts has been determined in the hydrogenation of CO₂ to methanol and to dimethyl ether (DME). Bifunctional catalysts used for DME synthesis consist of the metallic part (CuZnAl and CuZnAl-ZrO₂ catalysts) and acidic component. The commercial montmorillonite K10, modified with heteropolyacid $H_3PW_{12}O_{40}$ was applied as the acidic component.

The Cu-Zn-Al-O and Cu-Zn-Al-ZrO₂ precursors were synthesized by co-precipitation method at pH=7 and pH=9. The XRD analysis revealed that precursors consist only of hydrotalcite-like phase. In order to obtain the active phase, the precursors were calcined at 550°C for 3h. XRD, BET, TPR, SEM, ICP and N₂O specific adsorption techniques were used physico-chemical characterization of the synthesized catalysts. The catalytic measurements were performed in the high pressure fixed-bed flow reactor in the temperature range 180-240°C under pressure of 80 bars for methanol, and in the temperature range 240-320°C, under pressure 40 bars for DME synthesis and GHSV = $3600h^{-1}$ for both reactions.

In methanol synthesis the investigated catalysts exhibit high selectivity to methanol. In case of CuZnAl-ZrO₂ at the reaction temperatures above 200°C the presence of CO and DME in the reaction products is observed. The appearance of DME in the reaction products shows that on catalyst where some amount of Al_2O_3 was replaced by ZrO_2 methanol undergoes dehydration at higher temperatures. Amongst CuZnAl catalysts obtained at pH=7, those having the smallest average CuO crystal size of (5,2nm), exhibits the highest yield to methanol, equal 20%.

The influence of composition modification and synthesis method of metallic function on the activity of final bifunctional catalysts was determined. The CuZnAl catalyst obtained at pH=7 was used as metallic components in optimization measurements of the composition of bifunctional catalyst. Based on the obtained results it was found that metallic (M)/acidic(A) volume ratio equal 1:1 is optimal for DME synthesis. Higher contents of acidic component do not improve the yield of DME.

The increase of the DME yield is connected with the decrease of the M:A and results from equilibrium shift towards DME formation. The increase in the numbers of acidic sites and hence the increase of the acidic component content leads to the higher rate of methanol dehydration. The performed catalytic tests suggest that each bifunctional catalyst with the ratio M:A = 1:1 exhibits similar yield peak to DME (6-7%) in the temperature range $280 - 295^{\circ}$ C. The modification of metallic component by the addition of ZrO₂ and change of pH synthesis has not considerable affected on the catalytic activity of the bifunctional catalysts in the CO₂ hydrogenation to DME.

Oxide-Silver Catalyst for Total Oxidation of Organic Compounds

(Tadeusz Machej, PhD, Wojciech Rojek, MSc)

Volatile organic compounds (VOC) can be destroyed by means of thermal or catalytic combustion. These compounds are combusted in air to CO_2 and water when VOCs contain carbon, hydrogen and oxygen. In the best case the heat of reaction sustains the combustion process.

Catalytic combustion takes place at much lower temperatures (200-500°C) than the thermal combustion (750-900°C). So, the catalytic combustion is a self-sustaining process taking place at much lower concentration of VOCs than in the case of thermal combustion. The catalytic combustion is a better economic solution because of low operation costs. The operation costs can be further decreased by using catalysts operating at the temperature range 100-200°C. Standard catalysts can not be used below 200°C because of the adsorption of VOCs and the reaction products.

Aim of the research was a development of the catalyst which would be effective in the VOCs combustion below 200°C, i.e. exhibiting high VOC conversion without adsorption of substrates and products.

The preliminary results showed that a standard $Pt//\gamma$ -Al₂O₃ catalyst has not been active in the total oxidation reaction of hydrocarbons, both aliphatic and aromatic because of the strong adsorption VOCs and CO₂ below 200°C which resulted low catalytic activity whereas organic compounds e.g. alcohols were converted to CO₂, H₂O and aldehydes.

Total oxidation of methanol and ethanol were therefore chosen as the test reactions.

Ag/MnO_x/ γ -Al₂O₃ catalyst was prepared. Total oxidation of methanol and ethanol was carried out in a flow reactor at GHSV=10.000 h⁻¹. The concentration of reagents were 0.2 g/m³. The conversion of alcohols was monitored by measuring the concentration of CO₂ in outlet of the reactor. It was found that the oxidation of methanol is effective below 200°C. The methanol conversion and selectivity to CO₂ were 100 % at the temperature 175°C.

Total oxidation of ethanol (Fig. 1) takes place at higher temperatures than in the case of methanol. 100 % of the conversion and selectivity to CO_2 are observed at 200°C. Acetaldehyde is not observed in the reaction products. At low temperatures ethanol is partially converted to acetaldehyde.

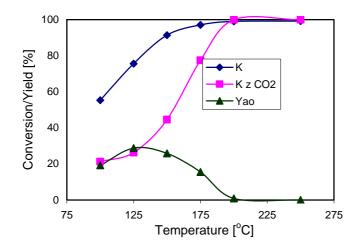


Fig. 1. Conversion of ethanol (K), and the yield of acetaldehyde $(Y_{ao}) \mbox{ as } a \mbox{ function of reaction temperature}$

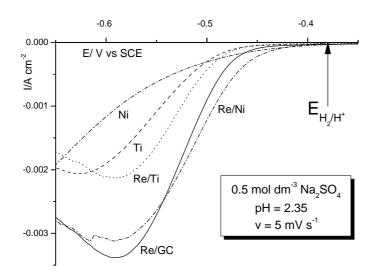
Alloys and Composites Consisting of Nickel, Cobalt, Iron, Rhenium and other Transition Metals as the Replacements of the Precious Metals in the Catalysts Used in Fuel Cells

(Assoc. Professor Paweł Nowak, Robert Socha, PhD, Grzegorz Mordarski, MScEng, Małgorzata Krzak, MScEng)

Growing deficit of energy caused that the research in the field of alternative energy sources becomes more and more important. One of the most intensively studied research area in the domain of catalysis is the synthesis of new catalysts for fuel cells and for hydrogen production by water electrolysis. Water electrolysis with the following storage and application of the produced hydrogen in the hydrogen fed fuel cells may be an attractive solution of the problem of decreasing energy consumption during the night and constant rate of energy production.

Serious problem hampering the development of fuel cells and hydrogen energetics is the deficit of some precious metals, used in the fuel cells as catalysts (platinum, palladium). The same metals show the best catalytic properties in the reaction of hydrogen evolution, but practically they are not used in the industry due to the very high price. So we have undertaken the investigations in the field of preparation and characterization of alloys and composites consisting of nickel, cobalt, iron, rhenium and other transition metals, which might replace precious metals as catalysts. Our interest in rhenium stems from the fact that Poland is one of the biggest producers of rhenium in the world and Polish industry is interested in finding new applications for rhenium.

The process of rhenium electrodeposition on different substrates: constructional steel, stainless steel, titanium, copper and nickel (materials used in the industry for construction of electrodes) and on glassy carbon (in view of the possible preparation of carbon/rhenium composites for the use in fuel cells) was investigated. Rhenium as well as their alloys with many metals may be easily deposited by electrolysis on different conducting substrates. The kinetics of hydrogen evolution on obtained materials was investigated with the application of polarization curve method (using Tafel relation) and with the use of electrochemical impedance method. The composition of the surface was investigated by X-ray photoelectron spectroscopy (XPS) method.



Rhenium and its alloys show low overpotential of hydrogen evolution (see Fig. 1). In alkaline solutions (such solutions are used in the industry) the lowest overpotential showed the rheniumcobalt alloy deposited on iron or nickel substrate. However low corrosion rhenium in resistance of oxidizing conditions may create problems in using rhenium.

Fig. 1. Polarization curves of several materials: nickel, titanium as well as rhenium deposited on nickel, titanium and glassy carbon, in acidic solution.

Some Reactions of the Monoterpene Hydrocarbons over Micro- and Mesoporous Materials

(Professor Bogdan Sulikowski, Ewa Włoch, PhD; Łukasz Mokrzycki, PhD; Urszula Filek, MScEng)

A whole series of hydrocarbons with various structures belongs to terpenes. They are still of paramount importance for chemical, food, cosmetics, and pharmaceutical industries. Manufacturing of such chemicals from crude oil or carbon resources, although theoretically possible, would have required a multi-stages production, and hence is unacceptable from economical point of view. In the period reported, our work was concerned with, first of all, on studies concerning the isomerization and oxidation processes proceeding in the liquid-phase.

 α -Pinene is the most abundant member of the monoterpene hydrocarbons as well as is the major component of the crude sulfate turpentine. That makes it inexpensive and convenient substrate which can be transformed to numerous compounds, of which the most important are camphene, limonene and *p*-cymene. Catalytic isomerization of α -pinene is nowadays carried out using TiO₂ impregnated with the sulphuric acid. The process has disadvantages, such as the low rate of the reaction and corrosion problems; we had therefore decided to study other viable, micro- and mesoporous catalysts. Isomerization of α -pinene was studied over synthetic zeolites: Y, ZSM-5, ZSM-12 and MCM-22. Proper hydrothermal conditions were chosen in order to form mesoporosity in the zeolite crystals. For example, ZSM-5 treated with concentrated NaOH solutions led to catalysts exhibiting significant higher conversion of α -pinene.

Dealuminated zeolite Y comprises another series of the samples investigated in the isomerization of α -pinene. These micro/mesoporous materials were obtained by a post synthesis treatment of zeolite Y, dealuminated chemically by means of ethylenediaminetetraacetic acid (H₄EDTA), what led to: (i) the change of Si/Al ratio, (ii) formation of mesoporous system, and (iii) brought about changes in the morphology of the crystals. Generally, dealumination improved the catalytic activity of the faujasite-based catalysts. For example, unmodified Y type zeolite exhibited conversion at the level of ca. 14 mol.%, whereas the 30%-dealuminated sample was 3-4 times more active. Physico-chemical characteristics of the samples were carried out using a number of methods (XRD, nitrogen sorption, NMR, SEM, and XPS). Moreover, the changes of acidity occurring in the desilicated zeolitic materials were followed qualitatively and quantitatively by means of pyridine sorption.

Physico-chemical characteristics of the $AlPW_{12}O_{40}$ salt was carried out, using first of all ¹H and ²⁷Al MAS NMR. The strongest dehydroxylation of the salt occurred at 473-573 K. The changes of the coordination of aluminium during dehydroxylation of the salt were demonstrated. Studies on oxidation of norbornene towards epoxynorbornane over heteropolyacids and their salts were continued.

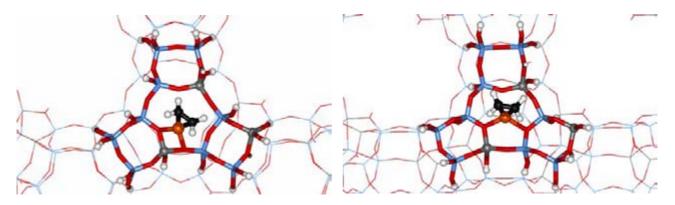
Finally, a series of ammonium perchlorate samples with various, and also full, degree of deuteration, was successfully prepared. Low-temperature, thermal decomposition of such samples was followed. It was found that thermal decomposition depends on the degree of deuteration and begins at the intersection of dislocations within the crystal and not on the crystal faces.

Adsorption and Catalytic Processes Modeling

Structural and Electronic Factors in Catalytic Activity of Metal Sites in Enzymes and Nanoporous Materials: Quantum Chemical Investigations

(Professor Ewa Brocławik, Assoc. Professor. Tomasz Borowski, Paweł Rejmak, PhD)

Ethene adsorption on Cu(I) sites in zeolites of faujasite and MFI types has been Subtask I: investigated. Ethene is employed as a probe molecule in infrared studies of zeolites; it is also a possible participant of the reactions of prospective practical importance, catalyzed by these systems (i.e. the selective catalytic reduction of nitrogen oxides). Optimal structures, interaction energies and harmonic vibrational frequencies have been calculated by means of the combined methods of quantum and molecular mechanics (QM/MM). For deeper insight into the nature of ethene - active site interactions the novel analysis of electron density flows and decomposition of interaction energies, called ETS-NOCV method, has been used in the quantum part of calculations. ETS-NOCV calculations have been performed in collaboration with the co-author of this approach, Dr. M. Mitoraj from Jagiellonian University. QM/MM calculations show, that C=C stretching frequencies of adsorbed ethene molecules are not site specific and depend almost exclusively on the type of copper ion coordination to framework O atoms, namely coordination either to single lattice tetrahedron (lower frequency band) or to two tetrahedrons (higher frequency band). Natural Orbitals for Chemical Valence, supported by energetic analysis, explain this effect as stemming from stronger π^* -back donation in the first type of adsorption complexes. In addition, their stronger stabilization despite the larger relaxation of Cu(I) site due to the adsorption, can be explained as the effect of more favorable electron interactions. Owed to our work experimental infrared spectra may be explained.



Two types of adsorption complexes (see Figure) were found within the same type of Cu(I) site (site II in faujasite): species with Cu(I) ion coordinated to single lattice tetrahedron (on left) and with Cu(I) bound to two lattice tetrahedrons (on right). The former complex is distinguished by the lower C=C stretching frequency.

<u>Subtask II</u>: Electronic and geometric structures of NO ('non-innocent ligand') complexes with Fe(II) center in various ligand environments (mimics of enzymatic sites) have been calculated by advanced, correlated quantum chemistry (CASPT2). Resolution of multiconfiguration wave function allowed for assessing the weights of Fe(II)-NO⁰, Fe(III)-NO⁻ or Fe(I)-NO⁺ electronic resonance structures, postulated for such complexes. It was shown that the last one is of minor importance while all studied complexes were described as mixtures of Fe(II)-NO⁰ and Fe(III)-NO⁻.

Sorption Processes Applied in the Environmental Protection: Theoretical Description of the Sorption Kinetics

(Professor Władysław Rudziński, Assoc. Professor Tomasz Pańczyk, Wojciech Płaziński, PhD)

Adsorption at a solid/solution interface is one of the most commonly used physical processes applied in the purification of waste waters. Understanding mechanisms of the kinetics of this process is crucial for designing sorption processes effectively.

The main results can be summarized as follows:

- 1. The adsorption kinetics at solid/solution interfaces has been described by using the kinetic model based on accepting the existence of the concentration gradient in the region of bulk solution close to the solid surface (external film-diffusion model). This model has also been adopted to explain some behaviors observed in the real adsorption systems.
- 2. The utility of the Weber-Morris and the Lagergren linear representations has been studied. The effects of different sorbent particle sizes, non-linear adsorption isotherm equations (including the non-linearity caused by the surface energetic heterogeneity) and the changing sorbate concentration in the bulk phase have been taken into account.
- 3. A novel two-resistance model for description of the sorption kinetics by porous particles has been proposed. The model takes into account two kinetic steps of different kinds which are involved in the overall sorption process rate: (i) the rate of solute diffusion in pores of the sorbent particles having uniform sizes and characterized by the homogeneous intraparticle diffusion coefficient and (ii) the rate of a direct adsorption/desorption process on the surface, described by applying the statistical rate theory (SRT) approach.
- 4. The connection between the most commonly applied mathematical expressions, such as the pseudo-second and the pseudo-first order equations and different models of sorption kinetics was investigated. These formulae do not correspond to any specific physical model but they approximate well the behaviours predicted by many different theoretical approaches.
- 5. The heavy metal ions biosorption processes were modeled. The origin of the commonly observed, so-called "heterogeneity effects" has been explained. Two most popular models were considered for that purpose: the adsorption model and the ion exchange model. The Condensation Approximation method has been used in order to develop the model describing the influence of surface heterogeneity on biosorption equilibria. As this model has been derived by accepting the ion exchange processes on the biosorbent surface, the obtained expressions are able to take into account also the pH effect.
- 6. Two stoichiometric assumptions (MX₂ and $M_{1/2}X$) have been compared for describing the divalent metal ions binding by the protonated sorbent/biosorbent. Mathematical expressions corresponding to these both models have been developed by applying the ion exchange model and methods of statistical thermodynamics. The $M_{1/2}X$ model appears to be unphysical because it does not take into account the basic fact that two sites binding one metal ion must be neighbouring.
- 7. The possible explanation for the applicability of NCAMs (Non-Competitive Adsorption Models, i.e. the models corresponding to the Langmuir model in which the maximum uptake value is pH-dependent) for biosorption systems is proposed. Both the metal ion speciation in the bulk solution and the formation of multidentate surface complexes are taken into account.

Electronic Structure of Model heteropolyacids - Cluster and Periodic Investigation

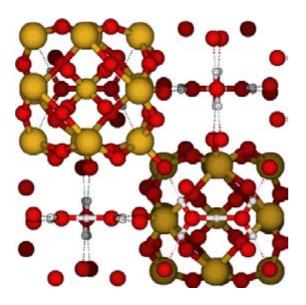
(Professor Małgorzata Witko, Renata Tokarz-Sobieraj, PhD, Dorota Rutkowska-Żbik, PhD, Robert Gryboś, PhD, Aneta Lewkowicz, MSc)

Heteropolyacids (HPA) have been known for almost 200 years, but wide employment in the industry did not start until the 70-ies of the last century. Heteropolyacids are catalyst (both in homoand heterogenous catalysis) for a number of reactions, e.g.: propene hydratation, oxidation of metacrolein, oligomerization of tetrahydrofurane and hydratation of n-butane. The structures of HPA vary greatly; in the present task only the most stable and useful types possesing the Keggin structure were investigated, the ones possesing phosphorus as the central ion, molibdenum and tungsten as addenda atoms and alkali metals as compensating cations.

Research was conducted along two paths: (i) using periodic models with the VASP code, and (ii) using cluster models with TURBOMOLE and StoBe codes. Periodic model is better suited to describing structure and properties of large crystallites present in heterogenous catalysis, while cluster approach is better for isolated Keggin structures active in homogenous catalysis.

Performed calculations had an initial and calibrating character - accuracy and adequacy of the methods used were tested, starting geometries for reaction simulations were prepared, reference energies for thermodynamic and kinetic calculations were established.

Calculations allowed for plotting electronic densities of states for pure HPAs, their salts (K, Rb, Cs) and hydrated structures.



Structure of the heteropolyacid crystallographic cell containing two $(H_5O_2)_3PW_{12}O_{40}$ units

Theoretical bond vibration frequencies were compared with the experimental spectra. Correspondence was attained only when all degrees of freedom were included. Simulations with blocked Hessian give band positions shifted by ca. 50 cm⁻¹ with respect to experiment.

The optimal structure of (001) surface, isomorphic with (010) and (100), was calculated for HPA (figure on the left) and for potassium, cesium and rubidium salts with different hydration levels.

Acid-base properties of different oxygen centers were compared for structures which differ only by the addenda atoms (Mo, W) as well as between molybdenum-based HPA and Mo oxides. **Dynamics of Nanoparticle and Colloid Systems**

Determination of Conformations of Globular Proteins in Electrolyte Solution and the Topology of their Layers at Heterogeneous Surfaces

(Professor Zbigniew Adamczyk, Monika Wasilewska, MSc, Barbara Jachimska, PhDEng, Jakub Barbasz, PhDEng)

Physicochemical properties of bovine fibrinogen (Fb) having the molecular mass of 340 kDa were characterized. The bulk characteristics of Fb suspensions involved the diffusion coefficient (hydrodynamic diameter), the electrophoretic mobility, and dynamic viscosity as a function of protein solution concentration, for various pH. Measurements of the hydrodynamic diameter revealed significant increase for the pH range 5 < pH < 7, which was interpreted in terms of molecule aggregation due to decreased surface charge. In order to test this hypothesis throughout measurements of the electrophoretic mobility of Fb as a function of ionic strength and pH have been carried out. Using the hydrodynamic diameter and the electrophoretic mobility data, the number of uncompensated (electrokinetic) charges on protein surfaces was determined. The electrophoretic mobility data were also converted to the zeta potential values, which allowed one to determine the isoelectric point (i.e.p.) of Fb (see Fig.1) It was found to be at pH = 5.8 in accordance with previous experimental data. Hence, for pH < 5.8 the Fb acquired a positive charge reaching the maximum value of 34 elementary charges for pH = 3. This important finding suggested that a significant adsorption in the side-on orientation of the molecule can take place on negatively charged substrate surfaces, e.g., mica. In order to determine in more detail conformations Fb at various pH, dynamic viscosity measurements were carried out as a function of their bulk volume concentration. The intrinsic viscosity derived from these measurements was interpreted in terms of the Brenner's model applicable for slender bodies. It was found that significant unfolding of the molecule took place at pH < 5 and pH > 7 due to high charge of Fb for these pH ranges. On the other hand, for pH near the isoelectric point, the Fb molecule assume a more compact shape with the side arms collapsed towards the core region.

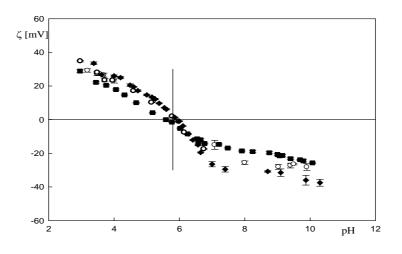


Fig.1. Zeta potential of fibrinogen as a function of pH, T = 298 K. The points denote experimental values determined for: $\bullet I = 1 \times 10^{-4}$ M, o $I = 1 \times 10^{-3}$ M, $\blacksquare I = 10^{-2}$ M. The vertical line shows the position of the isoelectric point (pH= 5.8)

Analysis of Influence of the Air Presence at Hydrophobic Solid Surface on Kinetic of the Three-phase Formation

(Professor Kazimierz Małysa, Marcel Krzan, PhD, Jan Zawała, PhD, Anna Niecikowska, MScEng)

The study was focused on importance of air presence at hydrophobic solid surfaces in formation of the three phase contact (TPC) - liquid/gas/solid - under dynamic conditions. Hydrophobic surfaces show high affinity to air and most studies of colloid chemistry is carried out in gas atmosphere - either in air or inert gas. Immersion into water phase does not guarantee that air is removed from the hydrophobic surface. Presence of air in a form of nano-bubbles is currently the most accepted explanation of the origin of the long range (up to hundreds nanometers) hydrophobic forces (LRHF), namely these long-ranged interactions are due to bridging of the nanobubbles attached to the interacting hydrophobic surfaces. High stability of the nanobubbles on hydrophobic surfaces is attributed to their flattened shape, which is caused by much larger values of the nanoscopic contact angle than the macroscopic ones, and/or due to the line tension action.

When the hydrophobic surface, contacted earlier with air, is immersed into water then air can be entrapped and stay attached to the surface. Amounts of gas entrapped can depend on properties of the solid surface being immersed into liquid phase. It was found that roughness of the hydrophobic solid surface had large influence on time of the three phase contact formation by the colliding bubble. Teflon is a model hydrophobic solid having advancing contact angle of ca. 120°, which did not vary significantly when the roughness of the Teflon plate was changed. However, roughness of the Teflon surface changed drastically the kinetics of the bubble attachment (time of TPC formation $- t_{TPC}$). The t_{TPC} was shortened from over 80 down to 2-3 ms when the roughness was increased from below 1µm to over 50µm. In the case of the Teflon surfaces having roughness below 1µm there were recorded four to five "approach-bounce" cycles prior to the bubble attachment. Simultaneously, after the first collision the bubble shape started to pulsate rapidly within time intervals of an order of fraction of millisecond. In the case of the Teflon plates having surface roughness of ca. 50 µm and larger, the attachment always occurred during the first collision - there was no bouncing observed and the time of the attachment was below 3 ms. This shortening of the time of the bubble attachment with increasing surface roughness was caused by larger amounts of air entrapped during the rougher Teflon plate immersion. The redistribution of the air entrapped caused that a larger area of the Teflon surface was covered either with sub-microscopic bubbles or air film, what led to facilitation of formation the TPC perimeter long enough for attachment of the colliding bubble. This was confirmed also by the findings that when the time of the plate immersion was prolonged the quicker attachment of the bubble to the Teflon surface was observed. Moreover, it was showed that during the bubble collisions the degree of the kinetic energy transfer into surface energy of the deformed bubble was significantly smaller in the case of Teflon hydrophobic surface than in the case of hydrophilic surfaces (mica and glass). Lower degree of the energy interchange at the hydrophobic solid surface indicates that some kinetic energy was dissipated due to larger shape deformation of the colliding bubble caused by nano- and microbubbles present there. Moreover, as the degree of the energy transfer decreased slightly with increasing surface roughness, so it is a further indication of air entrapment and presence at the Teflon plates immersed in water. Thus, one should be always aware that air can be attached to hydrophobic surfaces immersed into aqueous phase and the air presence can be of great importance in many processes.

Self-Organized Structures and Nanoparticles Monolayers

Thin Polymer Layers with Embedded Nanoparticles

(Professor Piotr Warszyński, Magdalena Elżbieciak, MSc, Jerzy Superata, MSc)

We investigated properties of multilayer polyelectrolyte films containing nanoparticles. Films were constructed by the Layer-by-Layer technique from the polyelectrolyte solutions of various composition. We used Prussian blue, metallic silver and Laponite clay nanoparticles to formed hybrid film films with various functionality. Multilayer containing polycations (PVP, PAH or chitosan) polyanion PSS and Prussian blue exhibited electroactive properties in solutions containing hydrogen peroxide, therefore they can be used as sensoric layers in the miniature H_2O_2 sensors. The PAH/PSS multilayers containing silver nanoparticles can be used as antimicrobial films at various surfaces.

In the reporting period most of work was devoted to determine thickness and barrier properties of films containing clay particles Laponite RD. We constructed multilayer films containing branched cationic polyelectrolyte polyethyleneimine (PEI) and Laponite RD nanoparticles at Si/SiO₂ surface of silicon wafers and compared their thickness with multilayer polyelectrolyte films formed of PEI and strong linear strong polyanion poly-4-styrenesulfonate (PSS). The same films were formed on surface of gold electrodes and cyclic voltamperometry was used as a simple method to determine their permeability for 1,2-naphthoquinone-4-sulfonic acid sodium salt and 9, 10-antraquinone-2, 6-disulfonic acid disodium salt.

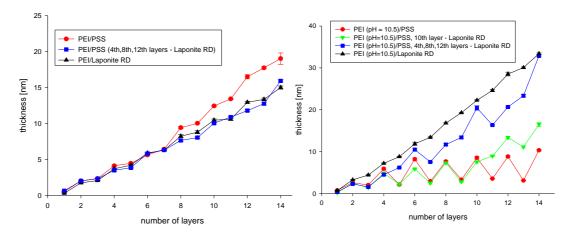


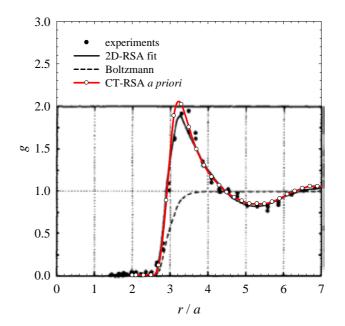
Fig. 1. Dependence of ellipsometric thickness of PEI/PSS films and films containing Loponite RD layers formed at various pH of PEI solution (left pH=6, right pH=10.5), on the number of deposited layers

Films were formed under conditions when PEI was strongly charged i.e. at pH=6 and at pH=10.5 when charge density of PEI was low. The results of ellipsometric measurements are collected in Figure 1. One can see that when films were formed at the conditions of strongly charged PEI, replacement of some or all polyanion layers by Laponite RD led to small decrease of multilayer film thickness. Much more pronounced effect of addition of Laponite can be observed for films formed at the conditions of weakly charged PEI. Here replacement of PSS by Laponite eliminates oscillation of film thickness with the number of adsorption cycles observed for PEI/PSS multilayers. One can see that films in which all PSS layers were replaced by Laponite ones (i.e. PEI/Laponite) formed at the conditions of weakly charged PEI are c.a. two times thicker than ones formed with strongly charged PEI. Replacement of polyanion layer by nanoparticles has also profound effect on films permeability.

Modeling of Thin Multilayers Structures of Controlled Architecture. Theoretical Description of Spherical Particle Systems Including Particle-particle and Particle-Substrate Interactions

(Assoc. Professor Paweł Weroński, Magdalena Nosek, MSc)

The theoretical model of multilayer adsorption of soft (electrostatically interacting) colloid particles CT-RSA, developed in our earlier research, has been generalized for polydisperse particles. The Brownian motion has been considered in the model as well. The new model was employed to simulate monolayer adsorption of spherical particles and the theoretical results were compared with results published in the literature. We have demonstrated a very good agreement of the theoretical and experimental results for spherical particles of the size of the order of micron. In particular, we have demonstrated that the developed theoretical model predicts *a priori* the monolayer structure without any fitting parameters. The results of our research will be published in one of the international colloid journals. In the figure below a comparison of the pair-correlation functions, determined experimentally and theoretically, is presented. In addition to the results obtained using the new CT-RSA model (without fitting parameters), the classical 2D-RSA model predictions are presented as well.



Physical Chemistry in the Protection of Cultural Heritage

Monitoring of Preservation State of Surfaces of Historic Objects Using Optical Methods

(Assoc. Professor Roman Kozłowski, Łukasz Bratas< PhDz, Michał Łukomski, PhD, Łukasz Lasyk, MSc)

The statutory task of 2008 was continued in 2009. It consisted in the development of optical methods for monitoring and tracing damage development on decorated surfaces of historic objects. The development of non-invasive, non-contact techniques is a very important task for conservation science, as works of art are usually both fragile and extremely valuable.

Further works on the development of interferometric methods for analysis of the surface preservation state were carried out in the reporting period. A speckle interferometer built and tested in the institute has proven to be a precise and effective laboratory tool for analyzing the damage development of decorative layers on the wooden supports. The next step in the development of the method was application of the interferometer *in situ*. This practical step is very important since valuable historic objects can rarely be transferred to the laboratory for scientific investigations. The biggest problem during the tests in historic buildings are vibrations of both the interferometer and the tested object. To minimize mechanical instability of the measuring system, the interferometer has been simplified and redesigned. The final system can be easily transported and mechanically connected with the object to minimize mutual vibration between the instrument and tested surface. The measurements performed in a historic church in Hedalen (Norway) and the National Museum in Krakow have confirmed the effectiveness of the applied technical solutions.

In addition, a computer program for the analysis and presentation of the results was expanded. Particular emphasis was laid on the development of a visualization of the spatial distribution of surface vibration. It resulted in a more intuitive presentation of the results which is extremely important in the communication between scientists and conservators.

RESEARCH PROJECTS

of the Ministry of Science and Higher Education

Research of Effectiveness of Hydrogen Activation of the Supported Catalysts Based on the Au Nanoparticles

Research project 1 T09A 094 30 [2006-2009] (project leader: Erwin Lalik, PhDEng)

The project seeks to investigate the influence of small amounts of noble gases on the process of hydrogen activation of Au based catalysts for the low temperature oxidation of CO to CO_2 . In the previous year it was found that an admixture of argon to hydrogen during the activation of Pd catalyst unexpectedly leads to thermokinetic oscillations which turned out to be purely a kinetic phenomenon that does not alter the thermodynamics of process. In 2009 the attempts were made to use the admixtures of other than argon inert gases, including He and N₂, for the hydrogen activation.

The kind of inert gas being used strongly influences the character of the thermokinetic oscillations. Figure 1a shows the oscillations during the reaction of deuterium with palladium respectively in the presence of helium, argon and nitrogen at the same partial pressure (Figure 1b shows the central part enlarged). It can be seen that the admixture of argon in deuterium causes oscillations with the highest amplitude, ca. 30 mW. Smaller oscillations are observed with N_2 , around 25 mW, and the smallest are those in the presence of helium, ca. 15 mW. The same order of variability of amplitude on going from He to Ar, can be observed also for different conditions of the partial pressures and the flow rates during the activation. It should be stressed that in spite of the dramatic differences in the character of oscillations, the total heat of reaction evolved during the process remains invariant. Therefore, the intervention of inert gases must be of kinetic nature, not changing the thermodynamic parameter of the process. Moreover, although the frequencies of oscillations during the same, that is, periodic.

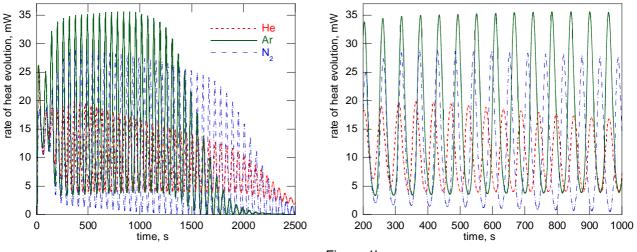


Figure 1a

Figure 1b

Hydrogen is the only component that is able to penetrate the bulk of palladium, and therefore a phenomenon that triggers the thermokinetic oscillations is located on the surface of palladium; hence it can be postulated that the rate limiting step of the reaction is related to the adsorption. In order to influence the kinetic of hydrogen activation as profoundly as it is observed, the coadsorbed gas cannot be only an inactive witness to the process, it can therefore be concluded that an interaction between the adsorbed forms of hydrogen and those of noble gases takes place on the surface. Hydrogen is adsorbed dissociatively on palladium, and so the postulated interactions on the surface are likely to involve the atomic hydrogen. This conclusion should also be valid for the Au supported catalysts for CO oxidation, thus confirming the existence of atomic forms of hydrogen adsorbed on the surface of Au nanoparticles.

The Mechanism of Formation of Polyelectrolyte Multilayer Films with Controlled Microarchitecture on Metallic Surfaces

Research project 1 T09A 123 30 [2006-2009] (project leader: Lilianna Szyk-Warszyńsk, a PhD)

The aim of his project was to perform theoretical and experimental work, which would allow to find the mechanism governing the phenomena occurring during multilayer adsorption of polyelectrolytes (PE), colloidal particles and microcapsules on heterogeneous and conducting surfaces under hydrodynamic and electric forces. As the result of theoretical works, the simulation algorithm of free PE chains in simple electrolyte solution was elaborated. It allowed to characterize the dependencies of effective polymeric chain length and degree of condensation of counter ion of PE used in this project and evaluate their effect of the build-up of PE multilayer films. The modeling of formation of polyanion- polycation complexes and adsorption of single chain on charged surface was also performed. The MC-RSA simulations of deposition of particles on heterogeneous surfaces proved the usefulness of this algorithm for description of experiments, in which model particles were deposited on surfaces with heterogeneity created by adsorption of PEI polycation on homogenous mica surface. These simulation allowed us to estimated the degree of heterogeneity of the metallic surfaces used in this project.

The initial experimental works was concerned the modification of homogeneous surfaces – silicon plates and then metallic surfaces of titanium, surgical stainless steel 316L and gold plates having various degree of heterogeneity, by sequential adsorption of multilayer PE films. We showed that on the homogeneous surfaces (silica plate) the initial deposition rate of capsules formed by encapsulation of latex particles by polyelectrolyte shells, was mainly depended on the electrostatic interaction between adsorbing surfaces and the external layer of the capsules shall. The initial rate did not depend on the thickness of the microcapsule shell or the number of layer covering the adsorbing surfaces and it was in a good agreement with convection-diffusion transport theory.

On heterogeneous metallic surfaces the results for initial deposition rate of latex particles or capsules were very scattered and irreproducible and usually the value of the rate was lower then one for the homogeneous surfaces (according to MC-RSA simulations). Modification of metallic surfaces by the adsorption of multilayer of PE films led to homogenization of surfaces and achieving the value of initial deposition rate in accordance with convective-diffusion mechanism.

Using electric field and electrophoretic mechanism for deposition of microparticles and microcapsules on metallic surfaces largely increased the rate of the deposition process, however, the layer obtained had three-dimensional structure because of the formation of surface aggregates, which were induced by electric field.

The reported period was the final year of this project. Its results were published in two publications having international circulation and were presented on the many international conferences.

The Effect of Cadmium and Selenium onto Surface Properties of Model Lipid Layers Formed on the Basis of Material Obtained from Natural Cell Membranes

Research project 1 T09A 122 30 [2006-2009] (project leader: Maria Zembala, DSc)

The aim of the project was to determine interaction of cadmium and selenium ions with monolayers of lipids of various structures, charge and polarity. The subject is related to a toxic action of cadmium compounds on plant material and to estimation of potential protective role played by small additions of selenates. Studies were performed on model systems selected in such a way to be used as a reference for results obtained for native material extracted from plants exposed to media containing studied ions. The changes of parameters important for plant functioning were determined and correlated with data obtained for model systems. The effect of selected hormones and antioxidants were also detected.

Surface properties (mechanical – surface pressure isotherms and electrical – zeta potentials of liposomes) of commercial lipids of defined structure, representing the basic components of cell membranes were determined. It was found, that surface pressure isotherms of single commercial lipids of various chemical character (DGDG, MGDG and PI) did not change after supplying the subphase with cadmium and selenium salts at the level of 1 mM. The phosphatidic acid (PA) monolayers were the only system reacting on the presence of calcium and cadmium ions which caused the shift of surface pressure value corresponding to the change of monolayer state. Electrokinetic properties of liposomes formed from studied lipids were more sensitive to the lipid/studied ions interactions. Interaction of cadmium ions with model lipids of various surface in pure buffer solution. Selenate ions modified very little a surface charge of lipids having relatively highest charge in pure buffer (PI and PA) causing its increase probably due to adsorption. A contact of DGDG liposomes with buffer solution containing selenate ions caused a small decrease of their effective surface charge.

It was found that hormones` presence, particularly IAA, caused a modification of structure of monolayers of lipids having surface charge of opposite sign.

For native elements of plant cells following properties were determined: fluidity and structural parameters of membranes, their permeability for ions, lipid peroxidation and the effect of cadmium and selenium ions on bio-mass rise and balance of micro- and macro- elements uptaken by plants from growing media of con trolled composition.

It was found that long-lasting action of cadmium and selenium ions during natural plant growth caused Re-building of cell membranes. Layers of lipids extracted from plants grown in Cd presence exhibited smaller packing density and largest effect was observed for MGDG. Selenate ions to much smaller degree modified character and properties of membrane lipids.

Performed experiments showed that addition of Se together with Cd ions partly counterbalanced the changes caused by Cd causing simultaneously a decrease of amount of Cd ions received by plant cells. The effect of Se onto chloroplasts` properties seems especially interesting when taking into account 200 times smaller concentration of selenate (in comparison to cadmium chloride) added to cultivate media,

Cadmium ions influenced accumulation of other compounds important for plant development. It was found that Cd effect was stronger in case of micro-elements (B, Mn, Fe, Zn) than for macro-elements (P, S, K, Ca, Mg).

Structure, Interfacial Properties and Permeability of Multilayer Polyelectrolyte Films for Surface Modification and Encapsulation of Chemicals

Research project N204 166 31/3734 [2006-2009] (project leader: Professor Piotr Warszyński)

The layer by layer (LbL) sequential adsorption of oppositely charged polyelectrolytes multilayers (PEM) is a method for forming ultrathin membrane shells with controlled thickness. Combination of different polyelectrolytes opens a wide range of applications, where PEM's can be used. Multilayers are used to modify surfaces of various materials improving their interfacial properties. They are of special importance in biomaterial area such as contact lens production or modification of surface of titanium implants. Coating of colloidal particles by polyelectrolyte multilayers and formation of hollow shell structures are of importance in case of encapsulation applications, e.g. in drug carrier systems or microreactors.

The aim of the project was to evaluate the influence of deposition conditions on permeability of the multilayer film. In particular, the effect of ionic strength of polyelectrolyte solution during film formation and treatment after deposition with solution of different pH were determined. Multilayers films were deposited at gold electrode surface. The cyclic voltamperograms for various numbers of deposited layers and size of electroactive compounds were obtained. As a electroactive compounds with different molecular mass: p-Benzoquinone, 1,2-Naphthoquinone-4-sulfonic acid sodium salt, and Manganese(III) 5,10,15,20-tetra(4-pyridyl)-21*H*,23*H*-porphine chloride tetrakis(methochloride), were chosen. For permeability measurements the model PAH/PSS polyelectrolytes were used.

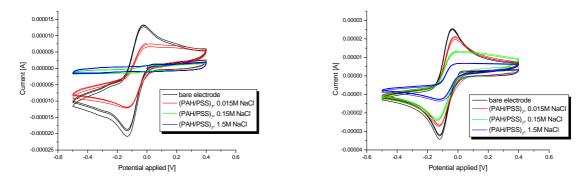


Fig.1. Comparison of voltamperometric curves for bare electrode and PAH/PSS multilayers deposited at different ionic strength obtained for 1,2-Naphthoquinone-4-sulfonic acid sodium salt (left) and *p*-Benzoquinone (right).

We demonstrated that CV current decreased with the number of deposited polyelectrolyte layers. That means that PAH/PSS films presented a barrier for transport of electroactive compounds to the electrode surface. In case of multilayer films deposited from solutions of different ionic strength, their permeability decreased when the ionic strength grew. The selection electroactive compounds of different molecular size permitted to identify differences in PAH/PSS films permeability and to estimate pores size in the films. There was no effect of multilayer films treatment with solution of three different pH: 3, 7, 11. The permeability of those films was the same as of untreated PAH/PSS films.

Heteropolyacids (HPA) as he Catalysts in Gas Phase Ethyl-*tert*-butyl Ether (ETBE) Synthesis

Research project N204 64 31/3689 [2006-2009] (project leader: Anna Micek-Ilnicka, PhDEng)

In this project our attention was focused on the determination of the mechanism and kinetics of ethyl-tert-butyl ether (ETBE) synthesis. Despite the broad literature on the heteropolyacids, the studies on the kinetics of catalytic reactions on heteropolyacids are scarce. Especially determination of reaction orders with respect to ethanol and isobutene are absent. On the basis of the kinetic experiments and physicochemical investigations, such as sorption of reagents investigated by spring-balance and IR spectroscopy we formulated detailed mechanism of ETBE synthesis. The main determinant of the Wells-Dawson $H_6P_2W_{18}O_{62}$ catalyst activity was the concentration of "free" protons, located between heteropolyacid anions and loosely bonded via hydrogen bonds. During ethanol sorption, a part of such protons form non-active protonated ethanol clusters which are absorbed into heteropolyacid crystallites.

Investigations of the influence of water on the rate of the ETBE synthesis were the main topics of this project. The explanation of this effect was crucial form the point of view of the industrial applications of heteropolyacids in the ETBE synthesis.

Experimental results pointed clearly that the presence of water vapour in the gas phase inhibited the ETBE formation. Water molecules similarly to ethanol, formed protonated clusters effectively reducing concentration of the "active" protons. Calculated reaction orders with respect to ethanol and isobutene were -2.0 and 1.6, respectively. Moreover, they were independent of the presence of water vapour in this system.

On the other hand, when water was present in the form of crystallization water in $H_6P_2W_{18}O_{62}$, the optimal amount of the crystalline water was determined. Maximum rate of etherification was obtained for the hydrate $H_6P_2W_{18}O_{62} \cdot 8H_2O$ (1.3 H_2O/H^+). For the same catalyst composition a maximum of ethanol sorption capacity has been has been found.

The sorption experiments on $H_6P_2W_{18}O_{62}$ allowed proposing the following mechanism of ETBE synthesis:

- 1. ethanol absorbs rapidly and in the large amounts into the bulk of heteropolyacid crystallites. In the IR spectrum, registered during ethanol sorption, the maximum at 1516 cm⁻¹ appeared, confirming the ethoxonium ions $C_2H_5OH_2^+$ formation,
- 2. isobutene adsorbs on the surface of heteropolyacid,
- 3. isobutene forms $C_4H_9^+$ carbocation,
- 4. sorption of ethanol prior to isobutene, does not change the amount of sorbed isobutene,
- 5. desorption of isobutene from the catalyst is possible after heating the heteropolyacid, indicating the formation of $(C_4H_8)_mH^+$ protonated oligomers,
- 6. reaction of the ETBE synthesis on heteropolyacid catalyst occurs between isobutene adsorbed at the surface and ethanol supplied from the bulk of heteropolyacid crystallites.

New Method of Producing Nano- and Microcapsular Systems Characterized by Targeted Functionality for a Selective Drug Delivery

Research project N205 022 31/1112 [2006-2009] (project leader: Professor Zbigniew Adamczyk)

The main goal of this studies was a quantitative determination of the deposition kinetics of model nano- and microcapsules on various interfaces. As reference systems in these investigations we have used monodisperse polystyrene latex particle suspensions having a positive surface charge and nominal fraction diameters of 100, 200, 500 and 800 nm. Particle size distribution in suspensions was determined using the dynamic light scattering and laser diffraction techniques. On the other hand, particle electrophoretic mobility (electrokinetic charge) was determined as a function of pH and the ionic strength by the microelectrophoretic method. It was shown that these latexes exhibited high, positive zeta potential for the high range of pH between 3 and 10. This enabled their efficient deposition on negatively charged substrates. The kinetics of particle deposition was quantitatively studied using the diffusion and convection transport conditions and the direct optical microscope particle counting under the in situ conditions. Independently, the coverage of particles was determined by the atomic force microscopy (AFM) both under the wet and dry conditions. In this way, best conditions for attaining uniform, high density layers of microcapsules have been determined. A typical micrograph showing the distribution of particles over the interfaces and the corresponding pair correlation function are shown in Fig.1. kinetic run obtained for the A800 latex (particle diameter of 800 nm) is shown in Fig.1. As can be seen, using the diffusion-controlled transport condition, a uniform monolayer of particles of a high coverage can be produced in a reliable way. It is also interesting to observe that the experimental data are in a quantitative agreement with theoretical predictions (depicted by the solid line in Fig.1) derived from the random sequential adsorption (RSA) model. It can be, therefore, concluded that the results obtained have a basic significance for calibration of microcapsule deposition kinetic runs performed under various transport conditions.

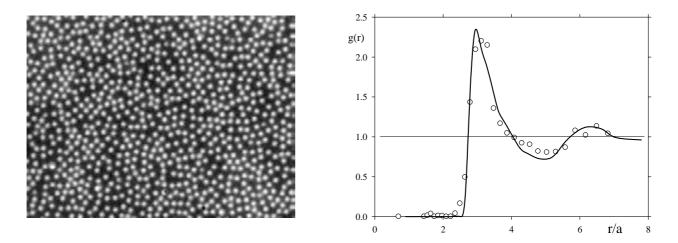


Fig.1. Part "a", the micrograph showing positive latex particles (averaged diameter 800 nm) deposited on bare mica, ionic strength $2x10^{-5}$ M, pH = 5.5, T = 298 K, $\Theta = 0.27$, part "b" the pair correlation function g(r/a) for the same parameters. The points denote experimental results derived from optical microscopy and the solid line denotes the theoretical results derived from the Monte-Carlo RSA simulations.

Theoretical Studies on Surface Heterogeneity of Catalysts Based upon Vanadium Pentoxide

Research project N204 024 31/0475 [2006-2009] (project leader: Professor Małgorzata Witko)

Clusters and periodic calculations performed by means of DFT approach have allowed obtaining structural characteristics of three low-index (010), (001) and (100) V₂O₅surfaces. Mixed ionic-covalent types of bonds characterize electronic structure of these surfaces. Vanadium centers at unsaturated (100) and (001) surfaces are less positive charged (less acidic) than vanadium centers that exist at saturated (010) surface. Nucleophilicity (negative charge) of surface oxygen centers scale with their coordination number. Total and atomic densities of states, for all studied surfaces, indicate O 2sp and V 3d hydride orbitals as responsible for conductive region whereas V 3d states as dominating a valence region. Electrostatic potentials above coordinatively saturated (010) surface is negative. In contrary for both unsaturated (100) and (001) surface it has both negative and positive values suggesting that both electrophilic and nucleophilic molecules could approach these surfaces. Hydrogen undergoes stabilization at all surface oxygen sites forming stable surface hydroxyl OH groups. Formation of surface oxygen vacancies is endo-energetic process that is facilitated by a presence of hydrogen i.e. on reduced surfaces. Formation of surface water type species (after adsorbing of two hydrogen atoms at one oxygen center) in some cases results in spontaneous vacancy creation. Reduced surfaces undergo easily re-oxidation. They accommodate more oxygen (two per one vacancy) than were at particular center before reduction. Adsorption of molecular oxygen from gaseous leads to its activation (elongation and weakening of O-O bond).

Analysis of energy stabilization for different cutting of Ti_2O_3 and V_2O_3 indicates M'OM, cutting as the most stable. For Cr_2O_3 the M'OM i OMM ' cuttings are energetically equivalent. Mixed ionic and covalent types of bonds characterize all X_2O_3 oxides. The nucleophilic character of oxygen centers increases with the increase of their coordination number. Surfaces of these oxides undergo modifications by adsorbing hydrogen, hydroxyls or by formation of surface vacancies.

The V-O-X systems (where X = V, P, Mg) are characterized by a wide spectrum of structures that are consequences of crystallographic differences, various building units, different ways of connecting building unit, changes of vanadium oxidation states as well as changes of V to third element ratio. As the result a large number of different surface-active centers are possible: singly coordinated P=O. Mg=O), doubly coordinated (V-O-V, (V=0, V-O-M, M-O-M; M=P, Mg), bridging, triply coordinated (2V-O-M; M=Mg, P) and also coordinated to four vanadium atoms (4V-O). All electronic and geometric parameters that have been collected for a family of compounds that are important in catalysis have been analyzed in order to find that responsible for catalytic activity.

Neuron networks have been applied as one of very first in heterogeneous catalysis to predict properties of surface-active center based upon its local environment. This in turn should allow preparing catalysts of defined selectivity for a particular process.

Mechanism of Catalytic Activity of "Nano-Au/Oxide" in Oxidation of CO

Research Project N204 089 32/2633 [2007-2010] (project leader: Professor Józef Korecki)

The studies of the defected substrate surfaces were performed for powder Au/MgO catalysts. The defect concentration at the substrate surface was changed by applying different annealing temperatures or by Li intercalation in both the substrate alone and the Au/MgO catalyst as well. The catalytic tests of activated (reduction in H₂) and inactivated powder catalysts were performed for CO oxidation reaction. The catalysts prepared at higher temperatures showed higher activity in CO oxidation. The reduction of the Au/MgO catalysts in hydrogen increases their activity at the temperatures up to 250°C. Lithium intercalation at the catalysts surface results in higher reaction rate constant for CO oxidation. This leads to higher activity of the Li intercalated systems at temperatures above 250°C. The highest activity per unit area was revealed for the catalyst annealed at 700°C. However, the catalyst prepared on the Li intercalated substrate showed the highest activity at 300°C.

In case of model catalysts, the XPS studies were performed on the catalysts obtained on MgO(001) monocrystalline substrates prepared by either cleaving the crystal in air and then Ar sputtering or by cleaving the crystal in water. Then Au was consecutively deposited in ultrahigh vacuum on the modified substrate surface. The electronic states of the substrate and Au deposit were studied after each step of Au deposition and after annealing at 150, 300 and 500°C. For both MgO substrate systems i.e. cleaved and then sputtered or cleaved in water, the electronic state of the deposited Au clusters was strongly dependent on substrate preparation. This effect was not observed after annealing at 500 °C. HR SEM technique was applied to study morphology of the annealed Au/MgO systems.

Synthesis of the Mesoporous Structures of the SBA-15 Type with Hierarchical Pore Distribution with the Use of Carbon or Latex Nanoparticles as the Hard Templates

Research project N204 166 32/4313 [2007-2009] (project leader: Malgorzata Zimowska, PhD)

In this project we concentrate on developing of the hierarchically, structured ordered SBA-15 porous materials containing two types of mesopores with the modified morphology controlled by addition of carbon black Pearl 2000 nano-particles or colloidal microspheres of ionic latex to the synthesis mixture. In this approach carbon black and latexes serves as a hard templates, which, when removed after synthesis by means of calcination, leaves empty voids in the material, in which it was embedded.

Addition of templates modifies the morphology of the silica materials and results in a decrease of the individual silica grains size. SEM analysis show presence of rod-like grains of standard SBA-15 material while modification of SBA-15 material by carbon causes formation of two coexisting phases with different grain size distribution: SBA-15 type phase and fine-grained, silica amorphous phase not visible in XRD.

Admixture of carbon black nanoparticles to the triblockcopolymer results in regular and elongated grains of SBA-15 type material similar to hexagonal prism, resembling hexagonal packed structure. Templating of parent SBA-15 material by moderate quantity of carbon black added to TEOS leads to the solid containing SBA-15 domains intergrown with foam-like amorphous silica generated by the hard template. In the presence of large quantity of carbon black the nucleation of SBA-15 component is hindered in favour of the formation of foamy amorphous silica. When the anionic latex microspheres (SBA-15/s-latex) are admixtured with the synthesis gel the more curved discoidlike silica grains are formed with the u-shaped parallel channels.

Modification of synthesis condition causes also small increase of the pores in SBA-15 type material. Addition of carbon black causes enlarging of the pores from 43,6 Å up to 53-56 Å, moreover exhibits a new broad maximum at 120Å, derived from amorphous silica. Addition of latex microspheres exhibits maximum around 63 Å.

Post-synthesis aluminated SBA-15 solids synthesized by standard and modified procedure were used as supports for cationic MnTMPyP manganese porphyrine and tested in the reaction of cyclohexene oxidation.

Oxidation of cyclohexene with hydrogen peroxide over cationic metalloporphyrin supported on mesoporous aluminosilicates proceeds in all cases along allylic oxidation pathways, by homolytic decomposition of hydrogen peroxide. The main reaction products were cyclohexanol and cyclohexanone.

In case of unsupported porphiryn the major reaction product (conversion of cyclohexane = 24%) was epoxide (89% yield), as a result of heterolytic decomposition of hydrogen peroxide. All modified by hard template SBA-15 type materials present higher conversion of cyclohexane in comparison with standard SBA-15 (with conversion of cyclohexane = 8%). The highest (25%) conversion of cyclohexane was observed for the sample with admixtured anionic latex microspheres (SBA-15/s-latex). It points to easier diffusion of substrates and products into u-shaped channels of mesoporous aluminosilicates.

Employment of Macrocyclic Metallocomplexes of Transition Metal Ions in Self-Assembling Systems for Catalytic Oxidation of Hydrocarbons

Research project N204 122 32/3142 [2007-2010] (project leader: Assoc. Professor Ryszard Grabowski)

Metallocomplexes are known as active and selective catalysts of organic compounds oxidation processes under mild conditions. Epoxidation of olefins in the presence of macrocyclic metallocomplexes like metalloporphyrins, metallophthalocyanines and metallosalens and oxygen donors is an important method of producing organic oxides.

In homogeneous oxidation reactions a macrocyclic catalyst can be the subject of dimerization which results in the decrease of its catalytic activity. The use of supports is the way to limit unfavourable effects. Recently, liposomes attract interest as supports of different compounds. Water solutions of liposoms with dissolved homogeneous catalysts constitute a kind of nanoreactors enabling the conversion of nonpolar reagents to polar reaction products. The products are withdrawn from the reaction place towards the surrounding water phase and the further conversion of desired products is limited.

In recent time a lot of articles concerning physico-chemical properties of liposomemetallocomplex systems have been published, but their practical use in chemical reactions refers only to the decomposition of hydrogen peroxide. However, in our laboratory for the first time in the world these catalysts have been applied in the oxidation of hydrocarbons. The aim of our work is the immobilization of anionic metallophthalocyanines and metalloporphyrins on liposomal supports and the determination of their catalytic activity in the oxidation of cyclohexene by an oxygen donor – sodium perchloride.

The catalytic oxidation of cyclohexene was carried out in 10 cm³ glass reactor at room temperature and under atmospheric pressure for two hours. The following series of anionic metallophthalocyanines: MnPcS, FePcS, CoPcS, CuPcS, NiPcS and anionic metalloporphyrins: MnTPPS, FeTPPS or CoTPPS as well as the same compounds immobilized on liposomes were applied as catalysts. The synthesized catalysts were characterized by means of UV-Vis spectroscopy, DLS analysis, zeta potential measurement and optical and fluorescent microscopy.

Characteristic Soret and Q bands are visible in UV-Vis spectra of metallophthalocyanines and metalloporphyrins confirming the synthesis of the catalysts. The products of cyclohexene epoxidation are: cyclohexene epoxide, cyclohexen-1-ol i 2-cyclohexen-1-one. All the catalysts are active in the studied reaction. Metallophthalocyanines demonstrate higher catalytic activity than the complexes of metalloporphyrins. The immobilization of complexes on liposomal support results in a substantial increase of their catalytic activity. The most active catalysts are manganese and copper complexes of phthalocyanine both before and after immobilization on liposomes.

The Influence of Kind of Central (P, Si) and Addenda Atoms (W, Mo) in the Silver Salts of Heteropolyacid with Keggin Structure on Direct Decomposition of Nitrogen Oxide NO_x

Research project N204 149433 [2007-2010] (projects leader: Tadeusz Machej, PhD)

In 2009 year the investigations of NO or NO₂ sorption on silver salts of heteropolyacid $Ag_3PW_{12}O_{40}$, $Ag_3PM_{012}O_{40}$, $Ag_4SiW_{12}O_{40}$ and $Ag_4SiM_{012}O_{40}$ were done by means of DSC/TG, high temperature XRD methods and FT-IR spectroscopy. The reaserch was performed in pure helium, helium with addition of 1% NO and helium with addition of 1%NO₂. All samples were heated from 293 to 573K, then cooled to 323K and again heated to 573K. The salts with tungsten as addenda atoms did not exhibit any endo or egzothermic effects. Contrarily, the salts with molibdenum as addenda atoms showed endothermic effects, the $Ag_3PM_{012}O_{40}$ salt about 475K, while the $Ag_4SiM_{012}O_{40}$ salt about 500K. In turn, the thermogravimetric analysis revealed the influence of gas composition on changes of samples mass (Table 1). Higher mass loss in atmosphere with NO or NO₂ addition can be explain by sorption of nitrogen oxides in room temperature. Precise calculations concerning amount of water in silver salts confirmed earlier observations that silver salts of $H_4SiW_{12}O_{40}$ and $H_4SiM_{012}O_{40}$ acids can contain only three hydrated silver ions.

	Ag ₃ PW ₁₂ O ₄₀			Ag ₃ PMo ₁₂ O ₄₀			Ag ₄ SiW ₁₂ O ₄₀			Ag ₄ SiMo ₁₂ O ₄₀		
Temperature range	He	1%NO	1%NO ₂	He	1%NO	1%NO ₂	He	1%NO	1%NO ₂	Не	1%NO	1%NO ₂
293-573K	-4.0	-1.6	-1.6	-3.0	-3.2	-4.1	-1.9	-2.3	-4.7	-1.9	-2.3	-5.6
573-323K	0	+1.0	-1.6	+2.5	+1.6	-4.7	+2.3	+1.2	-0.9	+2.2	+1.6	-4.3
323-573K	-0.7	-0.6	-1.2	-1.6	-0.4	-1.3	-1.0	-0.9	-1.5	-0.2	-0.1	-0.4
Total	-4.7	-1.2	-4.4	-2.1	-2.0	-10.1	-0.6	-2.0	-7.1	+0.1	-0.8	-10.3

Table 1. The changes of silver salts mass (%) in different atmospheres and temperature ranges observed by means of thermogravimetric analysis (TG).

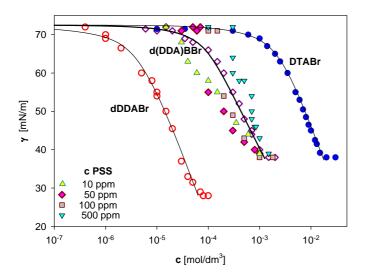
High temperature XRD investigations were performed in the same different atmospheres indicating how nitrogen oxides strongly influences the change of lattice parameter $\underline{\mathbf{a}}$ of some silver salts. For instance, in the case of Ag₃PW₁₂O₄₀ salt the heating performed in helium atmosphere resulted in a change of lattice parameter $\underline{\mathbf{a}}$ from 11.90Å at room temperature to 11.69Å at 573K. Addition of 1%NO or 1%NO₂ caused the same shortening of lattice parameter $\underline{\mathbf{a}}$ occuring already at 373K. Nature of this phenomena will be examine in the next investigations. The sorption of nitrogen oxides was also studied with FT-IR spectroscopy. At room temperature extra bands originating from NO or NO₂ sorption at 1358, 1384 and 2264 cm⁻¹ were observed. However, after the preheating of the samples at 423K and sorption of nitrogen oxides the band at 1358 cm⁻¹ disappeared.

The presented results of nitrogen oxides sorption clearly show that individual approach to their influence on the structure of investigated silver salts is necessary.

Multiple Cationic Surfactant Complexes with Polyelectrolytes as Elements of Nanostructures

Research project N N204 248634 [2008-2011] (project leader: Grażyna Para, PhD)

Understanding the mechanism of surfactant/polyion complex formation and the role of various parameters determining their structure and interfacial properties is fundamental for a proper design of the systems for their future applications. We investigated the interfacial behavior of aqueous solutions of dodecyltrialkylammonium bromides (DTX) with alkyl chain (X) having from 1-4 CH₃ groups, didodecyldimethyl-ammonium bromide (dDDABr) and gemini-type cationic surfactants d(DDA)XBr: having two dodecyldimethyl-amonium groups connected at the level of ammonium-head groups by a (-CH₂-)_n spacer and their interactions with polyanions: sodium poly(styrene sulfonate) (PSS) and poliacrylic acid (PAA), both of molecular weight about 70 kDa. For the interpretation of surface tension isotherms, we used the surface quasi-two dimensional electrolyte (STDE) model of ionic surfactant adsorption, modified to describe the adsorption isotherms of aqueous solutions of multivalent cationic surfactants and their interaction with polyanions. We demonstrated that the STDE model of adsorption of ionic surfactant with multiple valency can be successfully applied for the description of surface tension isotherms. Figure 1 shows the dependence of the surface tension on the surfactant concentration for d(DDA)BBr - in the presence of different polyelectrolyte concentrations. Influence of anionic polyelectrolytes on the surface tension isotherms was also investigated. It was found, according to the expectations, that the presence of polyelectrolyte decrease the surface tension of all investigated cationic surfactants due to formation of surfactant-polyelectrolyte complexes at the surface. This decrease is lower for high



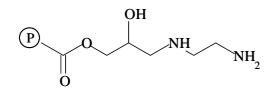
concentration of polyelectrolyte, which indicates that there is a competition between adsorption and formation of the surfactant – polyelectrolyte complex in the bulk.

Fig. 1. Dependence of the surface tension on the surfactant concentration for: d(DDA)BBr - in the presence of different PSS concentrations. Surface tension isotherms of DTABr and dDDABr are shown for comparison.

The Study on the Application of heterogenized chiral ruthenium complexes in enantioselective hydrogenation of ketones

Research project N N204 249034 [2008-2011] (project leader: Dorota Duraczyńska, PhD)

In this study a series of ruthenium catalysts supported on gel-type polymer FCN functionalized by C=O, NH₂ (Figure 1) groups were prepared. These catalysts contained different amounts of active phase e.g. 1%Ru/FCN, 2%Ru/FCN and 4%Ru/FCN. New materials were



characterized by the following techniques: FT-IR, XRD, SEM, EDS, TEM, XPS, and DSC. The results suggest the formation of ruthenium complex coordinated to NH₂ and NH polymer functional groups. SEM analysis revealed dramatic changes in morphology of parent carrier Fig. 1. The structure of FCN resin

FCN due to the entrapment of RuCl₃ and its subsequent reduction with NaBH₄. The catalytic properties of new materials were tested in hydrogenation of acetophenone (Figure 2). Secondary chiral alcohols formed as a result of ketones reduction are technologically important reagents and precursors, used mainly in the pharmaceutical industry in drugs or biologically active compounds manufacture. All tested catalysts were found to be active under very mild conditions of reactions: $T=40^{\circ}C$ and atmospheric pressure of hydrogen; isooctane/water (1:1 V/V) mixture was used as a solvent. These catalysts feature high selectivity towards PE (1-phenylethanol) as compared to 2%Ru/Al₂O₃.

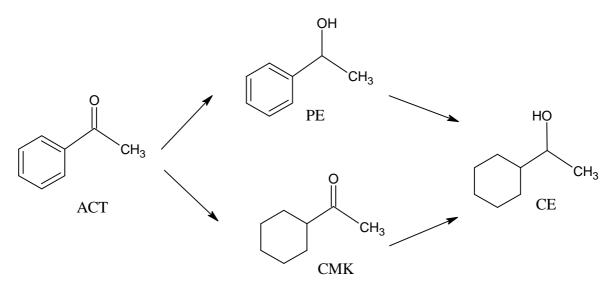


Fig. 2. Hydrogenation of acetophenone

Photocatalytic Water Dissociation outside Earth Atmosphere. Example of Catalysis Usage in Missions of Solar System Exploration

Research project N N204 217734 [2008-2009] (project leader: Robert. Gryboś, PhD)

Water dissociation is considered one of the most important processes for the future Moon base, which according to NASA plans should be ready by year 2020. The dissociation reaction can provide oxygen for life support, hydrogen for metal extraction from moon soil and oxygenhydrogen mixture for rocket engines and fuel cells. Recent NASA moon mission LCROSS (October 2009) confirmed existence of substantial, sub-surface water deposits. Additionally, transport and storage of water is much safer and easier than oxygen and hydrogen. Current method of choice for water dissociation is electrolysis powered by solar cells. Photocatalysis is a very promising alternative, as Moon soil contains large amounts of titanium dioxide, which is very efficient in water dissociation. Using Moon soil as a catalyst would be an ideal application of ISRU (In Situ Resource Utilization) principles. As the Moon surface receives full sunlight spectrum, there is no need to tune the photocatalyst to a particular wavelength window. On the other hand, extreme conditions on the Moon surface (low temperatures and pressures) present different challenges to the catalyst.

Two systems were chosen for catalytic tests:

(i) anatase - very efficient Earth catalyst, unfortunately not found in Moon soil samples

(ii) rutile - significantly less active than anatase, but present on the Moon

catalyst	H ₂ obtained after 45 minutes [mmol]
TiO ₂ 400/Ni	0,15
TiO ₂ 500/Ni	0,27
TiO ₂ 550/Ni	0,22
P 25/Ni	0,19
RD-5/Ni	_

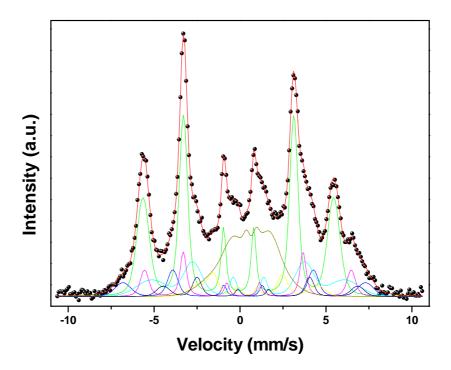
Results show, that catalytic activity is significantly increased when the catalyst contains nickel. It is a very interesting result, because Moon soil contains a variety of nickel compounds (mainly mixed oxides). This opens a possibility to produce locally (on the Moon surface) very efficient water dissociation photocatalysts.

Exchange-bias Effect in the Epitaxial Systems Obtained by Deposition of the 3d and 4f Metals on Polar and Non-polar Transition Metal oxide Substrates

Research project N N202 248535 [2008-2011] (project leader: Jacek Gurgul, PhD)

According to the project schedule several syntheses of epitaxial bilayers CoO/Fe/MgO(100) and Fe/CoO/MgO(100) were made. During the deposition process oxygen partial pressure, substrate temperature and the layer's deposition rate were optimized. The quality verification was performed by using LEED and AES. The best samples were chosen for magnetic characterization by VSM magnetometry, MOKE studies and Mössbauer effect measurements (⁵⁷Fe-CEMS).

The CEMS studies have shown (representative RT resonance spectrum for CoO/Fe/MgO(100) is presented below) that both measured systems have got interfaces with different chemical structures. The Fe layer is much more oxidized in CoO/Fe interface related to the Fe/CoO interface. This effect is connected to the procedure of cobalt reactive deposition under molecular oxygen atmosphere. The different chemical structures of interfaces imply completely different magnetic properties of both systems in the low temperatures. The Fe/CoO bilayer is characterized by large coercive fields, small exchange bias fields and relatively high blocking temperature $T_B = 290$ K, whereas in the CoO/Fe bilayer one can see small coercive fields, high exchange bias fields and lower blocking temperature $T_B = 257$ K. Moreover, in the case of CoO/Fe/MgO(100), enhancement of coercive field near T_N of CoO was observed what is in line with Stiles and McMichael theory.



Molecular Mechanisms Determining Product Specificity of 2-Oxoglutarate Dependent Oxygenases

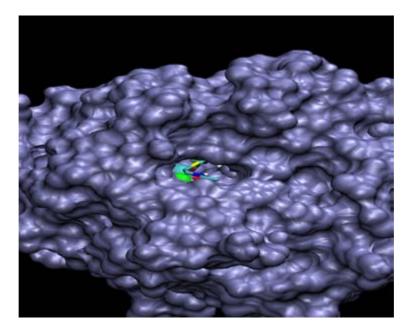
Research project N N301 093036 [2009-2011] (project leader: Professor Ewa Brocławik)

Within this project, which commenced in April 2009, it is planned that mechanisms of action of two groups of 2-oxoglutarate (2OG) dependent oxygenases will be investigated with theoretical methods. The first group, i.e. halogenases is represented by SyrB2, whereas the second comprises histone demethylases from the JMJD2 family.

Both groups of enzymes bind a single ferrous ion in their active sites and utilize a large difference of red-ox potential for 2OG and O_2 to carry out oxidative transformations of their primary organic substrates. These reactions involve: demethylation of lysine groups in the N-terminal fragment of histone protein H3 (JMJD2), and chlorination of threonine side chain (SyrB2). Biological significance of these chemical transformations stems from the fact that they are part of a machinery controlling gene expression (demethylases) or antibiotic synthesis (halogenases). It is planned that in this project we will conduct a series of molecular simulations with the purpose to shed new light on molecular mechanisms of the studied enzymes, especially on factors controlling the substrate and product specificity.

Since the structure of the SyrB2-substrate complex has not yet been determined experimentally, investigations on this system started with simulations of molecular docking. To this purpose models of both protein and organic substrate have been carefully prepared. A loop fragment which is missing in the crystal structure of SyrB2 has been modeled with simulated annealing procedures, whereas atomic charges of the ligand have been determined with the MK procedure. Molecular docking simulations have been performed with AutoDock program, and their results allow for identification of a probable structure for SyrB2-substrate complex (see Figure). This structure serves as a starting point for construction of smaller models of active site region used in currently being performed quantum-chemical studies on the reaction mechanism.

The necessary computer equipment has been purchased.



Overview of the SyrB2-subtrate complex. SyrB2 depicted as a solvent accessible surface, substrate in sticks representation.

Developing of the Methods of Formation Biologically Active Multilayers Contained Polyelectrolytes - Proteins - Dendrimers

Research Project N N204 028536 [2009-20012] (project leader: Barbara Jachimska, PhD)

The study of the adsorption process of colloidal particles, especially proteins at the solid/liquid interface plays a crucial role in many processes such as control of the separation of proteins and cells, immobilization of proteins and enzymes. The aim of the project is understand of the adsorption mechanisms of polyelectrolytes, proteins, and dendrimers for develop formation of multilayer structures with appropriate functionality in a controlled manner, which can be used as material for immunological tests, drug carriers and materials to accelerate the integration of implants. Despite the importance of cognitive and many promising practical applications of nano-layered material containing proteins, development of their formation is mainly limited by knowledge of the physical and chemical bases of these processes.

In particular, there are no systematic studies on the kinetic aspects of the multilayer with proteins formation processes and their stability under dynamic conditions. The first stage of the project was concerned on determined physicochemical characteristics of model polymers and biopolymers, and a fluorescent latex particle. The latex particles have defined surface properties what allows the control structure of nanoparticles layers. The next step is relating to the description of the kinetics of the monolayer and multilayer formation (proteins, dendrimers, polyelectrolytes) on the model surface and to determine their structure.

Investigation of the nanoparticles structure density (dendrimers, proteins), which are invisible under an optical microscope, will be possible by the adsorption of latex molecules which have the sizes allowing their observation under optical or fluorescent microscope. This series of the tests will lead to procedure of no-marker method of the replication the structure of the nanoparticles layers.

New Nanostructured Carbon Materials, Oxide Materials and their Composites as Components of Capacitors

Research project N N507 269936 [2009-2012] (project leader: Aleksandra Pacula, PhDEng)

- 1. Ni-Co-Al layered double hydroxides (LDHs) with molar ratio Ni : Co : Al = 1 : 1 : 1 was synthesized. Ni-Co-Al LDHs was prepared by co-precipitation method by addition of aqueous solution of sodium hydroxide to aqueous solution of nickel, cobalt and aluminum nitrates at 60° C with constant stirring. Elemental composition of Ni-Co-Al sample was confirmed by chemical analysis using ICP OES.
- 2. A series of composites were synthesized via chemical vapour deposition (CVD) method. CVD experiments were carried out in horizontal tube furnace under a flow of inert gas. Ni-Co-Al LDHs was heated for 3 hours at various temperatures of 600, 700, 800, 900°C, under a flow of argon saturated with acetonitrile (CH₃CN) vapour.
- 3. A series of carbon materials was obtained from composites via treatment with acid. Carbon materials were extracted from composites with the use of dilute hydrochloric acid during 2-day process.
- 4. Several techniques including X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS), scanning electron microscopy, nitrogen sorption, thermogravimetric analysis were employed to characterize physicochemical properties of newly prepared materials.
- 5. Composites have similar composition. They are composed of multiwall carbon nanotubes, metallic particles (Co, Ni) and metal oxides (CoO, Co₃O₄, CoAl₂O₄, NiO, NiAl₂O₄, Al₂O₃). Depending on synthesis temperature components of composites indicate different degree of crystallinity. Composites prepared at higher temperatures contain bigger crystallites therefore they have lower specific surface area. Specific surface area of composites prepared at 600, 700, 800, 900°C is 132, 100, 90, 44 m²/g, respectively.
- 6. Carbon materials have similar composition. They are mainly composed of multiwall carbon nanotubes but also contain small amount of metallic particles (Co, Ni) and metal oxides (CoO, Co₃O₄, CoAl₂O₄, NiO, NiAl₂O₄, Al₂O₃). Carbon nanotubes are defective and have a spaghetti-like appearance. Part of carbon nanotubes indicates bamboo-like shape. The diameter of carbon nanotubes prepared at lower temperatures is distributed in the range of 15-50 nm, whereas the diameter of carbon nanotubes prepared at higher temperatures is distributed in the range of 20-120 nm. The average diameter of carbon nanotubes increases with increasing CVD temperature. The use of acetonitrile as a carbon precursor results in incorporation of nitrogen atoms in the carbon framework. Nitrogen concentration in carbon nanotubes synthesized at various temperatures is ca. 3 wt %. Specific surface area of carbon materials prepared at temperatures of 600, 700, 800, 900°C is 158, 165, 112, 64 m²/g, respectively.

Acoustic Emission for Monitoring Museum Objects as a Universal Method of the Preventive Conservation

Research project N N105 278536 [2009-2012] (project leader: Łukasz Bratasz, PhD)

During the first period of the project, which started on May 5, 2009, scientific equipment for the measurement of acoustic emission generated during crack propagation in cultural heritage objects was developed. It was achieved by an acquisition of a high quality system for acoustic emission recording, a low-noise amplifier and a set of sensors sensitive to a frequency range from 10 to 1000 kHz. These pieces of equipment were integrated into one set up and programmed to effectively analyse the recorded signals. Development of an automatic module for the analysis, classification and recording of the acoustic events on site i.e. in museum display conditions, has been started and will be continued in the coming months. The entire system was tested in the laboratory by using wooden specimens and by the standard Nielson test. Currently, the developed equipment is in the process of optimization to achieve a proper amplification of the signals and to filter out undesired frequencies.

It is planned that all work necessary to finalize the development of the equipment and to carry out all necessary tests will be finished in March 2010.

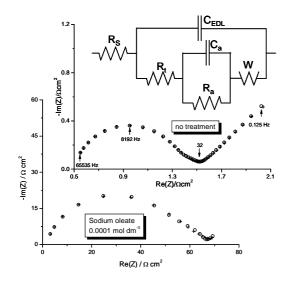
Within the second work package, the preparation of wooden specimens cut in the radial and tangential directions, imitating cultural heritage objects, has been started. Mechanical characterization, encompassing important, selected parameters, was accomplished for several types of wood.

Controlled Adsorption as a Method of Protection Against the Environment Contamination Caused by the Oxidation of Metal Sulfides

Research project N N523 451336 [2009-2011] (Assoc. Professor Paweł Nowak)

Mineral and metal industry produces large quantities of wastes. For example Polish copper industry during fifty years of its activity left on the depository places approximately 600 millions tons of flotation wastes, occupying for that purpose 2300 hectares of ground. Wastes from the mineral and non-ferrous metal industry contain frequently metals in the form of metal sulfides. The solubility of metal sulfides is very low, however when left on the depository places they oxidize to better soluble compounds, liberating metal ions. Weathering of metal sulfides may be considered as a corrosion process and, like in the case of metals it may be prevented by the application of inhibitors. Contrary to most world's copper ore deposits, where the main copper mineral is chalcopyrite, in Polish copper ores the main copper-bearing constituents are copper sulfides. So, the adsorption of several prospectus inhibitors on the surface of copper sulfides was studied in the aim to find an effective copper sulfide oxidation depressant.

Copper sulfide of the composition $Cu_{1.87}S$, synthesized from high purity copper and sulfur, similar in the composition to many non-stoichiometric copper (I) sulfides occurring in Polish copper ores as well as several pieces of natural copper sulfides isolated from the ores were used in the experiments. Copper sulfides are good electrical conductors, so electrochemical methods (mainly electrochemical impedance spectroscopy) were used in the investigations. Two types of measurements were performed: 1 - the capacitance of the electrical double layer in the solution of the potential inhibitor was measured and compared to the capacitance measured in pure base electrolyte solution (the decrease of the capacitance being the measure of the surface coverage by adsorbed molecules), 2 – the polarization resistance of the reaction of the exchange of copper ions between the sulfide surface and the solution of copper (II) salt was measured for untreated copper sulfide and the sulfide previously treated by the solution of potential inhibitor (the increase in the polarization resistance being the measure of inhibition – see Fig. 1). The composition of the surface of sulfide samples was investigated by X-ray photoelectron spectroscopy, X-ray diffraction was use to identify the phase composition of the sulfide samples.



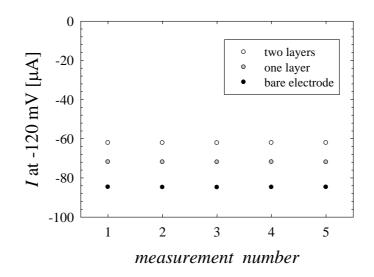
Among already tested substances the best inhibitor appeared to be sodium oleate, however strong inhibiting action showed also humic acid sodium salt, which is a very interesting result because humic substances are the natural components of the environment.

Fig. 1. Impedance spectra of an untreated $Cu_{1.87}S$ electrode and the electrode treated in sodium oleate solution, measured at the equilibrium potential in the solution containing 0.1 mol dm⁻³ CuSO₄ and 0.5 mol dm⁻³ H₂SO₄, as well as the equivalent electrical circuit used to describe the data.

Theoretical and Experimental Analysis of Multilayered Spherical Colloidal Particles Porosity

Research project N N204 347737 [2009-2012] (project leader: Assoc. Professor Paweł Weroński)

Preliminary measurements of surface roughness of gold rotating disk electrode have been conducted. The electrode has been polished with corundum powder (grain size 300 nm). We have determined that the polished surface roughness is of the order of 10% of the corundum grain. We have also verified that a few abrasive grains can attach permanently to the electrode surface during the polishing process. That can have a meaningful effect on electrochemical measurements conducted with such prepared electrode. We have measured the size and surface potential of colloid latex particles used in our experiments on adsorption at a gold rotating disk electrode. Preliminary measurements of the diffusive current at the disk electrode, rotating at various speed, were conducted. We have demonstrated that our experimental results are in a good agreement with Levich theory. We have also conducted preliminary measurements of the diffusive current as a function of the electrode surface coverage, both for the first and second adsorption layer of colloid particles. The agreement between our experimental results and approximate theoretical results is good. A further systematic research will be done based on these preliminary results. In the figure below we present a comparison of diffusive currents at the gold electrode surface potential of -120 mV. The black, grey, and open symbols correspond to the bar electrode, electrode with one layer of colloid particles of the size of 900 nm, and with two layers of particles of the size of 900 and 820 nm, respectively.



Ph.D. RESEARCH PROJECTS

of the Ministry of Science and Higher Education

Synthesis and Characterization of Composite Multimodal Materials with Enhanced Acidic Properties Prepared by the Recrystallization Method

PhD research project N205 048 31/2120 [2006-2009] (Assoc. Professor Mirosław Derewiński, Veronika Pashkova, MSc)

The recrystallization method was studied as a strategy for the preparation of the multimodal composites containing domains of the zeolitic character aside at least partially retained meso-/macroporous system of the used parent materials. Such combination of the features of zeolites (highly active acidic sites, shape-selectivity) with the advantages of meso-macroporous structures (accessibility of the interior) provides catalytically active materials with the acidic and diffusion properties enhanced. Application of various parent materials along with the selection of zeolitic structure introduced into the initial system allows tuning of the catalytic and surface properties of the prepared composites.

Standard amorphous aluminosilicates (with different Si/Al ratio), mesoporous molecular sieves (MCM-41, SBA-15) and the pre-shaped silica and silica-alumina monolithic structures were studied as the parent materials for the preparation of the multimodal composites. Zeolitic structures FAU, BEA and MFI were generated in these materials. In order to optimize synthesis procedure, variation of the basic synthesis parameters (such as synthesis time, temperature, amount and concentration of the template, method of homogenization of the reaction mixture) was performed.

Applied methods of the characterization (XRD, FT IR spectroscopy combined with the investigations of acidic properties using probe molecules, nitrogen sorption method for the investigations of porous properties, SEM and TEM techniques, ²⁷Al and ²⁹Si MAS NMR) provided the comprehensive studies of the prepared composite materials qualities along with the observation of the changes in the parent materials properties, introduced by the recrystallization. The last issue served as a basis for the revealing of the regular patterns of the recrystallization process in the different systems.

Determination of the Influence of Adsorbed Polyelectrolytes on the Kinetics of Deposition and Topology of Particle Monolayers

PhD research project N204 150 32/3822 [2007-2009] (*Professor Zbigniew Adamczyk, Anna Bratek-Skicki, MSc*)

The main goal of this work was determination of the correlation between the electrokinetic potential of surfaces covered by bilayers of polyelectrolytes and the kinetic of colloid particle deposition. The model colloid suspensions used in this work were the positively charged polystyrene latex particles. The measurements have been carried out in the diffusion and convection cells (impinging-jet cells) for mica covered by bilayer of PEI/PAA poly(ethylene imine), PEI of molecular mass of 75 kDa and poly(acrylic acid), of the average molecular mass of 12 and 70 kDa). For a quantitative determination of the bilayer formation the electrokinetic, streaming potential method was used, based on the parallel-plate, channel flow principle. The composition of bilayers, their density and surface charge have been regulated by changing the transport conditions and the concentration of polyelectrolyte, pH and the ionic strength. The bilayers formed under the in situ conditions, have been used as substrates of controlled heterogeneity in further colloid particle deposition experiments. The kinetic experiments enabled one to determine the initial deposition rate of particles on the polyelectrolyte bilayer, as well as the maximum coverage as a function of bilayer composition and density. The results of these experiments have been presented in the form of the dependence of reduced, initial deposition rate k_c/k_c^0 on the average zeta potential of the interface. It has been shown that this dependence is well reflected by the theoretical model formulated by us, based on the concept of the surface charge fluctuation. On the other hand, the widely used DLVO theory postulating a homogeneous charge distribution was proved completely inadequate. It was, therefore, concluded that that our results furnish a direct support of the surface heterogeneity hypothesis used qualitatively to interpret colloid particle deposition under a barrier-controlled deposition regime. These experimental results have a practical implication, proving that colloid particle deposition can be exploited as a sensitive tool for detecting the local charge fluctuations in the nanometer range scale.

Deposition of Model Microcapsules on Metallic Surfaces Modified by Adsorption of Polyelectrolyte Mulitayers

PhD research project N N204 214533 [2007-2009] (Professor Piotr Warszyński, Anna Trybała, MSc)

Modification of metallic surfaces by adhesion of nano- or microcapsules can be of importance for biomedical applications (e.g. bone implants or stents covered with capsules contatning anti inflammation drugs) or corrosion prevention. Therefore, the aim of the project is to investigation of the deposition of model microcapsules at bare metallic surface and ones modified with polyelectrolyte multilayers (PEM). PEM films are often used for surface modification and as adhesion promoters. The objectives of our studies performed in 2009 were as following:

- formation by 'layer by layer' (LbL) method of multilayers composed of polyelectrolytes (polycations PAH* poly(allylamine hydrochloride) fluorescein labeled, PEI poly(ethyleneimine), polyanion PSS poly(sodium 4-styrenesulfonate)) on silicon waffers;
- characterization of PE multilayers deposited in various conditions by elipsometric film thickness and fluorescence intensity measurements.

To investigate the effect of PEI first layer on the thickness of polyelectrolyte multilayers we prepared films (PAH*/PSS)_n, PEI(PSS/PAH*)_n of various thickness. The results presented in Fig. 1 indicate that films with given number of layers built up on PEI anchoring layer were thicker than structures having the same number of PE layers but consisting of PAH/PSS only. Branched PEI used as a first layer acts as an anchoring network for the consecutive layers' formation. We also investigated the effect of ionic strength of PE solution on film thickness. For that purpose polyelectrolytes were deposited from the solutions of various ionic strength: 1.5M, 0.15M, 0.015M regulated by addition of NaCl. PE concentration was 100 ppm in all experiments. The results depicted in Fig. 2 show that film thickness is growing with increasing the ionic strength of PE solution. For high ionic strength of the solution, the electric charges along PE chains are screened, therefore, chains can assume more coiled conformation. This conformation is preserved during PE adsorption and that favors thicker layers. Moreover, as a result of stronger screening the electrostatic repulsions between adsorbing PE molecules are diminished. The results of measurements of fluorescent intensity, which is proportional to the amount of adsorbed polycation indicated on similar effect of PEI and ionic strength of the PE solution.

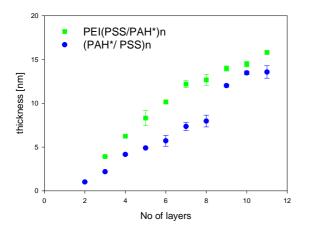


Fig. 1 Ellipsometric thickness of (PAH/PSS) and PEI(PSS/PAH) multilayer films deposited from solutions of I=0,15M.

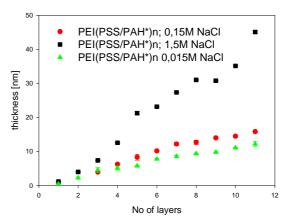


Fig. 2. Ellipsometric thickness of PEI(PSS/PAH) multilayer films deposited from solutions of various ionic strength.

Transformations of Terpene Hydrocarbons over Zeolite Catalysts

PhD research project N N204 123833 [2007-2009] (Professor Bogdan Sulikowski, Łukasz Mokrzycki, MSc)

The objective of this work was to study the desilication process as: (i) a direct modification procedure, and (ii) one of the stages in a modification/synthesis procedure. The "down approach", where mesoporous and acidity changes can be introduced to a system by the post synthesis modification is combined with the "bottom-up" method - here the changes are introduced at the synthesis level (or close to it). Next, physico-chemical characteristics of the materials obtained, coupled with the catalytic tests, were carried out. Desilication was performed both under atmospheric pressure and under hydrothermal conditions. The latter conditions were also applied for the synthesis of hybrid materials of the type *zeolite-mesoporous material*. MCM-22 zeolite was additionally modified by the delamination technique. The following zeolites were used as parent materials: commercial ZSM-5 from Zeolyst Inc., and ZSM-12 & MCM-22 type zeolites obtained by proprietary syntheses. All the materials studied had Si/Al=40.

ZSM-12, MCM-22 and ZSM-5 were modified by partial desilication using NaOH solutions of various concentrations (0.05-1.0 M) in the temperature range of 65-100°C. Hydrothermal conditions were used for the first time for desilication – the process was carried out in autoclaves under autogenic pressure at 100°C. All the samples were characterised by XRD, sorption of nitrogen, NMR and SEM. Changes of acidity in the desilicated zeolite matrices were followed by IR spectroscopy.

The XRD data revealed clearly how modification with basic solutions had affected the structure and different behaviour of the samples during the process. These data were then transformed into the crystallinity maps of the samples, showing the changes occurring during the modification. In the case of ZSM-5 and ZSM-12 their behaviour was similar, and the MFI and MTW structure was fully preserved in the range of the concentrations applied, temperatures and other conditions. On the contrary, the MCM-22 type zeolite was less stable and its structure was destroyed under treatment with 0.5 and 1.0 M NaOH. The SEM microphotographs revealed morphology of the samples studied, what was helpful for interpretation of the desilication process. It was also rationalized how modification affected the pore system of the samples. The fraction of mesopores was clearly increased both with temperature and concentration of NaOH, however, the effect of temperature was less pronounced. The ²⁹Si and ²⁷Al MAS NMR spectra showed that two other processes, dealumination and/or realumination, accompanied desilication, what was independently confirmed by XPS and EDX. Detailed analysis of the pyridine sorption revealed that desilication process has also affected the acidity of the zeolite matrices.

The modified samples obtained were tested in the liquid-phase isomerization of α -pinene, at temperatures 45-90°C. Isomerization of α -pinene is an important industrial process. The acid-impregnated TiO₂ is currently used as an industrial catalyst in batch reactors, but because of the low rate of the industrial process and corrosion problems, there is a great interest in finding novel catalysts exhibiting higher activity and selectivity towards camphene, limonene and *p*-cymene. The effect of zeolite structure, its pore system and acidity on the α -pinene isomerization course was well documented. It was shown that desilication of the ZSM-12 and ZSM-5 type zeolites led to materials with higher catalytic activity. In the case of MFI modified with 1.0 M NaOH increase in conversion from ca. 15 mol.% to ca. 70 mol.% was observed.

Influence of Modification of Electron Density of Catalysts Active Centre on its Activity in Hydrocarbons Oxidation

PhD research project N N205 018934 [2008-2010] (Professor Jerzy Haber, Robert Karcz, MScEng)

Catalysis by heteropolyacids (HPA) and related compounds is drawing since a long time a lot of attention. Heteropolyacids can be used as homogeneous or heterogeneous catalysts due to their useful properties like acidity and ability to catalyze redox reactions. The most stable HPAs are those of Keggin structure consisting of central tetrahedra containing phosphor atom surrounded by twelve metal-oxygen octahedras. It is well known that by a modification of its structure HPA properties can be tuned to the requirements of catalytic reaction. In our research tungstophosphoric and molybdophosphoric acids were modified by the exchange of counter cations or by the formation of lacunary anions followed by introduction of the transition metal cations into the lacunary position. Lacunary anions are formed by removal of one metal-oxygen species from Keggin anion and formation of empty coordination site. Such site can incorporate transition metal cation and form transition metal substituted polyoxometalate (TMSP). Hill and co-workers in their paper compared lacunary Keggin anion containing transition metal cations with metalloporphyrins due to the similarity in coordination of transition metals and possibility of the removal one by one of two electrons, controlled modification of the redox potential by using different atoms of the addenda and well defined active sites. To improve solubility in organic media phase transfer agent was used to prepare tetraalkylammonium salts. Organic salts of lacunary anions containing iron, manganese or cobalt were prepared. These metals were chosen on the basis of literature data concerning oxidation of hydrocarbons in presence of porphyrin catalysts. Especially Co complexes are highly active in autooxidation reactions. These reactions are multistep processes. The catalyst must perform two functions: activate the initial reagents and accelerate decomposition of intermediates. To compare catalytic activity of cobalt in different positions salts of parent heteropolyacids with cobalt as compensating cation also were prepared.

Studies of these catalysts allowed us to conclude how the type, position of transition metal and solubility of catalyst affects its activity in hydrocarbon oxidation reaction. As model reaction liquid phase cyclohexene oxidation was chosen. Activity and selectivity were studied as function of temperature and reagent concentrations.

The synthesized lacunary catalysts were characterized by XRD, DSC-TG, FTIR and UV-Vis spectroscopy. Physicochemical methods confirmed the introduction of transition metal atom into the addenda position of the Keggin structure. Salts containing cobalt as compensating cation were characterized by XRD and DSC-TG. The introduction of TBA cations results in the decrease of acidity with simultaneous improvement of both solubility in organic solvents and selectivity in oxidation of cyclohexene. Catalyst containing cobalt cations and tungsten in addenda position is the most active in this reaction. The Co salts prepared from tungstophosphoric acid known to be strong acid are more active catalyst in oxidation of cyclohexene than Co salts of molybdophosphoric acid, which is known to possess redox properties.

Encapsulation of active compounds in emulsions cores.

PhD research project N204 255934 [2008-2010] (Professor Piotr Warszyński, Krzysztof Szczepanowicz, MSc)

Microencapsulation is the process, in which colloidal particles or droplets are being coated by shells of various materials to obtain capsules. The layer-by-layer adsorption of polyelectrolytes is considered as a convenient method to obtain microcapsules' shells on colloidal cores. Use of emulsions droplets as liquid cores give us possibility to encapsulate oil soluble active components with control of size and shell properties of obtained capsules that opens perspectives for application in many fields such as cosmetic, medicine, pharmacy and food industry.

In 2009 we concentrated on preparation of active agent containing emulsions, which were stabilized by surfactant/polyelectrolyte complex. The obtained emulsion were used as core to produce nano and microcapsules. Emulsions, which contained oil soluble fluorescent dye (Cumarine 6) in octane, stabilized by AOT/PDADMAC complex were preapared. Emulsions were obtained with average droplets size 100nm. The multilayer shells on such prepared emulsion were constructed by subsequent adsorption of polyelectrolytes from their solutions without the intermediate rinsing step. Volumes of polyelectrolyte solution used to form each layer were chosen empirically by analyzing the results of simultaneous zeta potential measurements. Figure 1 illustrates typical zig-zag dependence of zeta potential of capsules on the adsorption of subsequent PAH and PSS layers. To confirm encapsulation, model active agent fluorescent dye coumarine 6 was dissolved in oil phase prior to emulsification. Fluorescent micrograph, shown in Figure 2, confirms encapsulation of this model substance.

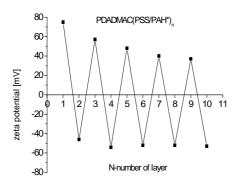


Fig.1 Zeta potential of capsules on formation of the consecutive layers of polyelectrolyte shell.

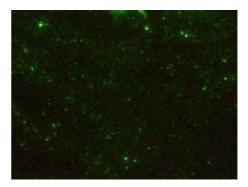


Fig.2 Image of AOT/PDADMAC(PSS/PAH) $_n$ capsules containing oil soluble fluorescent due cumarine-6.

Emulsion droplets containing silane derivatives, which can undergo hydrolysis and condensation on the drop surface that leads to formation of silica shell around liquid or semi-liquid core were also prepared. We used DTSACl (dimethyloctadecyl[3-(trimethoxysilyl)propyl] ammonium chloride) as surface active silica source in emulsions of chloroform in water. We prepared emulsions containing DTSACl, without any additional surfactant, with the average drop size 150 nm and we examined their zeta potential and stability. Progress of shell formation by hydrolysis of silica sources were observed using NMR-spectroscopy. The surface of such formed hard shell capsules was then modified by sequential adsorption of polyelectrolytes.

Conformations of Selected Globular Proteins in Aqueous Solutions and on Interfaces of Controlled Heterogeneity

PhD research project N N204 166336

(Professor Zbigniew Adamczyk, Monika Wasilewska, MSc)

Conformations of proteins in electrolyte solutions determine kinetics of aggregation processes, and mechanisms of their interactions with interfaces leading to mono- and multilayer adsorption. These con formations can be determined via measurements of diffusion coefficients of protein molecules, usually carried out by applying the dynamic light scattering method (DLS). Because of complicated shapes of proteins, deviating significantly from the spherical shape, theoretical interpretation of such measurements poses considerable difficulties. The aim of this research work was elaborating a new, efficient hydrodynamic model of the fibrinogen (Fb) molecule model, based on the bead concept (see Fig.1.). According to this model, the real shape of the molecule is replaced by a constellation of beads of various size, having the same volume as the molecule. Using this model, extensive calculations have been carried out, aimed at calculations of the hydrodynamic resistance matrix and the diffusion tensor. In this calculations the exact and efficient multiple expansion method was used. The influence of the angle φ formed between the side arms of Fb and its major axis of symmetry and the number of beads in these arms on the hydrodynamic resistance coefficients has been studied systematically. A comparison of theoretical calculations with experimental data acquired by DLS has shown that for pH < 5 and pH > 7, the diffusion coefficient of Fb assumed a maximum value, equal to $2x10^{-7}$ cm² s⁻¹. This was interpreted, using the theoretical model, as the effect of extension of the side arms leading to the increase in the φ angle above 90°. For pH in the range 5 - 6, the diffusion coefficient of Fb decreased, which indicates that the side arms were collapsing towards the Fb core region.

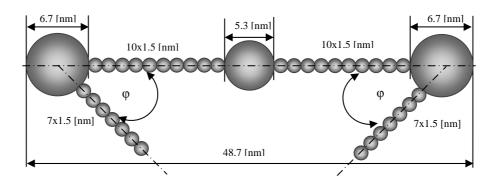


Fig. 1. The

fibrinogen used for calculating its hydrodynamic properties in the bulk.

bead model of

The Physico-chemical Properties of Bifunctional Catalysts and their Catalytic Activity in Direct Syngas-to-Dimethyleter Process

PhD research project N N204 279937 [2009-2011] (Assoc. Professor Ryszard Grabowski, Michał Śliwa, MSc)

The synthesis of metalic components of the bifunctionanal catalystys has been perfmormed. The obtained oxide catalysts composed of: 66 wt% CuO, 23 wt% ZnO, 9 wt% ZrO₂ or Al₂O₃ and 3 wt% of additives MnO or Ga₂O₃. The first series of methanol catalysts were obtained by coprecipitation method at pH 8. The solution of cation nitrates and Na₂CO₃ were added simultaneously into demineralised water under vigorous stirring. Afterwards, the precipitates of mixed basic carbonates were washed, dried at 373K and calcined in air for 3h at 623K. The second series of oxide precursors were synthesized by decomposition of the citrate complexes of metals. The mixture of citric acid and cation nitrates was evaporated in a rotating flask in vacuo, next the obtained precipitate was dried at 473K and calcined in air for 5h at 623K. The catalysts have been characterised using the following methods: BET, XRD. The N₂O decomposition technique was employed to evaluate the Cu surface area. The crystallites sizes of CuO in the fresh catalysts were determined by Rietveld method.

The physico-chemical properties of synthesised methanol catalysts are listed in Table 1. The XRD measurements were performed for the precursors and calcined samples in order to define theirs phase compositions. The recorded diffractograms show that the catalysts precursors obtained by co-precipitation consist mainly of malachite: $Cu_2(OH)_2CO_3$. The hydrotalcite is present, as a second phase, only in samples CZA and CZAG. The reflection peaks of CuO and ZnO phase appears in all samples after calcination. Due to higher temperature of drying no reflections peaks derived from hydroxycarbonates and hydrotalcite-like structures are present in the diffractograms of the samples synthesised by the decomposition of citrate complexes of metals. In this case, the peaks from CuO, Cu₂O and ZnO appear in the diffractograms.

Metallic function	Specific surface area (m ² /g)	Copper surface area (m^2/g)	CuO crystal size (nm)
CZA°	64,73	14,8	9,2
CZAM°	88,45	23,8	7,3
CZAG°	64,58	14,9	7,7
CZZ°	59,55	16,1	9,2
CZZM°	87,05	21,4	7,4
CZZG°	59,92	14,4	9,1
CZA•	19,71	6,7	20,5
CZAM•	16,1	10,3	14,4
CZAG•	21,68	7,7	17,5
CZZ•	20,85	4,8	20,5
CZZM•	16,2	7,5	20,7
CZZG•	31,91	7,8	20,7

Table1: The physico-chemical properties of metallic components

°-coprecipitation, •-decomposition of the citrate complexes of metals

The method of synthesis has a significant influence on the physico – chemical properties of the metallic component. The co-precipitation method leads to the catalysts with the higher specific area and higher copper surface area as compared to the citric method. Observed higher copper surface area for the catalysts containing manganese oxide (CZAM and CZZM) obtained by co-precipitation can arise from the possible reduction of MnO which accompanies reduction of CuO.

Mechanisms of Irreversible Adsorption of Nanoparticles and Selected Proteins on Interfaces Determined by Electrokientic Methods and the AFM

PhD research project N 204 137537 [2009-2011] (Professor Zbigniew Adamczyk, Maria Zaucha, MSc)

The goal of these studies was to determine the range of applicability of the new theoretical model, enabling calculations of the streaming potential for surfaces covered by nanoparticles, polymers and proteins. This has an essential significance for elaborating of an sensitive method for detection of trace amounts of proteins in solutions, working for the range below 1 ppm. The first stage of this work was a full physicochemical characteristics of the model polystyrene latex suspension having a controlled surface charge and dimensions. These model colloid particles will be used in the next stage as reliable reference systems in streaking potential measurements of protein covered surfaces, in particular to determine the antigen/antibody interactions. The physicochemical characteristics of latex suspensions involved particle size measurements carried out using the laser diffractometry technique and the DLS (dynamic light scattering) method and the zeta potential of as a function of the ionic strength and pH (isoelectric point), measured by microelecrophoresis. In Fig.1 results are presented, showing the dependence of the zeta potential of polystyrene latex suspension having the diameter of 520 nm on pH. These measurements enabled one to prove that the zeta potential of this latex attains high positive values for higher ionic strength and pH < 6. This corresponds to the best conditions for an efficient deposition of particles on negatively charge substrate surfaces, such as mica. Another useful property of this latex suspension is its amphoteric character, which allow one to regulate zeta potential of particles within broad limits, by the change In pH and the ionic strength.

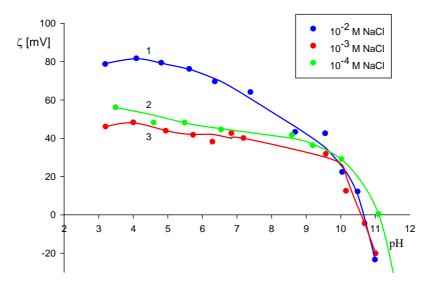


Fig. 1. The dependence of the zeta potential of positively charge polystyrene latex particles on the pH of the suspension; $1 - \text{ionic strength } 10^{-2} \text{ M}, 2 - \text{ionic strength } 10^{-4} \text{ M}, 3 - \text{ionic strength } 10^{-3} \text{ M}.$

POL-POSTDOC PROJECTS

of the Ministry of Science and Higher Education

Epitaxial Oxide Nanostructures on Metallic Single Crystalline Substrates

POL-POSTDOC II project PBZ/MEiN/01/2006/30 [2006-2009] (Dorota Wilgocka-Ślęzak, PhDEng)

According to the project schedule, the following task was accomplished: "Modifications of surface of selected oxide and its atomic scale characterization". In the frame of this task, STM measurements of 1ML FeO/Pt(997) surface modified by nanoparticles (0.1ML) and thicker layers (5ML) of gold were undertaken.

Au nanoparticles deposited at room temperature create islands of thickness corresponding to 1ML of Au(111) (~0.25nm). Particles with 2-3nm diameter dominate and show clear tendency to locate themselves on the terraces. Their distribution indicates correlation with Moire's FeO superstructure. Smaller particles (with diameter < 1nm) decorate mainly FeO steps.

In the case of thicker gold layers three types of surfaces were investigated: (I) Au deposited at room temperature, (II) Au deposited at room temperature and annealed at 500°C (III) Au deposited at 550°C. At all types of surfaces, islands elongated in the direction of FeO steps were present. STM images show strong dependence of the islands morphology on the preparation conditions. Surface I is created by flat, adjoining one to another, mosaic islands that cover FeO substrate entirely. Their typical width is 6-8 nm, what corresponds to the width of 3-4 FeO terraces. After annealing (surface II) the islands transform into huge three-dimensional objects and around 50% of FeO is exposed. Irregular, elongated islands, up to 8nm high (~35ML Au) have terrace structure with high density of regular monoatomic steps. Such gold morphology can be interesting from the model catalysis point of view. Steps as the places with lower coordination can act as adsorption centers. At the next stage of the project adsorption properties of this surface are to be investigated. In the case of surface III, regular, flat islands are present with height up to 3nm (10ML). They cover around 60% of iron oxide layer.

Establishing Standards for Allowable Microclimatic Variations for Polychrome Wood

POL-POSTDOC II project PBZ/MEiN/2006 [2006-2009] (Michał Łukomski, PhD)

The main aim of the project has been establishing precisely and quantitatively variations of air parameters that are safe for painted wood surfaces. During the third year of the project, the emphasis was on the analysis of fatigue processes caused by repeating microclimatic fluctuations.

Samples mimicking painted wood were periodically stretched and released in the universal testing machine. During mechanical deformation, the micro-cracking of gesso layer was observed and analyzed using speckle interferometry. The tests were performed at 50% of relative humidity (RH) (value recommended by most of the museums) and the amplitude of stretching corresponded to variations of RH not bigger than 20%. The mechanical cycles were performed not faster than one per second and the total number of cycles for a particular specimen did not exceed 36000. This value, for diurnal microclimatic fluctuation, corresponds to a display of an object for 100 years. The obtained results show that no damage is observed below a certain critical value of stretching (about 0.2% relative dimensional change) even for 36000 cycles. In contrast, the damage development is proportional to the number of mechanical cycles for bigger stretching amplitudes. For the critical value of 0.4%, damage is observed after first three cycles.

The conducted measurements show that the damage to polychromy on wooden supports increases proportionally to the number of humidity variations in the environment of the object. Such mechanism of damage is observed only above a certain critical value of the relative humidity change. Humidity variations of the amplitude smaller than the critical one are harmless to the object and do not lead to an increase in damage.

Synthesis and Immobilization of New Chiral Ru(II) Complexes onto Mesoporous Supports. Application in Asymmetric Catalysis

POL-POSTDOC II project D037/H03/2006 [2006-2009] (Dorota Duraczyńska, PhD)

In this study three new ruthenium coordination compounds containing chiral phosphine DuPhos were synthesized (Figure 1). These compounds are air stable both in solid and in liquid states; they form yellow crystals.

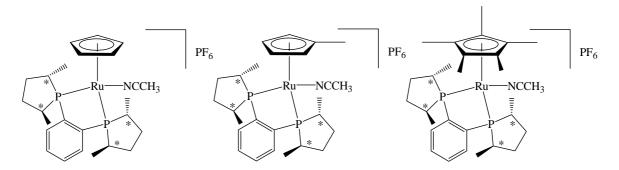


Fig. 1. The structures of ruthenium compounds containing DuPhos

They were characterized by NMR (${}^{1}H$, ${}^{13}C{1H}$, ${}^{31}P{1H}$) and UV-Vis spectroscopies, elemental analysis and X-ray crystallography.

These compounds were heterogenized on mesoporous molecular sieves MCM-40 using ionic exchange method. Characteristic included IR, XPS, UV-Vis, NMR spectroscopies and XRD analysis. Both the specific surface areas and pore volumes were determined for a carrier before and after immobilization process. All new catalysts were tested in hydrogenation of acetophenone (Figure 2).

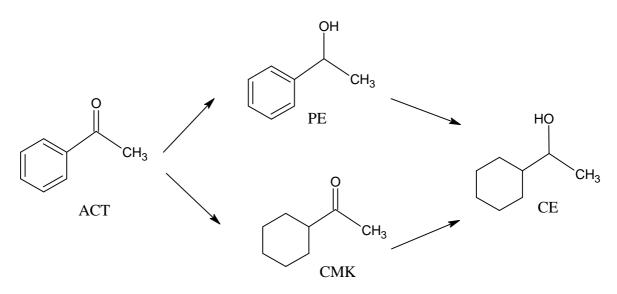


Fig. 2. Hydrogenation of acetophenone

SCIENTIFIC NETWORKS of the Ministry of Science and Higher Education

SURUZ Surfactants and Dispersed Systems in Theory and Practice

(project coordinators: Professor Piotr Warszyński, Professor Kazimierz Małysa)

The main aim of the Scientific Network SURUZ is the development of scientific cooperation and taking advantage of apparatus backup present in different research centres in Poland, which carry on studies on the surface active agents (surfactants) and colloidal systems. The teams of Scientific Network work on both basic and applicable researches on the new surfactants and technologies employing dispersed systems, thin films, adsorptive layers and so forth. The SURUZ project is accomplished by the teams from 11 research centres in Poland and the coordinator of the Network is prof. P. Warszyński (IkiFP PAN).

The tasks planned to carry on within the activity of the Network can be divided as following:

- The implementation of mutual research topics, supplemental or complementary to the tasks pursued within the basic, statutory activity of the units creating Scientific Network. The aim of these research is presented below.
- The exchange of scientific personnel as well as PhD students for optimalization of using the research infrastructure accesible for several participants of the Network. The extend of the possibility of using complementary research methods aiming at the solution of scientific problems.
- The organization of workshops intended mainly for young scientists, which will give them an opportunity to discuss about their research results and to solve emerging problems with more experienced scientists.
- The organization of meetings of the teams participating in the project for the presentation of main achievements, as well as for the discussions.
- The extend of the research area which aims at the preparation of mutual scientific projects, both national and international, especially within the 7th Framework Programme of the European Union.
- The establishing, controlling and coordinating of the researches performed within the Network; planning expenses for several tasks and controlling if the granted funds are utilized in a correct way.

The research proposed within the Scientific Network SURUZ include three priority scientific topics:

- I. Surfactants and dispersed systems synthesis and physicochemical characteristic.
- II. Dispersed systems of importance for biomedicine.
- III. Dispersed systems of importance for technology.

PV-TECH Development of New Technologies and Research Techniques in Area of Photovoltaic Cells

(subject leader: Robert Socha, PhD)

The increase of industrial and household demand on electricity and the necessity to fulfill the CO_2 regulations result with growing importance of the 'pure' energy obtained from photovoltaic cell. Additionally, the silicon shortage in the global market, followed by increasing silicon price, results in the need to utilize silicon waste from the electronic industry that leads to 'multicrystalline silicon ribbon' production, which can be applied in photovoltaic. PV-Tech network should help solution of above mentioned problems. Therefore, the project has following tasks:

- a. development of new technologies in photovoltaic cell production i.e. dedicated mainly to multicrystalline silicon ribbon cells,
- b. development of the research techniques dedicated to production and to analysis of the final product.

In the frame of the project, ICSC PAS performs analytical survey of intermediate and final stages of the silicon wafer/ribbon preparation. The stages consist: catalysts deposition for silicon etching, silicon surface etching and electric contacts formation. In these studies, the surface characterization of the delivered samples is performed by the means of microscopy (SEM) and spectroscopy (XPS, AES, EDX). Moreover, ICSC PAS delivers the test samples of the chemicals that can be applied as catalysts for silicon etching process or as materials that increase the internal reflection of light in the photovoltaic cell body.

BIONAN Molecular Mechanisms of Interactions in Biological Nanosystems and in Biologically Active Systems modified with Nanoparticles

(subjects leaders: Professor Piotr Warszyński, Professor Józef Korecki)

Understanding of molecular mechanisms responsible for alterations in the liquidity of biological membranes is of primary importance for both biologists and physicians, pharmacists as well as for physicists and chemists working on catalytic processes. It is known that destructive processes due to oxidative stress take place in living organisms. As a result singlet oxygen or free oxygen radicals are produced, causing the destruction of lipid and protein structures. Thus, the recognition of molecular mechanism which enables controlling these processes is crucial for designing of new generation drugs. These medicines exploit the encapsulation of pharmaceutically active substances. Such created capsules should be specifically delivered to the target in the organism. Moreover, the dosage of drug released should also be controlled. Therefore, a non-invasive method of controlling the transport and the permeability of the capsules is required. The implementation of nanosensors/nanomarkers based on modified carbon nanofibers, whose minor toxicity for living organisms has been proved, might significantly improve the selectivity and effectiveness of encapsuled drugs.

The researches proposed within BIONAN project are meant for studying of the activity of biologically active systems depending on their interaction with the surfaces with well defined physicochemical properties or with the nanoparticles in solution. Understanding of adhesive mechanisms at molecular level (interactions at membrane - nanolayer interface) will contribute to the conscious modelling of systems of required molecular dynamic and its affinity to specified structures.

The aim of research carried on within BIONAN project is:

- I. A built-in actions of physicians, chemists and biologists in the study of natural photosyntetic membranes and their artificial model equivalents suspended in solutions or moved onto defined solid surfaces.
- II. The study of physicochemical properties of natural and artificial systems under the influence of controlled, variable exterior factors.
- III. The establishment of optimal environment conditions and properties of the surfaces for obtaining the highest possible activity of examined, biologically active systems, or for obtaining the biochemical activity in model systems.
- IV. The development of manufacturing methods of biologically active systems with specific surface properties.
- V. The evaluation of biological nanosystems (components of photosyntetic membranes) as a platform for the construction of nanosystems that converts light energy into chemical energy.

The BIONAN network is coordinated by the Institute of Physics and Applied Informatics of AGH University of Technology.

ARTMAG Magnetic Nanostructures for Spintronic Applications

(subject leader: N. Spiridis PhD, Professor Józef Korecki))

The main activity of the network ARTMAG in 2009 concentrated on preparation of application of the project: PO IG 2.2. [2009-2011] " SPINLAB National Center of Nanostructures for Spintronic Applications".

The project coordinated by the Institute of Molecular Physics PAN has been accepted for funding (the contract with Ministry of Science and Higher Education was signed in November 2009), and the ICSC tasks, including purchase and implementation on PEEM-LEEM microscope in laboratory and synchrotron environment commence in January 2010.

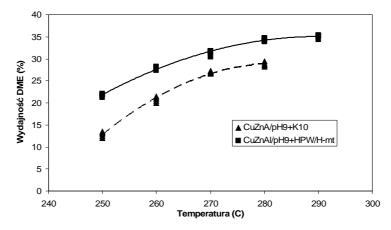
COMMISSIONED RESEARCH PROJECTS of the Ministry of Science and Higher Education

Chemistry of Perspective Carbon Transformation Multifunctional Catalysts Derived from Layered Minerals for Single Stage Process Syngas→Methanol→Dimethyether

Commissioned research project PBZ-MEiN-2/2/2006 [2006-2010] (project leader: Professor Ewa Serwicka-Bahranowska)

Project aims at development of novel bifunctional catalyst for direct transformation of syngas to dimethylether (STD), via methanol as an intermediate.

Design of both components of bifunctional catalyst was based on the use of synthetic or natural layered minerals. The catalysts for methanol synthesis from syngas were prepared from Cu-Zn-Al hydrotalcites doped with zirconium or gallium. The tests were performed in the high pressure fixed bed flow reactor. The dehydration reaction of methanol to dimethylether (DME) was tested over catalysts obtained by modification of montmorillonite clay. The measurements were performed over commercially available montmorillonite K10, the hydrogen form of Milowice montmorillonite (H-mt) and supported systems, in which heteropolyacid H₃PW₁₂O₄₀ was deposited onto K10 and Hmt carriers. The experiments resulted in the selection of a set of most active hydrotalcite-derived catalysts for methanol synthesis and a set of silicate catalysts most active in methanol dehydration, as potential components of a hybrid STD catalyst. The hybrid catalyst was prepared by mechanical mixing of redox and acid components in the ratio 2:1. Reaction was carried out at in a fixed bed flow reactor at 40 bar pressure, using syngas of composition H₂:CO:CO₂=65:30:5, in the temperature range 250-290°C and GHSV=3600 h⁻¹. The experiments demonstrated the importance of the nature of acid component. Attached Figure compares the performance of two hybrid catalysts, which both contained the same redox component (CuZnAl/pH9), but different acid components: either commercial acid catalyst montmorillonite K10, or laboratory prepared catalyst HPW/H-mt. In the whole tested temperature range the sample containing HPW/H-mt performed better. Detailed analysis of the data demonstrated that in the range of highest yields the effect was due to the increased CO conversion rather than increased selectivity to DME. On both hybrid catalysts, next to DME, emission of significant amounts of methanol was observed, which indicates that further optimization of the catalyst should be possible by adjusting the ratio of redox:acid component.



Investigations of the Physicochemical Properties of the Passive Layers on Aluminum, Magnesium and Titanium Alloys and Zn-Ni, Zn-Co, Sn-Zn, Co-P and Ni-P Alloy Coatings on Steel Impedance analysis of the process of corrosion of magnesium alloys

Commissioned research project PBZ-MNiSW-01/I/2007 [2008-2010] (project leader: Assoc. Professor Pawel Nowak)

Titanium and its alloys show very high corrosion resistance and excellent mechanical properties. So, they found wide application both in the aerospace industry, as a construction materials, as well as in medicine, as a material for implants (due to their biocompatibility). Corrosion of titanium and its alloys at the temperature of 37°C degree and in the solutions similar to physiological solutions is widely described in the literature. However from the point of view of the aerospace industry much more important would be the corrosion resistance in more aggressive media, that the parts of the machines are subjected to during their exploitation (salt brines present in airports during the winter). 5% NaCl solution may be used as a medium simulating such aggressive environment. The goal of the works, conducted jointly with the team from Silesian Technical University in 2009 year was the development of new procedures enabling the preparation of the protective layers on the surface of titanium and its alloys, which would increase the corrosion resistance of those materials in aggressive environments occurring during the exploitation of airplanes.

Samples prepared by the team from Silesian Technical University were used in the measurements. Five series of samples were investigated. Samples were covered by the protective layers, increasing their corrosion resistance. Different passivation baths (fluorosilicate, phosphate and silicate) and passivation conditions (time, temperature and voltage of passivation) were applied in sample preparations. The measurements were performed in 5% NaCl solution at the temperature of 30°C. For each sample a series of impedance spectra at the predetermined time intervals was registered to follow changes in the corrosion resistance. The equation describing the impedance of a selected electrical equivalent circuit was least-squares fitted to measured impedance data which enabled the calculation of the corrosion resistance.

The corrosion resistance of the samples differed strongly, depending on the procedure of preparation. The best corrosion resistance showed samples prepared in the silicate bath. Corrosion resistance of some samples from that series practically did not change for months. Protective layers obtained using phosphate bath showed good resistance for 40 to 840 hours, the samples obtained at higher voltage were more stable. Finally, the samples prepared using fluoroborate bath showed the lowest corrosion resistance, practically similar to pure titanium.

Investigations of the magnesium alloys, commenced in the year 2008 were continued in the year 2009 too. Basing on the experience gained during last year new procedures of the preparation of phosphate-permanganate baths were proposed and tested. The protective layers prepared using those baths had corrosion resistance higher by an order of magnitudes than the previously prepared ones, and higher than commonly used chromate layers which are considered to be the best.

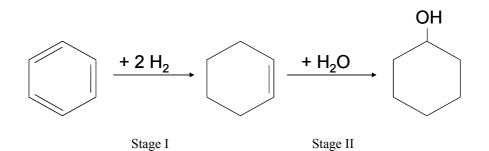
RESEARCH PROJECT "TECHNOLOGICAL INITIATIVE I" of the Ministry of Science and Higher Education

Development of a New, Wasteless Technology of Cyclohexanol/Cyclohexanone Synthesis (Intermediates in Nylon 6 and Nylon 66 Synthesis) from Benzene *via* Cyclohexene



Research Project Technological Initiative 1 KB/69/13848/IT1-B/U/08 [2008-2010] (project leader: Professor Ewa Serwicka-Bahranowska)

The project aims at development of scientific bases for the technological process of cyclohexanol synthesis from benzene, with efficiency allowing for its implementation in practice at a pilot scale. The method is attractive because virtually no side-products are generated - the cyclohexane which forms together with cyclohexene is a valuable commercial chemical. Research consisted of two stages. The first one involved synthesis of the catalyst for benzene to cyclohexene hydrogenation and optimization of the process conditions. Among a number of studied catalysts the most effective and easy to prepare was metallic ruthenium in the form of ruthenium black. It was found that the catalytic performance depended strongly on the degree of Ru particles dispersion. The reaction was carried in a four – phase system: liquid organic phase (benzene), liquid water phase, gas phase (hydrogen) and solid phase (catalyst suspension). Optimum conditions to conduct the reaction are: pressure 5 MPa, temperature 150°C. The reaction selectivity can be improved by using special additives, in particular some inorganic metal salts.



Second stage of our work dealt with the design of a catalyst for hydration of cyclohexene to cyclohexanol and optimization of the process parameters. The studies of the cyclohexene hydration were performed in a three phase system consisting of liquid organic phase, liquid water phase and solid phase – the catalyst. In the latter capacity various zeolite catalysts were used. The optimum process temperature was established at 120 - 150° C. Among optimized factors were the type of zeolite structure, the degree of zeolite alumination and the quantity of the catalyst. It was found that in the presence of zeolite ZSM-5 the selectivity of hydration to cyclohexanol is higher than in the case of mordenite. On mordenite a certain amount of dicyclohexyl ether was formed in addition to cyclohexanol. For ZSM-5 zeolite the dependence of yield of cyclohexanol on Si/Al ratio showed a maximum in the range 60 < Si/Al < 100. Indirect hydration of cyclohexene via ester formation with acetic, sulfonic and sulfuric was also investigated. Best results were obtained for reaction of sulfuric acid with cyclohexene to cyclohexyl sulfonate, which was in the next step hydrolyzed to cyclohexanol and sulfuric acid.

EU 6th FRAMEWORK PROGRAMME PROJECTS

IDECAT Integrated Design of Catalytic Materials for a Sustainable Production



UE NoE Project IDECAT NMP2-011730-2 [2005-2010] (project leader: Professor Malgorzata Witko)

IDECAT (Integrated Design of Catalytic Naomaterials for a Sustainable Production) is a Network of Excellence (NoE) launched in 2005 within 6th EU Framework Programme for Research and Technical Development. The Network is coordinated by the Consorzio Interuniversitario per la Scienza e Tecnologia dei Materiali, Italy and integrates 37 laboratories from 17 institutions: Consorzio Interuniversitario per la Scienza e Tecnologia dei Materiali, Italy; Ghent University, Belgium; Katholieke University Leuven, Belgium; J. Heyrovsky Institute of Physical Chemistry, Czech Republic; Helsinki University of Technology, Finland; Centre Nationale de la Recherche Scientifique, France; Max-Planck-Gesellschaft, Germany; Leibnitz Institute of Organic Catalysis, Germany; Technische University Munchen, Germany; Consiglio Nazionale delle Ricerche, Italy; NRSC-Catalysis, Tha Netherlands; Instytut Katalizy i Fizykochemii Powierzchni, Poland; Consejo Superior de Investigaciones Cientificas, Spain; Stockholm University, Sweden; University St. Andrews, The United Kingdom; University of Southampton, The United Kingdom; Ecole Polytechnique Federale de Lausanne, Switzerland.

The aim of the IDECAT NoE is to achieve a lasting integration between the main European Institutions in the area of heterogeneous, homogeneous and bio-catalysis via creating a coherent framework of research, know-how, training, promotion of science, spreading of excellence and technology transfer between those various catalysis communities.

The Network is divided into 5 programmes (Joint Programme of Integration, Joint Programme of Research, Joint Programme of Spreading Excellence, Joint Programme of Science and Technology Transfer and Management) and 13 Workpackages.

Joint Programme of Integration has been aimed to achieve long-term integration within European Research Institute of Catalysis (ERIC) which has been elished as a legal entity under the Law of Belgium in the form of a non profit association - A.I.S.B.L (Association International Sans Bout Lucratif). ERIC is basically composed of the vast majority of IDECAT partners, integrating the remaining institutions with the Framework Agreement. According to the Mission, ERIC aims to gather highly specialised expertise and know-how at the excellence level and serve as interface and platform to communicate industrial and societal needs in the field. Moreover, within the JPI, electronic tools to facilitate the gathering of information and allow better communication between the integrated groups were have been further improved. As regards gender issues, encouragement activities to increase women participation in research were continued, mainly in the form of the workshop "Gender, Science and technology".

The Joint Programme of Research focuses on four research tasks: 1) creating and mastering nano-objects and nano-organisation, 2) creating and designing new multifunctional and nano-structured materials, 3) bridging the gap in multi-disciplinary approaches, 4) eco-processes and sustainable energy and production. Activities within these tasks have been continued by the ICSC research groups in the frames of collaborations started within the project.

In the frames of The Joint Programme of Spreading Excellence ICSC was actively involved in the Festival of Science and The Open Door of the Institute was organised.

The objective of the Joint Programme of Science and Technology Transfer is to transfer to the industrial sectors and in general terms to Society of the scientific results and breakthrough arising from the network. The Industrial Liaison Office organized two IDECAT Industrial Board meeting and workshops "Industry – Academia Partnerships in catalysis" with the invited lecture of prof. Piotr Warszyński for the ICSC.

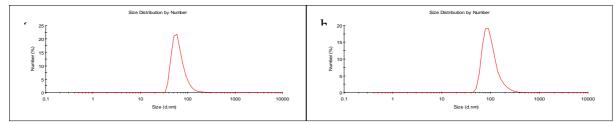
ICSC was actively participating in the vast majority of the Network activities and tasks.

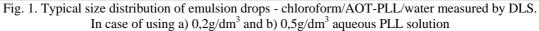
NANOMEDPART Multifunctional Particulate System for Nanomedicine

NANOMEDPART [2007-2010] in frameworks of ERA-NET MATERA project (project coordinator: Professor Piotr Warszyński)

The objective of the project is to develop effective methods for preparation of nanoparticles/nanocapsules and modification of their surfaces for targeted drug delivery (anticancereous, antimicrobial). Various methodologies of capsule core and shell formation and surface modification are investigated and optimised for selected applications. The main scientific and technical challenges of the project are the formation of stable particles and capsules in the nanosized range containing active compounds with shells tailored for cancer therapy and antimicrobial applications. For the formation of functionalised shells various adsorption or polymerisation techniques are used, combined with surface modification. The project is realized by the consortium consisting of: Institute of Catalysis and Surface Chemistry PAS, SINTEF Materials and Chemistry Oslo, Hebrew University Jerusalem, Institute for Cancer Research at the Norwegian Radium Hospital Oslo and Chemistry Department of Wrocław University of Technology.

In 2009 we concentrated on preparation of dye and model drug (Vitamin A and β -carotene) containing capsules based on emulsion cores with PE shell. Our results demonstrate that layer-by-layer technique can be successfully used to build polyelectrolytes multilayers on nano emulsion droplets stabilized by ionic surfactant (AOT) and biocompatible polycation (PLL), Multilayer shell were formed from biocompatible polyelectrolytes (PLL, PGA). Obtained capsules give a possibility to encapsulate active, hydrophobic substance in one step process, without dissolving sacrificial core and refilling the shell. The average size of capsules is 50-100 nm (Fig. 1). Usage of chloroform core, which can be easily evaporated from suspension and biocompatible polyelectrolytes as PLL and PGA to form a shell, should make capsules useful in biological or medical application.





A simple and economic method of synthesis of silver colloid nanoparticles with controlled size was developed. We produced nanoparticles with size below 20 nm (Fig. 2). Silver nanoparticles showed high activity against Gram-positive and Gram-negative bacteria (lab isolated strains), and clinical isolated strains included highly multiresistant strains such as *Staphylococcus epidermidis*, *Staphylococcus aureus* and *Pseudomonas aeruginosa*.

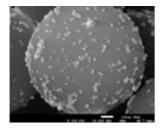


Fig. 2. SEM micrograph of silver nanoparticles reduced with hydrazine monohydrate deposited on opposite charge Latex particles.

PARFUN Nanoscale Surface Treatments to Functionalise Polymer Particles for Electronic Applications Controlled attachment of polystyrene particles at PEI modified surfaces

PARFUN [2007-2010] in frameworks of ERA-NET MNT project (project leader: Professor Piotr Warszyński)

The main objective of the project is to develop a new generation of spacer particles for LCD based on new concepts for surface treatment of polymer particles, and thereby also greatly improve the manufacturing process of LCD screens. There is a strong need for spacers with a careful control of the particle size and size distribution together with suitable surface properties. The introduction of flexible displays that can be bend and twisted during their use will need better adhesion between the spacers and the inner surface of the display. Also new and improved techniques for distribution of the spacers in the display is needed.

The aim of our activities in the project was to develop methods for surface modification of glass and/or plastic plates (poliamid - PI) in order to obtain better adhesion of micron size polystyrene particles in well controlled patterns. For surface modification cationic polyelectrolyte polyethyleneimine (PEI) was used. PEI of molecular weight 700 kD was adsorbed from 500 ppm aqueous, ethanol, and isopropanol solutions by dipping the glass plate in the solution. First attempt to selectively adsorbed PEI by microcontact printing were made. The adhesion of to types of latex particles, provided by Conpart was examined from ethanol and isopropanol suspension.

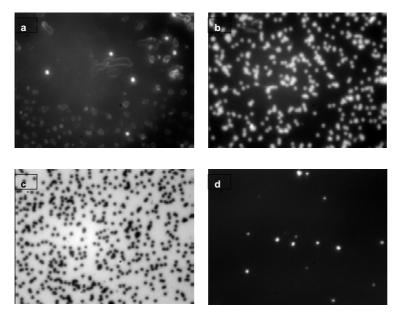


Fig. 1. Microscopic image of 4 μ m latex particles deposited at PEI surfaces from isopropanol solution. Bare PI plate a – negative particles, b – positive particles; PI plate modified with PEI, c – negative particles, d – positive particles.

The stability of coating was examined. PEI modified plates with spacer particles were soaked by 48 hours in the isopropanol solution. They were also contacted (for 1 week) with liquid cristal with liquid cristal. No detachment of particles was observed. The adhesion of particles in the shear flow in the "oblique impinging jet" was also checked. The applied shear stress up to 3 Pa did not caused detachment of spacer particles.

In order to obtain conductive spacer particles they were coated by either silver nanoparticles or by layer of metallic silver (electroless coating). The surface properties of coated particles were examined.

NANOREP II Multifunctional Surface Coatings and Highly Scratch Resistant Plastic Parts Surface Modification of Nano- and Micron Size Plastic Modifiers

NANOREP II [2007-2010] in frameworks of ERA-NET MNT project (project leader: Professor P. Warszyński)

The main objective of the NANOREP II project is the development of technology that will allow application of nanosized particles and nanostructured coating materials with multifunctional properties for (near)future high quality polymer products. The project is aiming to create a strong transnational network of research activities in the field of nanoparticle materials and bring the partners a step forward into implementation of nanotechnology in their products and applications.

The aim of our investigation was to determine the conditions to obtain the surface treated glass beads with good adhesion and compatibility with termo- and fluoroplastics.

Adhesion of microspheres was examined by colloidal probe AFM, based on the force measurement between glass beads attached to the cantilever of AFM probe and different surfaces (glass, glass modified with same chemical as the bead, silicon). The interaction force F was calculated from Hooke's law:

$$F = kD$$

where k is the cantilever spring constant determined from thermal tune method and D is deflection of cantilever.

An example of the force deflection curve obtained with colloidal force AFM is shown in Fig. 1.

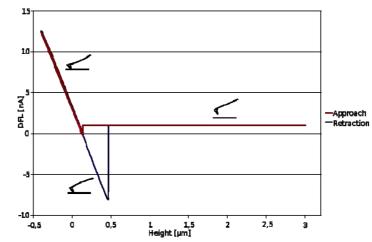


Fig. 1. An example of the force deflection curve obtained for interaction between glass bead modified with 1% APS (-3-Aminopropyl)triethoxysilane and glass surface.

We found that measured adhesion forces were in the range from 8.2·10-7 N to 1.6·10-5N. Adhesion to silicon was always weaker than adhesion to the surface modified with same chemical as glass beads. In the case of glass bead covered with 1% APS there were no significant differences in interaction with different surfaces, which proved previous finding that APS did not hydrophobize glass beads.

EU 7th FRAMEWORK PROGRAMME PROJECTS

InGAS Integrated Gas Powertrain – Low Emission, CO₂ Optimised and Efficient CNG Engines for Passenger Cars and Light Duty Vehicles



EU 7th FP InGAS Collaborative Project [2008-2011] (project leader: Professor Ewa Serwicka-Bahranowska, principal investigator: Tadeusz Machej, PhD)

InGAS is a large-scale international collaborative research project launched within 7th EU Framework Programme for Research and Technical Development. The project integrates 28 partners from 11 European Countries: Austria, Czech Republic, Denmark, Finland, France, Luxemburg, Germany, Poland, Sweden, Switzerland and Italy. The Consortium is coordinated by Centro Ricerche Fiat SCpA. Starting date of the project is 1st October 2008.

Main objective of the InGAS project is to deploy custom designed CNG engine integrated with specific aftertreatment systems applied to passenger cars and light duty vehicles able to achieve 10% higher fuel conversion efficiency than that of a corresponding 2006 Diesel vehicle and complying with an emission level lower that Euro 6.

ICSC is involved in Sub-project B2 "Aftertreatment for passengers cars CNG engines", Workpackage B2.2 "Advanced Catalysts Development". Research aims at design, synthesis, characterization and catalytic testing of new catalyst formulations for methane oxidation in CNG fueled engine exhausts, based on mixed transition metal oxides.

n 2009 several sereies of catalysts have been investigated: a) derived from hydrotalcite-like precursors, b) spinels and perovskites prepared by sol-gel procedure from citrate precursors, c) hexaaluminates, d) pillared clays. The synthesized solids were characterized with chemical analysis, XRD, SEM, XPS, FT-IR, thermal analysis, nitrogen adsorption at 77 K, and TPR in the hydrogen flow. The most active catalysts were found among hydrotalcite-derived mixed oxide catalysts containing Cu and/or Mn. A strong synergetic effect was observed in these solids, consisting in a better reducibility of mixed oxides containing both Cu and Mn, than in systems containing only one of the two transition metals. XPS spectroscopy demonstrated that these solids contained redox couples involving transition metal ions, between which mutually linked electron transfers occurred. Structure of mixed oxides was strongly amorphous, with traces of nucleating Cu-Mn spinel phases. Influence of various promoters was also investigated. Selected citrate-derived mixed oxides and pillared clays also display appreciable activity. Hexaaluminates formed the least active group. The obtained results show that a catalyst of high activity in low temperature methane combustion can be prepared based on mixed oxides of Mn and/or Cu, with addition of appropriate promoters.

NEXT-GTL Innovative Catalytic Technologies & Materials for Next Gas to Liquid Processes

EU 7th FP NEXT-GTL Large-scale Integrating Project [2008-2011] (project leaders: Professor Malgorzata Witko, Professor Ewa Serwicka-Bahranowska)

The main goal of the NEXT-GTL research project is to explore new technologies for conversion of methane to metanol, dimethyl ether (DME) and aromatics. The objective is to develop new processes which may be particularly suited for remote areas to facilitate transport of liquid products.

The kick-off meeting of the project took place in Brussels in the first half of November. During the meeting, project leaders defined the specific focus points for the first six months of collaborative research. In the mean time, the group of researchers from Institute of Catalysis and Surface Chemistry PAS was formed who will be pursuing the scientific work to meet NEXT-GTL objectives. It is anticipated that the research done at the Institute will be of theoretical and practical character.

Firstly, already published results of research on heteropoliacids and their reactivity as catalysts as well as carbene complexes with noble metals were gathered and analyzed. These will be the main groups of catalysts which are selected to focus on within the project by researchers from ICSC.

Acording to the state of the art of nowadays quantum chemistry, theoretical methods were chosen to adress the goals of the project. It was decided to use Density Functional Theory (DFT) within two non-local functionals (Becke-Perdew and Perdew-Young) and to apply both cluster and periodic approachs.

Next, the energetic effects of selected methane and its derrivatives transformations were explored:

Reaction	dE [eV]
$CH_4 \rightarrow CH_3 + H$	4.87
$CH_3SCH_3 + O \rightarrow CH_3OCH_3 + S$	-1.53
$CH_4 + O \rightarrow CH_3OH$	-4.08
$2CH_4 + S \rightarrow CH_3SCH_3 + 2H$	3.37
$CH_3SCH_3 \rightarrow CH_3S + CH_3$	3.15
$CH_3 + S \rightarrow CH_3S$	-3.21
$CH_3 + O \rightarrow CH_3O$	-4.26
$CH_3 + OH \rightarrow CH_3OH$	-4.18
$CH_3S + O \rightarrow CH_3O + S$	-1.06

Lastly, the preliminary calculations of geometry and electronic structure of $H_3PW_{12}O_{40}$ and N-heterocyclic platinum complex were done.

MUST Multi-level protection of materials for vehicles by "smart" nanocontainers Investigation of the interaction of model anticorrosive coatings with the corrosive environment by IR ATR spectrometry, XPS spectrometry and electrochemical methods



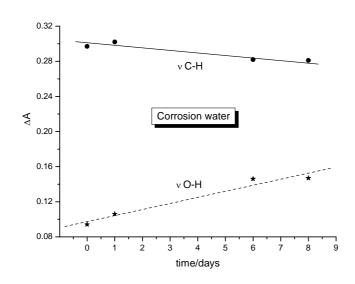
EU 7th FP MUST Collaborative Project [2008-2011] (project leader: Professor Piotr Warszyński))

The objective of the MUST project is the development of new active multi-level protective systems for future vehicle materials based on "smart" release nanocontainers incorporated in the polymer matrix, containing anticorrosion agents. Main object of the investigations at the ICSC in 2009 year was the interaction of the model anticorrosive coatings with the corrosive environment by Infrared (IR) attenuated total reflection (ATR) spectrometry. Additional investigations were performed using X-ray photoelectron spectrometry (XPS), fluorescence microscopy as well as electrochemical methods.

Two model coatings were chosen for the investigations: one organic solvent based epoxy resin and one water based epoxy resin, both supplied by other partners of the project. The model coatings were deposited on the surface of germanium reflection elements in the form of a thin layer and contacted with the corrosive solutions: so called "corrosion water" (ASTM standard D1384-7: NaHCO₃ 138 mg*dcm⁻³, Na₂SO₄ 148 mg dcm⁻³, NaCl 165 mg dcm⁻³), or 5% NaCl solution, or just pure water were applied. The IR ATR spectra were then registered after the predetermined periods of time.

Penetration of water into the organic coating was observed as the increase in the intensity of the absorption band of water at the same time delamination of the layer of polymer from the substrate caused the decrease in the intensity of the IR absorption band characteristic for the C-H bond (see figure 1 below). Diffusion of water in the polymer layer may also easily be investigated using IR ATR spectrometry.

XPS spectrometry enabled the investigations of the interaction of the metallic substrates with the so called pretreatment media, which are applied on the surface of metallic parts before the layer of the metallic coating is deposited. The influence of different additives on the corrosion resistance of the coatings was investigated by electrochemical methods, mainly electrochemical



impedance spectroscopy. Several different anticorrosive agents, selected jointly by the partners of the project were tested, mainly in the form of capsules added to the organic coatings.

Fig. 1. Changes in the intensity of the absorption band of liquid water (v_{O-H}) and of polymer (v_{C-H}) during the interaction of organic solvent based epoxy resin, deposited on the surface of the germanium reaction element, with "corrosion water".

Smart Monitoring of Historic Structures

EU 7th FP SMOOHS Collaborative Project [2008-2011] (project leader: Michał Łukomski PhD)



The main aim of the project is to develop a wireless monitoring system of historic structures which could be used by conservation practitioners in the field. It will have a modular structure so that one can select monitoring modules and combinations of sensors to answer specific questions arising at an object.

An acoustic emission (AE) sensor will be utilized for monitoring the progress of damage for historic objects. It is necessary to elaborate a measuring methodology which will allow a continuous monitoring of materials for a long period of time (up to two years) especially that the system will be power supplied by a battery. The most important task is defining characteristics of the acoustic emission signals related to the damaging processes for different historic materials, as well as the elaboration of a method capable of separating the relevant signal from the environmental noise.

The AE signals from different wood species were measured at this institute during compression tests in a universal testing machine. Furthermore, restrained wood specimens were monitored during the water sorption processes. The results were compared with the AE signals measured during non-destructive processes like free swelling of samples during the water uptake or rubbing two wooden samples one against another. Similar tests were conducted for sandstone from a historic portal in the Schwäbisch Gmünd Church in Germany. As a result, the time and frequency characteristics of the AE signals for destructive and non-destructive processes for two different types of materials were obtained. The preliminary analysis shows that acoustic waves with frequency higher than 100 kHz are released during the destructive processes. Therefore, it may be assumed that the frequency filtering can serve as a good method to separate signals from the environmental noise for the developed sensor.

Selection of the best sensor and the detection scheme is also a part of the conducted research. Therefore, a comparative testing of the AE signals released from wood and sandstone was performed using the equipment built in this institute and in the Stuttgart University. The results will be processed at the beginning of the next year.

ROCARE ROman Cements for Architectural REstoration to New High Standards

EU 7th FP 226898 ROCARE Collaborative Project [2008-2011] (project leader: Assoc. Professor Roman Kozłowski))

Highly hydraulic binders, known as natural or Roman cements, were key materials to cover façades of buildings of during the nineteenth and early twentieth centuries, a period of rapid urban growth in Europe. The conservation, restoration and reconstruction of historic Roman cement façades form therefore an important issue in Europe's efforts to preserve its architectural heritage. The former EU FP5-project ROCEM (2003-06, coordinated by the institute) has re-established manufacturing of Roman cements at a pilot scale and initiated their use in conservation practice. The main steps of the new ROCARE project planned for 2009-2011 are: scaling up of the RC technology to a commercial level, laboratory studies to fully understand cement hydration and property development as well as optimum conditions of mortar processing and handling in the conservation practice, broad dissemination measures which will allow the concept of low-energy Roman cements to establish itself on the European market of building construction.

The project started on September 1, 2009 with a kick-off meeting of the project consortium as well as the meeting of End-User Advisory Panel - comprising over 20 experts representing all relevant European countries. The institute leads Workpackage 3 "Dissemination and marketing" and has implemented Task 3.1 during the reporting period: preparing a questionnaire for the EAP meeting and drafting the first EAP report giving opinions, expectations and recommendations of the end-users which will be used in planning the laboratory work and field tests, and facilitate local conservation projects.

The implementation of the key research task of the institute – establishing the mechanism of shrinkage cracking in the Roman cement repair mortars by determining water vapour desorption curves, pore structure and desiccation shrinkage as a function of hydration period – has started with the preparation of the samples and the experimental set up to determine the shrinkage of mortars, composed of several displacement transducers and reference quartz crystal to quantify temperature-induced dimensional changes.

F3 Factory Flexible, Fast and Future Factory – European Chemistry Consortium Begins the Journey into the Future of Production

EC 7th FP CP-IP 228867-2 F3 Large-scale Integrating Project [2009-2013] (*project leader: Professor Piotr Warszyński*)

The F3 consortium's vision is that the EU's chemical industry's competitive position would be strongly enhanced if it could operate modular continuous plant (F3 plant), which combines world scale continuous plant efficiency, consistency and scalability with the versatility of batch operation. Our project will deliver such a radically new production mode based on:

- Plug-and-play modular chemical production technology, capable of widespread implementation throughout the chemical industry. This technology uses generic backbone facilities designed for rapid interfacing with standardized process equipment containers (PEC). The PEC house process equipment assemblies (PEA) composed of intensified process equipment for fast, flexible future chemical production
- Holistic process design methodology applying process intensification concepts and innovative decision tools. This will accelerate process development and provides a substantial reduction in energy consumption, raw material usage and plant volumes.

The F3 consortium of leading academic & research institutions and 7 major synthetic chemical producing industrial companies has 3 main goals:

- 1. To prove the technical feasibility of the F3 mode of manufacturing by building and operating a 0.1 to 30 kg/hr demonstration facility,
- 2. To demonstrate that operation of F3 plant will be more economical, eco-efficient and more sustainable than conventional production modes like large scale continuous or small to medium scale batch processing.
- 3. To drive a step change in the technology available to EU chemical production and engineering companies by designing intensified equipment for reaction and down stream processing, dissemination of standards for plug and play modular plant and providing open access to the backbone facility.

The main task of ICSC in the F3 project is to model properties of polyelectrolytes and copolymers based on the polyacrylic acids and to evaluate their adsorption properties, formation of thin polymer films and validation of performance of copolymers produced by project partners for the encapsulation of active compounds. This activity will start in late 2010.

EU COST ACTIONS PROJECTS

Molecular Structure-Performance Relationships at the Surface of Functional Materials

Interfacial functionalization of (bi)-metallic nanoparticles to prepare highly active and selective catalysts: understanding synergy and/or promotion effect

EC COST Action D36 research project [2006-2011] (Polish representative: Professor Józef Korecki)

Model catalysts

Bi-metalic Au-Fe nanoclusters deposited at ultrahigh vacuum conditions on a single crystalline rutile $TiO_2(110)$ substrate were studied using STM and XPS, in view of the influence of the deposition sequence (Au,Fe or Fe,Au) on the cluster morphology and electronic properties. The most interesting and innovative result demonstrating the impact of the deposition conditions on the cluster assembly is shown in Fig. 1. The diameter (and height) of these bimetallic clusters can be tuned by annealing temperature and preparation recipe, which will be used for modeling real catalysts.

Real catalysts

Au-Ni supported systems were investigated in catalytic CO oxidation. Three types of catalysts were prepared:

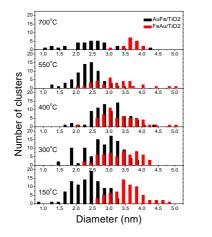
i. Au-Ni nanoalloys supported on titania (Ni-Au/TiO₂).

ii. Au nanoparticles deposited on Ni clusters or films and next supported on titania (Au/NiTiO₃Ti).

iii. Au and Ni nanoparticles subsequently deposited on titania (Au/Ni/TiO₂)

The activity of the synthesized catalysts was tested in the CO oxidation. It was shown that the lower the oxidation state of nickel, the lower is the catalytic activity. Such a behavior is universal and independent of the preparation method, regardless of the system bimetallic or alloy character.

Fig. 2 shows the catalytic performances of the catalysts (Ni oxidation state - 0) and their precursors (Ni oxidation state +2). In each case the activity of the catalyst is lower than that of the precursor. The promotion of the Au nanoparticles by nickel on the 0-oxidation state does not improve the activity of the catalytic system in the CO oxidation reaction.



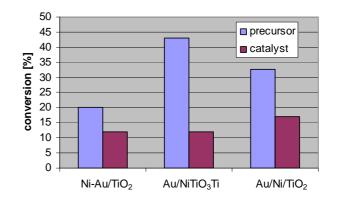


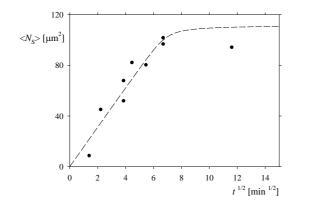
Fig. 1. Size distribution of the bimetallic goldiron clusters as a function of the deposition sequence and annealing temperature.

Fig. 2. Catalytic performances of the catalysts and their precursors in the CO oxidation reaction

Colloid and Interface Chemistry for Nanotechnology

EU COST Action D43 research project [2006-2011] (Polish representative: Professor Zbigniew Adamczyk)

Monodisperse silver core/shell particles were synthesized according to the method of Creighton et al. by reduction of AgNO₃ solutions with NaBH₄ in the presence of polyvinyl alcohol as the stabilizing agent. Bulk characteristics of silver nanoparticles in aqueous solutions were carried out by measuring their plasmon absorption spectrum, fluorescence, diffusion coefficients using the PCS method and the electrophoretic mobilities. The maximum absorbance occurred at the wavelength of 393 nm, increasing linearly with concentration of sols. It was also shown that the silver suspensions did not exhibit measurable fluorescence. The average hydrodynamic diameter of PVA covered silver particles was 44 nm, being fairly independent of ionic strength (changed in the range $I=10^{-5}-10^{-2}$ M) and pH in the range of 3-9. It was also shown that the hydrodynamic radius did not change within prolonged storage of suspensions (up to 75 days), indicating that the sols were quite stable. A similar value of 45 nm \pm 8 nm was determined from SEM measurements (see Fig. 1, part "b"). The electrophoretic mobility measurements showed that the zeta potential of silver nanoparticles was insensitive to pH an decreased with the ionic strength, attaining -45 mV for $I=10^{-5}$ M and -25 mV for $I=10^{-2}$ M. The well characterized silver sol was used for determining the kinetics of nanoparticle deposition on mica modified by adsorption of a saturated layer of poly(ethylene imine) (PEI). The surface coverage of particles was determined directly by counting the number of particles over various surface areas using the atomic force microscopy working in the semi-contact mode. The maximum surface concentration of particles for $I=10^{-3}$ M was 102 μ m⁻², which corresponds to the coverage degree of 17%. The kinetic run and the maximum coverage value was in a good agreement with theoretical predictions stemming from the random sequential adsorption (RSA) mode (see Fig. 1). This kinetic run allowed one to determine the size of Ag core, which was 20 nm and the thickness of the PVA layer, equal to 12 nm. These investigations confirmed that it is feasible to produce uniform silver nanoparticle monolayers on solid substrates, characterized by controlled density and structure using the colloid self-assembly from aqueous sols.



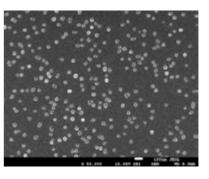


Fig. 1. Part "a": the kinetics of silver nanoparticle deposition at mica modified by the saturated PEI monolayer, determined by the AFM. Particle deposition conditions: pH=5.5, $I=10^{-3}$ M, T = 293 K. Full points represent the experimental data and the solid line shows the theoretical results obtained by numerical solution of the diffusion equation with the RSA blocking function. Part "b": silver particle monolayer (SEM picture, average particle size 45 nm).

Wood Science for Conservation of Cultural Heritage

EU COST Action IE0601 research project [2006-2011] (Polish representative: Assoc. Professor Roman Kozłowski)

Assoc Professor Roman Kozłowski of the ICSC, representing Poland in the Action's Management Committee, is member of the eight-person Steering Committee as leader of Working Group 2 "Assessment and Diagnosis". He participated in two Steering Committee meetings organized in 2009, co-edited the proceedings of the Action's conference in Braga, Portugal in 2008 and became member of the Scientific Committee of the next Action's conference organized in Hamburg in 2009. During the conference, members of staff of the institute presented 1 oral presentation and 1 poster. Mr Kozłowski organized in Krakow Action's workshop "Diagnosis and Conservation of Wooden Cultural Heritage: Necessary European Standardization within CEN Technical Committee 346 'Conservation of Cultural Heritage".

Management of the Museum Collection Based on Computer Modeling of the Impact of the Microclimatic Fluctuations on Historic Objects

(Assoc Professor Roman Kozłowski, Bartosz Rachwał, MSc, in cooperation with National Museums in Krakow and Warsaw)

The project concerns development of management strategies for the microclimatic control for museum collections consisting of wooden objects. Partners of the project – the National Museum in Krakow and the National Museum in Warsaw are particularly interested in the optimum microclimatic control in their galleries and storage rooms as they own the most important collections of medieval and modern art consisting of wooden objects in Poland. The main aim of the project is to develop management strategies for the microclimate control, which will on the one hand reduce the costs and on the other ensure the effective protection of art objects.

Fulfillment of this objective requires a scientific assessment of the impact of microclimatic parameter fluctuations on the process of destruction of historic objects. The tools for achieving this objective will be computer simulations, supported by measurements of the physical properties of materials and monitoring of the micro damage of objects caused by fluctuations of the air temperature and humidity.

The project started in December 2009 with a kick-off meeting of the project consortium. Specific tasks for the partners and the time schedule for their execution were decided.

Physics of Droplets

UE COST Action P21 research project [2006-2010] (Polish representative: Professor Kazimierz Małysa)

The COST P21 "Physics of droplets" is the project, currently joining 27 research teams from various scientific institutions of 17 European countries, and the Ian Wark Research Institute of University of South Australia, Australia. Research teams from the Institute of Catalysis and Surface Chemistry Polish Academy of Sciences (ICSC PAS0, Institute of Fundamental Technological Research Polish Academy of Sciences (IFTR PAS) and the Warsaw University of Technology, Department of Chemical and Process Engineering (WUT-CPE) are taking part in the project and Professor Kazimierz Małysa from ICSC PAS is the Polish representative in the Management Committee. Professor N.Vandewalle, GRASP, Institut de Physique B5a Sart Tilman, Université de Liège, B-4000 Liège, Belgium is the project coordinator.

Two official meetings of the Management Committee (MC) there were held in 2009. The 7th MC Meeting was held on May 5, 2009 during the Joint Meeting of the Working Groups WG1, WG2 and WG3 organized in Bucharest, Romania on May 4-6, 2009. The 8th Management Committee Meeting was held in Thessaloniki, Greece on September 23 and 25, 2009 during the 4th International Workshop "Bubble and Drop Interfaces", coupled with the joint meeting of the COST P21 Working Groups. Prof. K.Małysa attended the both MC Meetings. Prof. K.Małysa and prof. P.Warszyński attended the Joint Meeting of the WG1, WG2 and WG3 in Bucharest and presented the following oral contributions: 1) A.Niecikowska, M.Krasowska, J.Ralston, K.Małysa "Influence of Surface Charge and Hydrophobicity on Kinetics of the Three Phase Contact Formation During Bubble Collision with TiO₂ Surface", and 2) K.Szczepanowicz, D.Dronka-Góra, L.Szyk-Warszynska, P.Warszyński "Encapsulation of Emulsion Drops". COST P21 supported financially participation of Dr. M.Krzan, prof. K.Małysa and prof. P.Warszyński at the Thessaloniki Conference, where they presented two orals: 1) A.Niecikowska, J.Zawała, R.Miller, K.Małysa "Influence of the Motion Induced Adsorption Layer Architecture on Time of the Three Phase Contact formation at Mica Surface in C_nTABr Solutions", 2) W.Barzyk, T.Jasiński, E.Jarek, P.Warszyński "Surface Potential Isotherms of Nonionic and Ionic Surfactants; Experiment and Molecular Dynamic Simulations", and the poster: A.Olszewska, M.Krzan, E.Jarek, P.Warszyński, K.Małysa "Influence of pH on Adsorption and the Bubble Velocity in n-Alkanoic Acid Solutions" contributions.

Four scientists from ICSC PAS applied and obtained financing for the STSM visits in 2009. A.Niecikowska, M.Sc STSM was entitled "Influence of carbon chain length on adsorption coverage of n-alkyl-trimethylammonium bromides at liquid/gas interface and its role in time of the three phase contact formation" and carried out in Max Planck Institute of Colloids and Interfaces, Germany. Dr. J.Zawała STSM was entitled "Bouncing of droplets and bubbles from various interfaces - similarities and differences" and carried out University of Liege, Institut de Physique – GRASP, Belgium. Dr M.Krzan STSM was entitled "Foamability of protein-based surfactants" and carried out University of Liege, Institut de Physique – GRASP, Belgium. Prof. K.Małysa STSM entitled "Effect of surface charge and hydrophobicity in wetting film stability" and carried out at University of South Australia in Adelaide, Australia.

ICSC PAS was the host institution for the STSM awarded by Australian Governental Agency to M.Krasowska from the Ian Wark Research Institute of University of South Australia in Adelaide, Australia. Dr. M.Krasowska STSM was entitled "Influence of wetting film radius on rate of film drainage and time of three phase contact line formation" and was devoted to studies on influence of the size of the colliding bubble and the composition of the surrounding aqueous phase on the time of the three phase contact line formation at titania surface.

Dynamics of Interactions of the Moving Micro-objects (Particles, Droplets, Bubbles) with Interfaces

Modification of the bubble motion parameters due to adsorption of ionic, nonionic surfactants and polyelectrolyte (poly-sodium 4-styrene sulfonate - Na-PSS), as well as dynamics of the collisions and kinetics of the three phase contact (TPC) formation - gas/liquid/solid - were further studied during the reporting period. In clean water, devoid of any contaminants, the bubble interface is mobile and therefore the bubble local and terminal velocities are significantly higher than in solutions of surface active substances. Adsorption of surface active substances and establishment of the motion induced architecture of the dynamic adsorption layer (DAL) causes retardation of the bubble surface mobility (Marangoni effect), which leads to lowering the velocity of the rising bubbles. Formation of the DAL means that nonuniform distribution of surfactant is induced over the bubble surface what in the case of high surface active agents means that the top pole of the rising bubble is almost completely devoid of the adsorbed surfactant molecules. It was found in studies on influence of Na-PSS that the polyelectrolyte affected the bubble motion parameters, i.e. local and terminal velocities, dimensions and degree of the shape deformation, similarly like the typical surfactants. With increasing Na-PSS concentration the bubble terminal velocity was lowered of from 34.8±0.3 cm/s in distilled water to ca. 15 cm/s at 500ppm solution concentration. Moreover, it was found that additional purification of the electrolyte solutions using the "surface purification method" caused that higher concentration of the polyelectrolyte was needed for similar lowering the bubble velocity. After this additional purification the terminal velocity of the bubbles was lowered to 15cm/s only at the Na-PSS solutions of concentration 1000ppm. These results show that the commercial samples of polyelectrolyte contained some highly surface active contaminants and this hypothesis was confirmed by additional surface tension measurements for commercial and purified Na-PSS solutions.

Adsorption of cationic surfactants on the bubble surface can cause a reversal of the bubble electric charge from negative to positive. As a result of this charge reversal the three phase contact can be formed even on hydrophilic surface of the negatively charged mica – attractive electrostatic interaction cause destabilization and rupture of the wetting film. The TPC formation at mica surface was studied in n-dodecyl-trimethylammonium and n-cetyl-trimethylammonium bromides solutions of different concentrations. Phenomena occurring during the bubble collisions with mica plates located at different distances (3mm - location "close", and "far" - 100mm) from the point of the bubble detachment and the kinetics of TPC formation were monitored using high-speed camera (frame frequency over 1000Hz). It was shown that the three phase contact was formed in the solutions studied and the time of the TPC formation (t_{TPC}) was significantly shorter when the mica plate was at the location "close". Longer the t_{TPC} values at the mica location "far" were attributed to formation of the dynamic adsorption layer (DAL) over interface of the rising bubble. As a result of the DAL establishment the time of the TPC formation was longer at the mica location "far" because there was needed an additional time to restore such degree of cationic surfactant coverage at the bubble top pole, which caused the electric charge reversal from negative to positive. In the case of the mica location "close" the DAL has not yet been induced and the attractive electrostatic interaction between negatively charged mica and positively charged bubble surfaces caused the wetting film rupture and faster formation of the three phase contact.

EEA FINANCIAL MECHANISM PROJECTS

NOMRemove Effective Photocatalytic-Membrane Methods of Removal of Organic Contaminants for Water Treatment Removal of humic substances from water by simultaneous application of photocatysis and chemical oxidation



EEA PL0084 NOMRemove [2007-2010] (project leader: Professor Piotr Warszyński)

The aim of the project is the elaboration of the new, highly effective method of the removal of natural organic matter (NOM) from water for utilization in farming and industry. One of the main sources of NOM in natural waters is the decomposition of humic substances, widely distributed in the environment. The main object of the investigations in the year 2009 was the application of photocatalytic process in combination with the chemical oxidation for the decomposition of humic substances.

The apparatus built previously was applied for testing the activity of the catalysts, and commercial humic acid sodium salt (Aldrich) was used as a model substance. Several commercial preparations of titanium dioxide were tested, but the best results were obtained using Degussa-Evonic P-25 preparation. Two oxidizing agents were tested: hydrogen peroxide and sodium persulfate.

Titanium dioxide appeared to be a very effective catalyst for the photocatalytic decomposition of humic acid, however much better results were obtained when titanium dioxide was applied in combination with the oxidant. Much better results were obtained for sodium persulfate than for hydrogen peroxide, so most of the experiments were performed using the former compound. Synergetic effect was observed when both substances were applied together. In such a case the decomposition rate of humic acid was much higher than the sum of the decomposition rates measured when both substances (sodium persulfate and titanium dioxide) were applied separately (see Table 1 below).

The works on the improvement of the apparatus for testing the effectiveness of the photocatalysts were continued. Apparatus was further developed for the use in testing the performance of membranes and the photocatalysts deposited on the surface of membranes. Membranes obtained from the Norwegian partner of the project were successfully tested.

Table 1. Comparison of the effectiveness of the processes of photochemical decomposition and the decomposition by chemical oxidation of humic acid sodium salt (HANa) with the effectiveness of combined process

with the check veness of combined process.								
Composition of investigated solution	50 mg/l HANa + $1 \text{x} 10^{-3} \text{ mol/l Na}_2 \text{S}_2 \text{O}_8$		50 mg/l HANa + 100 mg/l TiO ₂		$50 \text{ mg/l HANa} + 1x10^{-3} \text{ mol/l Na}_2\text{S}_2\text{O}_8 + 100 \text{ mg/l TiO}_2$			
Type of radiation	VIS	UV	VIS	UV	VIS	UV		
Conversion degree (%)	2.6	18.5	1.5	36.1	34.2	91.1		

Establishing Standards for Allowable Microclimatic Variations for Polychrome Wood



EEA PL0086 [2007-2010]

(project leader: Assoc. Professor Roman Kozłowski)

Panel paintings, polychrome wooden sculpture and paintings decorating wooden architectural surfaces are an important part of the artistic heritage. The protection of this heritage is particularly important for Poland and Norway in which a group of polychrome wooden churches, unique on a world scale, has been preserved. The most valuable of them are inscribed on the UNESCO World Heritage List.

Uncontrolled variations of the air parameters in the environment of polychrome surfaces, leading to the deformation and mechanical damage of wood, gesso and paint layers, are principal hazards to their preservation. The main aim of the project is establishing precisely and quantitatively the variations of air parameters that are safe for the painted wooden surfaces. The main research tool is direct monitoring of the climate-induced delamination of the decorative layers from the wooden support and their mechanical damage using optical methods, acoustic emission and computer modeling.

During the third year of the project, the major emphasis was put on the measurements in historic buildings. Analysis of the preservation state of polychromy on the wooden altarpiece in the Hedalen Church in Norway was performed. The measuring campaign aimed at the detection of environmentally-induced damage progress for representative parts of the altarpiece. The measurements followed previous investigations performed in autumn 2008 – before the heating season.

Two types of analysis were carried out:

- 1. monitoring of the acoustic emission signal from a wing of the altarpiece as well as from the central sculpture of Christ. A correlation between the acoustic emission level and the microclimatic fluctuations in the church was analyzed. The results were compared with a condition survey made by Norwegian conservators
- 2. analysis of the preservation state of the polychromy on the same altarpiece wing before and after the heating season. The measurements were performed using a speckle interferometer. The interpretation of results was made, as for the acoustic emission, using the outcome of the condition survey by the conservators.

The obtained results allowed assessing the influence of a heating system in the church on the progress of damage of the polychrome wood in the church.

A computer model to simulate the response of a multi-layered structure representing painted wood to changes in temperature and humidity was developed. Modules for calculation of heat and moisture diffusion and for calculation of stress and deformation were merged into one computer program. It allowed simulating stress- and deformation-fields for the panel painting for various, time dependant, variations of temperature and humidity.

PROJECTS OF SECTORIAL OPERATIONAL PROGRAMMES OF EU STRUCTURAL FUNDS

MPD Krakow Interdisciplinary PhD Projects in Nanoscience and Advanced Nanostructures

PO IG 1.1.2. MPD [2008-2012] (project leader: Professor Józef Korecki)

Project "Krakow Interdisciplinary PhD Projects in Nanoscience and Advanced Nanostructures" is implemented within the Innovative Economy Operational Programme, Priority 1. Research and development of new technologies, Measure 1.2 - Strengthening the human potential of science, International PhD Projects Programme. The Project Coordinator is Dr. B.Szafran affiliated to AGH University of Science and Technology, a member of this Project. The project is also carried out in cooperation with the following foreign partners: Universieit Antwerpen (Belgium), University of Leeds (The United Kingdom), National Research Centre INFM-CNR (Włochy), University of Stockholm (Sweden), Max Plank Institute für Molekulare Pflanzenphysiologie (Germany), Max Plank Instytut für Kolloid und Grenzflächenforschung (Germany), Basque Country University (Spain), SINTEF Materia land Chemistry (Norway), VŠB-Technical University of Ostrawa, (Czechy), Institute of Physical Chemistry, Bulgarian Academy of Sciences (Bulgaria).

Project started on November 1st, 2008 and aims to promote 12 PhD thesis within 12 individually defined PhD projects. Recruitment procedure of the PhD students is in the form of the open call. In 2009 the recruitment procedure was completed and since that time in the ICSC PAS the following 5 individual PhD project are being implemented:

- 1. A.Wójcik, MSc, supervisors: Professor E.Brocławik and Assoc. Professor T.Borowski, project title: "Computer Modeling of Biological Nanostructures"
- 2. M.Adamczak, MSc, supervisor: Professor P.Warszyński, project title: "Surfactants, Polyelectrolytes and Nanoparticles as Building Blocks for Surface Nanostructures"
- 3. D.Kosior, MSc, supervisor: Professor K.Małysa, project title: "Nanostructures and Stability of Thin Liquid Layers"
- 4. K.Luberda-Durnaś, MSc, supervisor: Professor W.Łasocha, project title: "Hybrid Organic-Inorganic Layered Materials - Precursors of Semiconducting Nanostructures"
- 5. E.Młyńczak, MSc, supervisor: Professor J.Korecki, project title: "Surface and Interface Properties of Metal-Oxide Magnetic Nanostructures"

ISD Advanced Materials for the New Technologies and Energy of the Future

PO KL 4.1.1 ISD [2008-2012] (project leader: Professor Józef Korecki)

The Interdisciplinary PhD Project entitled "Advanced Materials for the New Technologies and Energy of the Future" is implemented within the Human Capital Operative Programme funded form the European Union Structural Funds and the national budget sources. The project is coordinated by the Faculty of Physics & Applied Computer Science AGH University of Science and Technology and carried out in the partnership with the Institute of Catalysis and Surface Chemistry, Polish Academy of Sciences and the Institute of the Nuclear Physics of the Polish Academy of Sciences.

The aim of the Project is to educate high level specialist with the PhD diploma in the disciplines considered as strategic in the European and national research policy, i.e. Info, Bio, Nano, Techno. Research and education within the project involve physical, chemical and technological aspects of material science and modern energy science.

Implementation of the Project will be based on the 3 recruitment editions, starting form 2009. In each recruitment edition about 20 students will be recruited for the 4 years PhD studies.

The first recruitment was launched in 2009 and as a result the following individual PhD projects are being implemented in the ICSC PAS:

- 1. A.Lewkowicz, MSc, supervisor: Professor M.Witko, project title: "Modeling of the Physicochemical Properties of the Heteropoliacids – Catalysts for the Biodiesel Synthesis"
- 2. M.Pancerz MSc, supervisor: Assoc. Professor P.Weroński, project title: "Theoretical and Experimental Analysis of the Porosity of the Multilayered Colloidal Materials of the Controlled Architecture"
- 3. A.Pajor, MSc, supervisor: Professor P.Warszyński, project title: "Modification of the Surfaces of Materials with the Use of Polymer Layers Containing Nanoparticles"
- 4. M.Krzak, MSc, supervisor: Assoc. Professor P.Nowak, project title: "Composite Alloys and Materials based on Transition Metals with the Low Reaction Overpotential of the Emission/Ionization of Hydrogen
- 5. M.Oćwieja, MSc, supervisor: Professor Z.Adamczyk, project title: "New Layered Materials of Controlled Architecture and Functionality Containing Silver Nanoparticles – Biomedical Applications"

SPINLAB National Centre of Nanostructures for Spintronic Applications

PO IG 2.2. SPINLAB [2009-2011] (projct leader: Professor Józef Korecki))

This project coordinated by the Institute of Molecular Physics PAS has been accepted for funding (the contract with Ministry of Science and Higher Education was signed in November 2009), and the ICSC PAS tasks, including purchase and implementation of PEEM-LEEM microscope in laboratory and synchrotron environment, commence in January 2010.

OTHER INTERNATIONAL PROJECTS

Enzymes as Nanotools - Development of a New, Enzyme-Based Technology for Engineering Selectivity Permeable, Nano-Structured Membranes

GRDE France [2007-2010]

(project leader: Professor Piotr Warszyński)

Lipases and phospholipases play a key role in living organisms and biotechnological processes, therefore, their properties are widely investigated. However, some of their properties have not yet been fully understood due to difficulties in analyzing of complex processes occurring in these multicomponent and multiphase systems. One has to use various experimental characterization techniques to achieve reliable description of these processes. The aim of the project is to acquire new results and knowledge, which allow application of enzymes as "nanotools" for obtaining new materials with nanostructured surfaces for biomedical and/or sensor applications.

In 2009 our research concentrated on determination of the stability of formation of phospholipid films at various surfaces. Changes of properties films deposited at various surfaces (mica, silicon wafers, glassy carbon) either by solvent evaporation or Langmuir-Blodgett method, were monitored by contact angle, ellipsometry, streaming potential and three phase contact formation.

We found that the thickness of DLPC layer deposited on silicon wafer by LB method was 1 nm, while for DPPC 3 nm. They were unstable and the decrease of thickness and value of the contact angle was observed with time. On the other hand the same phospholipid layers deposited by solvent evaporation were stable. The same trend was observed at glass surfaces. For example the dynamic contact angle of the DPPE layer deposited by LB technique, decreased from c.a. 100 degrees to 40 degrees after 20 minutes immersion in water. Moreover, the layer was destroyed during three phase contact formation measurements. The layers formed by solvent evaporation were much more stable. The observed decrease of the contact angle did not exceed 20 degree and the three phase contact was always formed. At the mica surface the films formed by both methods were stable were stable, which was indicated by the kinetics of the three phase contact formation.

IUP Casein Polyelectrolyte Multilayer Films

ECO-NET France IUP [2007-2008] (project leader: Professor Piotr Warszyński)

The aim of this project is to establish basis mechnisms and parameters controlling buildup of the polyelectrolyte multilayer films with casein embedded. Such films could create the skeleton and matrix for immobilization proteins, which do not change their properties and intrinsic structure, assuring correct function of proteins and protect them from loosing biological activity.

Fig. 1 shows the example of the results of ellipsometric measurments of thickness of (PLL/CAS)n films exposed to the solution of calcium ions. One can see that together with the increasing the number of adsorbed layers the thickness of the multilayer PE film increases. Growth of the film thickness after contacting it with the calcium ions indicate their binding by casein embedded in the multilayer film.

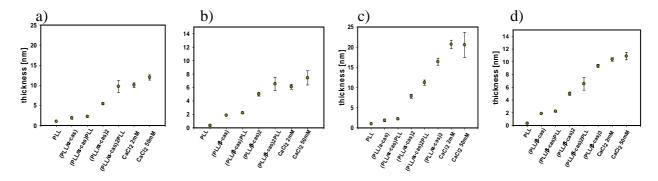


Fig. 1. Ellipsometric thickness of the (PLL/CAS)n multilayer films treated with calcium ions.

XPS measurement allowed to determine presence and quantity of calcium ions in investigated (PLL/CAS)n and (PLL/CAS)nPLL multilayer films. Data obtained by XPS were in accordance with ellipsometric measurments and showed the bigger concentration of calcium ions in the films build up with α -casein.

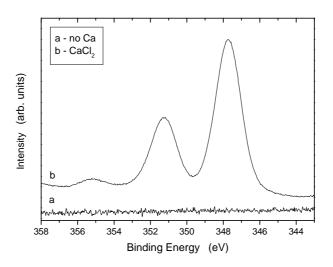


Fig.2. XPS spectrum for silicon plate covered with multilayer films containing casein treated and untreated with calcium ions CaCl₂.

Nanostructural Micro/Mesoporous Materials as New Catalysts for the Production of Environmentally Friendly Diesel Fuel

Intergovernmental Polish-Spanish project [2007-2009] (project leader: Assoc. Professor M. Derewiński)

Liquid fuels obtained from natural gas through the well-known Fischer-Tropsch synthesis (FTS) offer significant advantages over those derived from crude oil. In particular, FTS diesel fuels have high cetane values and are almost free of contaminants such as sulfur, nitrogen, and (poly) aromatics. Catalysts used for hydrocracking waxes (which are an important fraction of the FTS products) typically comprise a noble metal (Pt, Pd) supported on microporous molecular sieves (zeolites) or amorphous silica-alumina (ASA) as the acidic components. Catalysts based on zeolites are much more active but less selective towards diesel than those based on ASA owing to the presence of stronger acid sites and to the microporous environment favoring re-cracking of diesel-range molecules into lighter products (gasoline and gases). However, the presence of micropores may in some cases limit their use, because of restriction imposed on the transport of reacting molecules inside the zeolite crystals.

We proposed to use new nanomestructured materials with combined micro and meso/macroporosity and controlled pore size distribution as the acidic component of bifunctional hydrocracking catalysts. These materials may overcome the diffusion limitations of catalysts based on zeolites for hydrocracking of Fischer-Tropsch waxes.

Preparation of nanostructured materials (hierarchically micro-meso-macro) with combined micro and mesoporosity, controlled pore size distribution, strong acidic properties and high thermal stability was carried out by using different approaches:

- 1. Simultaneous or sequential synthesis of zeolite/meso/macroporous composites. The new nanomaterials were obtained by the controlled transformation of aluminosilicates with irregular meso/macropores into composite materials which retain the original porosity, while at least part of starting material was converted into nanocrystalline zeolites. Prepared Pt-modified BEA composites were tested in the hydrocracking of n-C₃₆. Higher activity (comparing to the Pt-modified Al-MCM-41 used as a reference catalyst) as well as shift of the selectivity towards lighter fraction observed for the composite based bifunctional catalysts, indicated a deeper cracking, caused by the presence of strong Brønsted sites in the zeolitic domains generated during the formation of composite material.
- 2. Growing zeolite crystallites on carbon or polymer nanoparticles, followed by the removal of the carbon or polymer "template" by controlled calcinations. Carbon nanoparticles used were commercial Black Pearls 2000 and polydispersed suspensions of active carbon.

Supporting Pd (2 wt%) on a hierarchical carbon-templated MFI zeolite allowed to improve the metal dispersion as compared to a conventional MFI sample with the same Si/Al ratio and having equivalent microporosity and acidity. The bifunctional catalysts based on the hierarchical and the conventional zeolites were shown to be well balanced in terms of metal/acid ratio, despite the better metal dispersion in the former, resulting in almost the same activity and selectivity during the hydroconversion of *n*-octane (used as a model bifunctional reaction) in the absence of sulfur. However, in the presence of sulfur accompanying the *n*-C₈ feed the carbon templated catalysts displayed a superior sulfur resistance with respect to that of the reference catalyst. TEM characterization of spent samples after the sulfur-containing experiments revealed that this effect is likely due to a limited sintering of the metal nanoparticles when confined within the intracrystalline mesovoids of the hierarchical zeolite, leaving more metal sites unpoisoned that helped in keeping more acid sites active and preventing extensive coking under the sulfur conditions.

Adsorbates on Fe Monolayer on W (110)

Intergovernmental Polish-Austrian project [2009-2010] (project leader: Professor Józef Korecki)

Oxygen adsorption on Fe monolayer on W(110)

Electric quadrupole interactions in an iron monolayer on W(110) upon oxygen adsorption have been studied by means of nuclear resonant scattering (NRS) of synchrotron radiation in grazing incidence geometry. Our earlier studies showed extreme reactivity of the Fe monolayer on W(110) in adsorption of residual gases at ultrahigh vacuum conditions. In the present contribution the adsorption of oxygen and its influence on electric quadrupole interactions is of main interest. On the atomically clean W(110) surface, one pseudomorphic iron monolayer was deposited at 450°C. The prepared 1 ML film displaying a (1×1) LEED pattern was exposed at RT to a partial pressure of 1×10^{-9} and 5×10^{-9} torr 5.0N purity O₂ for exposure of 0.05, 0.1, 0.2L and 1L, respectively. Already at exposure of 0.1L a very weak $p(2 \times 2)$ LEED pattern appeared and transferred to $p(3 \times 2)$ pattern with increasing oxygen coverage. After each exposure to oxygen, an NRS time spectrum has been registered. The NRS spectrum for a pure Fe monolayer shows a simple exponential decay indicating that the main axis of the electric field gradient is perpendicular to the wave vector of incoming radiation and its polarization, i.e perpendicular to the surface. Adsorption of oxygen atoms causes an appearance of a beat pattern that becomes more pronounced as the oxygen coverage increases. A tentative numerical analysis with the CONUSS software suggests a strong influence of oxygen atoms on the direction of a main EFG axis, whereas the quadrupole splitting does not vary significantly with increasing oxygen coverage.

SCIENTIFIC OUTPUT OF THE INSTITUTE IN 2009

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- M.Zimowska "Otrzymywanie mezoporowatych struktur ypu SBA-15 o hierarchicznym systemie porów przy użyciu nanocząstek węgla jako szablonów strukturalnych", Materiały.
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- 141. M.Zimowska, B.D.Napruszewska, R.Dula, A.Michalik-Zym, E.M.Serwicka "Badanie rekonstrukcji struktury hydrotalkitów cynkowo-glinowych", 41. Ogólnopolskie Kolokwium Katalityczne, IKiFP PAN, Kraków 2009
- 142. M.Zimowska, J.Połtowicz, Z.Olejniczak "Synteza, właściwości strukturalne i katalityczne aluminowanych mezoporowatych sit molekularnych typu SBA-15 o hierarchicznym systemie porów", 41. Ogólnopolskie Kolokwium Katalityczne, IKiFP PAN, Kraków 2009
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LECTURES IN SCIENTIFIC INSTITUTIONS AND SOCIETIES

- 1. T.Borowski "Preliminary Results for SyrB2 Halogenase", Stockholm University, Stockholm 2009
- 2. M.Derewiński "Materiały hierarchiczne na bazie sit molekularnych", Instytut Inżynierii Chemicznej PAN, Gliwice 2009
- 3. A.Drelinkiewicz, <u>R.Kosydar</u> "Hydrogenation of Amylanthraquinone in the Presence of Palladium Catalysts; Synthesis of Catalysts for Direct Synthesis of H₂O₂", Solvay Research and Technology Centre, Brussels 2009
- 4. J.Haber "Kataliza Alchemia XXI wieku", wykład im. prof. A.Basińskiego, Wydział Chemii UMK, Toruń 2009
- 5. R.Kozłowski "Cement romański w architekturze XIX wieku i dziś", Wydział Chemii Uniwersytetu Warszawskiego, 2009
- 6. W.Łasocha, A.Rafalska-Łasocha "Zastosowania strukturalne dyfraktometrii proszkowej", Instytut Fizyki PAN, Warszawa 2009
- 7. M.Łukomski "Analiza wpływu warunków mikroklimatycznych na materiały higroskopijne" Muzeum Narodowe, Kraków 2009
- 8. K.Małysa "Velocity of the Rising Bubbles as a Simple Method for Monitoring Organic Contaminants in Water Reservoirs", Ian Wark Research Institute, University of South Australia, Adelaide 2009
- 9. A.Pacuła_"N-Doped Carbon Materials Pepared via Chemical Vapour Deposition", Division of Chemistry, Graduate School of Science, Hokkaido University, 2009

- 11. P.Weroński "Modeling of Multilayer Random Sequential Adsorption of Charged Nanospheres", University of Alberta, Edmonton 2009
- 12. P.Weroński "The First Law of Thermodynamics", University of Alberta, Edmonton, 2009
- 13. M.Wierzchoń, <u>J.Barbasz</u> "Analizy kognitywistyczne na przykładzie badań uczenia mimowolnego", Wydział Fizki, Astronomii i Informatyki Stosowanej UJ, Kraków 2009
- 14. M.Witko "Molekularny świat katalizatora", Wydział Chemiczny Politechniki Gdańskiej, 2009

LECTURES FOR STUDENTS AN PhD STUDENTS

- 1. T.Borowski, "Chemia kwantowa makrocząsteczek" zajęcia pracowni komputerowej, Wydział Chemii UJ (30 godz.)
- 2. Ł.Bratasz "Konserwacja, kształtowanie architektury i aranżacja wnętrz obiektów sakralnych" wykłady w ramach studium podyplomowego Politechniki Krakowskiej (4 godz.)
- 3. E.Brocławik "Struktura, spektroskopia i oddziaływania" wykłady w ramach Międzynarodowego Studium Doktoranckiego IKiFP PAN (14 godz.)
- 4. E.Brocławik, koordynacja wykładu z chemii fizycznej
- 5. E.Brocławik "Modelowanie Molekularne Materiałów", wykład monograficzny dla panelu 'nowe materiały i kataliza', na zaproszenie Dziekana Wydziału Chemii UJ (45 godz.)
- E.Brocławik, "Chemia Kwantowa Makrocząsteczek", wykład kursowy dla kierunku 'biofizyka', na zaproszenie Dziekana Wydziału Biochemii, Biofizyki i Biotechnologii UJ (30 godz.)
- 7. E.Brocławik, "Stosowana Chemia Kwantowa", wykład monograficzny dla doktorantów Środowiskowego Studium Doktoranckiego przy Wydziale Chemii UJ, na zaproszenie Kierownika Studium (30 godz.)
- 8. M.Derewiński "Wstęp do spektroskopii w podczerwieni i Ramana", wykłady w ramach Międzynarodowego Studium Doktoranckiego IKiFP PAN (4 godz.)
- 9. A.Drelinkiewicz "Chemia fizyczna, termodynamika", wykłady dla doktorantów Międzynarodowego Studium Doktoranckiego IKiFP PAN (20 godz.)
- 10. R.Grabowski "Zjawisko adsorpcji i jego zastosowanie w badaniach", wykłady dla doktorantów Międzynarodowego Studium Doktoranckiego IKiFP PAN (2 godz.)
- 11. J.Gurgul "Electron spectroscopy for chemical analysis (ESCE)", wykład dla studentów i doktorantów, Wydział Fizyki i Informatyki Stosowanej AGH (2 godz.)
- 12. B.Jachimska "Metoda Dynamicznego Rozpraszania Światła (DLS) pomiary wielkości cząstek koloidalnych", wykład w ramach przedmiotu specjalistycznego Metody Analizy Instrumentalnej, 4. rok, Wydział Inżynierii Materiałowej i Ceramiki AGH (2 godz.

- 13. B.Jachimska "Laser Doppler Velocimetry Pomiary Ruchliwości Elektroforetycznej cząstek koloidalnych", wykład w ramach przedmiotu specjalistycznego Metody Analizy Instrumentalnej, 4. rok, Wydział Inżynierii Materiałowej i Ceramiki AGH (2 godz.)
- 14. J.Korecki, pensum dydaktyczne AGH (pełny etat)
- 15. J.Korecki, "Metody badań katalizatorów spektoskpia Mössbauera" wykłady dla doktorantów Międzynarodowego Studium Doktoranckiego IKiFP PAN
- 16. R.Kozłowski "Nowoczesne techniki analityczne dla konserwacji obiektów zabytkowych", wykłady w ramach Studium Podyplomowego Wydziału Chemii UJ (4 godz.)
- 17. R.Kozłowski "Vulnerability of Cultural Heritage to Climate Change", wykład i ćwiczenia w ramach kursu European Youth Center, Council of Europe, Strasbourg 2009 (8 godz.)
- 18. W.Łasocha "Chemia fizyczna rentgenowska analiza strukturalna", wykłady w ramach Międzynarodowego Studium Doktoranckiego IKiFP PAN (4 godz.)
- 19. W.Łasocha "Metody badań kataliz\atorów rentgenowska analiza strukturalna", wykłady w ramach Międzynarodowego Studium Doktoranckiego IKiFP PAN (4 godz.)
- 20. P.Nowak "Chemia fizyczna elektrochemia", wykłady dla doktorantów Międzynarodowego Studium Doktoranckiego IKiFP PAN (4 godz.)
- 21. P.Nowak "Metody badań katalizatorów elektrochemia", wykłady dla doktorantów Międzynarodowego Studium Doktoranckiego IKiFP PAN (4 godz.)
- 22. E.M.Serwicka-Bahranowska "Zastosowanie EPR do badania minerałów ilastych", wykłady dla doktorantów Międzynarodowego Studium Doktoranckiego IKiFP PAN (4 godz.)
- 23. M.Szaleniec "Analiza tłuszczów i lipidów", wykłady w ramach Studiów podyplomowych z Biologii Molekularnej z Elementami Biotechnologii, Uniwersytet Pedagogiczny w Krakowie (3 godz.)
- 24. M.Szaleniec "Analiza tłuszczów i lipidów", ćwiczenia laboratoryjne w ramach Studiów podyplomowych z Biologii Molekularnej z Elementami Biotechnologii, Uniwersytet Pedagogiczny w Krakowie (20 godz.)
- 25. P.Warszyński "Chemia Fizyczna", wykłady dla doktorantów Międzynarodowego Studium Doktoranckiego IKiFP PAN (14 godz.)

SCIENTIFIC SEMINAS OF THE INSTITUTE

LECTURES OF INVITED GUESTS

- 1. L.Berreau (Department of Chemistry and Biochemistry, Utah State University)"Synthetic and Mechanistic Investigations of CO-releasing Divalent Nickel Acireductone Complexes"
- 2. W.Duch (Wydział Fizyki, Astronomii i Informatyki Stosowanej UMK) "Czy jesteśmy automatami? Wolna wola, podmiotowość i mózg" [wykład Wielkanocny]

- 3. S.Kędracka-Krok (Wydział Biochemii, Biofizyki i Biotechnologii UJ) "Fluorescencyjne metody badania białek. Część I"
- 4. S.Kędracka-Krok (Wydział Biochemii, Biofizyki i Biotechnologii UJ) "Fluorescencyjne metody badania białek. Część II"
- 5. T.Kornaś (Wydział Polonistyki UJ) "Teatr wobec rytuału i liturgii" [wykład Bożonarodzeniowy]
- 6. M.Kosmulski "(Wydział Elektrotechniki i Informatyki PL) "Ładunki powierzchniowe na granicy faz tlenek metalu roztwór elektrolitu i punkty zerowego ładunku"
- 7. K.Sadlej (Instytut Podstawowych Problemów Techniki PAN) "Modelowanie współczynników oporu oraz promieni hydrodynamicznych podstawowych białek"

LECTURES OF INSTITUTE STAFF

- 1. J.Barbasz "Świat w nanoskali"
- 2. J.Barbasz "Kognitywistyka zrozumieć siebie"
- 3. A.Bielański "Kilka kartek z historii chemii prorocy i szczęściarze"
- 4. T.Borowski "Odrobina chemii w filiżance"
- 5. M.Czerwenka "Finansowanie nauki (Instytutu) ze środków budżetowych krótki kurs"
- 6. M.Czerwenka "Odpowiedzialność prawna naukowców za upowszechnianie materialnych i niematerialnych wyników badań"
- 7. A.Drelinkiewicz "Konwersja biomasy. Część II: paliwa, chemikalia"
- 8. R.Gryboś "Kataliza w Kosmosie"
- 9. R.Gryboś "Selektywna redukcja tlenku azotu metanem na palladzie osadzonym w mordenicie. Obliczenia kwantowo-chemiczne"
- 10. E.Jarek "Mikrokapsułki jakonowoczesne nośniki leków"
- 11. J.Korecki "Małe jest inne: Nanocząsteczki złota i ich niezwykłe właściwości"
- 12. R.Kozłowski "Zabytki z laki japońskiej mechanizm niszczenia i strategia ochrony"
- 13. M.Krzan "Rheology and Stability of Particle Laden Aqueous Foams
- 14. A.Michna"Otrzymywanie i charakterystyka uporządkowanych struktur tworzonych przez nanocząstki koloidalne"
- 15. D.Rutkowska-Żbik "Samoorganizacja, czyli 'czy bałagan może się sam posprzątać?"

16. M.Szaleniec "Zrozumieć i stworzyć - jak projektujemy leki"

LECTURES AT "OPEN DOORS DAY" OF THE INSTITUTE

- 1. J.Barbasz "Kognitywistyka zrozumieć siebie"
- 2. T.Borowski "Odrobina chemii w filiżance"
- 3. R.Gryboś "Paliwa rakietowe"
- 4. E.Jarek "Mikrokapsułki jakonowoczesne nośniki leków"
- 5. M.Łukomski "Obrazy mistrzów w zbliżeniu"
- 6. D.Rutkowska-Żbik "Samoorganizacja, czyli 'czy bałagan może się sam posprzątać?"
- 7. M.Szaleniec "Sekrety laboratorium chemicznego"

POPULARIZED SCENTFIC LECTURES

- 1. A.Bielański "Kilka kartek z historii chemii: prorocy i szczęściarze", Zjazd Absolwentów Chemii UJ, Kraków 2009
- 2. T.Borowski "Odrobina chemii w filiżance", Beskidzkie Centrum Onkologii, Bielsko-Biała 2009
- 3. R.Gryboś, wywiad radiowy z okazji przyznania nagrody im. W.Kołosa
- 4. B.Jachimska "Natura inspiruje", Szkoła Podstawowa Nr 41 im. Jana Kochanowskiego, Kraków 2009
- 5. R.Kozłowski "Cement romański tajemnica krakowskich kamienic", Uniwersytet Otwarty AGH, Kraków 2009
- 6. M.Łukomski, udział w programie telewizyjnym TVP W.Nidzickiego 'Laboratorium XXI wieku',
- 7. M.Witko "Ekspedycja w świat mikro katalizatora", Uniwersytet Otwarty, AGH, Kraków 2009

SCIENTIFIC DEGREES AWARDS

DSc IN CHEMISTRY

1. T.Borowski "Mechanizmy reakcji na mononuklearnych cntrach żelazowych i manganowych w wybranych niehemowych enzymach aktywujących tlen - badania teoretyczne"

PhD IN CHMISTRY

1. A.Bratek-Skicki "Influence of Adsorbed Polyelectrolytes on the inetics of Colloid Particle Deposition and the Topology of their Monolayers" (promotor Z.Adamczk)

- 2. A.Burkat-Dulak "Nowe metody syntezy kryształów zeolitów typu MFI, FAU i BEA z multimodalnym systemem porów i ich wykorzystanie jako nowe nanomateriały dla procesów katalitycznych" (promotor M.Derewiński)
- 3. Ł.Mokrzycki "Transformation of α-Pinene over Modified ZSM-5, ZSM-12, and MCM-22 Type Zeolites" (promotor: B.Sulikowski)
- 4. V.Pashkova "Synthesis and Characterization of Composite Multimodal Materials with Enhanced Acidic Properties Prepared by the Recrystallization Method" (promotor M.Derewiński)
- 5. W.Płaziński "Teoretyczny opis kinetyki adsorpcji na granicy faz roztwór/ciało stałe: zastosowanie statystycznej teorii transportu międzyfazowego (na UMCS w Lublinie, promotor W.Rudziński)

CONFERENCES AND SCIENTIFIC EVENTS ORGANIZED IN THE INITUTE

- 41. Ogólnopolskie Kolokwium Katalityczne, Kraków, 30 marca-1 kwietnia 2009 (B.Sulikowski)
- 2. Workshop COST Action IE0601 on Diagnosis and Conservation of Wooden Cultural Heritage: Necessary European Standardisation within CEN Technical Committee 346 'Conservation of Cultural Heritage', Krakow, April 2nd-3rd, 2009 (R.Kozłowski)
- 3. Festiwal Nauki, Rynek Główny w Krakowie, 14-17 maja 2008 (P.Warszyński, M.Barańska)
- 4. 2nd International Symposium on Surface Imaging/Spectroscopy at Solid/Liquid Interface, ISSIS 2009, Krakow, May 31st-June 3rd, 2009 (P.Nowak)
- 7th International Symposium on Surface Heterogeneity Effects in Adsorption and Catalysis on Solids ISSHAC-7, Kazimierz Dolny, July 5th-11th, 2009 (wspólnie z Uniwersytetem Marii Curie-Skłodowskiej) (W.Rudziński)
- 6. 2nd International Environmental Best Practices Conference and AGFES Educational Workshop, Krakow, September 14th-18th, 2009, [wspólnie z Uniwersytetem Jagiellońskim i Uniwersytetem Warmińsko-Mazurskim] (M.Nattich, D.Dronka-Góra)
- 7. Dzień Otwarty IKiFP PAN, Kraków, 20 października 2009 (M.Derewiński)