



**BORESKOV INSTITUTE OF CATALYSIS**

**SIBERIAN BRANCH OF THE RUSSIAN ACADEMY OF SCIENCES**

**ANNUAL REVIEW 2011**



<http://catalysis.ru>



# ANNUAL REVIEW

## of Activities in Basic Research Areas

### 2010



**BORESKOV INSTITUTE OF CATALYSIS**  
**SIBERIAN BRANCH OF THE RUSSIAN ACADEMY OF SCIENCES**

**Novosibirsk**

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Dear Colleagues,

This issue of the Annual Review of the Boreskov Institute of Catalysis in the main fields of its academic and R&D activities covers the year 2010 and follows the scheme which has been accepted for such kinds of reviews many years ago.

The Boreskov Institute of Catalysis, or BIC, is well known to experts in both academic and industrial catalysis not only in Russia and the fSU countries, but also in many western and oriental countries. It is the world oldest and world largest institution which was organized to provide any kind of R&D in the area of catalysis. Since 1960's it remains the largest Russian chemical research institution in the Asian part of Russia and had many deep and stable traditions in providing basic and applied research in nearly all fields of catalysis. The interests of BIC spread from the sophisticated areas of purely academic research to the real applied problems, including development of commercial catalysts and catalytic technologies.



The current features of BIC are reflecting indeed Russia entering into the market economy. BIC is affiliated to the Siberian Branch of the Russian Academy of Sciences. However, according to realities of new Russia, a lot deal of BIC's activity is devoted not only to basic research, but also to numerous industrially oriented topics. The intensity and quality of the research and engineering activity at the Institute became at the new period of the Russian history even higher as before. According to the Russian and international data bases of citation, for several years BIC has been positioned as a leading Russian chemical institution from the point of view. Fortunately, the Institute gained in research in catalysis fundamentals, thus continuing the old traditions originated from the beginning of the Institute due to a very powerful starting impulse of its creator and first director Academician Georgii K. Boreskov, as well as the master ideas of the second director Academician Kirill I. Zamaraev. Hope, the readers will feel that themselves, when comparing the materials of this and previous issues of the Annual Review, which can be easily found at the BIC website.

The BIC has a strategic collaboration with some industrial companies, both Russian and foreign, which is nowadays, undoubtedly, the very important feature of the current scientific activity of the Boreskov Institute.

Since 1998 the Institute has been united with few other R&D chemical institutions of the Siberian Branch of the Russian Academy of Sciences in the frame of a now informal association which was called the United Institute of Catalysis (UIC). In 2004 there were some

rearrangements in the UIC structure, since the Omsk Division of the BIC has separated from BIC as a new legal entity, the Institute of Hydrocarbons Processing (IHP), and the Volgograd Scientific center with large flexible pilot facilities for fine organic synthesis has joined BIC as its Volgograd Division. Thus, now UIC consists of two large legal entities: BIC and IHP. In addition to the Volgograd pilot plant, UIC's facilities include two extra flexible and powerful semi-industrial plants capable of manufacturing the first industrial batches of various catalysts and specialty chemicals. Thus, BIC together with UIC is now one of the most powerful Russian specialized R&D organizations in the industry-oriented chemistry with unique facilities for scaling up numerous innovations for that industry. Indeed, the current management of the Institute realizes the existing problems of the large R&D structure operation and tries to take care of the maintenance of high R&D activity and productivity of BIC and UIC in both fundamental and applied areas as the goal of the highest priority.

Hope, the current issue of the Annual Review of the Boreskov Institute of Catalysis will succeed in exhibiting many sides of the Institute potentialities in basic research on catalysis, catalysts and catalytic technologies. As to the activity of the Institute in the applied areas, which is now much more active than ever in the past, we publish special leaflets. Also, we invite the readers to visit BIC's website at **[www.catalysis.ru](http://www.catalysis.ru)**.



Valentin N. Parmon

## **BORESKOV INSTITUTE OF CATALYSIS OF THE SIBERIAN BRANCH OF THE RUSSIAN ACADEMY OF SCIENCES**

*More than the 50-year-long history of the Institute is a glowing example of fruitful interaction between fundamental science and industry and of the profound realization that solving scientific problems should be aimed at some practical result. The accomplishments of the Institute embody its original conception, which includes studies at the atomic/molecular level and commercialization of the results. It is due to this approach that the Institute has made a significant contribution to the material and technical basis necessary for the rapid development of the Russian chemical industry and became one of the world's most prominent research centers in chemical catalysis.*

The Institute was founded in the summer of 1958 as a part of Siberian Branch of the USSR Academy of Sciences. Initiator of the Institute and its permanent director up to 1984 was Academician Georgii K. Boreskov (1907-1984), an eminent scientist with international recognition, an outstanding physical chemist and chemical technologist. In 1992 the Institute of Catalysis was named after Academician G.K. Boreskov.

In 1969 the Institute of Catalysis was awarded The Red Banner of labor for the contribution to development of chemical science and for training high-skilled personnel.

In 1976 Professor Kirill I. Zamaraev has been invited by Academician G.K. Boreskov to join the Institute of Catalysis as a Deputy Director. Kirill Ilyich has come to Novosibirsk at early 1977 together with a large team of young chemical physicists, thus largely widening the studies of elementary catalytic reactions on molecular-atomic level through application of modern physical and kinetic instrumental methods of investigation.

In 1984 Professor Zamaraev became the Director of the Institute of Catalysis, and since that time he was the real leader of all research and development activity of this huge Institute. K.I. Zamaraev, the former President of the International Union of Pure and Applied Chemistry, Foreign Fellow of the Indian National Science Academy and of the Korean Academy of Science and Technology, holder of the Silver Medal of the Royal Chemical Society of Great Britain as a centenary lecture, has passed away in the prime of his brilliant scientific career on 26 June 1996.

In 1995 Professor Valentin N. Parmon, prominent specialist in the field of catalysis, chemical kinetics and photocatalysis, headed the third generation of the directorate of the Institute.

Since 1998 the Institute has been united with some other R&D chemical institutions of the Siberian Branch of the Russian Academy of Sciences in the frame of a new R&D association: the United Institute of Catalysis headed by the director of the Boreskov Institute of Catalysis. Now the United Institute of Catalysis (UIC) affiliates the Boreskov Institute of Catalysis with its St. Petersburg and Volgograd Departments, the Institute of Hydrocarbon Processing in Omsk and three pilot plants.

The scope of the Institute's activities is extremely wide, ranging from solving fundamental problems of catalysis to creating new catalytic technologies and organizing commercial scale production of catalysts.

The high level of the fundamental research carried out by the Institute is reflected by numerous monographs, as well as articles published in prestigious international journals and collections of works. The Institute publishes more than 300 works per year in peer-reviewed journals. According to the interdisciplinary server [www.scientific.ru](http://www.scientific.ru), the Institute has the highest total citation index among the Russian chemical institutions over the last 8 years. The average citation index of one article is above 6 and is high on the citation index list of articles from chemical instructions of the Siberian Branch of the Russian Academy of Sciences.

The Institute patents over 40 discoveries and maintains more than 30 foreign patents annually. The Institute is the avowed intellectual property protection leader among the Russian academic institutions.

The Institute is designing new generations of catalysts and technologies to meet the present-day global trends. Throughout its more than 50-year-long history, the Institute has been among the leaders in developing new technologies for the chemical and petrochemical industries, power engineering, and environmental protection.

The numerous exploratory and prospective studies of the Institute in the field of catalytic technologies include advancement of a large complex of environmental and energy-saving technologies, use of nontraditional feedstock in the production of various motor fuels and oil processing products, fine synthesis of medicines and plant protection chemicals, etc.

The Institute designed original devices for studying the properties of catalysts and has organized small-scale production of automated devices and installations for investigation and control of the physicochemical properties of various substances and materials. The novel, high-efficiency catalysts and technologies developed by the Institute satisfy the present-day requirements and are in demand in Russian markets and around the world.

The Institute is widely known abroad for its achievements and is a partner of many Russian and foreign companies.

## STRUCTURE OF THE INSTITUTE

The Boreskov Institute of Catalysis unites about 1000 employees, among its 400 researchers being 1 academician, 3 Corresponding Members RAS, about 70 Drs. Sci. and more than 200 PhD's. The Institute is a unique association of specialists in various fields of science and technology that are capable of solving any catalytic problems from fundamentals to design of industrial catalysts and processes.

The Institute structure comprises 9 research departments. Supporting and service departments provide the smooth work of the Institute.

The successful research activities are based on the overall access to the latest and reliable scientific data. There is the **Information and Library Center** in the Institute, and the library is one of the best scientific libraries in Siberia.

The **Scientific Organization Department** deals with scientific events such as conferences, seminars, schools, meetings, presentations of various companies and equipment producers, discussions. There is a museum where the information on catalytic science is collected.

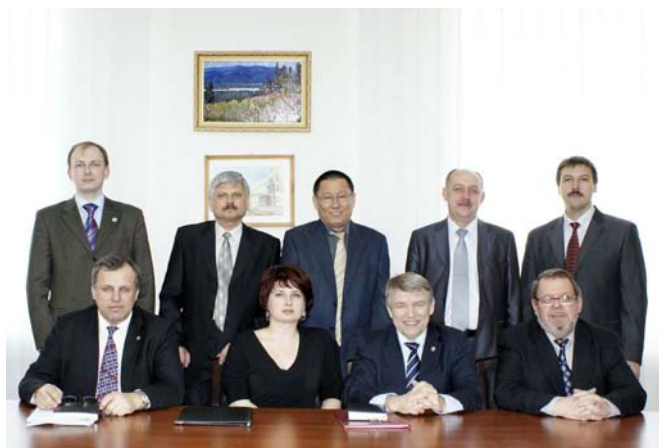
**Advertising and Exhibits Department** of the Institute is active in competence, perfect service in the field of organization of exhibitions of the new inventions and successive achievements of the Institute.

The activities of the **Group of Science, Engineering and Innovation Programs** are aimed at the participation of the Institute in State priority competitive programs for creation of technologies and materials to make Russia an economically independent country.

The **Publishing Department** concentrates its activities on supporting the R&D process inside the Institute, as well as on public information about the BIC's advances. Among the published items are Annual Reports, Proceedings of conferences organized by the Institute, information bulletins, manuals, etc.

**Production departments** provide engineering and technical support of the research activities, exploitation and modernization of the available facilities.





# STRUCTURE OF THE INSTITUTE

## Directorate:

A.A. Vedyagin, I.A. Kamolkin, B.S. Bal'zhinimaev,  
S.E. Glaznev, O.N. Martyanov

V.I. Bukhtiyarov, N.V. Mukhina, V.N. Parmon,  
A.S. Noskov

## DIRECTOR

### Directorate

### Scientific Council

## Research Departments



DEPARTMENT OF PHYSICOCHEMICAL METHODS FOR  
CATALYST INVESTIGATION  
Head: Prof. Oleg N. Martyanov



DEPARTMENT OF NONTRADITIONAL CATALYTIC PROCESSES  
AND TECHNOLOGIES  
Head: Acad. Valentin N. Parmon



DEPARTMENT OF HETEROGENEOUS CATALYSIS  
Head: Corr. Member RAS Valerii I. Bukhtiyarov



DEPARTMENT OF EXPLORATORY AND APPLIED INVESTIGATIONS  
Head: Prof. Bair S. Bal'zhinimaev



DEPARTMENT OF CATALYTIC PROCESS ENGINEERING  
Head: Prof. Alexander S. Noskov



DEPARTMENT OF CATALYTIC PROCESSES OF FINE ORGANIC  
AND BIOORGANIC SYNTHESIS  
Head: Prof. Zinaida P. Pai



SCIENTIFIC AND TECHNICAL DEPARTMENT OF APPLIED CATALYSIS  
Head: Prof. Lubov A. Isupova



St. PETERSBURG DEPARTMENT OF THE BORESKOV INSTITUTE OF CATALYSIS  
Head: Dr. Nikolai A. Pakhomov



VOLGOGRAD DEPARTMENT OF THE BORESKOV INSTITUTE OF CATALYSIS  
Scientific Director Dr. Aleksandr M. Beskopylnyi



Scientific-Supportive Units



Manufacturing Facilities



Administration and Services

## **DEPARTMENT OF PHYSICOCHEMICAL METHODS FOR CATALYSTS INVESTIGATION**

**HEAD OF THE DEPARTMENT PROF. OLEG N. MARTYANOV**



**Laboratory of Adsorption  
Head: Prof. Oleg N. Martyanov**



**Laboratory of Analytical Chemistry  
Head: Prof. Vladimir N. Sidelnikov**



**Laboratory of Investigation of the Mechanisms of  
Catalytic Reactions  
Head: Prof. Eugenio P. Talsi**



**Laboratory of Catalyst Texture Studies  
Head: Dr. Maxim S. Melgunov**



**Laboratory of Quantum Chemistry  
Head: Dr. Igor L. Zilberberg**



**Laboratory of Spectral Methods  
Head: Prof. Dimitrii I. Kochubey**



**Laboratory of Structural Methods  
Head: Prof. Sergey V. Tsybulya**



**Group of Low Temperature Catalysis by Metals  
Head: Prof. Vladimir V. Gorodetsky**



**Group of Advanced Developments  
Head: Prof. Oleg N. Martyanov**



**Group of Solid-State NMR Spectroscopy**  
**Head: Prof. Olga B. Lapina**



**Group of NMR Spectroscopy for Catalytic Hydrocarbon Conversion**  
**Head: Prof. Alexander G. Stepanov**



**Group of Nanostructural Analysis**  
**Head: Prof. Fedor V. Tuzikov**

## **DEPARTMENT OF NONTRADITIONAL CATALYTIC PROCESSES**

**HEAD OF THE DEPARTMENT ACADEMICIAN VALENTIN N. PARMON**



**Laboratory of Catalytic Methods of Solar Energy Conversion**  
**Head: Academician Valentin N. Parmon**



**Laboratory of Hydride Compounds Studying**  
**Head: Prof. Valentina I. Simagina**



**Laboratory of Catalytic Processes for Desulfurization**  
**Head: Dr. Olga N. Kovalenko**



**Laboratory of Energy Accumulating Processes and Materials**  
**Head: Prof. Yuriy I. Aristov**



**Group of Adsorption-Catalytic Processes for Fuel Cells**  
**Head: Dr. Alexey G. Okunev**



**Group of Aerosol Catalysis**  
**Head: Dr. Valerii N. Snytnikov**



**Group of Biocatalysis**  
**Head: Prof. Galina A. Kovalenko**



**Group of Catalysts on Carbon Supports**  
**Head: Dr. Irina L. Simakova**



**Group of Metal Complex Catalysis**  
**Head: Prof. Nina I. Kuznetsova**



**Group of Sulfide Catalysts**  
**Head: Prof. Anatolii N. Startsev**



**Group of Photocatalysis on Semiconductors**  
**Head: Prof. Alexandr V. Vorontsov**



**Group of Energy-Chemical Processes and Technologies**  
**Head: Prof. Vladimir I. Anikeev**



**Group of Biodegradable Polymers**  
**Head: Dr. Aleksandr G. Potapov**



**Group of Experimental Kinetics**  
**Head: Dr. Ilya Yu. Pakharukov**

## **DEPARTMENT OF HETEROGENEOUS CATALYSIS**

### **HEAD OF THE DEPARTMENT CORR. MEMBER RAS VALERII I. BUKHTIYAROV**



**Surface Science Laboratory**  
**Head: Corr. Member RAS Valerii I. Bukhtiyarov**



**Laboratory of Catalytic Processes in Fuel Cells**  
**Head: Prof. Vladimir A. Sobyenin**



**Laboratory of Heterogeneous Selective Oxidation**  
**Head: Dr. Vladimir I. Sobolev**



**Laboratory of Dehydrogenation**  
**Head: Prof. Vladimir V. Chesnokov**



**Laboratory of Catalysts for Deep Oxidation**  
**Head: Prof. Vladislav A. Sadykov**



**Laboratory of Oxidative Catalysis over Zeolites**  
**Head: Prof. Aleksandr S. Kharitonov**



**Laboratory of Catalysts Preparation**  
**Head: Prof. Aleksandra S. Ivanova**



**Laboratory of Ecological Catalysis**  
**Head: Prof. Zinifer R. Ismagilov**



**Laboratory of Catalytic Conversion of Carbon Oxides**  
**Head: Prof. Aleksandr A. Khassin**



**Group of Supported Metal Oxide Catalysts Investigation**  
**Head: Prof. Andrey I. Boronin**



**Group of Noble Metal Based Heterogeneous Catalysts**  
**Head: Prof. Anatolii V. Romanenko**



**Group of Surface Compounds Synthesis**  
**Head: Dr. Vladimir L. Kuznetsov**

## **DEPARTMENT OF CATALYTIC PROCESSES ENGINEERING**

### **HEAD OF THE DEPARTMENT PROF. ALEKSANDR S. NOSKOV**



**Laboratory of Unsteady-State Catalytic Methods for Gas Purification**

**Head: Prof. Aleksandr S. Noskov**



**Laboratory of Multiphase Processes Modeling**

**Head: Prof. Valerii A. Kirillov**



**Laboratory of Catalytic Hydrocarbon Conversion**

**Head: Prof. Gennadii V. Echevsky**



**Laboratory of Catalytic Polymerization**

**Head: Prof. Vladimir A. Zakharov**



**Laboratory for Catalytic Processing of Renewable Sources**

**Head: Dr. Vadim A. Yakovlev**



**Group of Kinetics of Catalytic Processes**

**Head: Prof. Sergei I. Reshetnikov**



**Group of Development and Optimization of Catalytic Processes with Catalyst Deactivation**

**Head: Dr. Vitalii N. Kashkin**



**Group of Membrane-Catalytic Processes**

**Head: Dr. Ilya V. Mishakov**





**Group of Complex Engineering Processes**  
**Head: Dr. Victor A. Chumachenko**



**Group of Mathematical Methods for Catalytic and Adsorption Processes Modeling**  
**Head: Dr. Natalia A. Chumakova**



**Group of Hydrogenation Processes**  
**Head: Dr. Galina A. Bukhtiyarova**

**DEPARTMENT OF EXPLORATORY AND APPLIED  
INVESTIGATIONS**

**HEAD OF THE DEPARTMENT PROF. BAIR S. BAL'ZHINIMAEV**



**Laboratory for Characterization and Testing of New Materials for  
Catalysis**

**Head: Prof. Bair S. Bal'zhinimaev**



**Laboratory of Zeolites and Acid Base Catalysis**

**Head: Prof. Eugenio A. Paukshtis**



**Group of Catalytic Conversion of Sulfur-Containing Compounds**

**Head: Prof. Anna V. Mashkina**

**DEPARTMENT OF CATALYTIC PROCESSES OF  
FINE ORGANIC AND BIOORGANIC SYNTHESIS**

**HEAD OF THE DEPARTMENT PROF. ZINAIDA P. PAI**



**Laboratory of Liquid-Phase Catalytic Oxidation of Organic  
Compounds**

**Head: Prof. Zinaida P. Pai**



**Group of Catalysts and Processes Based on Heteropoly Acids**

**Head: Prof. Elena G. Zhizhina**



**Group of Heterogeneous Catalysts for Selective Liquid-Phase  
Oxidation**

**Head: Prof. Oxana A. Kholdeeva**

**SCIENTIFIC AND TECHNICAL DEPARTMENT  
OF APPLIED CATALYSIS**

**HEAD OF THE DEPARTMENT PROF. LYUBOV A. ISUPOVA**



**Group of Catalysts and Supports for High Temperature Processes  
Head: Prof. Lyubov A. Isupova**



**Group of Aerogel Materials  
Head: Dr. Alexander F. Danilyuk**



**Assurance Group  
Head: Sergey A. Lopatin**



**Group of Catalysts Preparation  
Head: Vasilii Yu. Kruglyakov**



**Group of Catalysts Testing  
Head: Alexander S. Bobrin**



**Group of Pilot Plants and High Pressure  
Head: Victor N. Korotkikh**



**Group of Preparative Synthesis  
Head: Dr. Yurii I. Amosov**

**ST. PETERSBURG DEPARTMENT  
OF THE BORESKOV INSTITUTE OF CATALYSIS**



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**VICE-DIRECTOR DR. VALERY P. TULMANKOV**

**VOLGOGRAD DEPARTMENT  
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**SCIENTIFIC DIRECTOR  
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**CHIEF EXECUTIVE IGOR V. TERTISHNIKOV**

## **SCHOOLS OF THE BORESKOV INSTITUTE OF CATALYSIS**

The successful development of the Institute has been possible only due to the existence of scientific schools headed by the Institute's leading scientists in a variety of important research areas.

### **Academician G.K. Boreskov**

Theory of heterogeneous catalysis. The nature of the interaction between the reactants and the catalyst. The effect of the reactant–catalyst bonding energy on the catalytic activity and selectivity. The concepts of stepwise and associative mechanisms of redox catalytic reactions. Unsteady-state catalysis. Development of industrial catalysts.

### **Academician K.I. Zamaraev**

Theory of tunneling transfer of electrons in solids. The coordination chemistry of metal complex solutions. Studies of stepwise mechanisms of homogeneous and heterogeneous catalytic reactions, active site structure at the atomic/molecular level. Search for novel areas in catalysis.

### **Academician V.N. Parmon**

Catalysis and photocatalysis in renewable and non-traditional energetics. Photochemical and thermochemical methods of solar energy conversion. Radiation and thermal catalysis. The role of photocatalysis in the global chemistry of the terrestrial atmosphere. Abiogenic catalysis in the nature. Synthesis of nanomaterials. New types of chemical reactors.

### **M.G. Slin'ko, Corresponding Member of the Russian Academy of Sciences**

Methods of mathematical modeling of catalytic processes and reactors based on hierarchical models. Optimization and modeling of processes in fixed and fluidized beds; liquid-phase, multiphase, and unsteady-state processes. Design of industrial reactors.

### **R.A. Buyanov, Corresponding Member of the Russian Academy of Sciences**

Scientific basis of catalyst preparation and technology. Enhancing the reactivity of solid phases. Mechanochemistry in catalysis. Catalyst deactivation. Theoretical basis of the synthesis of carbon–mineral composites. Theory of nonbranched-chain free-radical catalytic reactions. Development of industrial catalysts.

### **V.A. Likholobov, Corresponding Member of the Russian Academy of Sciences**

Molecular design of catalytic compositions for organic syntheses. “Tailoring” of active sites of metal complex catalysts. Synthesis of porous supports with the desired textural and surface properties.

### **Professor Yu.I. Yermakov**

Targeted synthesis of supported catalysts with the desired composition and dispersion using immobilized organometallic compounds. Catalytic polymerization of olefins. Development of industrial catalysts.

**Professor V.A. Dzis'ko**

Formation of oxide catalysts of complex composition. Control of the chemical and phase compositions, acid–base surface properties, textural and strength characteristics, and thermal stability of the catalysts. Development of industrial catalysts and supports.

**Professor G.M. Zhidomirov**

Heterogeneous catalysis theory. Principles of the IR spectroscopy of the surface. Quantum chemical modeling of catalytic systems. Calculation of the electronic structure and properties of the acid and redox sites of zeolite, oxide, and metal catalysts.

## R&D ACTIVITY AREAS

### Basic research

- ◆ *Fundamentals of catalysis science, prediction of catalytic action*
- ◆ *Scientific and technological bases of catalyst preparation and synthesis of new materials including functional nanomaterials*
- ◆ *In situ studies of catalysts and mechanisms of catalytic reactions*
- ◆ *Theoretical basis of developing catalytic processes*
- ◆ *Mechanisms and kinetics of practically important catalytic reactions*
- ◆ *Mathematical modeling and optimization of parameters of reactors and catalytic processes*

### Applied catalysis

- *Efficient catalysts and catalytic technologies for oil & gas processing, for production of key chemicals*
- *Catalysts and catalytic technologies for synthesis of polymers with required properties, including biodegradable polymers*
- *Catalysts and processes for fine synthesis for pharmaceutical industry*
- *Sorbents, catalysts and technologies for environmental protection*
- *Reactors and processes based on membrane catalysts. Microchannel catalytic reactors*
- *Catalysts and processes for hydrogen energetics*
- *Catalytic technologies for heat power generation*
- *Catalysis for non-traditional energetics and processing of renewable resources*
- *Methods and facilities for catalyst testing*

### *Some BIC's proprietary technologies*

- **Reverse process** for gas purification
- **Zeoforming processes** for synthesis of gasoline
- **Alphox technology** for one step phenol from benzene production (in cooperation with Solutia)
- Honeycomb catalysts for oxidation of ammonia in the production of **nitric acid**
- New technology of **loading catalysts to reactors**
- **BICYCLAR** technology for production of aromatic hydrocarbons
- Direct catalytic **oxidation of H<sub>2</sub>S** to elemental sulfur
- Treatment of mixed **radioactive organic wastes**
- Liquid-phase oxidative **treatment of industrial wastewater**
- Technology for **formaldehyde** production
- Technology for **one step formic acid** production
- Technology for **reductant atmospheres** production over honeycomb catalysts

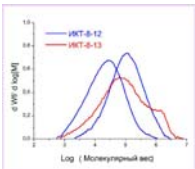


- Catalytic technologies for production of **carbon nanofiber materials and carbon nanotubes**
- Technology for synthesis of **Nafion-type polymer materials**
- Technology for synthesis of **polymer hydrogel materials for soft contact lenses**
- Technologies for generation of **synthesis gas from hydrocarbon fuel** (natural gas, gasoline, diesel and biofuel)
- Catalytic technologies for processing of **bio resources to produce biofuel**

*Some BIC's catalysts for industrial applications*



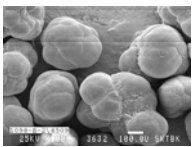
Vanadium pentoxide catalysts for sulfuric acid production



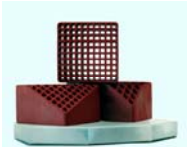
Supported catalysts ICT-8-12 and ICT-8-13 for polyethylene production



Catalyst ICT-8-20 for production of ultrahigh molecular weight polyethylene



Supported Ti-Mg catalyst IC-8-21 for polypropylene production



IC-42 series non-platinum catalysts for oxidation of ammonia in the production of nitric acid



High silica zeolite catalysts



Palladium catalyst for hydrogenation of distilled fatty acids in the production of stearine



Thermostable catalyst ICT-12-40 for gas purification



Glass fiber catalysts for treatment of waste gases of the production of synthetic rubber

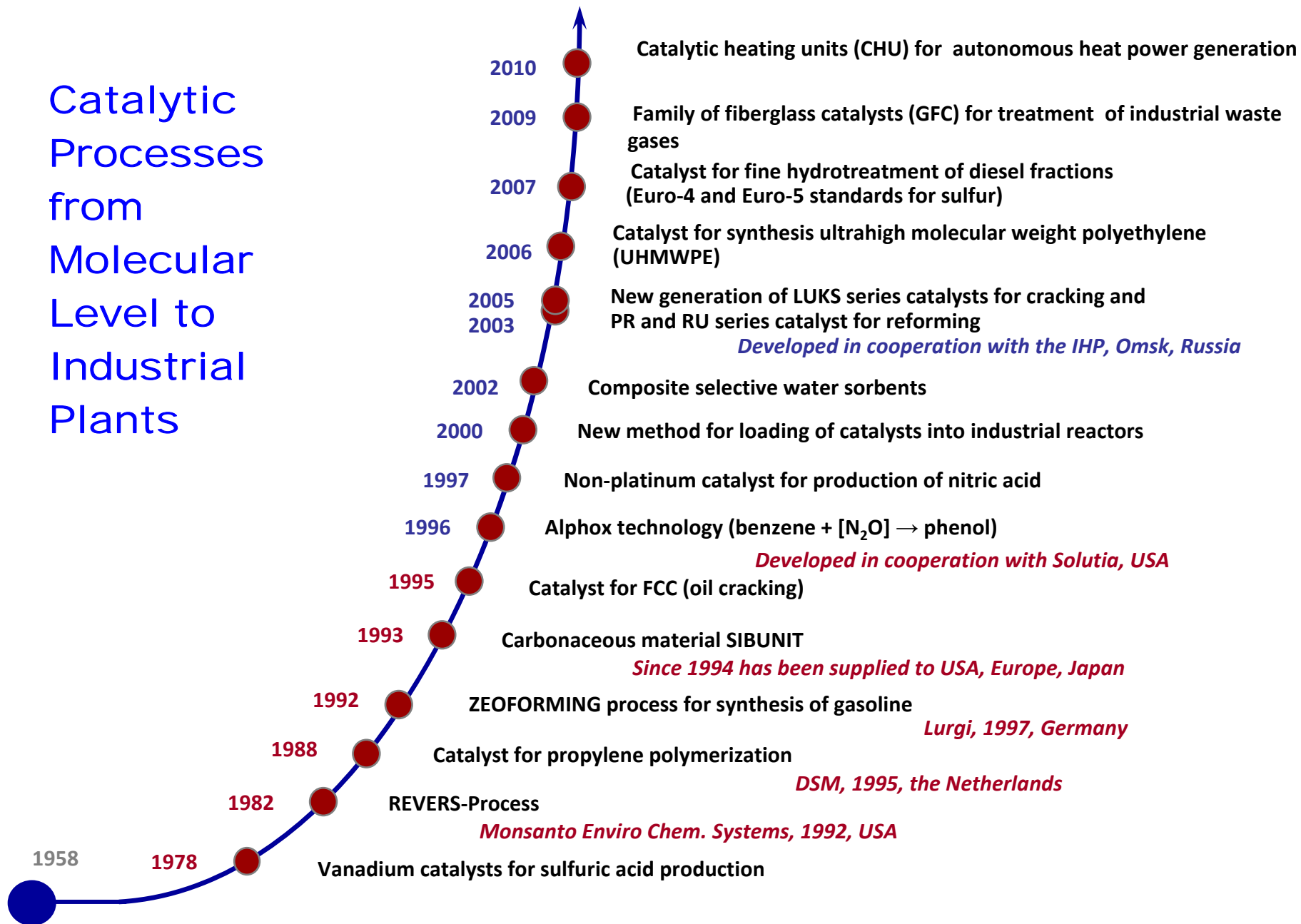


Photocatalytic systems for air cleaning and detoxication



CDM series dehydrogenation catalysts

# Catalytic Processes from Molecular Level to Industrial Plants



## INSTRUMENTATION FACILITIES

The Institute engages the leading position in the world in the improvement and design of new physicochemical methods for catalyst study. The Boreskov Institute of Catalysis has specialized groups united into Department of Physicochemical Methods for Catalyst Investigation oriented at analytical and scientific studies. At the Department there are many specialists of high qualification specializing in the investigation of catalysts and nanomaterials.

Modern and, in some cases, unique physicochemical methods provide a basis for fundamental studies at the atomic-molecular level.

The types of analytical and research studies performed at the Institute and methods used for them are listed below.

**Analytical** (composition of catalysts and catalytic reaction products)

### ***Bulk composition***

Atomic emission spectrometry

Atomic absorption spectroscopy

X-Ray fluorescence analysis of macro- and microprobes

### ***Phase analysis***

X-Ray diffraction, including *in situ* diffraction

Differential dissolution

Thermally programmed reduction, oxidation, desorption

X-Ray diffusion scattering

EXAFS spectroscopy (for amorphous materials)

### ***Morphology***

Transmission electron microscopy

High resolution transmission electron microscopy (HRTEM)

Scanning electron microscopy

Scanning tunneling microscopy

X-Ray small-angle scattering

### ***Surface***

X-Ray photoelectron spectroscopy (XPS)

Secondary ion mass-spectrometry (SIMS)

Determination of the surface acidity using IR spectroscopy of probe molecules

### ***Molecular composition of individual compounds and their mixtures***

Gas, gas-liquid and liquid chromatography on packed, capillary and polycapillary columns

Superrapid chromatography

Mass-spectrometry

### ***Adsorptive*** (specific surface area, pore structure, adsorption heat)

Porosimetry

Calorimetry



## **Kinetic**

Gradientless and integral differential reactors  
Fast relaxation technique  
Stop flow technique  
Radiochemical and isotopic methods  
Mass-spectrometric detection of free radicals

## **Spectral methods**

NMR spectroscopy on different nuclei, including high-temperature and *in situ*  
ESR spectroscopy, including *in situ*  
FMR spectroscopy, including *in situ*  
VUV electron spectroscopy  
UV-VIS electron spectroscopy  
Vibrational spectroscopies (IR, including *in situ*, and RAMAN)  
HREELS  
LEED  
X-Ray and NMR tomography

## INTERNATIONAL COOPERATION

The scientific cooperation of the Boreskov Institute of Catalysis with the catalytic communities from various countries is effected in accordance with various forms of cooperation: conducting joint seminars on catalysis, exchanging the information and appropriate materials, exchanging research fellows, visiting scientific centers, and participating in congresses and symposia on theoretical and applied catalysis.

According to research programs, projects and grants, the fundamentals of catalysis are studied jointly with researchers from various universities, institutions, research laboratories and companies. BIC collaborates fruitfully on a commercial basis with the leading companies from more than 20 countries, sells licenses, know-how and performs research projects according to client requests.

Academician Valentin N. Parmon is the Russian representative in the European Federation of Catalytic Societies (EFCATS), Member of the International Association of the Catalysis Societies (IACS).

### Visits of the specialists from the Boreskov Institute of Catalysis to foreign institutions in 2010

Austria	32	Germany	47	Saudi Arabia	4
Azerbaijan	13	Hungary	1	Serbia	2
Belarus	3	India	1	Singapore	2
Belgium	9	Israel	1	Slovenia	6
Brazil	2	Italy	13	Spain	6
China	19	Japan	7	Sweden	4
Czech Republic	7	Kazakhstan	10	Turkey	7
Denmark	3	Netherlands	8	UAE	4
Egypt	2	Poland	4	Ukraine	7
Finland	5	Portugal	1	United Kingdom	4
France	23	Republic of Korea	1	USA	5

**Visits of foreign specialists to the Boreskov Institute of Catalysis in 2010**

Australia	1	Indonesia	1	Republic of Korea	2
Austria	1	Italy	2	Saudi Arabia	1
China	21	Japan	1	Spain	2
France	30	Kazakhstan	5	Taiwan	2
Germany	3	Mongolia	1	United Kingdom	5
India	2	Netherlands	7	USA	2

**SCIENTIFIC COOPERATION  
WITHIN THE FRAMEWORK OF CONTRACTS AND AGREEMENTS  
WITH FOREIGN ORGANIZATION**

**ITALY**

The cooperation in the frame of the agreement between **Russian Academy of Sciences** (RAS) and **National Council on the Scientific Research** of Italy with *The Istituto di Tecnologie Avanzate per l'Energia "Nicola Giordano"* (CNR Institute of Advanced Energy Technologies "Nicola Giordano"), Messina – *BIC*, Novosibirsk on the Project "**Materials with Enhanced Properties for Energy Conversion**". Coordinators: **Prof. Yu. Aristov** (*BIC*) and **Prof. G. Restuccia** (*Istituto di Tecnologie Avanzate per l'Energia "Nicola Giordano"*).

**FRANCE**

According to the agreement between **RAS** and **CNRS** *BIC* collaborates with the *Institute de Recherches sur la Catalyse et l'Environnement de Lyon* (Research Institute on Catalysis and Environment), Villeurbanne, in the frame of the Russian-French European associated Laboratory on Catalysis headed by **Prof. V. Parmon** and **Dr. M. Lacroix**. The Laboratory was established by an agreement signed December 6, 2004 in Moscow by RAS and CNRS. Four areas of research were identified:

- **Catalysis and Energy: Production of Syngas and Hydrogen**
- **Catalysis and Environment: Treatment of Waste Waters**
- **Catalysis and Fine Chemistry: Pollution-Free Catalytic Processes**
- **Advanced Spectroscopic Methods: Mobility of Species in Porous Medium.**

**INDIA**

In the frame of RAS-DST Long Term Programme of cooperation in science and technology *BIC* collaborates with *National Chemical Laboratory*, Pune. Coordinators on the Program "Catalysis" are **Prof. V. Parmon** and **Prof. S. Sivaram**.

**GERMANY**

The cooperation in the frame of the agreement between **RAS** and **German Scientific Research Society** (GSRS) with

– *Ruhr-Universität Bochum*, Bochum, on the Project "**Synthesis and Reactivity of Novel Heterogeneous Polyoxometallate Based Catalysts for Alcohol Oxidation Containing Gold and TiO<sub>2</sub>**". Coordinators: **Prof. O. Kholdeeva** (*BIC*) and **Prof. Klaus Merz** (*Ruhr-Universität Bochum*)

– *Fritz-Haber-Institut der Max-Planck-Gesellschaft*, Berlin, on the Project "**Development of in situ Methods for Study of Solid Surfaces**". Coordinators: **Prof. V. Bukhtiyarov** (*BIC*) and **Prof. R. Schlögl** (*Fritz-Haber-Institut der MPG*)



– *Universität Konstanz*, Konstanz on the Project “*Various Resting States of the Catalysts for Olefin Polymerization and Oligomerization – Studying of the Reaction Route by Methods of Optical, ESR and NMR Spectroscopy*”. Coordinators: **Prof. E. Talsi** (*BIC*) and **Prof. H. Brintzinger** (*Universität Konstanz*).

#### **CHINA**

The cooperation in the frame of *Associated Research Laboratory* which was established by an agreement signed December 4, 2004 by the Borskov Institute of Catalysis and Heilongjiang University, Harbin. Chief Executive officers of Laboratory are: **Prof. V. Bukhtiyarov**, **Prof. G. Echevsky** (*BIC*) and **Prof. Fu Hong-Gang**, **Prof. Wu Wei** (*Heilongjiang University*) on the Project “*Synthesis and Modification of ZSM-12 Zeolites. Zeolite ZSM-12 in Reaction of Naphthalene Alkylation with Methanol*”.

## COOPERATION IN THE FRAME OF PROJECTS FINANCED BY INTERNATIONAL FOUNDATIONS

### CRDF Project

#### Carbon Nanoreactor for Solid-State Synthesis of Novel Nanoscale Materials Based on Nanocrystalline Oxides

Project Coordinators:

**Prof. A. Volodin**, *Boreskov Institute of Catalysis*, Novosibirsk, Russia

**Prof. K.J. Klabunde**, *Kansas State University*, Manhattan, Kansas, USA.

### EUROPEAN COMMUNITY SIXTH FRAMEWORK PROGRAM

#### I. Co-Processing of Upgraded Bio-Liquids in Standard Refinery Units

Coordinator: **Dr. Y. Solantausta**, *VTT Processes*, Espoo, Finland

Partners:

*Rijksuniversiteit Groningen*, The Netherlands; *Boreskov Institute of Catalysis*, Novosibirsk, Russia (**Prof. V. Kirillov**); *Uhde Hochdrucktechnik GmbH*, Germany; *BTG Biomass Technology Group BV*, The Netherlands; *University of Twente*, The Netherlands; *STFI-PACKFORSK AG*, Sweden; *Institute of Wood Chemistry*, Hamburg, Germany; *Slovenian Institute of Chemistry*, Slovenia; *Arkema SA*, France; *Helsinki University of Technology*, Finland; *ALMA Consulting Group SAS*, France; *Centre National de la Recherche Scientifique*, France; *Chimar Hellas SA*, Greece; *Albermarle Catalysts Company BV*, The Netherlands; *Metabolic Explorer*, France; *Shell Global Solutions International*, The Netherlands.

#### II. Non-Noble Catalysts for Proton Exchange Membrane Fuel Cell Anodes

Coordinator:

**Dr. G. Tsotridis**, *Institute for Energy*, Joint Research Centre, Petten, The Netherlands

Partners:

*Technical University of Denmark*, Lyngby, Denmark; *Boreskov Institute of Catalysis*, Novosibirsk, Russia (**Prof. V. Parmon**, **Dr. O. Taran**); *Southampton University*, United Kingdom; *Technical University of Munich*, Germany; *Bavarian Center for Applied Energy Research*; *Umicore, AG & Co KG*, Germany.

#### III. Demonstration of SOFC Stack Technology for Operation at 600°C

Coordinator: **B. Rietveld**, *Energy Research Centre of The Netherlands*, Petten, The Netherlands

Partners:

*University St. Andrews*, UK; *HTceramix*, Yverdon, Switzerland; *Imperial College*, UK; *Karlsruhe University*, Germany; *Oxford University*, UK; *University of Leoben*, Austria;

*Boreskov Institute of Catalysis, Novosibirsk, Russia (Prof. V. Sadykov); Technical University of Denmark, Denmark; VTT Technical Research Centre of Finland, Finland; Dalian Institute of Chemical Physics, China.*

## **EUROPEAN COMMUNITY SEVENTH FRAMEWORK PROGRAM**

### **I. Reforming of Crude Glycerin in Supercritical Water to Produce Methanol for Re-Use in Biodiesel Plants**

Coordinator: **J. Vos**, *BTG BiomassTechnology Group BV*, The Netherlands

Partners:

*Acciona Servicios Urbanos, Spain; Boreskov Institute of Catalysis, Novosibirsk, Russia (Prof. V. Kirillov); Rijksuniversiteit Groningen, The Netherlands; University of Maribor, Slovenia; UHDE High Pressure Technologies GmbH, Germany; SPARQLE International BV, The Netherlands.*

### **II. Engine and Turbine Combustion of Bioliquids for Combined Heat and Power Production**

Coordinator: **J. Vos**, *BTG BiomassTechnology Group BV*, The Netherlands

Partners:

*AMO ZIL, Moscow, Russia; Aston University, Birmingham, UK; Boreskov Institute of Catalysis, Novosibirsk, Russia (Prof. V. Kirillov); BTG Biomass Technology Group BV; Central Scientific Automobile and Automotive Engines Institute, NAMI, Moscow, Russia; Encotech BV, The Netherlands; University of Florence, Florence, Italy.*

### **III. Oxidative Coupling of Methane Followed by Oligomerization to Liquids**

Coordinator: **G. Marin**, *Ghent University*, Belgium

Partners:

*Bayer Technology Service, Germany; Institute de Recherches sur la Catalyse et l'Environnement de Lyon, Villeurbanne, France; Ruhr-Universität Bochum, TECHEM, Bochum, Germany; Instituto de Tecnologia Quimica, Spain; Johnson Matthey, UK; SINTEF, Trondheim, Norway; Linde Engineering, Germany; Cepsa R&D Center, Spain; Haldor Topsoe, Denmark; Universitetet i Oslo, Oslo, Norway; University of Cambridge, UK; ALMA Consulting Group, France; Boreskov Institute of Catalysis, Novosibirsk, Russia (V. Sadykov); INEOS, Belgium; Institut für Mikrotechnik Mainz GmbH, Germany; Eni SpA, Italy.*

## **INTERNATIONAL SCIENCE AND TECHNOLOGY CENTER (ISTC)**

### **I. Synthesis and Investigation of the Metal Oxide Catalysts for Photocatalytic Degradation of Harmful Gases Resulted from Terrorist Acts and Man-Caused Catastrophes**

Project Manager from BIC **Prof. A. Vorontsov.**

### **II. Development of an Integrated Separator for Direct Reforming of Hydrocarbons in High-Temperature Fuel Cells**

Project Manager from BIC **Prof. Z. Ismagilov.**

### **III. Composite Nanocarbon Based Materials for Electromagnetic Applications**

Project Manager from BIC **Dr. V. Kuznetsov.**

### **IV. Small Capacity Fuel Cells**

Project Manager from BIC **Prof. V. Kirillov.**

### **V. Development of High-Performance Oxygen-Conducting Membranes and Compact Syn-Gas Generators on Their Base**

Project Manager from BIC **Prof. V. Sadykov.**

## CONFERENCE AND EXHIBITION ACTIVITIES

The advertising and information activities are universal marketing tools for establishing professional contacts. Again, this is a necessary part of the market examination to be settled in the world. Each year, achievements of the Boreskov Institute of Catalysis are demonstrated at the very prestigious exhibitions in Russia and abroad. The goal is to strengthen collaboration with the present partners and to attract attention of potential customers.

In 2010, the most important BIC's achievements in the area of applied catalysis were presented at the Exhibitions:

- ❖ *“Ecology of Big City”*, March 17-19, St. Petersburg, Russia
- ❖ *“Russian National Exhibition”*, June 12-16, Paris, France
- ❖ *“X Moscow International Innovations' and Investments' Show”*, September 7-10, Moscow, Russia  
**Golden Medal and Diploma** for *“Catalysts Based on Fiberglass Materials”* and *“Hydrocarbon Dehydrogenation Catalysts for Synthetic Rubber Production”*
- ❖ *“XIII International Trade Fair “Carbon Expo”*, September 14-17, Kemerovo, Russia  
**Golden Medal and Diploma** for *“Catalytic Heat Supply Installations”*  
**1<sup>st</sup> degree Diploma** for *“Catalytic Heat Generators”* and *“Fiber-Glass Based Catalysts”*
- ❖ *Exhibition “International Chemical Assembly”, “ICA-2010”*, September 27-30, Moscow, Russia
- ❖ *“SibPolyTech-2010”*, October 19-22, Novosibirsk, Russia.  
**Big Golden Medal and Diploma** *“For Development and Production of Catalytic Heat Supply Installations of High Energy Efficiency and Ecological Reliability”*
- ❖ *“Rusnanotech-2010”*, November 1-3, Moscow, Russia
- ❖ *Exhibition of Innovative Developments of SB RAS in China-Russian Technopark*, December 12-18, Changchun, China.

Solution of many specific problems requires joint efforts in order to promote its fastest accomplishing. International congresses, symposia, conferences, and seminars are the most productive to familiarize scientists with innovations outside their own research and to disseminate the information about the results achieved to a wider audience. One of the directions of the scientific-organizational work carried out in the Institute is aimed on conducting conferences and seminars with the participation not only of Russian scientists and researchers from fSU, but foreign participants as well.

**SECOND GERMAN-RUSSIAN SEMINAR ON CATALYSIS**  
**“BRIDGING THE GAP BETWEEN MODEL AND REAL CATALYSIS”**

*March 14-17, Kloster Seeon, Bavaria, Germany*



*Seminar organizers:*

- Fritz-Haber-Institut der Max-Planck-Gesellschaft, Berlin, Germany
- Borekov Institute of Catalysis, Novosibirsk, Russia.

The Seminar provided a platform for discussing the progress in understanding catalytic function with the help of model *in situ* study. The Program consisted of 6 invited plenary lectures and 32 oral presentations on the three bases:

- ❖ *Study of the Mechanisms of Heterogeneous Catalytic Reactions at Atomic-Molecular Level;*
- ❖ *Application of the Surface Sensitive Methods to Study the Formation, Activation and Degradation of the Active Sites in Heterogeneous Catalysts;*
- ❖ *Computational Chemistry to Study the Catalytic Reactions.*

*Plenary lectures:*

- **A. Stepanov** (*Borekov Institute of Catalysis, Novosibirsk, Russia*) – “*In situ* NMR in Catalysis: A Study of Small Alkanes Activation and Aromatization on Zn and Ga-Modified Zeolites BEA”
- **I. Yudanov** (*Borekov Institute of Catalysis, Novosibirsk, Russia*) – “Catalytic Activity of Metal Nanocrystallites: Structure Sensitivity and Particle Size Effect from DFT Modeling”
- **J. Lercher** (*Technical University of Munich, Munich, Germany*) – “Bifunctional and Concerted Catalysis for Hydrocarbon Conversion”

- **J. Sauer** (*Humboldt University of Berlin, Berlin, Germany*) – “C-H Activation by (Transition) Metal Oxides”
- **R. Fischer** (*Süd-Chemie AG, Bruckmühl, Germany*) – “Innovation as Catalyst for Success - Some Recent Examples from Süd-Chemie”
- **M. Muhler** (*Ruhr-University Bochum, Germany*) – “On the Active Site of Methanol Synthesis over ZnO”.

Among 43 participants were 15 scientists from Germany, 27 from Russia and 1 from Azerbaijan.

**IV MEMORIAL SEMINAR**  
**“MOLECULAR DESIGN OF CATALYSTS AND CATALYSIS IN THE PROCESSES OF**  
**HYDROCARBON CONVERSION AND POLYMERIZATION”**  
**dedicated to the 75th birthday of Professor Yu.I. Yermakov**

*April 13-16, Listvyanka village, Russia*



*Seminar organizers:*

- Borekov Institute of Catalysis, Novosibirsk
- Institute for Hydrocarbons Processing, Omsk
- Institute of Chemistry and Chemical Technology, Krasnoyarsk
- Scientific Council on Catalysis, Branch of Chemical Sciences of RAS
- Baikal Limnological Museum, Listvyanka, Russia.

The Seminar Program was scheduled in 6 sessions:

- ❖ *Catalysis for Hydrocarbon Material Processing*
- ❖ *Catalytic Olefins Polymerization*
- ❖ *Catalysis by Metal Complexes.*

The Program of the Seminar consisted of 11 key lectures, 18 oral presentations and 25 oral reports.

*Key lectures:*

- **V. Anannikov** (*Zelinsky Institute of Organic Chemistry, Moscow, Russia*) – “Homogeneous, Heterogeneous and Nanosized Catalytic Systems for Organic Synthesis: Carbon-Carbon Bond Formation”
- **V. Bukhtiyarov, I. Beck** (*Boreskov Institute of Catalysis, Novosibirsk, Russia*) – “Catalysts with Nanostructured Active Component on the Basis of Noble Metals: Synthesis and Study of Size Effects”
- **M. Vargaftic, N. Kozitsyna, S. Nefedov, N. Akhmadullina, N. Cherkashina, I. Yakushev, I. Moiseev** (*Kurnakov Institute of General and Inorganic Chemistry, Moscow, Russia*) – “Heterometal Palladium Carboxylates – from Molecular Complexes to Catalytically Active Materials”
- **V. Zakharov** (*Boreskov Institute of Catalysis, Novosibirsk, Russia*) – “Hydrides of Transition Metals as Active Intermediates in the Processes of Olefin Polymerization”
- **A. Lavrenov** (*Institute for Hydrocarbons Processing, Omsk, Russia*) – “Novel Catalyst for Ethylene Oligomerization Based on NiO/B<sub>2</sub>O<sub>3</sub>-Al<sub>2</sub>O<sub>3</sub> System: Composition, Structure, Properties”
- **G. Lisichkin** (*Moscow Lomonosov State University, Moscow, Russia*) – “Grafted Surface Compounds: Synthesis, Properties, Application”
- **V. Likholobov** (*Institute for Hydrocarbons Processing, Omsk, Russia*) – “Development of the Ideology for Supported Metal Catalysts Synthesis by Anchoring Metal Complex Precursor”
- **L. Novokshenova, I. Meshkova, T. Ushakova, V. Krashenninnikov, T. Ladygina, I. Leipunskii\*, A. Zhigach\*** (*Semenov Institute of Chemical Physics, Moscow, Russia; \*Institute for Energy Problems of Chemical Physics, Moscow, Russia*) – “Immobilization of Metallocene Catalysts for Olefin Polymerization and Analysis of Heterogeneity of Active Centers”
- **A. Startsev** (*Boreskov Institute of Catalysis, Novosibirsk, Russia*) – “Concept of Acid-Base Catalysis by Metal Sulphides”
- **E. Talsi** (*Boreskov Institute of Catalysis, Novosibirsk, Russia*) – “Active Centers of Catalysts for Ethylene Polymerization and Trimerization Based on Cobalt, Vanadium and Chromium Compounds”
- **N. Chesnokov, N. Mikova, B. Kuznetsov** (*Institute of Chemistry and Chemical Technology, Krasnoyarsk, Russia*) – “Synthesis and Properties of Nanoporous Carbon Supports Prepared from Natural Carbonaceous Material and Catalysts on Their Base”.

A Round Table on the subject “*Catalysts for Oil Treatment and Petrochemistry*” took place in the frames of the Seminar.

There were 77 participants from 10 Russian cities and 1 scientist from Azerbaijan.



**XX ALL-RUSSIAN SCIENTIFIC CONFERENCE  
“X-RAY AND ELECTRONIC SPECTRA AND CHEMICAL BOND”**

*May 24-27, Novosibirsk, Russia*



*Conference organizers:*

- Borekov Institute of Catalysis, Novosibirsk
- Nikolaev Institute of Inorganic Chemistry, Novosibirsk
- Novosibirsk State University, Novosibirsk.

Conference Program included 9 invited plenary lectures, 7 key lectures, 52 oral and 100 poster presentations on the following topics:

- ❖ *Novel Theoretical Approaches to Description of the Electronic Structure. Novel Design of Laboratory Equipment. Development of Laboratory Devices*
- ❖ *Photoelectron Spectroscopy and Auger Electron Spectroscopy. Photoelectron Diffraction*
- ❖ *X-Ray Emission Spectroscopy. EXAFS and NEXAFS (XANES)*
- ❖ *Application of Photoelectron Spectroscopy in Investigation of Surface, Catalysts and Semiconductors*
- ❖ *Application of Photoelectron Spectroscopy in Investigation of Nanostructured and Functional Materials.*

*Plenary lectures*

- **K. Eltsov** (*Prokhorov General Physics Institute, Moscow, Russia*) – “Scanning Tunneling Spectroscopy – Possibilities and Limitations”
- **A. Kochur** (*Rostov State University of Transport Communications, Rostov-on-Don, Russia*) – “Cascade Effects in Electronic and Emission Spectra”
- **M. Kuznetsov** (*Institute of Solid State Chemistry, Yekaterinburg, Russia*) – “Photoelectron Diffraction and Surface Spectroscopy”
- **Yu. Mikhlin** (*Institute of Chemistry and Chemical Technology, Krasnoyarsk, Russia*) - “Application of Photoelectron and X-Ray Spectroscopy and Probe Microscopy for Studying Mineral Surface”
- **S. Molodtsov** (*European XFEL, Hamburg, Germany*) – “Possibilities of Electron and X-Ray Spectroscopy: From Synchrotrons to Free-Electron Lasers”
- **A. Soldatov, G. Smolentsev, V. Mazalova, A. Kravtsova** (*Southern Federal University, Rostov-on-Don, Russia*) – “X-Ray Absorption Spectroscopy as a Method for Analysis of Nanosized Substances Structure”
- **A. Stakheev** (*Zelinsky Institute of Organic Chemistry, Moscow, Russia*) – “X-Ray Photoelectron Spectroscopy for Studying Supported Metal and Oxide Catalysts”
- **Yu. Chizhov** (*St. Petersburg State University, St. Petersburg, Russia*) – “Photoemission of Metal Complexes”
- **A. Shikin** (*St. Petersburg State University, St. Petersburg, Russia*) – “Peculiarities of Electronic Structure and Physico-Chemical Properties of 2D, 1D and 0D Small Metal Systems”

About 180 participants from Russia and fSU Countries took part in the event.

## INTERNATIONAL CONFERENCE “CATALYSIS FOR RENEWABLE SOURCES: FUEL, ENERGY, CHEMICALS”

*June 28 – July 2, Tsars Village, St. Petersburg suburb, Russia*



### *Conference organizers*

- Boreskov Institute of Catalysis, Novosibirsk, Russia
- Missouri University of Science and Technology, USA
- Petrobras, Brazil
- St. Petersburg Scientific Center RAS, Russia
- Scientific Council on Catalysis RAS, Russia.

The principal objective of the Conference was to give a wide overview of the recent achievements in the development of catalytic technologies for renewable sources processing, and to discuss the latest advances.

The Conference scientific Program comprised 6 invited lectures of leading specialists, 8 key lectures, 2 presentations, 48 oral and 78 poster presentations on the following topics:

- ❖ *Catalysis for Biofuel Production*
- ❖ *Catalysis for Biochemical Generation*
- ❖ *Catalytic Gasification of Bioproducts*
- ❖ *Biocatalysis for Biomass Processing.*

### *Invited speakers:*

- **O. Martyanov, V. Parmon** (*Boreskov Institute of Catalysis, Novosibirsk, Russia*) – “Design of Catalysts and Catalytic Processes for Biofuels Production”
- **P. Vasudevan, R. Davison** (*University of New Hampshire, NH, Durham, USA*) – “Environmentally Sustainable Biofuels: Advances in Biodiesel Research”

- **D. Murzin** (*Åbo Akademi University (Turku), Finland*) – “Catalytic Transformations for Production of Biofuels, Specialty Chemicals and Pharmaceuticals from Woody Biomass”
- **W. Prins** (*University of Ghent, Faculty of Bioscience Engineering, Ghent, Belgium*) – “Progress in the Development of Biomass Fast Pyrolysis Technology and Its Application”
- **D. Aranda, L. Rocha, A. Ramos\*, N. Antoniosi-Filho\*\*** (*Greentec-Federal University of Rio de Janeiro, Brazil; \*DEQ/UFS, Brazil; \*\* LAMES/UFG, Brasil*) - “Transesterification and Hydroesterification: Theoretical and Experimental Analysis of Biodiesel Production Using Heterogeneous Catalysis”
- **M. Al-Dahhan** (*Missouri University of Science and Technology, Rolla, USA*) – “Advancement of Catalytic Fischer-Tropsch Slurry Bubble Columns via Advanced Measurement Techniques for Renewable Energy and Chemicals Production”.

200 scientists from 33 countries participated in the Conference.

## XIX INTERNATIONAL CONFERENCE ON CHEMICAL REACTORS “CHEMREACTOR-19”

*September 5-9, Vienna, Austria*



*Conference organizers:*

- Boreskov Institute of Catalysis, Novosibirsk, Russia
- Russian Scientific and Cultural Center in Vienna, Austria
- Federal Agency ROSSOTRUDNICHESTVO, Moscow, Russia
- Ministry of Education and Science of the Russian Federation
- European Federation on Chemical Engineering
- Scientific Council on Theoretical Foundations of Chemical Technology RAS
- Scientific Council on Catalysis RAS.

Four-day scientific Program comprised 6 plenary invited lectures, 7 key-note presentations, 71 oral presentations, 4 presentation lectures and 125 posters on the following topics:

- ❖ *Advances in Chemical Reactors Fundamentals* (Section I):
  - Chemical Reactions Kinetics
  - Fundamentals of Chemical Reactors Simulation

- Heat & Mass Transfer in Chemical Reactors
- Hydrodynamics and CFD Studies in Chemical Reactors
- ❖ *Chemical Reaction Engineering and Reactors Design – Novel Approaches, Modeling, Scale-Up, Optimization* (Section II):
  - New Designs of Chemical Reactors (Membrane Reactors, Microreactors, Structured Reactors etc)
  - Novel Approaches in Chemical Reaction Processes Engineering (Unsteady-state and Transient Processes, Reverse-Flow Operation, Sorption-Enhanced Reaction Processes, Multifunctional Reactors, Reaction-Separation Processes etc)
- ❖ *Chemical Reactors and Technologies for Emerging Applications* (Section III):
  - Processing of Biomass and Renewable Feedstock's
  - Environmental Protection and Utilization of Wastes
  - Production of Hydrogen and Green Fuels
  - Advanced Processing of Natural Gas and Oil.

*Plenary lectures:*

- **Yu. Matros** (*Matros Technologies, Inc., USA*) – “How to Design Optimal Catalytic Reactor” (*Professor Mikhail Slin'ko Honorary Lecture*)
- **J.C. Schouten, M. de Croon, E. Rebrov, J. van der Schaaf, X. Nijhuis** (*Eindhoven University of Technology, The Netherlands*) – “Multifunctional Devices for Intensified Chemicals Processing: From Microreactors to Spinning Disks”
- **V. Yakovlev** (*Boreskov Institute of Catalysis, Novosibirsk, Russia*) – “Design of Catalytic Processes for Biofuels Production”
- **V. Likholobov** (*Institute of Hydrocarbons Processing, Omsk, Russia*) – “High Temperature Processes of Nanostructural Carbon Materials Production”
- **H. Stitt, Dan Enache, S. Pollington, M. Winterbottom\*** (*Johnson Matthey PLC, UK; \*University of Birmingham, UK*) – “Multiphase Catalytic Reactions in Reactors Structured at the Meso-Scale”
- **L. Giorno** (*Institute on Membrane Technology, ITM-CNR, Italy*) – “Membrane Reactors: State of the Art and Perspectives in Biotechnology and Chemical Production”.

More than 200 specialists from 37 countries took part in the Conference.

**AZERBAIJAN-RUSSIAN SYMPOSIUM WITH INTERNATIONAL PARTICIPATION  
“CATALYSIS FOR SOLVING THE PROBLEMS OF PETROCHEMISTRY AND  
OIL REFINING”**

*September 28–30, Baku, Azerbaijan Republic*



*Symposium organaizers:*

- Azerbaijan National Academy of Sciences
- Siberian Branch of the Russian Academy of Sciences
- Mamedaliyev Institute of Petrochemical Processes ANAS
- Boreskov Institute of Catalysis, Novosibirsk
- Branch of Chemical Sciences ANAS
- Scientific Council on Catalysis, Branch of Chemical Sciences of RAS.

Scientific Program of the Symposium comprised 51 oral presentations and 94 posters delivered at two parallel sections:

*Section I.*

- ❖ *Catalysts for the Processes of Petrochemistry and Oil Refining*
- ❖ *Physical and Chemical Aspects of Catalytic Processes and Catalysts in Petrochemistry and Oil Refining*

*Section II.*

- ❖ *Nano-Technologies in Catalysis*
- ❖ *Environmental Problems of Catalysis.*

160 specialists from 8 countries took part in the Symposium.

## EDUCATIONAL ACTIVITIES

With that deep belief that people are a decisive factor and that the best way to educate is to teach others, many researchers of the Institute participate in the training of specialists in chemistry and, particularly, in catalysis, ensuring closest and time-tested links of the Institute with the main sources of researchers – Novosibirsk State University (NSU), Novosibirsk State Technical University (NSTU), Tomsk State University (TSU), Tomsk Polytechnical University (TPU), Kazan State University (KSU). Students studying at the following chairs realize their graduate works in the Boreskov Institute of Catalysis:

- Catalysis and Adsorption, Department of Natural Sciences, NSU
- Physical Chemistry, Department of Natural Sciences, NSU
- General Chemistry, Department of Natural Sciences, NSU
- Environmental Chemistry, Department of Natural Sciences, NSU
- Chemical and Biological Physics, Department of Physics, NSU
- Physical Methods to Study Solids, Department of Physics, NSU
- Differential Equations, Mechanico-Mathematical Department, NSU
- Environmental Engineering, Aircraft Department, NSTU
- Technological Processes and Apparatuses, Department of Mechanics and Technology, NSTU
- Physical and Colloid Chemistry, Chemical Department, TSU
- Fuel Chemical Technology and Chemical Cybernetics, Chemical-Technological Department, TPU.

About 70 scientists combine teaching and their job at the Research Institute. They present lectures, give seminars and practical classes; participate in the organization and renovation of the educational process.

Since the earliest years, the strategy of the training of students has been built up by leading scientists of BIC and combined in harmony two aspects: curricula provide the students with deep theoretical and practical knowledge in catalysis, i.e. theory of the prevision of catalytic action of substances, kinetics of catalytic reactions, scientific bases of catalyst preparation, engineering of catalytic processes, application of modern physical and mathematical methods for investigation of catalysts and catalysis; and, on the other hand, the educational process arrangement allows the scope of postgraduate activities of the students to be expanded significantly. They can work as technologists, production managers, researchers at various research organizations, teach at universities.

All these features allow students to prepare their diploma at the highest scientific level and become widely informed specialists in chemistry and catalysis. The graduates from the Chair of Adsorption and Catalysis are the principal source of recruitment of the personnel of the Institute.

### ***BIC – NSU***

The Novosibirsk State University was established as an essential part of the Siberian Branch of the Academy of Sciences; it embodies the idea of the close integration of education and

science. The chief mission of the University is to train professionals for scientific institutions of the country.

From its earliest age, the University pursues *three fundamental principles*. The *first* one is that the teachers are experts engaged in the real science. The intellectual basis of the University is more than 5000 scientists of 30 research institutes of the Siberian Branch. The *second* principle is a thorough mathematical background provided to students of all the departments. And the *third* principle means that the students master theoretical disciplines during their first three years and do their practical research in academic institutes of the Siberian Branch during the last years.

The Boreskov Institute of Catalysis is a key element in the training of specialists in chemistry at NSU. Graduation works of the students are conducted at the laboratories of the Institute and are supervised by experienced researchers. Their results are usually published in scientific journals and form the basis of their future Ph.D. theses.

The *Chair of Catalysis and Adsorption* (Head Prof. V. Bukhtiyarov) is of special importance for the Institute. It is only 5 years younger than Novosibirsk State University itself. The organization of the Chair was initiated in 1964 by Academician G.K. Boreskov – the founder and first director of the Boreskov Institute of Catalysis SB RAS. The Chair is located at the Institute, which provides it with everything required for efficient training of students specializing in catalysis and technology of catalytic processes.

*Courses of lectures:*

- ◆ Catalysis
- ◆ Adsorption and Porous Structure
- ◆ Scientific Bases for Catalysts Preparation
- ◆ Kinetics of Heterogeneous Catalytic Reactions
- ◆ Engineering Chemistry of Catalytic Processes
- ◆ Catalysis, Environment and Sustainable Development of Civilization
- ◆ Molecular Design of Catalysts
- ◆ Modern Technique of Catalytic Experiments
- ◆ Analytical Methods
- ◆ Quantum-Chemical Methods in Catalysis
- ◆ Magnetic Resonance Spectroscopy
- ◆ Optical Spectroscopy
- ◆ X-Ray Technique
- ◆ Thermodynamics of Working Catalyst
- ◆ Physical Methods in Catalysis
- ◆ Computer Application for Catalytic Studies.

The *Chair of Physical Chemistry* (Head Prof. V. Parmon) prepares highly skilled specialists in the field of chemical kinetics and thermodynamics, application of physical methods for catalysis.



*Courses of lectures:*

- ◆ Physical Chemistry
- ◆ Chemical Thermodynamics
- ◆ Chemical Kinetics
- ◆ Nonequilibrium Thermodynamics
- ◆ Matter Structure
- ◆ Terminal System: Computer Modeling of Processes and Physical-Chemical Phenomena
- ◆ NMR and ESR Spectroscopy for Catalysis.

The main objective of the *Chair of General Chemistry* (Head Prof. V. Sobyenin) is teaching first year students in general and inorganic chemistry.

*Courses of lectures:*

- ◆ Physical Chemistry
- ◆ Inorganic Chemistry
- ◆ General and Inorganic Chemistry.

The *Chair of Physical Methods to Study Solids* (Head Prof. S. Tsybulya) prepares specialists dealing with development, design and application of physical methods for investigation of structure of solids, including functional materials of various purpose and nanosystems.

*Courses of lectures:*

- ◆ Crystallography
- ◆ X-Ray Diffraction
- ◆ Electron Microscopy
- ◆ XPS Spectroscopy
- ◆ Magnetic Resonance
- ◆ Thermal Analysis
- ◆ Synchrotron Radiation.

***BIC – NSTU***

The Novosibirsk State Technical University is one of the largest research and educational centers in Russia. The University trains and retrain qualified specialists for research and industrial complex of Siberia and the Far East. Joint training of specialists at NSTU and institutes of the Siberian Branch of RAS in the field of technology of catalytic processes is of special importance for practical application of fundamental knowledge in the field of catalysis. The basic training is conducted by the *Chair of Environmental Engineering* (Head Prof. V. Larichkin) founded at the Boreskov Institute of Catalysis.

*Courses of lectures:*

- ◆ Technology Bases for Environmental Protection
- ◆ Processes and Apparatuses for Environmental Protection
- ◆ Catalytic Methods for Environment Protection
- ◆ Methods and Devices for Environmental Control, Ecological Monitoring
- ◆ Mathematical Modeling of Chemical Processes and Reactors

- ◆ Organic Chemistry
- ◆ Industrial Ecology
- ◆ Technologies for Utilization of Industrial Wastes
- ◆ Oil-Gas Branch
- ◆ Analytic Chemistry and Physical-Chemical Methods of Analysis.

Chemical engineering is a science-intensive field. The engineers will be able to work successfully in the priority investment areas. The close co-operation of the *Chair of Technological Processes and Apparatuses* with research institutes of the Siberian Branch of Russian Academy of Science is the key to train high-skilled specialists.

### ***Joint Educational Scientific Centers and Laboratories***

***Educational Scientific Centers (ESC)*** were established according to the Government concept for the education modernization. Educational, scientific and innovational complexes give the possibility of improving professional education by providing the unity of educational, scientific and technical innovational activity at every stage of training specialists.

The Educational Scientific Center “*Catalysis*” was organized in 2008 jointly with *NSU* and *NSTU*. Executors: **Prof. Valerii I. Bukhtiyarov** (BIC), **Prof. Vladimir V. Sobyenin** (NSU), **Prof. Vladimir V. Larichkin** (NSTU).

#### *Specialization:*

- ◆ Fundamentals of Catalytic Systems Preparation and Application
- ◆ Hydrogen Energy
- ◆ Technology for Fuel and Energy Production from Organic Raw Materials
- ◆ Treatment and Utilization of Technogenic Wastes Including Catalytic Methods of Environmental Protection
- ◆ Renewable Energy Sources
- ◆ Nanotechnologies and Nanomaterials
- ◆ Energy-Saving Processes and Apparatuses
- ◆ Catalytic Reactors Modeling.

The Educational Scientific Center “*Catalytic Hydrocarbon Processing*” is organized jointly with the *Gubkin Russian State University of Oil and Gas*, Moscow, in 2009. Executors: **Prof. Aleksandr S. Noskov** (BIC) and **Prof. Vladimir M. Kapustin** (Gubkin University).

#### *Specialization:*

- ◆ Study and Development of Catalytic Processes for Oil Refining and Petrochemistry
- ◆ Design of Efficient Catalytic Reactors for Oil Refining and Petrochemistry
- ◆ Fundamentals of Nanocatalysts Synthesis for Basic Processes of Oil Refining and Petrochemistry.

The Educational Scientific Center “*Chemical Technologies and Catalysis*” was made on the basis of the *Kazan State Technological University*, Kazan, in 2008. Executors: **Prof. Aleksandr S. Noskov** (BIC) and **Prof. Kharlampii E. Kharlampidi** (KSTU).

*Specialization:*

- ◆ Study and Development of Catalytic Processes of Oil Refining and Petrochemistry
- ◆ Design of Efficient Catalytic Reactors for Oil Refining and Petrochemistry
- ◆ Fundamentals of Nanocatalysts Synthesis for Basic Processes of Oil Refining and Petrochemistry
- ◆ Design and Investigation of Composite Polymer Materials.

The Educational Scientific Center “*Processes and Apparatuses of Chemical Technology*” was organized jointly with *NSTU* and *Open Joint-Stock Company “Novosibirsk VNIPIET”*, Novosibirsk, in 2008. Executors: **Prof. Zinfer R. Ismagilov** (BIC), **Prof. Gennadii G. Kuvshinov** (NSTU), **Prof. Yurii V. Ostrovskii** (OJSC “Novosibirsky VNIPIET”).

*Specialization:*

- ◆ Energy- and Resource-Saving Processes and Apparatuses of Chemical Technology
- ◆ Development of Technology and Apparatuses for Treatment and Utilization of Technogenic Wastes, Including Catalytic Methods for Environmental Protection
- ◆ Fundamentals and Technology of Catalytic Systems Design and Application
- ◆ Fundamentals and Apparatuses of Hydrometallurgical, Thermal and Catalytic Processes
- ◆ Nanotechnologies and Nanomaterials Including Composite Nanomaterials
- ◆ Hydrogen Energy
- ◆ Catalytic Reactors Modeling.

The Educational Scientific Center “*Catalytic Membranes*” was organized jointly with *NSU* and the *Budker Institute of Nuclear Physics*, Novosibirsk, in 2010. Executors: **Prof. Vladislav A. Sadykov** (BIC), **Prof. Vladimir A. Sobyenin** (NSU), **Prof. Nikolai A. Mezentsev** (BINP).

*Specialization:*

- ◆ Fundamentals of Controllable Synthesis of Nanocomposites and Nanocrystals as Prospect Materials for Production of a New Generation of Membranes
- ◆ Regularities of Formation of Layers with Membrane Properties from Nanocomposite and Nanocrystal Materials
- ◆ Phase, Chemical, Thermal and Structural Stability of Membranes
- ◆ Modeling of Processes in Membrane Reactors and Fuel Cells
- ◆ Development of Processes and Reactors Based on Membrane Materials
- ◆ Development of the Method for Man-Caused Wastes Purification Using Membrane Reactors.

The Educational Scientific Center “*Catalysis for Atmosphere Protection*” was organized jointly with the *Biysk Technological Institute*, a structural division of the *Polzunov Altai State*

*Technical University*, Barnaul, in 2010. Executors: **Prof. Aleksandr S. Noskov** (BIC), **Prof. Vladimir N. Khmelev** (BTI).

*Specialization:*

- ◆ Fundamentals of Preparation of Stable and Highly Active Catalysts for Waste Gases Neutralization
- ◆ Studying of the Mechanism and Kinetics of Oxidative Processes of Gases Purification
- ◆ Development of Technology for Catalytic Gases Purification in the Presence of Catalysts Poisons
- ◆ Physico-Chemical Bases for Ultrasonic Effect on Catalytic and Topochemical Reactions
- ◆ Modeling of the Catalytic Processes for Man-Caused Waste Gases Purification
- ◆ Design of Catalytic Reactors for Gases Purification
- ◆ Study of Aero(gas)dynamic Processes in Catalytic Reactors
- ◆ Development of Scientific Bases of Technology of Sorption and Catalytic Purification of Man-Caused Waste Gases upon Ultrasound Exposure.

The Educational Scientific Center “*Functional and Structural Carbon Nanocomposites*” was organized jointly with the *Vladimir State University*, Vladimir, in 2010. Executors: **Dr. Alexey A. Vedyagin** (BIC), **Prof. Sergey M. Arakelyan** (VISU).

*Specialization:*

- ◆ Fundamental and Applied Studies in the Field of Physics and Synthesis of Carbon Nanostructures for Their Further Utilization for Development of Novel Materials and Innovation Productions
- ◆ Scientific Bases for Production and Application of Catalytic Systems for Hydrocarbon Raw Materials Processing
- ◆ Development of Technology for Catalytic Hydrocarbon Pyrolysis
- ◆ Development of Technology and Catalysts for Production of Carbon Nanomaterials of Various Modification
- ◆ Potentiality of Carbon Nanostructures Application.

The Educational Scientific Center “*Natural and Synthetic Nanomaterials*” was organized jointly with the *Far Eastern Federal University*, Vladivostok, in 2010. Executors: **Dr. Alexey A. Vedyagin** (BIC) and **Dr. Kirill S. Golokhvast** (FESTU).

*Specialization:*

- ◆ Scientific Bases for Diagnostics of Toxic Properties of Synthetic and Natural Nanomaterials
- ◆ Development of a Standardized Definition of Parameters of Biosafety Upon Production and Exploitation of Nanotechnology Products
- ◆ Development of the Methods for Nanomaterials Synthesis and Isolation and Investigation of Their Physico-Chemical, Biological and Catalytic Properties
- ◆ Development of Perspective Directions of Catalytic Oil and Gas Treatment Using Nanomaterials
- ◆ Development of Method for Natural and Synthetic Nanomaterials Biotesting
- ◆ Synthesis of Carbon Nanomaterials from Hydrocarbon Raw Materials and Investigation of Their Nanotoxic Characteristics.

The Educational Scientific Center “*Physical Methods for the Investigation of Functional Materials in situ*” was organized jointly with *NSU* in 2010. Executors: **Prof. Oleg N. Martyanov** (BIC) and **Prof. Sergey V. Tsybulya** (NSU).

*Specialization:*

- ◆ Experimental and Theoretical Investigation of Structure and Properties of Functional Materials (Catalysts, Sorbents, Nanomaterials) Using Modern Physico-Chemical Methods
- ◆ Development of New Experimental and Computational Approach for Investigation of Functional Materials, Including *in situ*
- ◆ Experimental and Theoretical Investigation on the Atomic-Molecular Level of the Mechanisms of Catalytic Reactions, Structure and Properties of Intermediates and Active Surface Centers Using Modern Physico-Chemical Methods, Including *in situ*.

The Educational Scientific Center “*Structured and Hybrid Nanomaterials for Catalytic Processes*” was organized jointly with the *Southwest State University*, Kursk, in 2010. Executors: **Dr. Alexey A. Vedyagin** (BIC), **Prof. Aleksandr A. Kuzmenko** (SSU).

*Specialization:*

- ◆ Scientific Bases for Synthesis of Nanosized Particles of Metal Hybrids from Solutions
- ◆ Development of Method for Synthesis of Nanosized Platinum Group Metal and Hybrids on Their Base
- ◆ Development of Method for Synthesis of Nanosized Nickel Particles and Hybrids on Their Base
- ◆ Development of Prospect Methods for Utilization of Anodic Sludge
- ◆ Investigation of Physico-Chemical and Catalytic Properties of Nanosized Particles of Metal Hybrids.

**3 Joint Laboratories** were created with the purpose of integration of high education and fundamental science to increase the efficiency of joint efforts aimed at the training of high skilled specialists.

**Joint Laboratory of Catalytic Processes and Apparatuses** of the *Borshchov Institute of Catalysis* and the *Novosibirsk State Technical University*, created in 2002. Scientific Coordinators are **Prof. Aleksandr S. Noskov** (BIC) and **Prof. Gennadii G. Kuvshinov** (NSTU).

Main areas of scientific research of the Laboratory are:

- ◆ Mass and Thermo Transport Processes in Apparatuses for Chemical Engineering
- ◆ Design and Development of New Processes and Apparatuses for Energy- and Resource-Saving Chemical Technologies
- ◆ Development of the Scientific Basis for the Technology of Filament Carbon Synthesis
- ◆ Simulation of Moving Bed Apparatuses.

Joint **Laboratory of Sorption and Catalytic Processes** of the *Borshkov Institute of Catalysis* (Novosibirsk) and the *Butlerov Institute of Chemistry, Kazan State University, Kazan*, created in 2003. Scientific Coordinators are **Prof. Aleksandr S. Noskov** (BIC) and **Prof. Aleksandr A. Lamberov** (KSU).

Main areas of scientific research of the Laboratory are:

- ◆ Design of the Catalysts and Sorbents for Petrochemistry and Oil Refinery in Russia
- ◆ Scientific Basis of the Support Texture Formation within Industrial Synthesis and Working-Off
- ◆ Comparative Testing of Supports, Sorbents and Catalysts
- ◆ Development of New Highly Efficient Energy- and Resource-Saving Technologies for Petrochemistry and Oil Processing.

Joint **Laboratory of Engineering Chemistry** of the *Borshkov Institute of Catalysis* (Novosibirsk) and the *Volgograd State Technical University, Volgograd*, created in 2004. Scientific Coordinator is **Prof. Yurii V. Popov** (VolSTU).

Main areas of scientific research of the Laboratory are:

- ◆ Development of Novel Efficient Energy- and Resource-Saving Chemical-Technological Processes
- ◆ Design of Scientific Bases and Technology for Synthesis of Physiologically Active Substances
- ◆ Design of Scientific Bases and Technology for Heterogeneous Catalytic Processes for Chemicals Production
- ◆ Design and Study of Fuel and Oil Additives.

### ***Dissertation Council Activities***

In addition to teaching students in many Russian universities, researchers of the Institute participate in the training of young scientists of the highest qualification capable of conducting original basic and applied research in the field of catalysis. Their training has priority importance for the Institute and is related directly to its development and well being. The Institute solves these problems through the postgraduate school of SB RAS and postgraduate school of NSU. Annually 20-30 postgraduates are trained at the graduate school working at the Institute. Supervised by the researchers of the Institute, postgraduate students conduct their Ph.D. studies in chemical kinetics and catalysis, physical chemistry, chemical engineering in the BIC laboratories.

Dissertation Council D 003.012.01 was confirmed by The Order of Rosobrnadzor on May, 2010. Dissertation Council is allowed to uphold a thesis to receive an academic degree of Doctor of Science and Ph.D. on specialities: 02.00.15 "Kinetics and Catalysis" within chemical sciences and 02.00.04 "Physical Chemistry" within chemical sciences. Head of the Dissertation Council is Prof. V. Parmon.

Dissertation Council DM 003.012.02 was confirmed by The Order of Rosobrnadzor on July, 2010. Dissertation Council is allowed to uphold a thesis to receive an academic degree of Doctor of Science and Ph.D. on specialties: 02.00.15 “Kinetics and Catalysis” within chemical and technical sciences and 05.17.08 “Processes and Apparatuses for Chemical Technology” within technical sciences. Head of the Dissertation Council is Prof. Vladimir A. Sobyenin.

10 Ph.D. theses were defended in 2010:

**Roman V. Gulyaev** – *“Interaction of Palladium with the Surface of Ce-Containing Supports and Role of Surface Phases in Low Temperature CO Oxidation”*

**Ivan N. Voropaev** – *“Development and Formation of Pt/C Catalysts for Low Temperature Fuel Cells”*

**Sophia A. Khromova** – *“Studying of Catalytic Deoxygenation of Model Biooil Compounds”*

**Vasilii Yu. Kruglyakov** – *“Iron Oxide Monolith Honeycomb Catalyst for Ammonia Oxidation and Technology of Its Production”*

**Oleg Yu. Lyakin** – *“Application of NMR and ESR Spectroscopy to Study Structure and Reactivity of Fe and Zr Complexes – Active Particles for Homogeneous Catalytic Oxidation and Polymerization”*

**Igor E. Soshnikov** – *“Formation and Nature of Active Centers of Vanadium and Cobalt Oxide Catalysts for Olefin Polymerization According to NMR and ESR Data”*

**Sergey A. Prikhodko** – *“Studying of the Hydrodefluorination of Pentafluoroaniline and Its Derivatives in the Presence of Nickel Complexes”*

**Olga A. Bulavchenko** – *“Structural Aspects of Oxide Alumocobalt and Alumomanganese Catalysts Activation”*

**Tatiana Yu. Kardash** – *“Structure and Physicochemical Properties of Multicomponent Catalysts Based on Polygonal-Cellular Molybdenum Oxides”*

**Sergey I. Moseenkov** – *“Synthesis and Investigation of the Onion-Like Carbon and Its Composites”*.

There is a great demand for specialists in catalysis trained at the Boreskov Institute of Catalysis. Many of them apply their knowledge in various research centers, universities and manufacturing companies.

## SCIENTIFIC SOCIAL LIFE

- ❖ According to Resolution of the SB RAS Presidium on the results of the *Lavrentiev competition of SB RAS* youth projects three projects from Boreskov Institute of Catalysis became the winners:

**E.A. Kozlova, D.V. Kozlov, P.A. Kolinko, M.N. Lyulyukin, D.S. Selishchev, D.E. Tsydenov** – “*New Composite TiO<sub>2</sub> Based Inorganic Materials for Solar Light Energy Utilization*”

**E.N. Gribov, O.V. Netskina, I.M. Krivobokov, A.M. Ozerova, A.I. Lysikov** – “*Development of Highly Efficient Catalysts and Membrane-Electrode Assemblies for Energy Generators Based on Borohydride Hydrogen Storage*”

**A.A. Lysova** (BIC, International Tomography Center) – “*Development of NMR Tomography for in situ Investigation of Catalysts and Catalytic Processes*”.

- ❖ **Dr. Anna Lysova** (BIC, International Tomography Center) was awarded a **Medal of the Russian Academy of Sciences for young scientists** for her work “*Development of the Applications of NMR Spectroscopy for Catalysis*”.
- ❖ **Dr. Ilya Mishakov** became one of the winners of **Competition for grants to young scientists and specialists in the field of innovation activity** for his work “*Synthesis of High Surface Area Carbon Nanofibres for Modification of Materials Applied for Road-Building*”.
- ❖ June, 9, 2010, Dmitry Medvedev signed Executive Order **On Awarding the 2009 Russian Federation National Awards in Science and Technology**.

The document states, in part: “Award the 2009 Russian Federation National Award in Science and Technology and the honorary title of laureate of the 2009 Russian Federation National Award in Science and Technology to:

**Academician Valentin Parmon**, director of the Boreskov Institute of Catalysis, for his significant contribution to the development of theory and practice of catalytic methods for deep processing of hydrocarbon feedstocks and use of renewable resources.”



- ❖ **Dr. Julia Knights**, the First Secretary and the Head of Science and Innovation Section, and **Mikhail Lachinov**, the Senior Counselor of the Science and Innovation Department of the



British Embassy in Moscow, visited Novosibirsk Akademgorodok on June 15, 2010. The program included a meeting with Academician V.M. Fomin – the Vice-President of the Siberian Branch of the Russian Academy of Sciences – to discuss the scientific capacity of Siberia and potential areas of the further cooperation. After the discussion, the British diplomats visited to the Budker Institute of Nuclear Physics and the Boreskov Institute of Catalysis.

- ❖ Scientific Coordination Councils of Programs “Telecommunications and Multimedia Resources of the SB RAS” and “The SB RAS Information Resources” announced a competition of official WEB sites of institutes and organizations of the Siberian Branch of the RAS. The Boreskov Institute of Catalysis has been announced **The Winner of WEB Site Competition** in the area of chemical sciences.



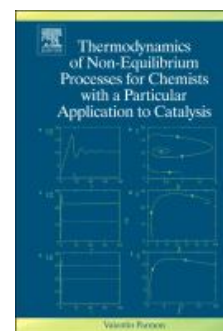
- ❖ As part of the Skolkovo Innograd Project, the Foundation for Development of the Center of Research and Commercializing of New Technologies was officially registered in Moscow. This is a nonprofit organization purposed to provide socially useful results in the development of innovations. **Academician Valentin Parmon** became a *member of the Scientific and Advisory Council* of the Foundation for Development of the Center of Research and Commercializing of New Technologies in Skolkovo.

- ❖ October, 22, a solemn ceremony of handing certificates to the winners of 2010 **competition on awarding grants of the President of the Russian Federation**, aimed at the state support of the young Russian scientists - PhDs and Doctors of Sciences – took place in the House of Sciences of the SB RAS on October 22. Among the winners – **Dr. Pavel V. Snytnikov (BIC)**.

- ❖ The Guardian Council and the Expert Committee of the Russian Science Support Foundation made public the names of the winners of “**The Best RAS Postgraduate Students**” Contest for fellowship in the field of humanities and sciences. The fellowship contest was organized for the postgraduate students of the RAS institutions and the leading Russian higher educational institutions. Among the 200 winners are two postgraduates from the Boreskov Institute of Catalysis: **Olga A. Nikolaeva** and **Mariya M. Zyryanova**.



- ❖ **Academician V. Parmon** published the synopsis of lectures on thermodynamics of non-equilibrium processes and a particular course on thermodynamics of operating catalysts “*Thermodynamics of Non-Equilibrium Processes for Chemists with a Particular Application to Catalysis*”, Elsevier Science, 2010, 340 pp.



“Thermodynamics of non-equilibrium processes is a comparatively new area of thermodynamics. Traditionally this discipline is taught only to chemistry students who have a very strong background in physics. The author of the present book has adapted his course of thermodynamics of non-equilibrium processes so that the subject can be treated in terms understandable to any chemist with a formal physicochemical education in the fields of classical thermodynamics of equilibrium processes and traditional chemical kinetics. The discipline combines thermodynamics and chemical kinetics and is helpful to researchers engaged in studying complex chemical transformations, in particular, catalytic transformations. For example, important concepts for such studies are conditions of kinetic irreversibility of complex stepwise stoichiometric reactions, rate-determining and rate-limiting stages, etc. In traditional chemical kinetics these concepts are not very clear and tend to be 'concealed' in courses. Fortunately, these concepts appear to be consistently and properly defined in terms of thermodynamics of non-equilibrium processes. The book applies simple approaches of non-equilibrium thermodynamics to analyzing properties of chemically reactive systems. It also covers systems far from equilibrium, allowing the consideration of most chemically reactive systems of a chemical or biological nature. This approach resolves many complicated problems in the teaching of chemical kinetics”. (Publisher's description).

- ❖ On December 6-7, the annual **Competition for the best scientific research work** took place at the Boreskov Institute of Catalysis.

**The first prize winner:**

**DEVELOPMENT OF AN ECOLOGICALLY SOUND TECHNOLOGY FOR  
CO-POLYMERIZATION OF PERFLUOROSULFO MONOMER WITH  
TETRAFLUOROETHYLENE FOR PROTON-CONDUCTING MEMBRANES  
OF FUEL CELLS**

**S.S. Ivanchev, O.N. Primachenko, S.Ya. Khaikin, V.P. Tyul'mankov,  
V.S. Likhomanov, V.S. Misin**

The current status of research in the field of preparation, structure modification and practical applications of polymer membranes for fuel cells is inspected. Particular attention is paid to physicochemical characteristics and specific features of the composition, microphase separation and structure of proton-conducting Nafion-type material and its analogues. On analyzing patent literature on the Nafion synthesis, the water-emulsion process for copolymerization of tetrafluoroethylene with perfluorine(3,6-dioxo-4-methyl-7-octene)sulfonyl fluoride (FS-141) seems of particular interest. As a result, we have developed a process for synthesis of Nafion with the optimal equivalent mass and high conversion (ca. 70 %) of the expensive sulfomonomer characteristic of it. The synthesized materials are applicable for manufacturing of proton conducting membranes that feature properties competitive to those of standard Nafion samples. The mechanism of the emulsion copolymerization of the fluorinated monomers under study is suggested, which involves topochemistry of the elementary reactions of copolymerization and formation of disperse latex particles.

The developed technology is tested at the Russian National Center “Prikladnaya Khimiya” using a pilot plant with a 5 L reactor to produce more than 7 kg of the copolymer. The technology is covered by

Russian Patent 2 348 649, the application for World Patent was filed; it was presented at international exhibitions (Paris, Minsk, Moscow, 2009–2010).

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*Zh. Prikladnoi Khimii*. **2008**. V. 81. No 4. pp. 529-545 (Russ.)
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Polymer Membranes for Fuel Cells: Preparation, Structure, Modification, Properties (Review paper),  
*Uspekhi Khimii*. **2010**. V. 79. No 2. pp. 117-134 (Russ.)
3. **S.S. Ivanchev, O.N. Primachenko, (V.N. Pavluchenko), S.Ya. Khaikin, (V.A. Trunov)**,  
Polymer Membranes Based on Multicomponent Copolymers,  
*Zh. Prikladnoi Khimii*. **2008**. V. 81. No 7. pp. 1134-1140 (Russ.)
4. **(V.A. Trunov, V.T. Lebedev, A.E. Sokolov, I.I. Ivanov, V.B. Rybakov), T.M. Yurieva, S.S. Ivanchev, (D. Torok)**,  
Some Potentialities of Neutron Methods for Studying Materials and Components of Devices Used in Hydrogen Energetics,  
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### The second prize winners:

#### MECHANISMS OF ACTIVATION AND TRANSFORMATION OF LIGHT ALKANES OVER Zn- AND Ga-CONTAINING $\beta$ -ZEOLITE (FROM *in situ* NMR DATA)

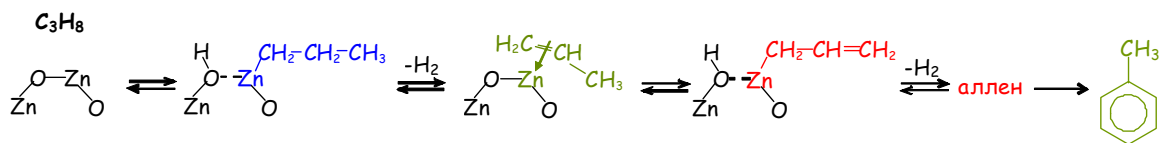
**A.A. Gabrienko, S.S. Arzumanov, A.V. Toktarev, M.V. Luzgin, A.G. Stepanov**

Zinc- and gallium-containing high-silica zeolites are effective catalysts for aromatization of light alkanes. While these alkanes are very stable, of particular interest for studies is the early stage of their transformation, viz. the activation stage. Establishment of the role of acidic sites and of zinc or gallium introduced in the zeolite is an important step towards understanding the mechanism and towards the further improvement of their activity and selectivity to the reaction of aromatization.

In the present work, *in situ* solid-state  $^1\text{H}$  and  $^{13}\text{C}$  NMR techniques were used for studying mechanisms of activation of C1-C4 alkanes over Zn/H-BEA and Ga/H-BEA zeolites, transformation of ethane and propane into aromatic products and into by-product (methane) over Zn/H-BEA zeolite. The mechanism of co-aromatization methane and propane over Ga/H-BEA zeolite was studied, too.

Zinc and gallium were shown to promote activation of the C–H bond of alkanes on acidic site of the zeolite. This influence was accounted for by the preliminary dissociative adsorption of the alkane molecules on zinc and gallium oxide species to form Zn- and Ga-alkyl fragments.

The use of  $^{13}\text{C}$ -labeled reactants allowed the intermediates and products of ethane and propane aromatization to be identified on the Zn/H-BEA zeolite. From the data obtained, the transformation of propane is mediated by formation of *n*-propyl-zinc,  $\pi$ -complexes of propylene with zinc and  $\sigma$ -allyl-zinc species.



It is established that the main route to the formation of methane as a by-product of hydrocarbon aromatization is the direct hydrogenolysis of initial alkanes.

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## THE ROLE OF PHASE AND CHEMICAL COMPOSITIONS OF THE MoVTeNb OXIDE SYSTEM IN OXIDATION AND OXIDATIVE AMMONOLYSIS OF PROPANE

**E.V. Ishchenko, T.V. Andrushkevich, G.Ya. Popova, G.I. Aleshina, L.M. Plyasova, V.V. Malakhov, T.Yu. Kardash, L.S. Dovlitova, Yu.A. Chesalov, A.V. Ishchenko**

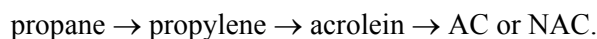
Selective transformation of propane into acrylic acid and acrylonitrile is a promising and economical process against the traditional industrial synthesis of acrylonitrile (NAC) and acrylic acid (AC) from propylene.

At present, mixed MoVTeNb oxide catalysts are the most promising for oxidation of propane to AC and oxidative ammonolysis to NAC.

The studies were aimed at the developing reproducible method for synthesis of the catalyst and to understanding the role of phase and chemical compositions of the MoVTenb oxide catalysts in oxidative transformations of propane.

The mechanism of the formation of the MoVTenb oxide catalyst was established, intermediate compositions and structures determined at every stages of the catalyst preparation, conditions of all the stages optimized.

The transformation of propane into AC or NAC follows a consecutive mechanism mediated by the formation of propylene and acrolein:



The highest activity and selectivity to these reactions are observed with the catalysts with maximal content of an orthorhombic M1 phase. The M1 phase is unique in the presence and optimal combination of active sites for the formation of intermediates and their transformation into the target product.

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### APPLICATION OF <sup>95</sup>Mo AND OTHER NUCLEI NMR FOR STUDYING SOLUTIONS AT SYNTHESIS OF MULTICOMPONENT CATALYSTS BASED ON Mo AND V OXIDES

**R.I. Maksimovskaya, V.M. Bondareva, G.M. Maksimov, G.I. Aleshina**

Multicomponent catalysts based on Mo and V oxides are used for oxidative transformations of various hydrocarbons. The MoVNb and MoVTenb oxide catalysts are most promising for transformations of light alkanes; their efficiency is determined by specific crystalline phases where the components at various valence states are mixed at the atomic level. The formation of these phases needs careful choice and accurate control of the conditions of multistage synthesis of the catalyst. <sup>125</sup>Te, <sup>95</sup>Mo, <sup>51</sup>V and <sup>17</sup>O NMR techniques were used for studying synthetic solutions in a wide range of conditions.

Aqueous molybdate solutions were pre-studied to determine potentialities of  $^{95}\text{Mo}$  NMR in examining systems containing  $\text{Mo}^{\text{VI}}$  as a predominant and structure-forming component. Vanadate and mixed  $\text{Mo}^{\text{VI}}\text{-V}^{\text{V}}$  solutions were studied earlier. In the present work, the studies were focused on changes in the solution compositions caused by addition, in different orders,  $\text{Nb}^{\text{V}}$  oxalate, oxalic acid, telluric acid. In triple  $\text{Mo}^{\text{VI}}\text{-V}^{\text{V}}\text{-Te}^{\text{VI}}$  solutions, there were observed complexes  $\text{TeMo}_6\text{O}_{24}^{6-}$ , identified for the first time  $\text{TeV}_5\text{Mo}_3\text{O}_{27}^{5-}$ ,  $\text{TeMo}_3\text{V}_3\text{O}_{24}^{9-}$ ,  $\text{Mo}_7\text{VO}_{26}^{5-}$  and  $\text{V}_9\text{TeO}_{28}^{5-}$ , and pH region of their stability established. On addition of Nb oxalate to the solution,  $\text{VO}_2^+$  and  $\text{MoO}_2^{2+}$  oxalates become observed. Nb is not comprised in the complexes but forms  $\text{Nb}_2\text{O}_5 \cdot n\text{H}_2\text{O}$  gel. When a special drying mode is provided, the gel prevents from isolation of individual phases but favors formation of a homogeneous product with all the components distributed homogeneously through it, that is necessary for the formation of the target mixed oxide phases during the further thermal treatment.

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❖ On August 4, 1997, **Zamaraev International Charitable Scientific Foundation** was organized in the memory and honor of Professor Kirill I. Zamaraev.

The main Founders of the Foundation from the Russian side are:

- Borekov Institute of Catalysis, Novosibirsk
- Institute of Semiconductors Physics, Novosibirsk
- Russian D.I. Mendeleev Chemical Society, Moscow
- Scientific-Industrial Company «Altay», Biysk
- «Ecogeochem Ltd.», Moscow
- Association «Russian House for International Scientific and Technological Cooperation»
- Other scientific, commercial and financial institutions.

The Foundation is supported by IUPAC and some Russian sponsors.

The Zamaraev Foundation is grateful to all sponsors for their contribution and invites Russian and foreign organizations (as well as wealthy persons) to become the honorary participants of the campaign.

The main aim of the Foundation is to support gifted Russian scientific youth working in the field of chemical catalysis, physical methods of its research and chemical physics.

Major part of Foundation budget is spent for Zamaraev post-graduate scholarships, prizes and grants. The Foundation plans to support international cooperation of scientific youth in the field of chemical catalysis and chemical physics, i.e., their participation in international scientific conferences and seminars. In 2010 **A. Gabrienko, D. Demidov, A. Miller** got the post-graduate scholarship.

- ❖ 17 Ph.D. students and reserach fellows of the Institute are members of the **Council of Scientific Youth**. The main principle of the Council is to represent interests of young scientists in the Institute. These are organization of competition of the youth projects, support of the young lecturers of higher educational establishments – employees of the Institute, organization of training for qualifying examinations for the Ph.D. degree, improvement of living conditions of the young scientists, and other activity areas to improve positions of young researchers in the Institute.

## Memorial Rooms of Academician Georgii K. Borekov and Academician Kirill I. Zamaraev

The **Memorial Room of Academician Georgii K. Borekov**, the founder and first director of the Institute of Catalysis, was opened in June 1987, on the occasion of his 80<sup>th</sup> anniversary. Georgii Konstantinovich always emphasized that the foundation of the Institute was the most important cause of his life. In 1991, the Institute of Catalysis was named after Georgii K. Borekov.



There is a small exhibition based on the documents, awards, books, Borekov's belongings in one of the rooms of Laboratory of Oxidation, which was headed by Georgii Konstantinovich.

A series of photos dated 1930's through 1960's tell about an extremely intense and fruitful activity of G.K. Borekov in the fields of research and science management. He headed the Laboratory of Catalysis at the Chemicoradiological Institute (Odessa) in 1932 through 1937 (the laboratory registry dated 1931 with records by Georgii Konstantinovich concerning the preparation and characterization of vanadium catalysts was kept); the Laboratory of Catalysis at the Research Institute for Fertilizers and Insecto-Fungicides (Moscow) in 1937 through 1946, the Laboratory of Technological Catalysis at the Karpov Institute of Physical Chemistry (Moscow), and in 1958 became the Director of the Institute of Catalysis (Novosibirsk).

For his contribution to the Russian science, distinguished scientific, educational, managing and social activities, his great role in establishing the Siberian Branch of the USSR Academy of Sciences, Georgii Borekov was awarded the Badge of Honor, three Orders of Lenin, Order of the Red Banner of Labor, Golden Medal of Hammer and Sickle, he was twice USSR State Prize winner, Ukrainian SSR State Prize winner, he was awarded the title of a Hero of Socialist Labor. Among the others, these decorations are exhibited in the Museum.

Borekov's activity in the area of catalysis was recognized worldwide. He was a foreign member of the National Academy of Sciences of German Democratic Republic, honorary member of the New York Academy of Sciences, Doctor HONORIS CAUSA of the Wroclaw Polytechnic Institute (Poland) and of the Poitiers University (France). Government of Bulgarian Republic awarded the Order of Kirill and Mepodium to G.K. Borekov. Visitors can see an unusual exposition displaying the full attributes of ceremonies of awarding the HONORIS CAUSA titles to Georgii Borekov in Wroclaw in 1976 and in Poitiers in 1981, these are black mantles and caps, an ermine scarf and leather tubules for storing the Diplomas.

Of particular interest among the exhibit is the personal book collection of Georgii Konstantinovich including more than 1000 scientific volumes and 15 sets of Russian and foreign scientific journals. There are especially memorable copies with dedicatory inscriptions by the authors and the rarities published in the end of XIX and beginning of XX century in the collection. The books capable of telling about Borekov's educatory activity are displayed in one of the showcases, these are course descriptions and lecture summaries, books used for preparing the lectures, and the like.

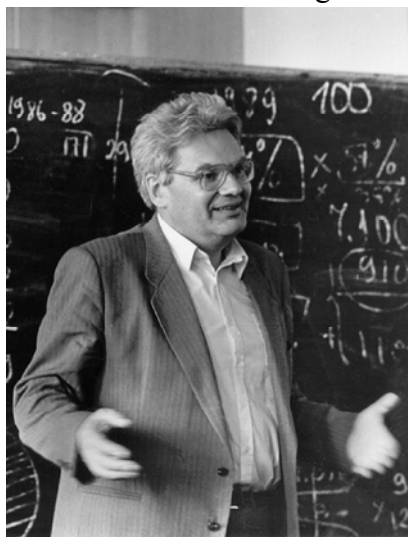




There is his writing table, case, bureau and an old push-button typewriter.

Albums, arranged by the colleagues and friends in different periods of time and presented by foreign colleagues, tell about numerous scientific events, where Boreskov presented lectures, about his keenness to winter fishing and to summer “mushroom hunting”, as well as about many other interesting events.

We see the same Georgii Konstantinovich in the portraits and photos, as he was – an intellectual and charming individual.



The **Memorial Room of Academician Kirill I. Zamaraev**, the Director of the Institute of Catalysis from 1984 through 1995, was opened on the 20<sup>th</sup> of May 1999, the day of 60<sup>th</sup> anniversary of K. Zamaraev. Establishment of the Memorial Room was promoted by the International Charitable Zamaraev Foundation and directly participated by Kirill Ilyich’s widow, Lyudmila Aleksandrovna Zamaraeva. This small museum exposition relates to the life and activities of Academician Kirill Ilyich Zamaraev, who was a hereditary scientist.

Diplomas, awards, photo archives and belongings of Kirill Ilyich are displayed in showcases. A unique scientific book collection gathered by him is also available here.

Kirill demonstrated his brilliant potentialities in sciences as early as his school years. He finished school as the Golden Medal winner; the medal is also stored in the Museum. He continued his education in the Mendeleev Institute of Chemical Technology in Moscow, but after the third year moved to the Moscow Physical and Technological Institute (MPhTI) in order to get a strong

background in chemical physics. In the Museum, there are laboratory registries of post-graduate student Kirill Zamaraev and EPR spectra recorded by him during his post-graduate years.

The Moscow period (1956-1976) of his life and work is illustrated by the photo expositions: here is Kirill Zamaraev as a junior, then senior researcher at the Institute of Chemical Physics of the USSR Academy of Sciences; he heads a laboratory and, at the same time, he is an assistant professor in MPhTI.

Early in 1977 Kirill Ilyich was invited by Academician G.K.Boreskov; with his family and a team of young MPhTI graduates, he moved to Novosibirsk. Owing to the activity of Kirill Zamaraev and his colleagues, investigations of mechanisms of catalytic reactions at the atomic and molecular levels were considerably extended and intensified in the Institute of Catalysis.

He combined his vast research and management activity with teaching in the Novosibirsk State University.



The decade from mid-1980's to mid-1990's was the time of international recognition of his scientific achievements. The exhibition demonstrates his state awards, honorary medals of the USSR Academy of Sciences, as well as the certificate of full member of the USSR Academy of Sciences. He became a foreign member of Indian and Korean Academies of Sciences, awarded the Silver Medal by the Royal Chemical Society as an "Outstanding Lecturer of the Century", and was elected a member of Academy of Europe. In 1994 K.I. Zamaraev became a President of IUPAC, International Union of Pure and Applied Chemistry, and in 1995 was honored with the German prestigious Karpinski Award for Russian Scientists. There are diplomas and medals, as well as a number of photos of that time in the Museum.

Everyone, who knew personally Kirill Ilyich, remember his exceptional ability not to loose courage at any, even very severe, circumstances. He had a really iron character, and it was not without vain that the sport he went in for was mountaineering.

A smiling, talented, brilliant and courageous man appears before visitors to the Memorial Room.

**IMPORTANT RESULTS  
OF THE R&D ACTIVITY**

# **RESEARCH ACTIVITY**

## Quantum-Chemical Investigations

### QUANTUM-CHEMICAL STUDY OF PROPANE TRANSFORMATION OVER ALUMINIUM AND COBALT CHLORIDES

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*Russ. Chem. Bull.*,  
10 (2010) pp. 1821-1830.

Density functional PBE/TZ2p quantum chemical calculations of activated complexes and pathways of model catalytic transformations of propane under the action of aluminum chloride - cobalt chloride ionic bimetallic complexes were carried out. The formation of an intermediate with a broken C-C bond can occur on the cationic cluster  $\text{CoAlCl}_4^+$  characterized by the strongest coordination of propane molecule. The activation barrier to the reaction is  $\Delta G = 25.0 \text{ kcal}\cdot\text{mol}^{-1}$ . Activation of alkane C-H bonds follows the alkyl pathway involving the formation of bimetallic alkyl complexes. The interaction of activated hydrocarbon fragments bound to transition metal atoms in cobalt-chloroaluminate clusters can result in alkane metathesis products (in this case, ethane and a polymetallic cluster containing an extended chain alkyl radical).

### ACTIVATION OF MOLECULAR OXYGEN IN TRIFLUOROACETIC ACID

M.V. Vishnetskaya\*, A.V. Vasin\*, V.N. Solkan\*\*, G.M. Zhidomirov, M.Ya. Mel'nikov\*\*\* (\*Gubkin Russian State University of Oil and Gas, Moscow, Russia; \*\*Zelinsky Institute of Organic Chemistry, Moscow, Russia; \*\*\*Moscow Lomonosov State University, Moscow, Russia)

*Russ. J. Phys. Chem. A*,  
84(11) (2010) pp. 1905-1907.

The interaction of molecular oxygen with aqueous trifluoroacetic acid (TFA) led to an increase in pH. This effect was explained by a decrease in the concentration of the protonated  $\text{CF}_3\text{CO}_2\text{H}_2^+$  and  $\text{H}_3\text{O}^+$  species after oxygen was fed in the reactor. Quantum-chemical calculations show that a radical pair can be formed in an activation-free exothermal reaction involving the radical residue of the acid, the  $\text{CF}_3\text{CO}_2\text{H}_2^+\cdots^3\text{O}_2\cdots\text{CF}_3\text{CO}_2^-$  peroxide radical, and the acid molecule in the  $\text{CF}_3\text{CO}_2\cdots\text{HOO}\cdot$  collision complex. It was assumed that the activation of molecular oxygen in aqueous TFA solutions, providing the activity of the system in oxidations of

various organic and inorganic substrates, is related to the formation of peroxide radicals in them.

### THE MECHANISM OF FORMATION OF THE HYDROPEROXYL RADICAL IN THE $\text{CF}_3\text{COOH} + ^3\text{O}_2$ SYSTEM: A QUANTUM-CHEMICAL STUDY

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*Russ. J. Phys. Chem. B*,  
4(5) (2010) pp. 705-708.

*Ab initio* quantum-chemical calculations of the  $(\text{CF}_3\text{CO}_2\text{H}_2^+\cdots^3\text{O}_2)$  and  $(\text{CF}_3\text{CO}_2^-\cdots^3\text{O}_2)$  complexes were performed by the MP2 method. It was found that these complexes were characterized by low complex formation energies, of 2.97 and 1.72 kcal/mol, respectively. According to the MP2(full)/6-311++G(d, p) calculation data, the bridge stabilization of oxygen by linking with both the  $\text{CF}_3\text{CO}_2\text{H}_2^+$  cation and  $\text{CF}_3\text{CO}_2^-$  anion is much more favorable energetically. A study of the potential energy surface of the joint molecular system  $(\text{CF}_3\text{CO}_2\text{H}_2^+\cdots^3\text{O}_2\cdots\text{CF}_3\text{CO}_2^-)$  shows that proton experiences activationless transfer from the cation to the  $^3\text{O}_2$  molecule accompanied by electron transfer from the  $\text{CF}_3\text{COO}^-$  anion. An analysis of spin density distribution shows that two radicals are stabilized in the  $(\text{CF}_3\text{CO}_2\cdots\text{OOH}\cdots\text{O}=\text{C}(\text{OH})\text{CF}_3)$  complex in the triplet state observed on the potential energy surface.

### ION-EXCHANGED BINUCLEAR $\text{Ca}_2\text{O}_x$ CLUSTERS, $x=1-4$ , AS ACTIVE SITES OF SELECTIVE OXIDATION OVER MOR AND FAU ZEOLITES

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*J. Comput. Chem.*,  
31(2) (2010) pp. 421-430.

A new series of calcium oxide clusters  $\text{Ca}_2\text{O}_x$  ( $x = 1-4$ ) at cationic positions of mordenite (MOR) and faujasite (FAU) is studied via the isolated cluster approach. Active oxide framework fragments are represented via 8-membered window (8R) in MOR, and two 6R and 4R windows (6R+4R) possessing one common Si-O-Si moiety in FAU. Structural similarities between the  $\text{Ca}_2\text{O}_x$  (8R) and  $\text{Ca}_2\text{O}_x$

(6R+4R) moieties are considered up to  $x = 4$ . High oxidation possibilities of the  $\text{Ca}_2\text{O}_2$  (nR) and  $\text{Ca}_2\text{O}_3$  (nR) systems are demonstrated relative to CO, whose oxidation over the Ca-exchanged zeolite forms is well studied experimentally. Relevance of the oxide cluster models with respect to trapping and desorption of singlet dioxygen is discussed.

#### STRUCTURE OF CHLORINE ON Ag(111): EVIDENCE OF THE (3×3) RECONSTRUCTION

**B.V. Andryushechkin\***, **V.V. Cherkez\*\*\*\***, **E.V. Gladchenko\*\*\*\***, **G.M. Zhidomirov**, **B. Kierren\*\***, **Y. Fagot-Revurat\*\***, **D. Malterre\*\***, **K.N. Eltsov\*\*\*\*** (\*International Joint Laboratory IMTAS, A.M. Prokhorov General Physics Institute, Moscow, Russia; \*\*International Joint Laboratory IMTAS, Université H. Poincaré–Nancy, Vandoeuvre les Nancy, France; \*\*\*Moscow Institute of Physics and Technology, Moscow Region, Russia)

*Phys. Rev. B*,  
81 (2010) 205434 (5 pages).

The structure of the chlorine induced reconstruction of Ag(111) has been studied by a combination of low-temperature scanning tunneling microscopy (STM), low-energy electron diffraction (LEED), and density-functional theory (DFT). It was demonstrated that previously observed mysterious LEED pattern arises as a result of diffraction from a system of small Å (15–30 triangular antiphase domains with a new (3×3) superstructure. In the author's model supported by DFT calculations, within a (3×3) unit cell the upper silver layer reconstructs forming a couple of three-atom triangles placed in fcc and hcp sites of the substrate. Chlorine atoms occupy fourfold hollow sites between these triangles. The corner holes, which look like depressions in the STM images, are also occupied by chlorine atoms.

#### ON MECHANOCHEMICAL DIMERIZATION OF ANTHRACENE. DIFFERENT POSSIBLE REACTION PATHWAYS

**V.M. Tapilin**, **N.N. Bulgakov**, **A.P. Chupakhin\***, **A.A. Politov\*\***, **A.G. Druganov\*\*\*** (\*Novosibirsk State University, Novosibirsk, Russia; \*\*Institute of Solid State Chemistry and Mechanochemistry, Novosibirsk, Russia; \*\*\*Novosibirsk Institute of Organic Chemistry, Novosibirsk, Russia)

*J. Struct. Chem.*,  
51(4) (2010) pp. 635–641.

Calculations of the anthracene crystal structure, 10% isotropic compression of the anthracene crystal and its two dimers linked not through the central atoms of central rings are performed in the density functional

theory approximation. Linear lattice parameters  $a$ ,  $b$ ,  $c$ , interatomic distances, and bond angles coincide with those determined by single crystal X-ray diffraction and previously calculated for an isolated anthracene molecule. The parameter  $\gamma$  is different by  $12^\circ$ , which is due to a weak dependence of the lattice energy on  $\gamma$  (the energy of only a few kcal per mole is required to turn the lattice at this angle). The calculated lattice energy (15 kcal/mol) is close to the enthalpy of sublimation. Dimers of another configuration than those linked through the central atoms of central rings are less energetically favorable. The formation of dimers at high pressure and shear deformation of “sandwiches” composed of anthracene molecules located above each other is shown, and a two-step dimerization scheme is proposed.

#### TO MECHANOCHEMICAL DIMERIZATION OF ANTHRACENE. CRYSTALLINE PHENANTHRENE UNDER HIGH PRESSURE AND SHEAR CONDITIONS

**A.A. Politov\***, **A.P. Chupakhin\*\***, **V.M. Tapilin**, **N.N. Bulgakov**, **A.G. Druganov\*\*\*** (\*Institute of Solid State Chemistry and Mechanochemistry, Novosibirsk, Russia; \*\*Novosibirsk State University, Novosibirsk, Russia; \*\*\*Novosibirsk Institute of Organic Chemistry, Novosibirsk, Russia)

*J. Struct. Chem.*,  
51(6) (2010) pp. 1064–1069.

Calculations at the density functional theory level show that for phenanthrene, unlike anthracene, stable dimeric structures are absent. A study of the absorption and photoluminescence spectra of crystalline phenanthrene under a pressure to 30 kbar and a simultaneous shear reveals reversible changes in the spectra: a shift of absorption bands and luminescence to the long wave region, a decrease in the intensity, and disappearance of the vibrational structure. Unlike anthracene, the dimerization (and other irreversible transformations) of phenanthrene under the simultaneous effect of high pressure and shear is not observed.

#### ELECTRONIC STRUCTURES OF MIXED IONIC-ELECTRONIC CONDUCTORS $\text{SrCoO}_x$

**V.M. Tapilin**, **A.R. Cholach**, **N.N. Bulgakov**

*J. Phys. Chem. Solids*,  
71(11) (2010) pp. 1581–1586.

Electronic structures of  $\text{SrCoO}_x$  with  $x=3$ , 2.875 and 2.75 were calculated by DFT technique in SLDA approximation. Two kinds of oxygen vacancy ordering with energies of 0.22 and 0.01 eV lower in comparison with random vacancy distribution were

revealed. The transition between these ordered vacancy systems with the activation energy 0.34 eV can be a step in the ionic conductivity mechanism. The calculated ion charges, magnetic moments and electron density of states were used to analyze chemical bonding in the crystals. All calculated compounds have metal electronic conductivity.

#### **WATER EFFECT ON THE ELECTRONIC STRUCTURE OF ACTIVE SITES OF SUPPORTED VANADIUM OXIDE CATALYST VO<sub>x</sub>/TiO<sub>2</sub>(001)**

**V.I. Avdeev, V.M. Tapilin**

*J. Phys. Chem. C*,  
114(8) (2010) pp. 3609-3613.

The interaction of water molecules with the reduced and fully oxidized surface sites of the supported vanadium oxide catalyst VO<sub>x</sub>/TiO<sub>2</sub> has been investigated by the Periodic DFT method. It has been found that the molecular structures of the surface VO<sub>x</sub> species are radically altered when adsorbed water is involved in the redox cycle. Water dissociates spontaneously on the reduced vanadium sites forming the surface hydroxyl groups OH. The following reoxidation by gas-phase oxygen results in the formation of active sites O=VO<sub>2</sub>(OH) including both the Bronsted acid sites OH and the vanadyl oxygen V=O more reactive than on the dehydrated surface. Gas-phase oxygen, embedded on the surface under oxidation, does not take part in the formation of surface hydroxyl groups. The hydroxylation-hydration reaction path on the fully oxidized VO<sub>x</sub>/TiO<sub>2</sub> surface has been calculated. It has been found that the recombination reaction of the two surface hydroxyl groups V-OH to form water with the following water desorption prevails over the reverse reaction of hydroxylation. In agreement with the experimental data it was concluded that lattice oxygen of surface vanadia species VO<sub>x</sub>, rather than gas-phase oxygen, undergoes isotope exchange with that of the adsorbed water.

#### **SPIN-POLARIZED SELF-CONSISTENT-FIELD EQUATIONS FOR PAIRED ORBITALS**

**I.L. Zilberberg, S.F. Ruzankin**

*Phys. Rev. A*,  
82(4) (2010) 042505.

Unrestricted Hartree-Fock-like equations are proposed to find multiple spin-symmetry-broken states of the molecular systems. Developed equations are pseudo-eigenvalue-type equations for the Fock-type operators constructed in such a way to

include an effective field which makes different-spin orbitals biorthogonal. The eigenvectors of these operators are noncanonical Hartree-Fock orbitals becoming Lowdin-Amos-Hall paired (corresponding) orbitals after self-consistency is achieved. The eigenvalues of the modified Fock operators appear to be the energies of the paired orbitals. Because the paired orbitals do not follow the spatial symmetry of the molecular nuclear core, the equations allow one to obtain the broken symmetry states with relative ease as demonstrated for the model H<sub>6</sub> hexagon molecule. For this molecule, the Čížek-Paldus instability matrix analysis predicts the existence of three spin-symmetry-broken states. All these solutions are systematically achieved by the paired equations, unlike the standard unrestricted equations which basically converge to a single solution. The proposed approach is also valid for the density functional theory in which the spin-polarized Kohn-Sham equations might be transformed to paired equations.

#### **PAIRED ORBITALS FOR DIFFERENT SPINS EQUATIONS**

**I.L. Zilberberg, S.F. Ruzankin**

*J. Comput. Chem.*,  
31(1) (2010) pp. 84-89.

Eigenvalue-type equations for Lowdin-Amos-Hall spin-paired (corresponding) orbitals are developed to provide an alternative to the standard spin-polarized Hartree-Fock or Kohn-Sham equations in dealing with broken-symmetry (BS) states. To derive paired orbitals for different spins (PODS) equations there has been applied Adams-Gilbert "localizing" operator approach. The PODS equations contain different operators for different spins the eigen-vectors of which are paired orbitals associated with the same eigenvalue for each pair. Preliminary applications to simple systems show viability of this approach. Although the spectrum of possible applications of the PODS equations seems to be quite wide, they would be especially useful for obtaining and analyzing the S-z = 0 BS solutions for the systems with antiferromagnetic structure.

**ADSORPTION SPECIES OF ETHYL BENZOATE IN MgCl<sub>2</sub>-SUPPORTED ZIEGLER-NATTA CATALYSTS. A DENSITY FUNCTIONAL THEORY STUDY**

**D.V. Stukalov, V.A. Zakharov, I.L. Zilberberg**

*J. Phys. Chem. C*,  
114(1) (2010) pp. 429-435.

Adsorption species of ethyl benzoate (EB) on the (104) and (110) MgCl<sub>2</sub> surfaces have been studied within DFT. As a result, monodentate and bidentate complexes of EB were obtained on both the MgCl<sub>2</sub> surfaces. The bidentate structures on the (104) MgCl<sub>2</sub> surface proved to be stabilized by the decreased distance between neighboring adsorption sites (surface Mg cations). The different affinity of EB for the five- and four-coordinated Mg cations predicted was suggested to be the cause of changing the equilibrium shape of MgCl<sub>2</sub> crystals upon growing in the presence of EB: EB chemisorption seems to stabilize the (110) MgCl<sub>2</sub> surface to a greater degree as compared to the (104) MgCl<sub>2</sub> surface. The influence of EB coordination mode on active site stereoselectivity is discussed.

**KOOPMANS'S THEOREM IN THE RESTRICTED OPEN-SHELL HARTREE-FOCK METHOD: II. THE SECOND CANONICAL SET FOR ORBITALS AND ORBITAL ENERGIES**

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*J. Chem. Phys.*,  
132(18) (2010) 184110.

A treatment of the validity of Koopmans's theorem (KT) in the restricted open-shell Hartree-Fock (ROHF) method can be separated into two essentially different cases. The first of them involves the one-electron processes  $X \rightarrow X_j^\pm$  in which the spin state of an ion  $X_j^\pm$  having a hole or an extra electron in the closed, open or virtual orbital  $\varphi_j$  is correctly described by a one-determinant wave function. This case was analyzed using different methods by Plakhutin et al. [*J. Chem. Phys.* 125, 204110 (2006)] and by Plakhutin and Davidson [*J. Phys. Chem. A* 113, 12386 (2009)]. In the present work the authors analyze more complex processes where the state of an ion cannot be described by a single determinant. An example of such processes is the removal of an  $\alpha$  electron from the closed shell of a high-spin half-filled open-shell system  $X$ . For this case it is given a slightly generalized formulation of KT in both the "frozen" orbital approximation (i.e., within the *canonical* ROHF method) and the limited configuration

interaction approach for ionized systems. The authors also show that a simultaneous treatment of KT for all one-electron ionization processes possibly leads to the necessity of introducing in the canonical ROHF method *two different sets of orbitals* and *two respective sets of orbital energies*. The theory developed is compared with the previous formulations of KT in the restricted (ROHF) and unrestricted Hartree-Fock methods, and in the unrestricted density functional theory. The practical applicability of the theory is verified by comparing the KT estimates of the vertical ionization potentials in molecules O<sub>2</sub> and NO<sub>2</sub> with the respective experimental data.

**C<sub>60</sub> FULLERENE AS A PSEUDOATOM OF THE ICOSAHEDRAL SYMMETRY**

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*Russ. Chem. Rev.*,  
79(9) (2010) pp. 729-755.

Unique features of the electronic structure and spectra of the C<sub>60</sub> ( $I_h$ ) fullerene and its ions related to the icosahedral symmetry are discussed. The so-called 'accidental' degeneracy of terms in the open electron shells  $g^N$  and  $h^N$  (corresponding to four- and fivefold degeneracy of molecular orbitals, respectively) and the abnormal ('non-Bethe') splitting of terms in the  $h^N$  configuration are considered. The need to describe these terms arises in studies of ionized and excited states of the C<sub>60</sub> molecule and its derivatives. A novel approach to the description of C<sub>60</sub><sup>m±</sup> ions based on representation of the C<sub>60</sub> ( $I_h$ ) fullerene as a (pseudo)atom of icosahedral symmetry is considered in detail. The approach involves an expansion of the molecular orbitals of C<sub>60</sub> in terms of the atomic orbitals centred at the origin (at the centre of the C<sub>60</sub> molecule) and a multipole expansion of the electron repulsion. The energy spectra of the fullerene and C<sub>60</sub><sup>m±</sup> ions, the magnetic moments and optical dipole transitions are considered. The results obtained are compared with those of *ab initio* calculations and with the available experimental data.

**STABILIZATION OF Au AT EDGES OF BIMETALLIC PdAu NANOCRYSTALLITES**

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*Phys. Chem. Chem. Phys.*,  
12(19) (2010) pp. 5094-5100.

Density functional calculations were performed to study the distribution of Au atoms in bimetallic PdAu



nanoparticles. A series of Pd<sub>79-n</sub>Au<sub>n</sub> clusters of truncated octahedral shape with different content of Au ranging from  $n = 1$  to 60 was used to model such bimetallic nanosystems. Segregation of Au to the particle surface is found to be thermodynamically favorable. The most stable sites for Au substitution are located at the edges of the PdAu nanoclusters. The stabilization at the edges is rationalized by their higher flexibility for surface relaxation which minimizes the strain induced by larger atomic radius of Au as compared to Pd. This stabilization of Au at the edges indicates the possibility to synthesize PdAu particles with Pd atoms located mainly on the facets, and edges “decorated” by Au atoms. Such nanocrystallites are expected to exhibit peculiar catalytic properties and, being thermodynamically stable, should be prone to retaining their initial shape under catalytic conditions.

#### **SIMULATION OF HETEROGENEOUS CATALYSTS AND CATALYTIC PROCESSES USING THE DENSITY FUNCTIONAL METHOD**

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*Kinet. Catal.*,  
51(6) (2010) pp. 832-842.

The review is devoted to the use of high-level quantum-chemical calculations by the density functional method for the simulation of heterogeneous catalytic systems based on transition metals. The following problems are considered: (1) the development of methods for simulating metal particles supported on the surfaces of ionic and covalent oxides; (2) the calculation of the properties of individual transition metal atoms and small clusters adsorbed on the surfaces of MgO,  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>,  $\gamma$ -Al<sub>2</sub>O<sub>3</sub>, and various modifications of SiO<sub>2</sub> and in the pores of zeolites; (3) the mechanisms of hydrogen activation and acrolein hydrogenation on the metallic and partially oxidized surface of silver; and (4) the mechanism of formation of carbon residues upon the decomposition of methanol on nanosized Pd particles.

#### **SMALL GOLD SPECIES SUPPORTED ON ALUMINA. A COMPUTATIONAL STUDY OF $\alpha$ -Al<sub>2</sub>O<sub>3</sub>(0001) AND $\gamma$ -Al<sub>2</sub>O<sub>3</sub>(001) USING AN EMBEDDED-CLUSTER APPROACH**

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*Phys. Status Solidi B*,  
247(5) (2010) pp. 1023-1031.

The authors calculated the structures of and analyzed the bonding in adsorption complexes of small gold species Au<sub>n</sub> on  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>(0001),  $n = 1-6$ , and  $\gamma$ -Al<sub>2</sub>O<sub>3</sub>(001),  $n = 1-5$ . A scalar-relativistic gradient-corrected density functional (DF) method was applied to cluster models of the support that were embedded in an extended elastic polarizable environment (EPE). The shortest Au—O distances, 204–211 pm, are consistent with extended X-ray adsorption fine structure (EXAFS) data for gold clusters on alumina surfaces. The calculated total adsorption energies increase with cluster nuclearity, up to  $n = 4$ , but drop for larger adsorbed species. In the gas phase, these small gold clusters exhibit a planar structure which they keep, oriented parallel to the surface, as adsorbates on  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>(0001). Unfavorable energy contributions result for larger clusters as their planar shape is notably distorted by the interaction with the support which amounts to 0.5–1.5 eV. On  $\gamma$ -Al<sub>2</sub>O<sub>3</sub>(001), also the larger gold clusters retain their intrinsic planar structure as they adsorb oriented perpendicular to the surface. The corresponding adsorption energies are slightly smaller, 0.3–1.2 eV.

#### **ON QUANTITATIVE DESCRIPTION OF METAL PARTICLES SIZE EFFECT IN CATALYTIC KINETICS**

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*Kinet. Catal.*,  
51(6) (2010) pp. 828-831.

Quantitative description for turnover frequency dependence on the metal cluster size for a two-step catalytic cycle was performed based on a thermodynamic approach, which accounts for changes of the chemical potential upon adsorption depending on the metal cluster size. Such analysis revealed a possibility for turnover frequency to exhibit a maximum. A very good

correspondence between calculated and experimental data in hydrogenation and decarboxylation reactions over palladium was achieved.

#### **ELECTRONIC AND GEOMETRIC STRUCTURE OF ISOMERS OF NITRIC ACID. DFT QUANTUM CHEMICAL CALCULATIONS**

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*J. Struct. Chem.*,  
50(5) (2009) pp. 805-813.

Based on the B3LYP/6-311++G(3df,3pd) density functional method, quantum chemical calculations of the electronic structure, geometry, and thermodynamic

parameters of eight isomers of nitric acid (three known isomers in the form of peroxyxynitrous acid ONOOH and five new isomers in the form of oxo-conformation OON(H)O) are presented in the work. The molecular structure of each isomer is characterized by a local minimum on the potential energy hypersurface of the HNO<sub>3</sub> molecular system and corresponds to one of its stationary states. A theoretical study of the reactivity of nitric acid oxo-isomers characterized for the first time can provide adequate explanation for experiments on the autocatalytic use of nitric acid vapors in binding molecular nitrogen. The results obtained can be a direction for developing principally new methods to bind atmospheric nitrogen and activate methane, which are fundamental problems in chemical science and technology.

### **Monte-Carlo Simulation in Surface Science, Kinetic Studies and Biophysics**

#### **EFFECT OF LATTICE STRAIN ON THE KINETICS OF HYDRIDE FORMATION IN METAL NANOPARTICLES**

**V.P. Zhdanov**

*Chem. Phys. Lett.*,  
492(1-3) (2010) pp. 77-81.

The kinetics of hydriding of metal nanoparticles in the generic case when the process is limited by diffusion of vacancies via the hydride shell was analysed. The treatment is focused on the role of lattice strain related to the misfit of the hydride and metal structures. In the absence of strain, the kinetics exhibits an initial fast stage followed by slowdown. Depending on the relative contribution of strain to the H-metal interaction in the ground and activated states for diffusion, the initial fast stage of the kinetics is either nearly not manifested or becomes shorter and occurs up to somewhat higher conversion.

#### **OVERHEATING AND UNDERCOOLING DURING MELTING AND CRYSTALLIZATION OF METAL NANOPARTICLES**

**V.P. Zhdanov, M. Schwind\*, I. Zoric\*, B. Kasemo\*** (*\*Chalmers University of Technology, Göteborg, Sweden*)

*Physica E*,  
42(7) (2010) pp. 1990-1994.

In analogy with macroscopic metal samples, crystallization of metal nanoparticles may occur appreciably below the thermodynamic melting temperature,  $T_m$  (this temperature depends on the

particle size), while melting occurs at  $T_m$ . If the surface melting is suppressed, nanoparticles can be overheated during melting. These effects are described by using the classical nucleation theory and assuming that the nucleation starts at the particle corners or edges. The corresponding undercooling or overheating temperature intervals are found to be about  $0.1 T_m$  for corners and  $0.15 T_m$  for edges. These values are, respectively, two and one and a half times smaller than that for macroscopic samples. Under certain conditions, crystallization and melting can be controlled by the propagation of the front of a new phase. The corresponding temperature interval is found to be very narrow (about  $0.02 T_m$ ).

#### **DIFFUSION-LIMITED KINETICS OF ADSORPTION OF BIOMOLECULES ON SUPPORTED NANOPARTICLES**

**V.P. Zhdanov, B. Kasemo\*** (*\*Chalmers University of Technology, Göteborg, Sweden*)

*Colloids Surf., B*,  
76(1) (2010) pp. 28-31.

General equations have been derived describing the diffusion-limited kinetics of irreversible adsorption of biomolecules on nanoparticles, fabricated on a flat surface, in the case of no hydrodynamic flow in the solution. Under such conditions, the gradients in the concentration of biomolecules occur near the surface, while in more remote regions the gradients may or may not be significant depending on the surface concentration and

size of nanoparticles and the bulk concentration of biomolecules. The equations obtained make it possible to understand the conditions of realization of various regimes of adsorption.

#### **APPARENT KINETICS OF HYDRIDING AND DEHYDRIDING OF METAL NANOPARTICLES**

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*Physica E*,  
42(5) (2010) pp. 1482-1486.

Hydriding and dehydriding kinetics of nanoparticles depend on the particle size. The calculations illustrate that the apparent (averaged over size) kinetics of an ensemble of particles can be dramatically different compared to those of single particles. Specifically, the authors analyze the hydriding kinetics, limited by diffusion of hydrogen atoms from the surface layer via the hydride shell to the metallic core, and the dehydriding kinetics limited by associative desorption of hydrogen from the surface layer. In both cases, the apparent kinetics are relatively slow in the later stage, their time scale for the given average size is much larger than that for a single particle of the same size, and some of the special features of the single-particle kinetics (e.g., the initial slowdown of dehydriding) can be partly or completely washed out. The scaling of the time scale of the kinetics with respect to the particle size, however, remains valid.

#### **ON THE USE OF METAL NANOPARTICLES FOR ENHANCEMENT OF LIGHT ABSORPTION IN DYE-SENSITIZED SOLAR CELLS**

**V.P. Zhdanov**

*Physica E*,  
43(1) (2010) pp. 494-497.

Metal nanoparticles can be used to enhance light absorption in photovoltaic nanocrystalline dye-sensitized solar cells. The calculations scrutinizing this idea indicate, however, that the ratio of the rate of the induced power absorption by dyes, surrounding a nanoparticle, and the rate of the power dissipation by a nanoparticle is typically much lower than 0.5. Thus, this enhancement of light absorption seems to be practically inefficient.

#### **SIMULATION OF DISSOCIATION OF DNA DUPLEXES ATTACHED TO THE SURFACE**

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(\*Chalmers University of Technology, Göteborg, Sweden)

*Central Eur. J. Phys.*,  
8(6) (2010) pp. 883-892.

Monte Carlo simulations are presented of dissociation of duplexes formed of complementary single-stranded DNAs with one of the strands attached to the surface. To describe the transition from the bound state to the unbound state of two strands located nearby, a lattice model is used taking DNA base-pair interactions and conformational changes into account. The results obtained are employed as a basis for a more coarse-grained model including strand backward association and diffusion resulting in complete dissociation. The distribution of the dissociation time is found to be exponential. This finding indicates that the non-exponential kinetic features observed in the corresponding experiments seem to be related to extrinsic factors, e.g., to the surface heterogeneity.

#### **SIZE-DEPENDENT KINETICS OF HYDRIDING AND DEHYDRIDING OF Pd NANOPARTICLES**

**Ch. Langhammer\*, V.P. Zhdanov, I. Zoric\*, B. Kasemo\*** (\*Chalmers University of Technology, Göteborg, Sweden)

*Phys. Rev. Lett.*,  
104(13) (2010) 135502.

Using a new indirect nanoplasmonic sensing method with subsecond resolution, hydriding and dehydriding kinetics of Pd nanoparticles in the size range 1.8-5.4 nm have been studied. Strong particle-size effects are observed. The scaling of the hydriding and dehydriding time scales satisfies power and power-exponential laws. The former (with an exponent of 2.9) is in perfect agreement with Monte Carlo simulations of diffusion-controlled hydriding kinetics. The latter is explained by the effect of surface tension on hydrogen desorption from the surface layer. The approach is generalizable to other reactant-nanoparticle systems.

### SIZE-DEPENDENT HYSTERESIS IN THE FORMATION AND DECOMPOSITION OF HYDRIDE IN METAL NANOPARTICLES

**Ch. Langhammer\***, **V.P. Zhdanov**, **I. Zoric\***,  
**B. Kasemo\*** (\*Chalmers University of Technology,  
Göteborg, Sweden)

*Chem. Phys. Lett.*,  
488(1-3) (2010) pp. 62-66.

Using a novel indirect nanoplasmonic sensing platform, hydrogen absorption/desorption isotherms have been measured in Pd nanoparticles with an average diameter from 1.8 to 8 nm at 30°C. The isotherms are fully reversible at relatively low and high pressures while at intermediate pressures there is hysteresis related to the hydride formation and decomposition. The hysteresis shrinks with decreasing particle size and becomes negligible for particles smaller than 2.5 nm. This feature has been explained in terms of a diminishing contribution of lattice strain to the free energy of hydride formation as the particle size goes down.

### LSPR STUDY OF THE KINETICS OF THE LIQUID-SOLID PHASE TRANSITION IN Sn NANOPARTICLES

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**B. Kasemo\*** (\*Chalmers University of Technology,  
Göteborg, Sweden)

*Nano Lett.*,  
10(3) (2010) pp. 931-936.

Using the localized surface plasmon resonance as a probe in solid and liquid Sn nanoparticles of 107 nm diameter and 52 nm height, the authors have studied their kinetics of melting and freezing at temperature ramps and, for the first time, at fixed temperatures. During temperature ramps, the kinetics exhibit distinct hysteresis. The melting occurs near the bulk melting point while the freezing is observed at much lower temperatures so that the undercooling interval is similar to 130 K. The time scale of the freezing kinetics measured at different fixed temperatures rapidly decreases as the latter are lowered. All these findings have been quantitatively described by assuming the nucleation to occur on the edges of nanoparticles and employing the classical nucleation theory with the corresponding modifications.

### PROTEIN ADSORPTION AND DESORPTION ON LIPID BILAYERS

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*Biophys. Chem.*,  
146(2-3) (2010) pp. 60-64.

The protein surface usually exhibits one or a few charged spots. If a lipid bilayer contains a significant amount of lipids with oppositely charged head groups, protein adsorption on a bilayer may be energetically favourable due to the protein-lipid electrostatic interaction. The specifics of this case are that the lipids are highly mobile and the protein adsorption is accompanied by the redistribution of lipids between the areas covered and not covered by protein. A kinetic model is presented illustrating that this effect is especially interesting if the fraction of the surface covered by charged lipids is relatively low. In this situation, with increasing protein coverage, the protein desorption rate constant rapidly increases while the adsorption rate constant drops, so that there is critical fraction of the area covered by protein. Adsorption above this fraction is hindered both kinetically and thermodynamically.

### ADSORPTION OF PROTEINS ON A LIPID BILAYER

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*Eur. Biophys. J. Biophys. Lett.*,  
39(11) (2010) pp. 1477-1482.

In analysis of protein adsorption on a lipid bilayer, the protein surface is considered to contain one or a few charged spots, and the bilayer contains a significant amount of lipids with oppositely charged head groups. After adsorption, a folded protein is assumed to change its shape slightly due to the electrostatic attraction, so that one of the spots forms a flat contact with the oppositely charged lipid heads of the lipid bilayer. With realistic parameters, this model predicts that the contribution of electrostatic interactions to the protein adsorption energy per charged amino acid-lipid pair is 16-25 kJ/mol. Thus, a few (four or five) pairs is sufficient for irreversible adsorption.

## **HIERARCHICAL GENETIC NETWORKS AND NONCODING RNAs**

**V.P. Zhdanov**

*Chaos*,  
20(4) (2010) 045112.

In eukaryotic cells, many genes are transcribed into noncoding RNAs. Such RNAs may associate with mRNAs and inhibit their translation and facilitate degradation. To clarify what may happen in this case, a kinetic model was proposed describing the effect of noncoding RNAs on a mRNA-protein network with the hierarchical three-layer architecture. For positive regulation of the layers, the model predicts either bistability with a fairly narrow hysteresis loop or a unique steady state. For negative or mixed regulation, the steady state is found to be unique.

## **INTERPLAY OF NONCODING RNAs, mRNAs, AND PROTEINS DURING THE GROWTH OF EUKARYOTIC CELLS**

**V.P. Zhdanov**

*J. Exp. Theor. Phys.*,  
111(4) (2010) pp. 699-705.

Numerous biological functions of noncoding RNAs (ncRNAs) in eukaryotic cells are based primarily on their ability to pair with target mRNAs and then either to prevent translation or to result in rapid degradation of the mRNA-ncRNA complex. Using a general model describing this scenario, it was shown that ncRNAs may help to maintain constant mRNA and protein concentrations during the growth of cells. The possibility of observation of this effect on the global scale is briefly discussed.

## **STOCHASTIC BURSTS IN THE KINETICS OF GENE EXPRESSION WITH REGULATION BY LONG NON-CODING RNAs**

**V.P. Zhdanov**

*JETP Letters*,  
92(6) (2010) pp. 410-415.

One of the main recent breakthroughs in cellular biology is a discovery of numerous non-coding RNAs (ncRNAs). The authors outline abilities of long ncRNAs and articulate that the corresponding kinetics may frequently exhibit stochastic bursts. For example, the authors scrutinize one of the generic cases when the gene transcription is regulated by competitive attachment of ncRNA and protein to a regulatory site. Monte Carlo simulations show that in this case one can observe huge long transcriptional bursts consisting of short bursts.

## **NON-CODING RNAs AND A LAYERED ARCHITECTURE OF GENETIC NETWORKS**

**V.P. Zhdanov**

*Central Eur. J. Phys.*,  
8(6) (2010) pp. 864-872.

In eukaryotic cells, protein-coding sequences constitute a relatively small part of the genome. The rest of the genome is transcribed to non-coding RNAs (ncRNAs). Such RNAs form the cornerstone of a regulatory network that operates in parallel with the protein network. Their biological functions are based primarily on the ability to pair with and deactivate target messenger RNAs (mRNAs). To clarify the likely role of ncRNAs in complex genetic networks, it is presented and comprehensively analyzed a kinetic model of one of the key counterparts of the network architectures. Specifically, the genes transcribed to ncRNAs are considered to interplay with a hierarchical two-layer set of genes transcribed to mRNAs. The genes forming the bottom layer are regulated from the top and negatively self-regulated. If the former regulation is positive, the dependence of the RNA populations on the governing parameters is found to be often non-monotonous. Specifically, the model predicts bistability. If the regulation is negative, the dependence of the RNA populations on the governing parameters is monotonous. In particular, the population of the mRNAs, corresponding to the genes forming the bottom layer, is nearly constant.

## **EFFECT OF NON-CODING RNA ON BISTABILITY AND OSCILLATIONS IN THE mRNA-PROTEIN INTERPLAY**

**V.P. Zhdanov**

*Biophys. Rev. Lett.*,  
5(2) (2010) pp. 89-107.

The feedbacks between the mRNA and protein synthesis may result in kinetic bistability and oscillations. Two generic models predicting bistability include, respectively, a gene with positive regulation of the mRNA production by protein and two genes with mutual suppression of the mRNA production due to negative regulation of the gene transcription by protein. The simplest model predicting oscillations describes a gene with negative regulation of the mRNA production by protein formed via mRNA translation and a few steps of conversion. The authors complement these models by the steps of non-coding RNA (ncRNA) formation and ncRNA-mRNA association and degradation. With this extension, the bistability can often be observed as well. Without and

with ncRNA, the biochemistry behind the steady states may be different. In the latter case, for example, ncRNA may control the mRNA population in the situations when this population is relatively small, and one can observe a switch in the mRNA, protein and ncRNA populations. The analysis of oscillatory kinetics of the mRNA-protein interplay shows that with ncRNA the oscillations may be observed in a wider range of parameters and the amplitude of oscillations may be larger.

### **ncRNA-MEDIATED BISTABILITY IN THE SYNTHESIS OF HUNDREDS OF DISTINCT mRNAs AND PROTEINS**

**V.P. Zhdanov**

*Physica A*,  
389(4) (2010) pp. 887-890.

The kinetics of gene expression can be bistable due to the feedback between the mRNA and protein formation. In eukaryotic cells, the interplay between mRNAs and proteins can be influenced by non-coding RNAs. Some of these RNAs, e.g., microRNAs, may target hundreds of distinct mRNAs. The model presented here shows how a non-coding RNA can be used as a mediator in order to involve numerous mRNAs and proteins into a bistable network.

## **Studying of Active Centers, Reaction Mechanism and Kinetics**

### **STUDY OF *n*-HEXANE ISOMERIZATION ON Pt/SO<sub>4</sub>/ZrO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> CATALYSTS: EFFECT OF THE STATE OF PLATINUM ON CATALYTIC AND ADSORPTION PROPERTIES**

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**E.V. Zatulokina\***, **D.I. Kir'yanov\***, **E.A. Paukshtis**,  
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*Kinet. Catal.*,  
51(4) (2010) pp. 584-594.

The state of surface Pt atoms in the Pt/SO<sub>4</sub>/ZrO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> catalyst and the effect of the state of platinum on its adsorption and catalytic properties in the reaction of *n*-hexane isomerization were studied. The Pt-X/Al<sub>2</sub>O<sub>3</sub> alumina-platinum catalysts modified with various halogens (X = Br, Cl, and F) and their mechanical mixtures with the SO<sub>4</sub>/ZrO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> superacid catalyst were used in this study. With the use of IR spectroscopy (CO<sub>ads</sub>), oxygen chemisorption, and oxygen-hydrogen titration, it was found that ionic platinum species were present on the reduced form of the catalysts. These species can adsorb to three hydrogen atoms per each surface platinum atom. The specific properties of ionic platinum manifested themselves in the formation of a hydride form of adsorbed hydrogen. It is believed that the catalytic activity and operational stability of the superacid system based on sulfated zirconium dioxide were due to the participation of ionic and metallic platinum in the activation of hydrogen for the reaction of *n*-hexane isomerization.

### **EFFECT OF CHROMIUM CONTENT ON THE PROPERTIES OF A MICROSFERICAL ALUMINA-CHROMIUM CATALYST FOR ISOBUTANE DEHYDROGENATION PREPARED WITH THE USE OF A CENTRIFUGAL THERMAL ACTIVATION PRODUCT OF GIBBSITE**

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**V.A. Rogov**, **V.I. Zaikovskiy**, **T.V. Larina**,  
**V.V. Molchanov**

*Kinet. Catal.*,  
51(6) (2010) pp. 898-906.

The states of chromium in both promoted and unpromoted alumina-chromium catalysts with various chromium contents prepared with the use of a centrifugal thermal activation product of gibbsite were studied in detail. The presence of five chromium species was found in the catalysts of this type: two Cr<sup>6+</sup> and three Cr<sup>3+</sup> species. The concentration of each particular chromium species depends on the total chromium content of the catalyst. Based on the experimental data, the authors hypothesized that highly disperse Cr<sup>3+</sup> particles, the formation of which was completed at a chromium content of ~7–9 wt %, are responsible for the catalytic activity of alumina-chromium samples in the reaction of isobutane dehydrogenation.

## NONHEME MANGANESE-CATALYZED ASYMMETRIC OXIDATION. A LEWIS ACID ACTIVATION VERSUS OXYGEN REBOUND MECHANISM: EVIDENCE FOR THE “THIRD OXIDANT”

R.V. Ottenbacher, K.P. Bryliakov, E.P. Talsi

*Inorg. Chem.*,  
49(18) (2010) pp. 8620-8628.

The catalytic properties of a series of chiral nonheme aminopyridinylmanganese(II) complexes [LMn<sup>II</sup>(OTf)<sub>2</sub>] were investigated. The above complexes were found to efficiently catalyze enantioselective olefin oxidation to the corresponding epoxides with different oxidants (peroxycarboxylic acids, alkyl hydroperoxides, iodosylarenes, etc.) with high conversions and selectivities (up to 100%) and enantiomeric excesses (up to 79%). The effect of the ligand structure on the catalytic performance was probed. Epoxidation enantioselectivities were found to be strongly dependent on the structure of the oxidants (performic, peracetic, and *m*-chloroperbenzoic acids; *tert*-butyl and cumyl hydroperoxides; iodosylbenzene and iodosylmesitylene), thus bearing evidence that the terminal oxidant molecule is incorporated in the structure of the oxygen-transferring intermediates. High-valence electron-paramagnetic-resonance-active manganese complexes [LMn<sup>IV</sup>=O]<sup>2+</sup> and [LMn<sup>IV</sup>(μ-O)<sub>2</sub>Mn<sup>III</sup>L]<sup>3+</sup> were detected upon interaction of the starting catalyst with the oxidants. The high-valence complexes did not epoxidize styrene and could themselves only contribute to minor olefin oxidation sideways. However, the oxomanganese(IV) species were found to perform the Lewis acid activation of the acyl and alkyl hydroperoxides or iodosylarenes to form the new type of oxidant [oxomanganese(IV) complex with a terminal oxidant], with the latter accounting for the predominant enantioselective epoxidation pathway in the nonheme manganese-catalyzed olefin epoxidations.

## RESULTS OF NMR SPECTROSCOPIC STUDIES OF HYDROCARBON CONVERSIONS ON SOLID ACID CATALYSTS IN THE LAST 25 YEARS

A.G. Stepanov

*Kinet. Catal.*,  
51(6) (2010) pp. 854-872.

High-resolution solid-state NMR studies of hydrocarbon conversions on the surface of solid acid catalysts are overviewed. The results of identification of hydrocarbons adsorbed on solid acid catalysts are presented. Alkane activation and hydrocarbon conversion

mechanisms and the nature of reactive intermediates are discussed. It is demonstrated that NMR spectroscopy can be used not only in the *in situ* analysis of hydrocarbons on the catalyst surface, but also as a method for seeking new hydrocarbon conversion routes.

## METAL-ALKYL SPECIES ARE FORMED ON INTERACTION OF SMALL ALKANES WITH GALLIUM OXIDE: EVIDENCE FROM SOLID-STATE NMR

A.A. Gabrienko, S.S. Arzumanov, A.V. Toktarev, A.G. Stepanov

*Chem. Phys. Lett.*,  
496(1-3) (2010) pp. 148-151.

<sup>13</sup>C CP MAS NMR analysis of the products of the interaction of methane, ethane and propane with α-Ga<sub>2</sub>O<sub>3</sub> or Ga-modified zeolite BEA at 523-623 K shows that dissociative adsorption of C<sub>1</sub>-C<sub>3</sub> alkanes on the surface of gallium oxide or Ga-modified zeolite BEA results to the formation of Ga-methyl, Ga-ethyl and Ga-propyl species. This observation allows one to conclude that Ga-alkyls, rather than earlier suggested alkoxy species, could be the intermediates in small alkane dehydrogenation and aromatization on these catalysts.

## PROPANE AROMATIZATION ON Zn-MODIFIED ZEOLITE BEA STUDIED BY SOLID-STATE NMR *in situ*

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*J. Phys. Chem. C*,  
114(29) (2010) pp. 12681-12688.

The conversion of propane (propane-1-<sup>13</sup>C and propane-2-<sup>13</sup>C) on Zn/H-BEA zeolite at 520–620 K has been studied by <sup>1</sup>H and <sup>13</sup>C (CP) MAS NMR. Propene adsorption complex with zinc sites (π-complex) and σ-allyl zinc species as intermediates have been identified in the course of propane conversion to aromatics. The mechanism leading to the formation of methane and ethane, which are constituents of an undesirable route in propane conversion, has been examined by kinetic modeling of the expected reaction network based on *in situ* <sup>1</sup>H MAS NMR kinetic measurements of the reaction performance. The pathways for propane aromatization and hydrogenolysis have been proposed. Hydrogenolysis of propane has been concluded to occur with the involvement of both Brønsted acid sites and Zn sites.

## H/D EXCHANGE OF MOLECULAR HYDROGEN WITH BRONSTED ACID SITES OF Zn- AND Ga-MODIFIED ZEOLITE BEA

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*Phys. Chem. Chem. Phys.*, 12(19) (2010) pp. 5149-5155.

Kinetics of hydrogen H/D exchange between Brønsted acid sites of pure acid-form and Zn- or Ga-modified zeolites beta (BEA) and deuterated hydrogen ( $D_2$ ) has been studied by  $^1H$  MAS NMR spectroscopy *in situ* within the temperature range of 383–548 K. A remarkable increase of the rate of the H/D exchange has been found for Zn- and Ga-modified zeolites compared to the pure acid-form zeolite. The rate of exchange for Zn-modified zeolite is one order of magnitude higher compared to the rate for Ga-modified zeolite and two orders of magnitude larger compared to the pure acid-form zeolite. This promoting effect of metal on the rate of H/D exchange was rationalized by a preliminary dissociative adsorption of molecular hydrogen on metal oxide species or metal cations. The adsorbed hydrogen is further involved in the exchange with the acid OH groups located in vicinity of metal species. The role of different metal species in the possible mechanisms of the exchange with involvement of zeolite Brønsted acid sites and metal species is discussed.

## STRONG ACIDITY OF SILANOL GROUPS OF ZEOLITE BETA: EVIDENCE FROM THE STUDIES BY IR SPECTROSCOPY OF ADSORBED CO AND $H^1$ MAS NMR

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*Microporous Mesoporous Mater.*, 131(1-3) (2010) pp. 210-216.

Acidic OH groups of acid-form and Zn and Ga-modified zeolite beta (BEA) have been characterized with  $^1H$  MAS NMR and by IR spectroscopy of adsorbed carbon monoxide. It is demonstrated that OH groups, which exhibit a vibration band at  $3740\text{ cm}^{-1}$ , reveal acidity, which is similar to that of the OH groups with the band at  $3610\text{ cm}^{-1}$  according to the value of the low frequency shift of OH vibrations with adsorbed CO ( $\Delta\nu_{OH/CO}=300\text{ cm}^{-1}$ ). The IR band  $3740\text{ cm}^{-1}$  corresponds to the signal at ca. 2.1 ppm in  $^1H$  MAS NMR spectrum. The OH groups with signal 2.1 ppm are involved in H/D exchange with methane- $d_4$  similar to acidic OH groups

with the signals 4.0–5.1 ppm. The signals at 2.1 ppm in  $^1H$  MAS NMR and at  $3740\text{ cm}^{-1}$  in IR are attributed to the strongly acidic silanol groups of the faulted structure of the zeolite. The silanols with the signals at 1.8 ppm and  $3745\text{ cm}^{-1}$  are weakly acidic ( $\Delta\nu_{OH/CO}=85\text{ cm}^{-1}$ ) and are not involved in the H/D exchange. Loading of the zeolite with Zn affords a notable decrease of the concentration of strongly acidic SiOHAl groups, whereas the quantity of these groups does not decrease upon loading the zeolite with Ga.

## THE “ALKYL” AND “CARBENIUM” PATHWAYS OF METHANE ACTIVATION ON GA-MODIFIED ZEOLITE BEA: $^{13}C$ SOLID-STATE NMR AND GC-MS STUDY OF METHANE AROMATIZATION IN THE PRESENCE OF HIGHER ALKANE

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*J. Phys. Chem. C*, 114(46) (2010) pp. 21555-21561.

By using  $^{13}C$  solid-state NMR spectroscopy and GC-MS analysis, the activation of methane and co-aromatization of methane and propane have been monitored on gallium-modified zeolite BEA at 573–823 K. A noticeable degree involvement of the  $^{13}C$ -label from methane- $^{13}C$  into the aromatic reaction products (benzene, toluene) has been demonstrated. The major intermediate of the methane activation represents gallium-methyl species, which are formed by methane dissociative adsorption on  $Ga_2O_3$  species of the zeolite. The minor species of methane activation, Ga-methoxy groups, provide the involvement of methane into aromatics by the methylation of aromatic molecules, which are generated exclusively from propane, by the mechanism of electrophilic substitution. Ga-methyl species can serve as methylating nucleophilic agent for the reaction of nucleophilic substitution with participation of aromatic molecules, which contain the electron-withdrawing substituents.



**CARBONYLATION OF DIMETHYL ETHER ON SOLID Rh-PROMOTED Cs-SALT OF KEGGIN 12-H<sub>3</sub>PW<sub>12</sub>O<sub>40</sub>: A SOLID-STATE NMR STUDY OF THE REACTION MECHANISM REACTIVITY**

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*J. Catal.*,  
278(2) (2010) pp. 353-361.

The carbonylation of dimethyl ether (DME) with carbon monoxide on Rh-promoted cesium salt of 12-tungstophosphoric acid, Rh/Cs<sub>2</sub>HPW<sub>12</sub>O<sub>40</sub> (HPA), has been studied with <sup>13</sup>C solid-state NMR. The bifunctional character of Rh/Cs<sub>2</sub>HPW<sub>12</sub>O<sub>40</sub> catalyst in halide-free carbonylation of DME has been directly demonstrated. The activation of the C-O bond of DME proceeds on Brønsted acid sites of HPA with the formation of the methyl group attached to the surface of HPA (methoxy species), whereas the role of rhodium consists in trapping carbon monoxide from gaseous phase and a transfer of CO to the center of DME activation, acidic OH-group of the catalyst, in the form of rhodium-carbonyls. The lattice of Cs<sub>2</sub>HPW<sub>12</sub>O<sub>40</sub> makes it possible to locate these two different active centers in close proximity to each other, e.g., on two adjacent oxygen atoms, terminal and bridging, of one Keggin anion, thus facilitating the insertion of carbon monoxide from rhodium carbonyl into the C-O bond of methoxy-group to produce the acetate group bound to the Keggin anion. The latter offers finally methyl acetate under the interaction with DME, the intermediate surface methoxy-groups being restored.

**EFFECT OF Pt NANOPARTICLE SIZE ON THE SPECIFIC CATALYTIC ACTIVITY OF Pt/SiO<sub>2</sub> AND Pt/TiO<sub>2</sub> IN THE TOTAL OXIDATION OF METHANE AND *n*-BUTANE**

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*Russ. Chem. Bull.*,  
9 (2010) pp. 1713-1719.

The dependence of the specific catalytic activity ( $A_{sp}$ ) of the catalysts Pt/SiO<sub>2</sub> and Pt/TiO<sub>2</sub> in the total oxidation of CH<sub>4</sub> and *n*-C<sub>4</sub>H<sub>10</sub> on the Pt nanoparticle size (in the range from 1 to 4 nm) was studied. The specific catalytic activity increases with an increase in the platinum nanoparticle size, indicating that the total oxidation is a structure-sensitive reaction. The structure sensitivity depends on the size of an oxidized molecule: it increases sharply on going from CH<sub>4</sub> to *n*-C<sub>4</sub>H<sub>10</sub>. The support also exerts a considerable effect

on the  $A_{sp}$  value: in the oxidation of both CH<sub>4</sub> and C<sub>4</sub>H<sub>10</sub> the specific catalytic activity for the catalysts Pt/TiO<sub>2</sub> is 3–4 times that for Pt/SiO<sub>2</sub>.

**OXYGEN SPECIES ON THE SILVER SURFACE OXIDIZED BY MW-DISCHARGE: STUDY BY PHOTOELECTRON SPECTROSCOPY AND DFT MODEL CALCULATIONS**

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*Surf. Sci.*,  
604(13-14) (2010) pp. 1185-1192.

A polycrystalline silver surface has been studied by synchrotron radiation photoelectron spectroscopy after deep oxidation by microwave discharge in an O<sub>2</sub> atmosphere. Oxidized structures with high oxygen content, AgO<sub>x</sub> with  $x > 1$ , have been found on the silver surface after oxidation at 300–400 K. The line shapes observed in the O1s spectra were decomposed into five components and indicated that complex oxidized species were formed. An analysis of the oxidized structures with binding energies,  $E_b(O1s)$ , greater than 530 eV pointed to the presence of both Ag–O and O–O bonds. A detailed experimental study of the valence band spectra in a wide spectral range (up to 35 eV) have been carried out, which has allowed to register the multicomponent structure of spectra below Ag4d band. These features were assigned to the formation of Ag–O and O–O bonds composed of molecular (associative) oxygen species. DFT model calculations showed that saturation of the defect oxidized silver surface with oxygen leads to the formation of associative oxygen species, such as superoxides, with electrophilic properties and covalent bonding. The high stability of oxygen-rich silver structures, AgO<sub>x</sub>, can be explained by the formation of small silver particles during the intensive MW oxidation, which can stabilize such oxygen species.

**THE INVESTIGATION OF OXIDIZED SILVER NANOPARTICLES PREPARED BY THERMAL EVAPORATION AND RADIO-FREQUENCY SPUTTERING OF METALLIC SILVER UNDER OXYGEN**

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*Appl. Surf. Sci.*,  
257(2) (2010) pp. 404-413.

The investigation of oxidized silver nanoparticles by the photoemission (XPS, UPS) and HRTEM methods was performed. The nanoparticles of oxidized silver were obtained in the vacuum chamber

by two methods of synthesis: thermal evaporation of silver nanoparticles followed by transferring in convective gas flow and sputtering of oxidized clusters under the action of plasma. Both methods indicated that oxygen interaction with silver nanoparticles depends strongly on its size. It was shown that the chemical bonding of oxygen species stabilized on small particles differs from the oxygen species adsorbed on bulk silver surfaces (monocrystals, foils and large particles). The low charged oxygen with molecular type of bonding stabilizes on particles of size approximately 5 nm and smaller. Increasing particle size leads to the dissociation of molecular oxygen species and the formation of strongly charged oxygen composed of oxide nanoparticles like Ag<sub>2</sub>O or AgO type. The presence of extended defects in the microdomain large nanoparticles facilitates the formation of Ag<sub>2</sub>O or AgO layers covering metallic nanosilver.

#### INVESTIGATION OF ACTIVE METAL SPECIES FORMATION IN Pd-PROMOTED SULFATED ZIRCONIA ISOMERIZATION CATALYST

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*Appl. Catal., A*,  
387(1) (2010) pp. 5-12.

The state of palladium in Pd/SO<sub>4</sub><sup>2-</sup>-ZrO<sub>2</sub> (Pd/SZ) isomerization catalyst was investigated by the temperature-programmed reduction (TPR), chemisorption technique, infrared spectroscopy of adsorbed carbon monoxide (FTIRS), X-ray photoelectron spectroscopy (XPS), diffuse-reflectance UV-vis spectroscopy (UV-vis DRS), and benzene hydrogenation as a test reaction. It has been stated that reduction temperature has a great impact on the metal function of Pd-promoted sulfated zirconia catalyst. Metal centers are formed at about 30–70°C and characterized by high palladium dispersion and activity in benzene hydrogenation. At temperatures above 200 °C, intensive sulfate decomposition occurs and products of sulfate reduction poison the metal function of the catalyst. According to XPS and FTIRS study, palladium particles in the poisoned samples are only partly oxidized, but the main part is presented by metallic phase without large amount of PdS. Reduction of Pd-containing catalyst at 150 °C (instead of 250°C) leads to higher conversion and 2,2-dimethylbutane yield in acid-catalyzed reaction of

n-hexane isomerization. Higher isomerization activity in this case is provided by prevention of active sulfate species decomposition due to the capability of palladium metallic particles formation at low reduction temperatures.

#### AN ACTIVE PHASE TRANSFORMATION ON SURFACE OF Ni-Au/Al<sub>2</sub>O<sub>3</sub> CATALYST DURING PARTIAL OXIDATION OF METHANE TO SYNTHESIS GAS

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*Kinet. Catal.*,  
51(4) (2010) pp. 573-578.

It was studied the influence of gold addition on physico-chemical properties and catalytic activity of bimetallic Ni-Au/Al<sub>2</sub>O<sub>3</sub> catalyst in partial oxidation of methane (POM). The reduction behavior in hydrogen, XRD crystal structure, XPS spectra and POM catalytic activity were investigated. The reduction of Ni-Au catalyst is a prerequisite condition to catalyze POM reaction. The formation of Ni-Au alloy during high temperature reduction in hydrogen and also in the conditions of POM reaction was experimentally proved. The addition of gold to Ni/Al<sub>2</sub>O<sub>3</sub> system improves catalyst stability and activity in POM reaction.

#### FeAl<sub>12</sub>-KEGGIN TYPE CATION AS AN ACTIVE SITE SOURCE FOR Fe,Al-SILICA MESOPOROUS CATALYSTS

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*Appl. Catal., B*,  
95(1-2) (2010) pp. 110-119.

Iron-containing mesoporous mesophase materials Fe,Al-MMM-2 have been synthesized according to a sol-mesophase route under mild acidic conditions (pH 2.3–4.4) using Keggin type cation [FeAl<sub>12</sub>O<sub>4</sub>(OH)<sub>24</sub>(H<sub>2</sub>O)<sub>12</sub>]<sup>7+</sup> (FeAl<sub>12</sub><sup>7+</sup>) as Al and Fe sources. Effect of pH of the synthetic solution on structural and physicochemical properties of Fe,Al-MMM-2 has been evaluated by means of small- and wide-angle XRD, FTIR, DR-UV-vis, and N<sub>2</sub>-adsorption/desorption analysis. It has been established that deviation of stability of FeAl<sub>12</sub><sup>7+</sup> cation at various pH determines Al/Fe ratio in the resulting material as 12/1 (material synthesized at pH 4.4), and 6/1 (pH 2.4 and 3.3). Correlation between incorporation of Al and Fe in different forms and resulting materials texture is discussed.

Fe,Al-MMM-2 materials have been tested as catalysts for wet phenol oxidation with H<sub>2</sub>O<sub>2</sub>. Insertion of Al species into the framework of the silicate matrix increases the activity comparing to a reference Fe,Al-pillared clay due to both higher surface acidity and lower diffusion limitations that correspond to Fe,Al-MMM-2.

#### **A 'DOUBLE PEAK' CATALYTIC ACTIVITY OF NANO-SIZED GOLD SUPPORTED ON TITANIA IN THE GAS-PHASE SELECTIVE OXIDATION OF ETHANOL**

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*ChemCatChem*,  
2(12) (2010) pp. 1535–1538.

Au/TiO<sub>2</sub>, in contrast to Au supported on silica and alumina, shows “double-peak” catalytic activity in partial oxidation of ethanol to acetaldehyde by molecular oxygen. The additional low-temperature peak probably corresponds to the specific participation of oxygen generated on Au/TiO<sub>2</sub> surface under mild reaction conditions.

#### **SULFATED ALUMINA AND ZIRCONIA IN ISOBUTANE/BUTENE ALKYLATION AND *n*-PENTANE ISOMERIZATION: CATALYSIS, ACIDITY, AND SURFACE SULFATE SPECIES**

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*Catal. Today*,  
152(1-4 sp. issue) (2010) pp. 17-23.

The catalytic behavior of sulfated alumina (SA) and zirconia (SZ) was compared for two acid-catalyzed reactions, i.e., isobutane/butene alkylation and isomerization of *n*-pentane. For the alkylation reaction, the SZ catalyst gave a higher C<sup>5+</sup> alkane yield and much lower selectivity for trimethylpentanes (TMPs) at 2 h TOS. However, TMPs selectivity was comparable for both catalysts at 5.5 h TOS. For the isomerization, high *n*-pentane conversion was obtained on SZ at the reaction temperature of 100°C when SA had no activity. Surface sulfate species were identified by IR spectroscopy. The acidity of sulfated oxides was compared by IR spectroscopy using pyridine and carbon monoxide as probe molecules and TPD-MS of ammonia. An effort was made to explain the difference between catalytic behavior of two sulfated oxides in terms of acidity, the nature of surface sulfate species and the formation of hydride species.

#### **THE EFFECT OF SUPPORT PROPERTIES ON THE ACTIVITY OF Pd/C CATALYSTS IN THE LIQUID-PHASE HYDRODECHLORINATION OF CHLOROBENZENE**

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*Appl. Catal., A*,  
379(1-2) (2010) pp. 87-94.

The activity of palladium catalysts prepared using active carbons (AG-2000, MeKS) and Sibunit type carbon in the liquid-phase hydrodechlorination of chlorobenzene has been studied. The use of AG-2000 activated carbon prepared from coal raw material provided the highest initial activity of the catalyst (9.4 mol<sub>C<sub>6</sub>H<sub>5</sub>Cl</sub>/mol<sub>Pd</sub> min) for the catalyst with particle size of 0.08–0.1 mm which not reduced within next 4 cycles. However, this activity cannot be explained only on the basis of support texture characteristics. From results of chemical analysis as well as X-ray photoelectron spectroscopy (XPS) and high-resolution transmission electron microscopy (HR TEM) data the high activity of the Pd/AG-2000 catalyst is explainable by the presence on the surface of the initial activated carbon of oxygen-containing compounds of iron, aluminum, silicon, calcium and magnesium. The overall ash content of the activated carbon was 11.9 wt.%. It can be assumed that the oxide compounds of iron act as anchoring centers for palladium, and that oxide containing impurities may interact with HCl decreasing deactivation. The experimental data of this work has shown that the main contribution to the Pd/AG-2000 catalyst activity is made by the palladium particles with a size of 3 nm and higher which remain stable under the aggressive conditions of the chlorobenzene hydrodechlorination process.

#### **COPPER-CERIUM OXIDE CATALYSTS PREPARED BY THE PECHINI METHOD FOR CO REMOVAL FROM HYDROGEN-CONTAINING MIXTURES**

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*Kinet. Catal.*,  
51(1) (2010) pp. 119-125.

A series of copper–cerium oxide catalysts was prepared by the Pechini method, and their physico-chemical and catalytic properties in CO oxidation in hydrogen-containing gas mixtures were studied. The method chosen for catalyst preparation yields finely dispersed copper and cerium oxides in the catalyst.

## MECHANISM OF THE OXYGEN INVOLVEMENT IN NICOTINIC ACID FORMATION UNDER $\beta$ -PICOLINE OXIDATION ON V-Ti-O CATALYST

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*Catal. Today*,  
157(1-4) (2010) pp. 39-43.

Mechanism of the oxygen involvement in nicotinic acid formation under  $\beta$ -picoline oxidation on vanadia-titania catalyst was studied by *in situ* FTIR spectroscopy and kinetic method in temperature range of 120–300°C. The formation of nicotinic acid proceeds via a consecutive transformation of the surface carbonyl-like and carboxylate complexes stabilized at reduced vanadium. Catalyst oxygen includes in formation of these complexes. Carboxylate is a direct precursor of nicotinic acid, it turns into nicotinic acid in the presence of the gas-phase oxygen in joint step of catalyst reoxidation – acid desorption. Significant concentration ratio of oxygen to  $\beta$ -picoline ( $C_{O_2}:C_{\beta P} > 16:1$ ) is necessary to effective running reaction. This factor can be explained by the reaction mechanism. The variety of oxygen functions and of oxygen species require the maximum oxidized state of the catalyst and explain the necessity of a high oxygen excess in the reaction mixture.

## KINETICS OF FORMALDEHYDE OXIDATION ON V-Ti-O CATALYST

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*Catal. Ind.*,  
2(4) (2010) pp. 320-328.

The heterogeneous catalytic oxidation of formaldehyde in the gas phase may be considered as an alternative to the multistep liquid phase synthesis of formic acid. Monolayer vanadia–titania catalysts are active and selective in the oxidation of formaldehyde to formic acid. Detailed investigation of kinetics of formaldehyde oxidation over a monolayer vanadia–titania catalyst was carried out. It was established that by-products are formed via a consecutive–parallel reaction network.  $CO_2$  results from formaldehyde oxidation via parallel pathways and from formic acid overoxidation via consecutive pathway; CO is produced from the formic acid via consecutive pathway. It was shown that oxygen and water accelerate formic acid formation and that water retards CO formation. Based on experimental data, a kinetic model of formaldehyde oxidation was developed. The kinetic model was used in the mathematical simulation of the formaldehyde

oxidation process and in the determination of dynamic and design parameters of the reactor. Formic acid production by the gas phase oxidation of formaldehyde is unique and does not have any analogue. As opposed to conventional technologies, it is energy saving, environmentally friendly, and technologically simple. An enlarged scale pilot plant using this technology is under construction.

## EXPERIMENTAL STUDY OF INTERMEDIATES AND WAVE PHENOMENA IN CO OXIDATION ON PLATINUM METAL (Pt, Pd) SURFACES

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*Kinet. Catal.*,  
51(6) (2010) pp. 873-884.

The mechanism of catalytic CO oxidation on Pt(100) and Pd(110) single-crystal surfaces and on Pt and Pd sharp tip ( $\sim 10^3$  Å) surfaces has been studied experimentally by temperature-programmed reaction, temperature desorption spectroscopy, field electron microscopy, and molecular beam techniques. Using the density functional theory the equilibrium states and stretching vibrations of oxygen atoms adsorbed on the Pt(100) surface have been calculated. The character of the mixed adsorption layer was established by high resolution electron energy loss spectroscopy—molecular adsorption ( $O_{2ads}$ ,  $CO_{ads}$ ) on Pt(100)-*hex* and dissociative adsorption ( $O_{ads}$ ,  $CO_{ads}$ ) on Pt(100)-(1 × 1). The origin of kinetic self-oscillations for the isothermal oxidation of CO *in situ* was studied in detail on the Pt and Pd tips by field electron microscopy. The initiating role of the reversible phase transition (*hex*)  $\leftrightarrow$  (1 × 1) of the Pt(100) nanoplane in the generation of regular chemical waves was established. The origination of self-oscillations and waves on the Pt(100) nanoplane was shown to be caused by the spontaneous periodical transition of the metal from the low-active state (*hex*) to the highly active catalytic state (1 × 1). A relationship between the reactivity of oxygen atoms ( $O_{ads}$ ) and the concentration of  $CO_{ads}$  molecules was revealed for the Pd(110) surface. Studies using the isotope label  $^{18}O_{ads}$  demonstrated that the low-temperature formation of  $CO_2$  at 150 K is a result of the reaction of CO with the highly reactive state of atomic oxygen ( $O_{ads}$ ). The possibility of the low-temperature oxidation of CO via interaction with the so-called “hot” oxygen atoms ( $O_{hot}$ ) appearing on the surface at the instant of dissociation of  $O_{2ads}$  molecules was studied by the molecular beam techniques.

## KINETICS OF THE H<sub>2</sub><sup>18</sup>O/H<sub>2</sub><sup>16</sup>O ISOTOPE EXCHANGE OVER VANADIA-TITANIA CATALYST

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Yu.K. Gulyaeva, G.Ya. Popova,  
T.V. Andrushkevich

*J. Mol. Catal. A: Chem.*,  
316(1-2) (2010) pp. 118-125.

Study of oxygen exchange between vanadia-titania catalyst, H<sub>2</sub>O and O<sub>2</sub> has been performed in isothermal (200°C) and thermo programmed (50-500°C) conditions. Experiments using H<sub>2</sub><sup>18</sup>O, <sup>18</sup>O<sub>2</sub> and monolayer vanadia-titania catalyst placed to the plug-flow reactor were carried out. Significant values of the rate of isotope exchange between O<sub>2</sub> and the catalyst were observed at T > 450°C, but catalyst reduction accompanied oxygen desorption into gas phase proceeded in this case. Unlike dioxygen, water can exchange quite readily its oxygen atoms with vanadia-titania catalyst even at room temperature. Surface vanadium sites coordinating the OH group show the highest activity in the oxygen exchange with water. The rate constant of oxygen exchange between adsorbed water molecule and V-OH is ca. 0.5 s<sup>-1</sup> at 200°C, with the activation energy close to zero. The rate of oxygen exchange with dehydrated vanadium complexes V-O-V and V=O is much lower. The rate

constant of this exchange is ca. 10<sup>-3</sup> s<sup>-1</sup> at 200°C, with the activation energy of ca. 70 kJ/mol. The interaction of adsorbed water with VO<sub>x</sub> species results in their slow hydrolysis to form the V-OH groups (characteristic time ca. 10<sup>4</sup> s), which recombination underlies the isotope exchange.

## LIQUID PHASE HYDROAMINATION OF CYCLOHEXANONE

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L.V. Piriyutko, S.V. Semikolenov

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Activity and selectivity of supported Ni, Pt, and Pd catalysts were studied in the liquid phase reductive amination of cyclohexanone at temperatures ranging from 100 to 150°C. The catalyst 20% Ni/SiO<sub>2</sub> is most active and selective providing a maximum yield of cyclohexylamine. The influence of the reaction conditions on the parameters of the catalytic process was studied. A detailed analysis of the reaction products was carried out using <sup>13</sup>C NMR spectroscopy and gas chromatography coupled with mass spectrometry (GC-MS). This made it possible to refine the reaction mechanism and to identify a new by-product earlier unknown in the literature

## Application of Physicochemical Methods for Characterization of Catalysts, Supports, Synthesized Substances and Materials

### MOBILITY OF n-BUTANE IN ZSM-5 ZEOLITE STUDIED BY <sup>2</sup>H NMR

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The mobility of linear alkane, n-butane, adsorbed in zeolite ZSM-5 was investigated by deuterium solid-state NMR (<sup>2</sup>H NMR). Analysis of the <sup>2</sup>H NMR spectra line shape and spin-lattice relaxation time permitted characterizing the influence of pore confinement on the dynamics of the molecule over a broad temperature range (123-413 K). It has been found, that at a loading of two molecules per unit cell, n-butane molecules are essentially located in both straight and zigzag channels and not the intersections. The adsorbed molecules are involved in fast intramolecular motions and reorientation as a whole. Intramolecular motions are methyl group rotation and

conformational exchange occurring at  $\tau \approx 4-8 \times 10^{-11}$  s (at 300 K) with an activation barrier of  $E_{ci} \approx 11-12$  kJ mol<sup>-1</sup>. The reorientation of the molecule as whole is a complex anisotropic motion, which consists of jump exchange between neighboring channels, occurring at  $\tau_D \approx 3.7 \times 10^{-10}$  s (at 300 K) with an activation barrier of  $E_D \approx 5$  kJ mol<sup>-1</sup> and of other rotational modes that may correspond to uniaxial rotation and librations of n-butane in the pores. Influence of pore confinement on the dynamics of the adsorbed alkane is manifested by the complete suppression of isotropic reorientation.

## DYNAMICS OF BENZENE RINGS IN MIL-53(Cr) AND MIL-47(V) FRAMEWORKS STUDIED BY $^2\text{H}$ NMR SPECTROSCOPY

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Rotational dynamics of benzene rings in the flexible MIL-53 and rigid MIL-47 frameworks was investigated by  $^2\text{H}$  NMR spectroscopy. In both cases,  $180^\circ$  flips are observed, and the flipping rate is higher in MIL-53. To simulate experimental spectra, consideration of the quadrupolar interaction alone is not sufficient; the paramagnetic effect must also be taken into account.

## COMPARISON OF THE DYNAMICS OF MIL-53 AND MIL-47 FRAMEWORKS BY NEUTRON SCATTERING AND DFT METHODS

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*Eur. Phys. J. Special Topics*,  
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The low-frequency modes of MIL-47(V) and MIL-53(Cr) frameworks have been measured by inelastic neutron scattering. The librational modes of benzene are very close in frequency in the two structures. First-principles calculations have been performed to assign the measured spectra. The calculated values of the rotational barriers of the benzene rings are in reasonable agreement with experimental data. The relatively large barriers (413-588 meV) explain the absence of quasi-elastic broadening on the neutron time scale.

## UNUSUAL CHAIN-LENGTH DEPENDENCE OF THE DIFFUSION OF n-ALKANES IN THE METAL-ORGANIC FRAMEWORK MIL-47(V): THE BLOWGUN EFFECT

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*Chem. Eur. J.*,  
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Joint QENS experiments/MD simulations strategy has proved to be a valuable tool for providing a complete picture of the diffusion mechanism for short-length alkanes in a MOF-type material. A detailed analysis of the translational and rotational dynamics from the QENS spectra highlighted that all alkanes follow a 1D-type diffusion gliding along the tunnel of the MIL-47(V) solid, with either isotropic or uniaxial rotation depending on the chain length. Spectacular blowgun-type diffusion has been further evidenced for n-butane from a higher diffusivity at low loading of this alkane compared with propane. The understanding gained here on the chain-length dependence of n-alkanes diffusion in a model rigid MOF could be of great interest in the field of the alkane separation processes that are known to be significantly governed by transport kinetic criteria.

## MODULAR, HOMOCHIRAL, POROUS COORDINATION POLYMERS: RATIONAL DESIGN, ENANTIOSELECTIVE GUEST EXCHANGE SORPTION AND *ab initio* CALCULATIONS OF HOST-GUEST INTERACTIONS

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*Chem. Eur. J.*,  
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Two new, homochiral, porous metal-organic coordination polymers [Zn<sub>2</sub>(ndc) {(R)-man}(dmf)]·3DMF and [Zn<sub>2</sub>(bpdc) {(R)-man}(dmf)]·2DMF (ndc=2,6-naphthalene-dicarboxylate; bpdc=4,4'-biphenyldicarboxylate; man=mandelate; dmf=N,N'-dimethylformamide) have

been synthesized by heating  $Zn^{II}$  nitrate,  $H_2ndc$  or  $H_2bpdC$  and chiral (*R*)-mandelic acid ( $H_2man$ ) in DMF. The colorless crystals were obtained and their structures were established by single-crystal X-ray diffraction. These isorecticular structures share the same topological features as the previously reported zinc(II) terephthalate lactate  $[Zn_2(bdc)\{(S)\text{-}lac\}(dmf)]\cdot DMF$  framework, but have larger pores and opposite absolute configuration of the chiral centers. The enhanced pores size results in differing stereoselective sorption properties: the new metal–organic frameworks effectively and stereoselectively (*ee* up to 62%) accommodate bulkier guest molecules (alkyl aryl sulfoxides) than the parent  $[Zn_2(bdc)\{(S)\text{-}lac\}(dmf)]\cdot DMF$ , while the latter demonstrates decent enantioselectivity toward precursor of chiral anticancer drug sulforaphane,  $CH_3SO(CH_2)_4OH$ . The new homochiral porous metal–organic coordination polymers are capable of catalyzing a highly selective oxidation of bulkier sulfides (2-NaphSMe (2- $C_{10}H_7SMe$ ) and PhSCH<sub>2</sub>Ph) that could not be achieved by the smaller-pore  $[Zn_2(bdc)\{(S)\text{-}lac\}(dmf)]\cdot DMF$ . The sorption of different guest molecules (both *R* and *S* isomers) into the chiral pores of  $[Zn_2(bdc)\{(S)\text{-}lac\}(dmf)]\cdot DMF$  was modeled by using *ab initio* calculations that provided a qualitative explanation for the observed sorption enantioselectivity. The high stereo-preference is accounted for by the presence of coordinated inner-pore DMF molecule that forms a weak C–H $\cdots$ O bond between the DMF methyl group and the (*S*)-PhSOCH<sub>3</sub> sulfinyl group.

#### HYDROLYSIS OF AMMONIUM OXOFLUOROTUNGSTATES: A $^{19}F$ , $^{17}O$ AND $^{183}W$ NMR STUDY

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*J. Fluorine Chem.*,  
131(5) (2010) pp. 612-615.

Fluorine-19 and natural abundance  $^{17}O$  and  $^{183}W$  NMR spectroscopy were employed for the characterization of aqueous solutions of  $(NH_4)_2WO_2F_4$  and  $(NH_4)_3WO_3F_3$ . Dissolution of the  $(NH_4)_2WO_2F_4$  complex is accompanied by its partial acid hydrolysis to give the *trans(mer)*-dimer,  $[W_2O_5F_6]^{4-}$ , and unreacted *cis*- $[WO_2F_4]^{2-}$ . The *cis(fac)*- $[W_2O_5F_6]^{4-}$  anion is the major soluble product resulting from the alkaline hydrolysis of  $(NH_4)_2WO_2F_4$  along with the isolation of the solid  $(NH_4)_2WO_3F_2$ . In addition, the edge-bridging dimer,  $[W_2O_6F_4]^{4-}$ , and the cyclic

trimer,  $[W_3O_9F_6]^{6-}$ , are also suggested as hydrolysis products. Decomposition of  $(NH_4)_3WO_3F_3$  occurs in aqueous solution to give  $NH_4WO_3F$ .

#### APPLICATION OF NMR SPECTROSCOPY AND IMAGING IN HETEROGENEOUS BIOCATALYSIS

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*Appl. Magn. Reson.*,  
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Heterogeneously catalyzed enzymatic glucose isomerization was considered as a model process to extend the application of nuclear magnetic resonance (NMR) and magnetic resonance imaging techniques to the studies of biocatalytic processes and heterogeneous biocatalysts. It has been demonstrated that the  $T_2$  times of glucose are different for its aqueous solution in the pores of an unmodified porous support and in a heterogeneous biocatalyst, comprising bacterial cells immobilized on the same support. This observation has been used to map the spatial distribution of the active component within a packed bed of biocatalyst in a model reactor.  $^{13}C$  NMR spectroscopy was applied to follow the progress of glucose isomerization catalyzed by the heterogeneous biocatalyst in a batch reactor. The utilization of proton spin decoupling and nuclear Overhauser effect was shown to be necessary to obtain high signal-to-noise ratio in the natural abundance  $^{13}C$  NMR spectra of a glucose–fructose syrup present in the packed bed of biocatalyst. The spectra thus obtained were suitable for the quantification of the glucose-to-fructose ratio achieved in the biocatalytic reaction.

#### MAGNETIC RESONANCE IMAGING METHODS FOR *in situ* STUDIES IN HETEROGENEOUS CATALYSIS

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*Chem. Soc. Rev.*,  
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Magnetic resonance imaging (MRI) is a very powerful instrument used extensively in modern medical diagnostics because of its ability to look inside a body in a non-invasive and non-destructive way. Furthermore, MRI is more than just a single tool for extracting structural information. It is more of a sophisticated and versatile toolkit able to provide all sorts of useful information about the internal properties of an object under study and various

processes within it, including heat and mass transport, composition and chemical transformations, in a spatially resolved mode. While a living body is different from a catalyst body or a reactor, the *in situ* and Operando studies in catalysis can clearly benefit from the use of this non-destructive toolkit as a powerful complement to other available spectroscopic tools. This *tutorial review* gives an introduction to the field and describes the examples of the applications of MRI to the studies of the preparation, deactivation and regeneration of solid catalysts and to the spectroscopy, thermometry and imaging studies of heterogeneous catalysts and model catalytic reactors performed during actual catalytic processes.

#### MAGNETIC RESONANCE IMAGING AS AN EMERGING TOOL FOR STUDYING THE PREPARATION OF SUPPORTED CATALYSTS

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374(1-2) (2010) pp. 126-136.

Several magnetic resonance imaging (MRI) approaches are presented that can be used to study transport processes when supported catalysts are prepared by impregnating mm-sized porous support bodies. Both diamagnetic and paramagnetic components of an impregnation solution such as the solvent, the metal-containing precursors of an active component, and various organic and inorganic additives can be visualized with MRI. Their spatial distributions within a catalyst support body and the evolution of these distributions in time can be characterized not only qualitatively but also quantitatively, if calibration procedures are used to convert the images into corresponding concentration profiles. In particular, the physicochemical interactions between  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> support bodies and the aqueous solutions containing Cu, Co, Mo, citrate and phosphate ions are investigated upon pore volume impregnation as a function of concentration and solution pH. Furthermore, it is shown that multinuclear and solid state MRI can be used to map the solid phase distribution of the active component precursors and additives within the impregnated support bodies after their drying. Based on the case studies discussed, it can be concluded that MRI is an emerging versatile tool for studying the transport processes in support bodies upon their impregnation and the distribution of the key components after drying of the impregnated bodies.

#### MODERN SOLID-STATE NMR SPECTROSCOPY OF QUADRUPOLAR NUCLEI

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The potential of modern solid-state NMR spectroscopy techniques applied to quadrupolar nuclei with half-integer spin is demonstrated. Correlations of NMR parameters with the local environment of nucleus (for 14 nuclei) are presented in the second part of the review.

#### SELF-AGGREGATION AND ORIENTATION OF THE ION CHANNEL-FORMING ZERVAMICIN IIA IN THE MEMBRANES OF ePC VESICLES STUDIED BY cw EPR AND ESEEM SPECTROSCOPY

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*Appl. Magn. Reson.*,  
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The ion channel-forming peptide antibiotic zervamicin A was studied in egg phosphocholin lipid membranes of large multilamellar vesicles (LMV) at 77 K. Continuous wave electron paramagnetic resonance (EPR) and electron spin echo envelope modulation (ESEEM) methods combined with site-specific electron spin labeling were used to study the aggregation and immersion depth of two analog molecules, i.e., each monolabeled either at the N- or C-terminal end of the helical molecule. Analysis of the shape of the EPR spectra indicates that zervamicin molecules form aggregates in which the dipolar interaction between the spin labels at the N-terminus is substantially larger than that between the labels at the C-terminus. The ESEEM method was used to study the interaction between the nitroxide radical spin labels of the zervamicin molecules and deuterium nuclei in LMV, which were prepared using a D<sub>2</sub>O buffer. It is established that the largest amplitude of deuterium modulation of the unpaired electron is observed for zervamicin molecules labeled at the N-terminus. Based on the analysis of the Fourier parameters of the deuterium modulated spectrum, a model of the immersion depth of the terminal ends of the zervamicin molecule in a lipid bilayer is



formulated. All of the spin labels at the N-terminus are grouped at the lipid–water interface, whereas 60% of labels at the C-terminus are located at the lipid–water interface and 40% are more deeply inserted into the lipid bilayer.

#### **FEATURES OF THE REAL STRUCTURE OF PSEUDOBOEHMITES: VIOLATIONS OF THE STRUCTURE AND LAYER PACKING CAUSED BY CRYSTALLIZATION WATER**

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*J. Struct. Chem.*,  
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Rietveld structure refinement and simulation of the diffraction patterns of partially disordered materials are used to study the real structure of nanoscale pseudoboehmite samples obtained by different technologies. The effect of various violations in the structure of these nanomaterials on diffraction patterns is analyzed. The introduction of corrections for the Lorentz and polarization factors in the determination of the cell parameter  $b$  by the position of the 020 diffraction peak in the pattern is shown to be important. A model for the atomic structure of pseudoboehmite is proposed. The model involves additional water molecules as compared to the structure of boehmite. The water molecules in the interlayer space of the layered boehmite structure are found to violate its regularity, which results in a decrease in the size of crystallites.

#### **STRUCTURAL FEATURES OF FINELY DISPERSED PSEUDOBOEHMITE OBTAINED BY A SOL-GEL METHOD**

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*J. Struct. Chem.*,  
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Simulation of diffraction patterns, radial distribution function analysis, and electron microscopy are applied to study the features of the structure of nanosized pseudoboehmite obtained by the sol-gel method. It is found to consist of plate-like particles with a thickness of one lattice constant in the [010] direction. Such a structure of the pseudoboehmite particles results in the absence of the 020 diffraction peak in the diffraction pattern.

#### **CHEMICAL AND STRUCTURAL TRANSFORMATIONS IN MANGANESE ALUMINUM SPINEL OF THE COMPOSITION $Mn_{1.5}Al_{1.5}O_4$ DURING HEATING AND COOLING IN AIR**

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*J. Struct. Chem.*,  
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Single phase cubic spinel of the composition  $Mn_{1.5}Al_{1.5}O_4$  is synthesized. Its crystal structure refinement shows that  $0.4Mn+0.6Al$  are in the octahedral sites and  $0.7Mn+0.3Al$  are in the tetrahedral sites. High temperature X-ray diffraction is used to analyze  $Mn_{1.5}Al_{1.5}O_4$  behavior during heating and cooling in air. In a temperature range of  $600^{\circ}C$  to  $700^{\circ}C$ , initial spinel splits into layers, and the sample represents a two-phase system: cubic spinel  $Mn_{0.4}Al_{2.4}O_4$  and a phase based on  $\beta-Mn_3O_4$ . Above  $900^{\circ}C$  the sample again turns into single phase cubic spinel. The role of oxidizing processes in the decomposition of  $Mn_{1.5}Al_{1.5}O_4$  caused by oxygenation and partial oxidation of  $Mn^{2+}$  to  $Mn^{3+}$  is shown. A scheme of structural transformations of manganese aluminum spinel during heating from room temperature and cooling from  $950^{\circ}C$  is proposed.

#### **HIGH-TEMPERATURE STUDY OF $SrFe_{1-x}Mo_xO_{3-z}$ PEROVSKITES**

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The structure of high-temperature  $SrFe_{1-x}Mo_xO_{3-z}$  ( $0 \leq x \leq 0.5$ ) phases was studied. Such studies are necessary to understand the mechanism of oxygen transport in membrane materials used for high-temperature oxygen separation.

## ULTRAFINE Si/Al<sub>2</sub>O<sub>3</sub> COMPOSITES OBTAINED BY COMBINING METHODS OF MECHANICAL ACTIVATION AND SELF-PROPAGATING HIGH-TEMPERATURE SYNTHESIS

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*Combust. Explosion Shock Waves*,  
46(1) (2010) pp. 36-40.

Mechanical activation of a mixture of silicon oxide and aluminum is studied by methods of infrared spectroscopy, differential thermal analysis, and X-ray diffraction. If a SiO<sub>2</sub>/Al mechanocomposite is used as a precursor, then a Si/Al<sub>2</sub>O<sub>3</sub> composite with a small grain size and a uniform distribution of the components can be obtained by the method of self-propagating high-temperature synthesis.

## ON THE PROBLEM OF REACTOR GRAPHITE LIFETIME

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Techn.*, 4(3) (2010) pp. 442-451.

Fine-grained dense graphites of different manufacturers were tested and studied for the purpose of predicting their lifetimes at high (~2000°C) temperatures. The tests consisted in sample heating by electric current to a destruction temperature. The study included a set of electron microscopy, X-ray diffraction, and electrical methods. X-ray diffraction data for initial samples were compared with data for domestic MPG graphite. Scanning electron microscopy was applied to both initial and heated samples. The lifetime prediction was based on the Zhurkov classical formula. It was shown that CGD, Carbone Lorraine, and MPG graphite composites can differ appreciably in both structural characteristics and high-temperature lifetimes.

## MICELLAR SYNTHESIS AND CHARACTERIZATION OF ULTRADISPERSED POWDERS OF AMMONIUM NITRATE

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*J. Struct. Chem.*,  
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Reverse micelles of oxyethylated nonylphenol (Triton N-42) were used to synthesize ultradispersed powder of ammonium nitrate with the yield 75% and content 79%. Its composition and morphology were examined by colorimetry, IR Fourier spectroscopy, scanning electron microscopy (SEM) and X-ray diffraction analysis (XRD). According to XRD, the sample is stable with time, although it is a mixture of three polymorphic forms of crystal ammonium nitrate — one stable and three metastable forms. The agglomerates consists of 20-50 nm nanoparticles; a part of nanoparticles form filaments of length 0.5-1.5 μm and thickness 25-30 nm, as estimated by SEM.

## TWO MECHANISMS OF THERMAL EXPANSION IN PEROVSKITE

SrCo<sub>0.6</sub>Fe<sub>0.2</sub>Nb<sub>0.2</sub>O<sub>3-z</sub>

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*J. Therm. Anal. Calorim.*,  
100(1) (2010) pp. 79-82.

Perovskite SrCo<sub>0.6</sub>Fe<sub>0.2</sub>Nb<sub>0.2</sub>O<sub>3-z</sub> attracts attention as a promising material with high oxygen conductivity. The sample was investigated by means of high-temperature X-ray powder diffraction and thermogravimetry. Phase transition was detected near 400°C and accompanied with significant mass loss. The phase transition affects oxygen mobility, important for the synthesis of oxygen permeable membranes. The unit cell parameters are proved to change with temperature after two effects (1) reversible conventional thermal expansion and (2) irreversible contraction-expansion due to the changes in the oxygen content. *In situ* high-temperature X-ray diffraction experiments allowed to separate the contributions and to measure them as a function of temperature.

**CORRELATION BETWEEN HIGH-TEMPERATURE STRUCTURAL REARRANGEMENTS AND OXYGEN OUTLET IN PEROVSKITE  $\text{SrCo}_{0.6}\text{Fe}_{0.2}\text{Nb}_{0.2}\text{O}_{3-z}$**

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*Solid State Phenom.*,  
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Strontium cobaltite doped with Fe and Nb,  $\text{SrCo}_{0.6}\text{Fe}_{0.2}\text{Nb}_{0.2}\text{O}_{3-z}$  with perovskite-like structure, being one of the promising materials for oxygen permeable membrane implementation, has been investigated via *in situ* high temperature X-ray powder diffraction on Bruker D8 Advance instrument and high-resolution powder diffraction at synchrotron radiation beamline. The oxygen outlet from the lattice has been found to stay in correlation with the structural rearrangement forming two phases with different oxygen deficiency. The activation energy of phase splitting process has been estimated to be  $E_{\text{activation}} \approx 0.63 \pm 0.06$  eV.

**USING *ex situ* X-RAY POWDER DIFFRACTION AT SYNCHROTRON RADIATION TO FOLLOW CHANGES IN THE PHASE COMPOSITION OF PEROVSKITE-LIKE STRONTIUM COBALTITES**

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Precision X-ray diffraction at synchrotron radiation was used to reveal the separation of perovskite-like oxides  $\text{SrCo}_{0.8-x}\text{Fe}_{0.2}\text{Nb}_x\text{O}_{3-z}$  ( $x = 0.2$  and  $0.3$ ) into two phases of a similar structure identical to initial perovskite structure, but having different unit cell parameters and supposedly different oxygen deficiency. The structural transformation is accompanied by oxygen outlet from the structure. The study of oxygen atoms intercalation from air into the oxygen-deficient structure showed that the structural changes are reversible: heating to  $400^\circ\text{C}$  in air restores the initial state of samples.

**CONDENSATION OF A TETRAHEDRA RIGID-BODY LIBRATION MODE IN  $\text{HoBaCo}_4\text{O}_7$ : THE ORIGIN OF PHASE TRANSITION AT 355 K**

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*New J. Phys.*,  
12 (2010) 043035.

Rietveld profiles, Mössbauer spectra and X-ray absorption fine structure (XAFS) were used to analyze the source of the structural phase transition at  $T_s=355$  K in  $\text{HoBaCo}_4\text{O}_7$ . Excess of oxygen content over 'O<sub>7</sub>' was avoided by annealing the samples in an argon flow at  $600^\circ\text{C}$ . Space groups (S.G.)  $Pbn2_1$  and  $P6_3mc$  were used to refine the structure parameters in the low- and high-temperature phases, respectively. Additionally, the  $Cmc2_1$  symmetry was considered as a concurrent model of structure of the low-temperature phase. In the high-temperature phase, severe anisotropy of thermal motion of the majority of the oxygen atoms was observed. The variation of quadrupole splitting  $\varepsilon$  near  $T_s$  is not similar to the step-like anomaly frequently seen at the charge-ordering transition. The authors observe instead a dip-like anomaly of the average  $\varepsilon$  near  $T_s$ . Narrow distribution of the electric field gradient (EFG) over different cobalt sites is observed. XAFS spectra show no evidence of significant difference between  $\text{YBaCo}_4\text{O}_7$  ( $T > T_s$ ) and  $\text{HoBaCo}_4\text{O}_7$  ( $T < T_s$ ). The origin of the transition at  $T_s$  is ascribed to the condensation of the libration phonon mode associated with the rigid-body rotational movements of tetrahedra within the star-like tetrahedral units, the building blocks of the Kagomé network. It is shown that the condensation of the libration mode is not compatible with translation symmetry for the hexagonal S.G., but compatible for the orthorhombic S.G. The orthorhombic lattice parameters and EFG components ( $V_{xx}$ ,  $V_{yy}$  and  $V_{zz}$ ) vary smoothly with temperature on approaching  $T_s$ . The authors argue that such a continuous variation supports the plausible scenario of librating tetrahedra, although the librational excitations propagate, most probably, differently than plane waves.

## CONSTRUCTION OF THE MODEL RADIAL DISTRIBUTION CURVES WITH REGARD TO THE FEATURES OF X-RAY DIFFRACTION EXPERIMENT

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*J. Struct. Chem.*,  
51(2) (2010) pp. 274-280.

A method to construct model radial distribution functions (RDFs) from the already known structural data is described. The method includes the procedures to calculate termination ripples that always appear on the experimental RDF because of the bounds of the integration limits in the Fourier transformation of the X-ray scattering curve. The introduction of this procedure increases the accuracy of the comparative RDF method used to elucidate the phase composition of nanodispersed materials and to determine the features of the local structure of the phases as compared to their well crystallized analogues. Cerium dioxide samples with different dispersion exemplify the applicability of this method to determine the features of the local structure.

## X-RAY PHOTOELECTRON SPECTRA OF PLATINUM COMPOUNDS EXCITED WITH MONOCHROMATIC $AgL_{\alpha}$ IRRADIATION

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*J. Electron. Spectrosc. Relat. Phenom.*,  
177(1) (2010) pp. 15-18.

Non-monochromatic  $MgK_{\alpha}$  and monochromatic  $AgL_{\alpha}$  irradiations were used to measure Pt4f and Pt3d<sub>5/2</sub> X-ray photoelectron spectra from platinum black and from several platinum compounds with different Pt oxidation states. The Pt3d<sub>5/2</sub> core level binding energies from platinum compounds were measured for the first time. Potential of these data to XPS study of Pt/Al<sub>2</sub>O<sub>3</sub> catalysts is demonstrated.

## AN *in situ* CELL FOR INVESTIGATION OF THE CATALYST STRUCTURE USING SYNCHROTRON RADIATION

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*J. Struct. Chem.*,  
51(suppl.) (2010) pp. S20-S27.

Design of a cell for *in situ* characterization of heterogeneous catalysts by X-ray absorption spectroscopy EXAFS/XANES and X-ray diffraction on synchrotron radiation at high temperatures in a controllable gas medium, which was implemented at the Structural Materials Science end-station installed at KCSRNT, is presented. First results on nanostructural evolution of Pt/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub>, Pd/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub> and other catalysts during various treatments — oxidation in oxygen, reduction in H<sub>2</sub>/N<sub>2</sub>, and annealing in vacuum — are reported.

## ALLOY CATALYST IN A REACTIVE ENVIRONMENT: THE EXAMPLE OF Ag-Cu PARTICLES FOR ETHYLENE EPOXIDATION

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*Phys. Rev. Lett.*,  
104(3) (2010) 035503 (4 pp.)

Combining first-principles calculations and *in situ* photoelectron spectroscopy, it was shown how the composition and structure of the surface of an alloy catalyst is affected by the temperature and pressure of the reagents. The Ag-Cu alloy, recently proposed as an improved catalyst for ethylene epoxidation, forms a thin Cu-O surface oxide, while a Ag-Cu surface alloy is found not to be stable. Several possible surface structures are identified, among which the catalyst surface is likely to dynamically evolve under reaction conditions.

## ELECTRONIC STRUCTURE OF ALUMINUM OXIDE: *ab initio* SIMULATIONS OF $\alpha$ AND $\gamma$ PHASES AND COMPARISON WITH EXPERIMENT FOR AMORPHOUS FILMS

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*Eur. Phys. J. – Appl. Phys.*,  
52(3) (2010) pp. 30501-1-7.

The electronic structure of  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> and  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> has been investigated by means of the density functional theory. A comparison of the calculation results with experimental data for amorphous alumina films is also presented. The electronic structure is described in terms of band structure and density of states. It has been found that  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> have similar electronic structure with  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> and amorphous Al<sub>2</sub>O<sub>3</sub>. Effective electron masses in  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> as well as in  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> equal to  $m^*_e \approx 0.4m_0$  that is in a good agreement with the experimentally found tunnel electron mass in amorphous Al<sub>2</sub>O<sub>3</sub>. The heavy holes in both alumina crystals are explained by the valence band top forming by O  $2p_\pi$  nonbonding orbitals.

## MOCVD AND PHYSICOCHEMICAL CHARACTERIZATION OF (HfO)<sub>x</sub>(Al<sub>2</sub>O<sub>3</sub>)<sub>1-x</sub> THIN FILMS

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*Chem. Vap. Deposition*,  
16(4-6) (2010) pp. 185-190.

Investigation of the thermal behavior of Hf(dpm)<sub>4</sub> vapor in vacuum is carried out. An approach for obtaining films of (HfO)<sub>x</sub>(Al<sub>2</sub>O<sub>3</sub>)<sub>1-x</sub> alloys is developed. The use of a source containing a mixture of two precursors differing from each other in volatility, notably Hf(dpm)<sub>4</sub> (dpm=dipivaloyl-methanate, 2,2,6,6-tetramethylheptane-3,5-dionate) and Al(acac)<sub>3</sub> (acac=pentane-2,4-dionate), allows to employ the possibilities of combinatorial chemistry, and to obtain and analyze a number of different compositions of alloys. Film characterization is performed using a set of analytical methods. The character of Al and Hf distributions across the film thickness, depending on their content in the source, is investigated. The use of laser null ellipsometry allows to determine the distribution of the refractive index across the thickness of the (HfO)<sub>x</sub>(Al<sub>2</sub>O<sub>3</sub>)<sub>1-x</sub> film. A comparison of the ellipsometric data with the X-ray photoelectron

spectroscopy (XPS) data shows that the character of the refractive index variation reproduces the variation of the chemical composition across the film thickness. A uniform distribution of the elements across the film thickness is observed in the cases when separated sources are used. The structure of the film depends on the molar fraction of Al<sub>2</sub>O<sub>3</sub> in alloys. For Al $\geq$ 30 at.% amorphization of the structure occurs.

## INTERFACES ANALYSIS OF THE HfO<sub>2</sub>/SiO<sub>2</sub>/Si STRUCTURE

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*J. Phys. Chem. Solids*,  
71(5) (2010) pp. 836-840.

The physical and chemical properties of the HfO<sub>2</sub>/SiO<sub>2</sub>/Si stack have been analyzed using cross-section HR TEM, XPS, IR-spectroscopy and ellipsometry. HfO<sub>2</sub> films were deposited by the MOCVD method using as precursors the tetrakis 2,2,6,6 tetramethyl-3,5 heptanedionate hafnium—Hf(dpm)<sub>4</sub> and dicyclopentadienyl-hafnium-bis-diethylamide—Cp<sub>2</sub>Hf(N(C<sub>2</sub>H<sub>5</sub>)<sub>2</sub>)<sub>2</sub>.

The amorphous interface layer (IL) between HfO<sub>2</sub> and silicon native oxide has been observed by the HRTEM method. The interface layer comprises hafnium silicate with a smooth varying of chemical composition through the IL thickness. The interface layer formation occurs both during HfO<sub>2</sub> synthesis, and at the annealing of the HfO<sub>2</sub>/SiO<sub>2</sub>/Si stack. It was concluded from the XPS, and the IR-spectroscopy that the hafnium silicate formation occurs via a solid-state reaction at the HfO<sub>2</sub>/SiO<sub>2</sub> interface, and its chemical structure depends on the thickness of the SiO<sub>2</sub> underlayer.

**FORMATION OF THIN NANOSTRUCTURED LAYERS DURING HETEROGENEOUS GAS-PHASE SYNTHESIS FROM SMALL-SIZE VOLATILE METAL COMPLEXES ON THE SURFACE OF SEMICONDUCTORS AND DIELECTRICS**

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*Techn. Phys. Lett.*,  
36(3) (2010) pp. 265-268.

A new method for the formation of thin nanostructured layers on semiconductor and dielectric substrates by heterogeneous gas-phase synthesis from small-sized volatile metal complexes has been developed and verified. Thin nanostructured layers of copper on silicon and silica substrates were successfully formed from small-sized volatile formate metal complexes using a combined synthesis-transfer process. It is established that copper in the layers deposited by this method predominantly occurs in the metallic ( $\text{Cu}^0$ ) state in nanosized grains with a characteristic close-packed structure.

**COMPOSITION AND MORPHOLOGY OF FLUORINATED ANODIC OXIDES ON InAs (111)A SURFACE**

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*Appl. Surf. Sci.*,  
256(19) (2010) pp. 5722-5726.

The composition and morphology of fluorinated anodic oxide (FAO) films grown on InAs (1 1 1)A in alkaline aqueous (pH 11.5) and acid waterless (pH 1.5) electrolytes are studied by means of X-ray photoelectron spectroscopy (XPS), atomic force microscopy (AFM) and high resolution transmission electron microscopy (HRTEM) in order to reveal the passivation mechanism of fluorine on the FAO/InAs(1 1 1)A interface. The formation of the highest oxidation form of  $\text{As}^{+5}$  and passivation of defects in the FAO layers during the fluorination process explain the reduction of the density of surface states and unpinning of the Fermi level on the fluorinated AO/InAs(1 1 1)A interface.

**THE ESCA ANALYSIS OF THE  $\text{B}_3\text{H}_8^-$  ANION**

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*Russ. J. Gen. Chem.*,  
80(8) (2010) pp. 1550-1556.

Compounds  $[\text{Et}_4\text{N}]_2\text{B}_3\text{H}_8$  and  $\text{CsB}_3\text{H}_8$  are studied using the ESCA method. The results of analysis of the B1s electron spectra and estimation of the effective charge differences in  $[\text{Et}_4\text{N}]_2\text{B}_3\text{H}_8$  are compared to the data of theoretical calculations of the  $\text{B}_3\text{H}_8^-$  anion.

**STRUCTURE OF Ir AND Ir- $\text{Al}_2\text{O}_3$  COATINGS OBTAINED BY CHEMICAL VAPOR DEPOSITION IN THE PRESENCE OF OXYGEN**

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*J. Struct. Chem.*,  
51(1) (2010) pp. 82-91.

By chemical vapor deposition Ir and Ir- $\text{Al}_2\text{O}_3$  coatings are obtained with a thickness of up to 40  $\mu\text{m}$  on steel substrates precoated with a layer of  $\text{Al}_2\text{O}_3$ . Tris-acetylacetonates of iridium(III) and aluminium(III) are used as precursors. The deposition processes are carried out at atmospheric pressure in the presence of oxygen. The obtained coatings are studied by X-ray diffraction, X-ray photoelectron spectroscopy, and scanning electron microscopy. The dependences of coating structures and compositions on the preparation conditions are found. An increase in the deposition temperature results in the formation of Ir coatings with loose discontinuous structure, an increase in the size of metal crystallites, and the growth of the oxygen concentration in their composition. An increase in the concentration of precursor vapors in the deposition zone at a constant deposition temperature results in the formation of Ir coatings that consist of differently structured layers (compact, columnar, and granular). Mixed Ir- $\text{Al}_2\text{O}_3$  coatings which composed of metal Ir and amorphous  $\text{Al}_2\text{O}_3$  crystallites, which exhibit a pronounced iridium texture in the [111] direction, have the most perfect compact structure. The introduction of the oxide phase in the coating composition halves the Ir crystallite size.

### COMPARATIVE XPS STUDY OF Rh/Al<sub>2</sub>O<sub>3</sub> AND Rh/TiO<sub>2</sub> AS CATALYSTS FOR NaBH<sub>4</sub> HYDROLYSIS

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*Int. J. Hydrogen Energy*,  
35(13, sp. issue) (2010) pp. 6501-6507.

The catalysts Rh/Al<sub>2</sub>O<sub>3</sub> and Rh/TiO<sub>2</sub> for hydrogen production from NaBH<sub>4</sub> were prepared by deposition technique from RhCl<sub>3</sub> reduced by NaBH<sub>4</sub> and were studied by XPS and TEM. It was found that the RhCl<sub>3</sub>/Al<sub>2</sub>O<sub>3</sub> system is more stable comparing to RhCl<sub>3</sub>/TiO<sub>2</sub> which starts to decompose by weak heat treatment. It was shown that NaBH<sub>4</sub> reduced RhCl<sub>3</sub>/TiO<sub>2</sub> (Al<sub>2</sub>O<sub>3</sub>) to supported metal Rh nanoparticles in both cases. In the case of Rh/TiO<sub>2</sub> SMSI effect was found after RT reduction. The SMSI (Strong Metal-Support Interaction) effect gave an explanation for the difference of activity between Rh/TiO<sub>2</sub> and Rh/Al<sub>2</sub>O<sub>3</sub> catalysts in hydrolysis reaction of NaBH<sub>4</sub>.

### RHODIUM CATALYSTS FOR THE HYDROGENATION OF BENZENE AND ITS HOMOLOGUES IN VARIOUS SOLVENTS

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*Pet. Chem.*,  
50(1) (2010) pp. 47-50.

The creation of active hydrogenation catalysts for small amounts of benzene can solve the problem of the complete removal of gasoline from benzene. A set of rhodium catalysts supported on Sibunit, wood (birch) activated carbon (BAU), and fruit-stone activated carbon KAU is proposed. It has been shown that the catalyst activity depends to a large degree on the nature of the support, on the structure of the aromatic compound, and on the reaction medium. The characteristics of the charged state of rhodium on the surface of these studied catalysts have been determined by means of X-ray photoelectron spectroscopy.

### INFLUENCE OF POLOXAMER 407 ON FRACTIONAL AND SUBFRACTIONAL COMPOSITION OF SERUM LIPOPROTEINS OF MICE

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*HealthMED*,  
2(7) (2010) pp. 722-730.

Using a novel small-angle X-ray scattering (SAXS) method for determination of fractional and subfractional composition of lipoproteins (LPs), a significant elevation of total cholesterol-lipoproteins (C-LP) and, especially, total triglyceride-lipoproteins (TG-LP), was shown in this work.

### FRACTIONAL COMPOSITION OF BLOOD SERUM LIPOPROTEINS IN MICE AND RATS WITH TRITON WR 1339-INDUCED LIPEMIA

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*Bull. Exp. Biol. Med.*,  
149(5) (2010) pp. 567-570.

Fractional composition of blood serum lipoproteins (LP) in female ICR mice and Wistar rats induced by single administration of a nonionic detergent Triton WR 1339 in doses of 300 and 500 mg/kg were compared. Lipemia in animals of both species was characterized by a sharp increase in the concentration of cholesterol and, particularly, of triglycerides in blood serum lipoproteins by the 24th hour after administration of the detergent. A significant increase in the concentrations of atherogenic VLDL cholesterol (due to VLDL<sub>2</sub>), intermediate density lipoproteins, and LDL was revealed. These changes were more pronounced in rats. The model of lipemia can be used to study the role of fractional composition of lipoproteins and, particularly, of triglycerides in the pathogenesis of atherosclerosis. Moreover, this model holds much promise for evaluation of the efficiency of hypolipidemic drugs (statins and fibrates) in normalizing the increased level of atherogenic cholesterol of VLDL and LDL.

### INFLUENCE OF POLOXAMER 407 ON FRACTIONAL AND SUBFRACTIONAL COMPOSITION OF SERUM LIPOPROTEINS OF MICE

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*Bulletin SB RAMS,*  
30(5) (2010) pp. 70-75.

Nonionic detergent poloxamer 407 (Pluronic F-127) is suggested to be used in pharmaceutical industry as nanoparticles for delivering of medicinal substances. The composition of lipoproteins (LPs) has been determined with a small-angle X-ray scattering method.

### A NEW METHOD FOR ESTIMATING THE DISPERSITY OF DEPOSITED METALLIC NANOPARTICLES AND EXTENT OF THEIR INTERACTION WITH THE SUPPORT MATRICES

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*J. Struct. Chem.,*  
51(suppl.) (2010) pp. S7-S16.

A new technical approach to investigation of heterogeneous catalysts with deposited metallic nanoparticles of the active component is suggested. Small-angle X-ray scattering (SAXS) patterns for powder samples of the support matrices with deposited metal particles (Pt, Pd and others) taken at varying density contrast are shown to provide essential information on the structure and dispersity of the active components and their interaction with the support surface, which allows an integral assessment of the formation of strong chemical binding of deposited metal to the support matrix. The measured structural characteristics are compared with data obtained by other physicochemical methods, in particular, transmission electron microscopy (TEM). The suggested technique is verified using mathematical models and applied to a real heterogeneous catalyst Pt/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub>. The proposed method of SAXS data analysis makes it possible to reveal the formation of active sites in heterogeneous catalysts and optimize their preparation procedures.

### FTIR SPECTROSCOPY AND THERMODYNAMICS OF CO AND H<sub>2</sub> ADSORBED ON $\gamma$ -, $\delta$ - AND $\alpha$ -Al<sub>2</sub>O<sub>3</sub>

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*Phys. Chem. Chem. Phys.,*  
12(24) (2010) pp. 6474-6482.

The adsorption of CO and H<sub>2</sub> at the surface of transitional ( $\gamma$  and  $\delta$ ) and corundum ( $\alpha$ ) phases of Al<sub>2</sub>O<sub>3</sub> is studied by means of FTIR spectroscopy at temperature variable in the 293-60 K (CO) and 293-20 K (H<sub>2</sub>) intervals with the aims of better clarifying the nature of the surface Lewis centres and evaluate the thermodynamics of the adsorption process.

### EXPERIMENTAL STUDY BY THE IR SPECTROSCOPY METHOD OF THE INTERACTION BETWEEN ETHYLENE AND NANOPORES OF VARIOUS DENSITIES

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*Atmos. Oceanic Opt.,*  
23(4) 2010) pp. 266-269.

The combination vibrational bands  $\nu_5 + \nu_9$  and  $\nu_{11} + \nu_{11}$  of ethylene absorption adsorbed by silica aerogel nanopores of different densities have been studied for the first time in the 5700 to 6300 cm<sup>-1</sup> spectral region. The conducted measurements show significant differences between the spectra of ethylene in aerogels and ethylene in the gas phase, which consist of the change of the absorption band shapes, the shift of the band frequency, and the increase of the absorption intensity. It was concluded that in the studied pressure range of 88–952 mbar, the adsorbed ethylene is in the same structural state.



## PARTICLE IDENTIFICATION AEROGEL COUNTER WITH $n=1.13$ FOR p/K SEPARATION

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*Nucl. Instrum. Methods Phys. Res., Sect. A,*  
A623(1) (2010) pp. 336-338.

The paper is devoted to the cosmic muon tests of particle identification system for the SND detector at the new VEPP-2000  $e^+e^-$  collider. the goal of the tests is to measure average signal for the relativistic particles. the measurements of the average signal were done at five different points of the counter. obtained values of the signal are close to 10 photoelectrons. preliminary results for misidentification probability for  $\pi$  and K mesons with the energy of 1000 MeV are presented.

## LIGHT SCATTERING IN A SURFACE GLASS LAYER PRODUCED BY A SURFACE ION EXCHANGE PROCESS

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*Glass Technol.: A,*  
51(6) (2010) pp. 241-247.

The optical properties of glass with a surface layer modified by a surface ion exchange process are investigated. Using a surface ion exchange paste (SIEP) for glass surface modification leads to the formation of a special surface structure with system of microblocks ( $\sim 50$ - $150$   $\mu\text{m}$  in size) separated by microcracks ( $\sim 100$ - $200$  nm wide). This structure was investigated by scanning electron microscopy (SEM). It is shown that the formation of this surface glass structure determines the spectral characteristics of the scattered light, namely their angular dependences and extinction characteristics. The effectiveness of light propagation in the glass in the full internal reflection regime was investigated. Optical and morphological results obtained are essentially different from those for glasses that have not been treated and for glass with a surface layer produced by a well known and widely used glass chemical etching process. The results may

be very important for the use of glass in illumination and decoration.

## OXYGEN DESORPTION FROM POLYCRYSTALLINE PALLADIUM: THERMAL DESORPTION OF $\text{O}_2$ FROM A CHEMISORBED LAYER OF $\text{O}_{\text{ads}}$ IN THE COURSE OF THE DECOMPOSITION OF PdO SURFACE OXIDE AND IN THE RELEASE OF OXYGEN FROM THE BULK OF PALLADIUM

A.N. Salanov, E.A. Suprun

*Kinet. Catal.,*  
51(3) (2010) pp. 416-427.

The desorption of oxygen from polycrystalline palladium (Pd(poly)) was studied using temperature-programmed desorption (TPD) at 500–1300 K and the amounts of oxygen absorbed by palladium ( $n$ ) from 0.05 to 50 monolayers. It was found that the desorption of  $\text{O}_2$  from Pd(poly), which occurred from a chemisorbed oxygen layer ( $\text{O}_{\text{ads}}$ ), in the release of oxygen from a near-surface metal layer in the course of the decomposition of PdO surface oxide, and in the release of oxygen from the bulk of palladium ( $\text{O}_{\text{abs}}$ ), was governed by repulsive interactions between  $\text{O}_{\text{ads}}$  atoms and the formation and decomposition of  $\text{O}_{\text{ads}}\text{-Pd}^*\text{-O}_{\text{abs}}$  structures (Pd\* is a surface palladium atom). At  $\theta \leq 0.5$ , the repulsive interactions between  $\text{O}_{\text{ads}}$  atoms ( $\varepsilon_{\text{aa}} = 10$  kJ/mol) resulted in the desorption of  $\text{O}_2$  from Pd(poly) at 650–950 K. At  $0.5 \leq n \leq 1.0$ , the release of inserted oxygen from a near-surface palladium layer occurred during TPD in the course of the migration of  $\text{O}_{\text{abs}}$  atoms to the surface and the formation–decomposition of  $\text{O}_{\text{ads}}\text{-Pd}^*\text{-O}_{\text{abs}}$  structures. As a result, the desorption of  $\text{O}_2$  occurred in accordance with a first-order reaction with a thermal desorption (TD) peak at  $T_{\text{max}} \sim 700$  K. At  $1.0 \leq n \leq 2.0$ , the decomposition of PdO surface oxide occurred at a constant surface coverage with oxygen during TPD in the course of the formation–decomposition of  $\text{O}_{\text{ads}}\text{-Pd}^*\text{-O}_{\text{abs}}$  structures. Because of this, the desorption of  $\text{O}_2$  occurred in accordance with a zero-order reaction at low temperatures with a TD peak at  $T_{\text{max}} \sim 675$  K. At  $1.0 \leq n \leq 50$ , oxygen atoms diffused from deep palladium layers in the course of TPD and arrived at the surface at high temperatures. As a result,  $\text{O}_2$  was desorbed with a high-temperature TD peak at  $T > 750$  K.

**CHEMICAL COMPOSITION AND STRUCTURE OF THE SHELL OF FLY ASH NON-PERFORATED CENOSPHERES PRODUCED FROM THE COMBUSTION OF THE KUZNETSK COAL (RUSSIA)**

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*Fuel*,  
89(8) (2010) pp. 1849-1862.

The relationship between the composition and structure of the shell of low-density non-perforated cenospheres of fly ashes from the combustion of coal from the Kuznetsk Basin (Russia) at temperatures of 1500 and 1650°C has been investigated. Narrow fractions of cenospheres of constant composition have been separated from concentrates with the use of the technological scheme including stages of hydrodynamic gravitational separation, grain-size classification, and magnetic separation. It has been established that the concentrations of the major components of the chemical composition of the products obtained are related by linear regression equations. In particular, the aluminosilicate composition of the non-perforated cenospheres with a low Fe<sub>2</sub>O<sub>3</sub> concentration (2.5-3.5 wt.%) is described by two general regression equations  $[\text{SiO}_2] = 80 - 0.7 \cdot [\text{Al}_2\text{O}_3]$  and  $\text{SiO}_2/\text{Al}_2\text{O}_3 = 5.54 - 0.12 \cdot [\text{Al}_2\text{O}_3]$  with correlation coefficients of -0.98 and -0.99, respectively. In this case, narrow fractions of products with different Fe<sub>2</sub>O<sub>3</sub> contents are characterized by nearly constant SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> ratios. The shell structure has been investigated using scanning electron microscopy. It has been revealed that the outer and inner surfaces of globules are covered by a film with a thickness of 30-50 nm. The surface of magnetic cenospheres contains heterogeneous regions with extended linear ferrosphenel aggregates of crystallites with sizes ranging from 50 to 1000 nm. It has been shown that the size of globules and the thickness and porosity of the shell in each series depend on the composition of the melt from which they are formed and on its viscosity. Nonmagnetic cenospheres are formed from the high-silica K-Al-Si melt with the SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> ratio varying over a wide range (1.6-3.6). Narrow fractions of magnetic cenospheres formed at low temperatures (1500°C) are characterized by nearly constant SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> ratios. Globules of this type are formed from two immiscible high-silica K-Al-Si and Fe-Al-Si melts.

**MIDDLEINGS OF SELECTION OF HIGH FLY ASHES AND PREPARATION OF SPECIAL CEMENTS AND MAGNETIC MICROSPHERES ON ITS BASE**

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*Chem. Sustain. Devel.*,  
18(3) (2010) pp. 405-416.

High-calcium volatile ashes of brown coal from Berezovsk drill core of the Kansk-Achinsk coal field were studied. The samples were chosen from different ash-trapping points (convective mine, prechamber and each of four fields of electrofilters). Differences in chemical composition, dispersion, viscosity of these middleings were established, types determined in the system of chemical classification of these volatile ashes. The highest calcium contents were shown to be the distinctive feature of ashes from first to fourth electrofilters of Berezovsk and Nazarovsk coals. The phase diagram of CaO-Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub> demonstrates that hydraulically active phases similar to the main phases of portland cement clinker can be formed from electrofilter ashes, the phase proportions being varied due to different ash compositions. Along with their different dispersions, this allows portland cement specialty to be produced on their basis. Six fractions of high-purity magnetic microspheres were produced, their chemical, quantitative phase compositions and morphology (including the contribution of various type globules and structural-textural characteristics) studied, methods for their application, trends in varying compositions and morphology with changing the fraction size established.

**A STUDY OF THE TEMPERATURE EFFECT ON THE IR SPECTRA OF CRYSTALLINE AMINO ACIDS, DIPEPTIDS, AND POLYAMINO ACIDS: VI. L-ALANINE AND DL-ALANINE**

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*J. Struct. Chem.*,  
51(6) (2010) pp. 1052-1163.

The results of IR and single crystal X-ray diffraction studies on the dynamics of molecular groups and structural changes in L-alanine and DL-alanine (NH<sub>3</sub><sup>+</sup>-CH(CH<sub>3</sub>)-COO<sup>-</sup>) with temperature variation are given. An analysis of changes in the 4000–600 cm<sup>-1</sup> frequency range of the IR spectra with temperature variation reveals the occurrence of the

anomaly for the  $\sim 974\text{ cm}^{-1}$  band in *DL*-alanine, which is similar to the anomaly for the  $955\text{ cm}^{-1}$  band, previously described for *L*-alanine. The X-ray diffraction data for *L* and *DL*-alanine show that no dramatic changes in the unit cell parameters, conformations of amino acid molecules themselves, and hydrogen bond lengths occur with temperature variation, which would indicate the structural phase transition. Changes in the IR spectra of *L*-alanine and *DL*-alanine with temperature variation are compared to the changes in the vibrational spectra of other amino acids on cooling.

#### EFFECTIVE PLATINUM CATALYSTS FOR LOW-TEMPERATURE OXIDATION OF CO

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*Russ. J. Phys. Chem. A*,  
84(4) (2010) pp. 561-565.

A number of platinum-based catalysts bonded on carbon fiber karbopon were prepared using alcohol solutions of  $\text{H}_2\text{PtCl}_6$ . The catalytic activity of the samples was determined in the low-temperature oxidation of carbon monoxide by oxygen. The effect of the solvent nature of the precursor on the activity of the catalysts was determined. The best results were obtained in the presence of 1.5% Pt/karbopon catalyst prepared using isobutyl alcohol; 100% CO conversion was achieved in respiratory treatment ( $12000\text{ h}^{-1}$ , room temperature).

#### ESR STUDY OF ALUMINA NANOPOWDERS PREPARED BY AIR JET EVAPORATION METHOD

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*Vestnik NGU, ser: Fizika*,  
5(1) (2010) pp. 97-101.

The paper is devoted to experimental study of alumina and other nanopowders by electron paramagnetic resonance method. The experiments have showed the surface donor centers for  $\text{ZrO}_2 + \text{MgO}$  (9 % mol.) and  $\text{Al}_2\text{O}_3$  samples. The influence of  $\text{Fe}^{3+}$  dirt was determined in glacial  $\text{Al}_2\text{O}_3$  liquid melt, but  $\text{Al}_2\text{O}_3$  nanopowder was free from this impurity. The  $\text{Cr}^{5+}$  dirt were found in  $\text{ZrO}_2 + \text{MgO}$  (9 % mol.) nanopowder sample.

#### DETERMINATION OF THE LOCAL STRUCTURE OF A HIGHLY DISPERSED Pd-NANOSYSTEM LOCATED ON A TITANIUM DIOXIDE CARRIER

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**O.V. Bukhtenko\***, **M.V. Tsodikov\***,  
**N.Yu. Kozitsyna\*\***, **M.N. Vargaftik\*\***,  
**I.I. Moiseev\*\***, **E.A. Maksimovskii\*\*\***,  
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\*\**Kurnakov Institute of General and Inorganic Chemistry, Moscow, Russia*; \*\*\**Nikolaev Institute of Inorganic Chemistry, Novosibirsk, Russia*;  
\*\*\*\**Sobolev Institute of Geology and Mineralogy, Novosibirsk, Russia*; \*\*\*\*\**Universidad de Sevilla, Sevilla, Spain*; \*\*\*\*\**Netherlands Organization for Scientific Research, DUBBLE CRG/ESRF, Grenoble Cedex, France*)

*J. Surf. Invest. - X-Ray Synchrotron Neutron Techn.*,  
4(4) (2010) pp. 636-639.

This work is devoted to a structural study of a highly dispersed Pd nanosystem, which is stabilized in the  $\text{TiO}_2$  matrix, by XAFS spectroscopy. Nanocomposite was prepared from bimetallic  $\text{PdCo}(\mu\text{-OOCMe})_4(\text{NCMe})$  precursor followed by processing in several ways: calcination in air and in argon and microwave irradiation. The local structure of Pd catalysts formed by different methods was studied. Possible structural models were considered in detail.

#### THE EFFECT OF SUPPORTS ( $\text{Al}_2\text{O}_3$ , $\text{Al}_2\text{O}_3\text{-CeO}_2$ AND $\text{Al}_2\text{O}_3\text{-CeZrO}_2$ ) ON THE NATURE OF GOLD-SPECIES IN SUPPORTED GOLD CATALYSTS

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**E.V. Smolentseva\***, **F. Castillon\***, **M. Estrada\*\***,  
**E. Vargas\*\*\***, **E.P. Yakimchuk**, **D.P. Ivanov**,  
**D.G. Aksenov**, **D.V. Andreev**, **B.N. Novgorodov**,  
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*J. Surf. Invest. - X-Ray Synchrotron Neutron Techn.*,  
4(4) (2010) pp. 630-635.

This work is concerned with the study of Au specimens produced by gold deposition on nanosized mixed oxides (alumina, ceria, zirconia) prepared by the sol-gel method using organometallic precursors. According to X-ray absorption near edge structure, extended X-ray absorption fine structure, transmission

electron microscopy data, and ultraviolet-visible and X-ray photoelectron spectroscopy measurements, mixed Al-Ce-Zr oxides are quite effective for stabilization of different gold specimens. The samples pretreated in hydrogen at 150°C are characterized by the presence of gold Au<sup>3+</sup> cations located on the surface in slightly disordered octahedral oxygen coordination. Metallic gold nanoparticles with a size of about 2 nm and gold clusters were found in the samples treated in hydrogen at 300°C.

#### **STRUCTURAL STUDY OF CuCr<sub>1-x</sub>V<sub>x</sub>S<sub>2</sub> SUBSTITUTIONAL SOLID SOLUTIONS**

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*J. Surf. Invest. - X-Ray Synchrotron Neutron Techn.*, 4(4) (2010) pp. 640-644.

The local structure and charged state of metal atoms in the CuCrS<sub>2</sub> matrix and CuCr<sub>1-x</sub>V<sub>x</sub>S<sub>2</sub> solid solutions of substitution of vanadium for chromium (0 < x < 0.25) are studied by X-ray absorption spectroscopy (XAFS) using synchrotron radiation. According to XANES spectra (near-edge fine structures), introducing vanadium does not change the charged state of matrix elements and they exist as Cu<sup>1+</sup>, Cr<sup>3+</sup>, V<sup>3+</sup>, and S<sup>2-</sup> in the solid solution. According to EXAFS spectra (extended fine structures), introducing vanadium slightly distorts the local structures of both Cr and Cu atoms (to a greater extent for copper than for chromium). The XAFS analysis data show that vanadium only partially filled vacant positions (specified by the composition of the initial mixture) in the chromium sublattice, the majority of them being located in irregular positions of slightly distorted octahedrons consisting of sulphur atoms. It is shown that the substitution effect leads to deformation (compression) of the lattices of CuCr<sub>1-x</sub>V<sub>x</sub>S<sub>2</sub> samples without changing their crystallochemical structures and symmetries, but changes their magnetic structures.

#### **STUDY OF THIN GE FILMS WITH AMORPHOUS AND NANOCRYSTALLINE PHASES VIA THE TECHNIQUES OF EXAFS SPECTROSCOPY AND AFM**

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*J. Surf. Invest. - X-Ray Synchrotron Neutron Techn.*, 4(1) (2010) pp. 136-141.

The local atomic structure and surface morphology of thin semiconductor films of Ge have been studied via extended X-ray absorption fine structure spectroscopy and atomic force microscopy. The films have been obtained by thermal evaporation of a material in an ultrahigh vacuum at different substrate temperatures. The films contain both amorphous and nanocrystalline phases. The percentage of the phases depends on the condensation temperature. The classical linear dependence of grain sizes on condensation temperature *T* is violated at *T*=100°C.

#### **SYNTHESIS AND STRUCTURE STUDY OF ORDERED ARRAYS OF ZnSe NANODOTS**

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*J. Surf. Invest. - X-Ray Synchrotron Neutron Techn.*, 4(4) (2010) pp. 645-648.

A novel approach is presented for synthesis of ZnSe nanodot arrays by physical vapor deposition on porous aluminum oxide templates with ordered channels. The structure of nanodots was studied by scanning electron microscopy and EXAFS spectroscopy. Data were obtained for the sizes of nanodots in the array and local atomic structure parameters, i.e., the interatomic distances and coordination numbers, in comparison with the data for the ZnSe film synthesized on a smooth surface of nonporous Al<sub>2</sub>O<sub>3</sub>.

## SYNTHESIS AND STRUCTURAL STUDY OF THE ORDERED GERMANIUM NANOROD ARRAYS

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*J. Struct. Chem.*,  
51(suppl.) (2010) pp. S135-S139.

A new approach to synthesis of the ordered germanium nanorod arrays using thermal sputtering on the matrices of porous alumina with the ordered channel arrangement is presented. The synthesized filamentary nanostructures were examined by scanning electron microscopy (SEM), EXAFS and XANES spectroscopy. Data on nanorod geometry in arrays, parameters of the local atomic structure such as interatomic distances and coordination numbers for initial samples and those annealed at 450°C in the argon atmosphere, and data on changes in the electronic states near the absorption K-edge were acquired. A comparison was made with the data of EXAFS studies of a continuous Ge film synthesized on a smooth surface of non-porous Al<sub>2</sub>O<sub>3</sub>.

## SYNTHESIS OF ZnSe SEMICONDUCTOR NANODOTS ARRAYS BY TEMPLATED PVD

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*Phys. Status Solidi C*,  
7(6) (2010) pp. 1539-1541.

A novel approach for preparation of ZnSe nanodot arrays by physical vapor deposition on porous aluminum oxide templates possessing ordered system of channels is reported. The structure of nanodot arrays was investigated by scanning electron microscopy and EXAFS-spectroscopy. The geometry of nanodots and local atomic structure parameters like the interatomic distances and coordination numbers of Zn and Se atoms are reported.

## STUDY OF THE LOCAL STRUCTURE OF SUPPORTED NANOSTRUCTURAL PLATINUM CATALYSTS

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*J. Struct. Chem.*,  
51(suppl.) (2010) pp. S17-S25.

The possibility of controlling the state of platinum deposited on the support surface via minor changes in the catalyst preparation procedure is demonstrated using a series of highly dispersed Pt/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub> catalysts with different particle size of the active component. Dispersity, local structure and electronic state of supported platinum were examined by a combination of high resolution transmission electron microscopy and X-ray absorption spectroscopy (EXAFS/XANES). It was shown that various platinum species can be obtained on the surface of the support: bulk or surface Pt(II) or Pt(IV) oxides, mixed metal-oxide structures, bulk particles of metallic platinum, and two-dimensional surface Pt<sup>0</sup> particles strongly interacting with the support.

## MECHANOCHEMICAL SYNTHESIS OF AMORPHOUS AND CRYSTALLINE MAGNESIUM DIBORIDE

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*Inorg. Mater.*,  
46(1) (2010) pp. 22-27.

The authors have studied the phase composition of materials obtained by mechanochemical processing and subsequent heat treatment of mixtures of magnesium and boron powders in the atomic ratio 1:2. Differential dissolution, differential scanning calorimetry, and X-ray diffraction data indicate that, during mechanical processing, some of the magnesium reacts with boron to form amorphous magnesium diboride. During annealing of the activated powder mixture, X-ray amorphous magnesium diboride forms at 340°C and crystallizes at 480°C. As shown by high resolution transmission electron microscopy, the unreacted crystalline magnesium is covered with an amorphous layer consisting of magnesium diboride and boron. The amorphous material obtained by milling contains nuclei of MgB<sub>2</sub> crystallites 3–5 nm in size. During

subsequent heating of the activated mixture, magnesium and boron react further to form amorphous magnesium diboride and the amorphous phase crystallizes. Heating of mechanically activated mixtures to just below the crystallization temperature allow  $MgB_2$  nanoparticles to be produced. The formation of nanocrystalline magnesium diboride nuclei along with the amorphous phase during mechanochemical processing facilitates mechanochemical synthesis compared to thermal synthesis.

#### ***In situ* SYNTHESIS AND CATALYTIC ACTIVITY IN CO OXIDATION OF METAL NANOPARTICLES SUPPORTED ON POROUS NANOCRYSTALLINE SILICON**

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*J. Catal.*,  
271(1) (2010) pp. 59-66.

Reactive surface of mesoporous nanocrystalline silicon was used to synthesise noble metal nanoparticles via *in situ* reduction of the precursor salt solutions. The synthetic methodology for metal nanoparticle formation was systematically developed, and reaction conditions of metal salts reduction were optimised to prepare nanoparticles of controlled size distribution in the order 5-10 nm inside the mesoporous silicon template. CO oxidation was used as a test reaction for the synthesised Pt/porous silicon catalysts. Sharp reaction light-off was observed at about 120°C on the optimised catalysts. The catalysts were shown to be stable in the extended steady-state runs and in the catalysts re-use experiments. Metal nanoparticles were shown to be stable to sintering at elevated temperatures up to 1000°C. However, after thermal treatment on air, Pt nanoparticles were covered by a  $SiO_x$  layer and were less active in CO oxidation.

#### **OPTICAL CHARACTERISTICS OF CADMIUM SULFIDE NANOPARTICLES SYNTHESIZED IN POLYETHYLENE MATRIX AND ORTHO-XYLENE SOLUTION**

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*Opt. Spectrosc.*,  
109(2) (2010) pp. 154-161.

The properties of CdS nanoparticles synthesized in a polyethylene matrix and an ortho-xylene solution are compared. It is shown that these nanoparticles have different shapes, structures, and degrees of imperfection. Due to these differences, unaggregated nanoparticles in solution exhibit luminescence in contrast to nanoparticles in polyethylene. The stabilization of nanoparticles in polyethylene most probably occurs via OH groups, while nanoparticles in solution are stabilized due to partial interaction with dissolved sulfur. The absorption and luminescence spectra of samples with polyethylene are strongly distorted by reabsorption and scattering, while this effect in solution is absent due to a small concentration of nanoparticles.

#### **THE STRUCTURE OF CdS NANOPARTICLES**

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*J. Surf. Invest. - X-Ray Synchrotron Neutron Techn.*,  
4(4) (2010) pp. 654-657.

CdS nanoparticles in the high-pressure polyethylene (HPPE) matrix have been synthesized and investigated. Shift of the fundamental absorption edge has been shown to be absent with a variation in the nanoparticle size for the given samples and a small disordering of atoms in nanoparticles is observed.

## SYNTHESIS, X-RAY STRUCTURE AND REDOX PROPERTIES OF THE MACROBICYCLIC IRON(II) N-2- AND S-2-CONTAINING *vic*-DIOXIMATES

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*Inorg. Chim. Acta*,  
363(1) (2010) pp. 134-146.

Nucleophilic substitution of the reactive chlorine atoms of the boron-capped macrobicyclic *vic*-di- and hexahalogen-containing iron(II) precursors with 1,2-ethanedithiol and 1,2-benzenedithiol in dichloromethane as a solvent in the presence of triethylamine as a strong organic base afforded the corresponding di- and hexasulfide mono- and triribbed-functionalized clathrochelates, respectively, in relatively high yields. In the case of the low-reactive tin-capped clathrochelate  $[\text{Fe}(\text{Cl}_2\text{Gm})_3(\text{SnCl}_3)_2]^{2-}$  dianion this reaction was performed in DMF with the potassium salt of 1,2-benzenedithiol. The reaction of the dichlorine-containing  $\text{FeBd}_2(\text{Cl}_2\text{Gm})(\text{BF})_2$  precursor with an excess of ethylenediamine in DMF led to the clathrochelate with  $\text{N}_2$ -containing *vic*-dioximate ribbed fragment. The complexes obtained were characterized using elemental analysis, MALDI-TOF mass spectrometry, IR, UV-vis,  $^1\text{H}$  and  $^{13}\text{C}\{^1\text{H}\}$  NMR,  $^{57}\text{Fe}$  Mössbauer spectroscopies, and X-ray crystallography.

The nature and number of the ribbed substituents affect the geometry of a clathrochelate framework, first of all, the distortion of the trigonal prismatic-trigonal antiprismatic iron(II) coordination polyhedra, whereas the apical substituents at the capping boron atoms influence on the B–O distances in the apical  $\text{RBO}_3$  fragments. The geometry of the tin-capped hexasulfide clathrochelate complex was deduced from EXAFS data using the scattering both on the encapsulated iron(II) and capping tin(IV) ions.

## TRANSFORMATION OF PETROLEUM ASPHALTENES IN SUPERCRITICAL WATER

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*J. Supercrit. Fluids*,  
55(1) (2010) pp. 217-222.

The transformation of petroleum asphaltenes in supercritical water was studied. The experiments were performed in autoclave at temperature 380°C and

pressure 226 atm with stirring for 3 h, medium density was about 0.33 g/cm<sup>3</sup>. The reaction resulted in the formation of gas products, about 4.3%, and an insoluble residue (coke) with about 48.6% yield. The remaining products were separated into fractions by consecutive dissolution in hexane (30.0%), benzene (10.6%), and chloroform (5.7%). The properties of the obtained products were studied with FT-IR spectrometry and  $^1\text{H}$  NMR spectroscopy. The method of simulated distillation was used to demonstrate that the fractional composition of the hexane-soluble part of the products is close to the fractional composition of a mixture of the diesel fraction and vacuum gas oil of the corresponding oil in 1:1 ratio. The obtained data support the conclusion that asphaltene cracking proceeds in SCW, with most probable main processes being dealkylation of substituents in the aromatic fragments of molecules and aromatization. This leads to formation of gaseous products and hexane-soluble fraction consisting of lighter aliphatic and aromatic compounds, as well as carbonized solid residue.

## STATE OF IRON IN NANOPARTICLES PREPARED BY IMPREGNATION OF SILICA GEL AND ALUMINUM OXIDE WITH $\text{FeSO}_4$ SOLUTIONS

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*Phys. Solid State*,  
52(4) (2010) pp. 826-837.

The state of iron in nanoparticles prepared by impregnating silica gel and aluminum oxide with iron(II) sulfate solutions has been investigated using Mössbauer spectroscopy. It has been revealed that the state of iron depends on the nature of the support. Iron(III) hydroxysulfate and iron(III) oxysulfate nanoparticles are formed on the surface of silica gel, and iron oxide nanoparticles are formed on the surface of aluminum oxide. An increase in the concentration of iron ions or in the size of iron-containing particles leads to hydration of the nanoparticle surface. The calcination of the samples results in the formation of  $\epsilon\text{-Fe}_2\text{O}_3$  oxide in a strongly disordered or amorphous state in iron-containing particles on the surface of silica gel.

## FEATURES OF EPR APPLICATION TO SYSTEMS CONTAINING PARAMAGNETIC CENTERS AND FERROMAGNETIC NANOPARTICLES

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*Appl. Magn. Reson.*,  
38(4) (2010) pp. 495-500.

The scope of the electron paramagnetic resonance (EPR) technique in catalysis is often limited because of the presence of the ferromagnetic nanoparticles in many catalytic systems. The present work gives an effective method to discriminate solid-state EPR spectra in the microwave resonance absorption while investigating such systems. The approach is based on the different features of the EPR spectra and ferromagnetic resonance absorption that are simultaneously observed in the system.

## NONMONOTONIC BEHAVIOR OF MAGNETORESISTANCE, R(H) HYSTERESIS, AND LOW-TEMPERATURE HEAT CAPACITY OF THE $\text{BaPb}_{0.75}\text{Bi}_{0.25}\text{O}_3$ SUPERCONDUCTOR IN A MAGNETIC FIELD: POSSIBLE MANIFESTATIONS OF PHASE SEPARATION

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*J. Exp. Theor. Phys.*,  
110(4) (2010) pp. 584-593.

The transport properties (R(T) and R(H) dependences at various values of the transport current in magnetic fields up to 65 kOe) and low-temperature heat capacity in magnetic fields up to 90 kOe of the  $\text{BaPb}_{0.75}\text{Bi}_{0.25}\text{O}_3$  superconductor ( $T_c \approx 11.3$  K) are investigated with the goal of clarifying the mechanisms determining the nonmonotonic behavior and hysteresis of its magnetoresistance R(H). The type of R(H) hysteretic dependences for  $\text{BaPb}_{0.75}\text{Bi}_{0.25}\text{O}_3$  is analogous to that observed in granular high- $T_c$  superconductors (HTSCs); however, unlike classical HTSC systems, the field width of the magnetoresistance hysteresis loop for polycrystalline  $\text{BaPb}_{0.75}\text{Bi}_{0.25}\text{O}_3$  depends on the transport current. This means that although the mechanisms responsible for the magnetoresistance hysteresis (the influence of the magnetic flux trapped in superconducting regions on the effective field in Josephson interlayers) are identical in these objects, the transport current in  $\text{BaPb}_{0.75}\text{Bi}_{0.25}\text{O}_3$  may considerably affect the diamagnetic response of the superconductor.

A considerable effect of transport current on the field in which the R(H) dependences have a peak and exhibit hysteretic properties is observed. Such a behavior can be adequately interpreted using the model of the spatially inhomogeneous superconductor-insulator state proposed by Gorbatsevich et al. [JETP Lett. 52, 95 (1990)]. The nonmonotonic dependence of quantity C/T (C is the heat capacity) on the magnetic field discovered in the present study also agrees with the conclusions based on this model.

## APPLICATION OF SECONDARY ION MASS SPECTROMETRY FOR ANALYZING THE COMPOSITION AND STRUCTURE OF SURFACE LAYERS OF FINE MATERIALS

V.P. Ivanov

*J. Struct. Chem.*,  
51(suppl.) (2010) pp. S58-S63.

Fundamental and methodological aspects of secondary ion mass spectrometry applied to analysis of elemental/phase composition and structure of the surface layers of fine materials are discussed.

## EPR SPECTRA OF NITROGEN IN ULTRA-DISPERSED DIAMONDS

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*Appl. Magn. Reson.*,  
39(3) (2010) pp. 295-302.

The analysis of the electron paramagnetic resonance (EPR) line shape of ultra-dispersed diamond (UDD) obtained by conversion of trotyl and hexogen mixture and purified from other phases and metal compounds is carried out. The observed wide line with  $g = 2.0028$  and a line width of 8.7 G is shown to be formed by superposition of three lines with line widths of 15.3, 8.5 and 3 G and with a ratio of integral intensities of 70:30:1. The procedure of decomposition and subtraction of wide lines has revealed the resolved hyperfine structure (HFS) from donor nitrogen with parameters  $A = 40.8$  G and  $B = 29.2$  G. Experimentally obtained dependence of the line width of the exchange line on the concentration of donor nitrogen in synthetic diamonds assumed that variations in line widths of the EPR spectrum components are caused by different local concentration of donor nitrogen due to distribution of nitrogen impurity during crystallization of diamond



nanoparticles. EPR spectra of UDD after annealing in vacuum and at high pressures in the range of diamond phase stability are also discussed. At high-pressure annealing above 973 K, the areas with high concentration of defects are graphitized and a narrow Dyson-shape line from conductivity electrons and a resolved HFS from donor nitrogen can be observed without additional treatment of the EPR spectrum.

#### **PARTICLE SIZE DEPENDENT HEATS OF ADSORPTION OF CO ON SUPPORTED Pd NANOPARTICLES AS MEASURED WITH A SINGLE CRYSTAL MICROCALORIMETER**

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**J.M. Flores-Camacho\***, **A. Genest\*\*\***,  
**I.V. Yudanov**, **N. Rosch\*\*\***, **C.T. Campbell\*\***,  
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*Phys. Rev. B*,  
81(24) (2010) 241416 (4 pp.).

Calorimetric measurements are presented of the effect of cluster size on the adsorption enthalpy of carbon monoxide on Pd nanoclusters sized from 120 to 4900 Pd atoms per particle, which were grown *in situ* on a well-ordered Fe<sub>3</sub>O<sub>4</sub>/Pt(111) film. A substantial decrease of the initial heat of adsorption amounting to about 20-40 kJ·mol<sup>-1</sup> was observed on the smallest Pd nanoparticles as compared to the larger Pd clusters and the extended Pd(111) single crystal surface. This effect is presented in terms of the size-dependent properties of the Pd nanoparticles.

#### **DISTRIBUTION OF COPPER- AND NICKEL-CONTAINING MODIFIER COMPONENTS IN THE PORE SPACE OF HZSM-5 ZEOLITE**

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*Kinet. Catal.*,  
51(1) (2010) pp. 88-97.

The distribution of copper- and nickel-containing components in the pore space of HZSM-5 zeolite was quantitatively studied. It was found that the detailed distribution of a modifier in the micropore and mesopore volumes of the zeolite depends on both the chemical nature of the modifier and the conditions of supporting and the regime of M<sup>2+</sup> polycondensation in the pore space of the zeolite. The experimental data on the low-temperature

adsorption of nitrogen on Cu(*n*)ZSM-5 catalysts can be interpreted as the result of the partial filling of the zeolite micropore space (10 vol %) and the finest mesopores with *D* < 3 nm with the modifier. In the case of Ni(*n*)ZSM-5 catalysts, the penetration of the modifier into zeolite channels (micropores) in detectable amounts was not found, and it was arranged in mesopores on the surface of zeolite crystallites. The reason for differences between modifier distributions in the pore structure of the zeolite was explained from the standpoint of different structures of copper and nickel polyhydroxo complexes in impregnating solutions after polycondensation. It was found that, in the Cu(*n*)ZSM-5 and Ni(*n*)ZSM-5 catalysts, the modifier component contained copper and nickel only in a doubly charged state and mainly octahedral oxygen environments. In this case, three-dimensional nanoparticles or coarsely dispersed particles of CuO were not detected in the pore space of the support, whereas the presence of a small amount of sufficiently large NiO crystals with a coherent-scattering region of 80–100 nm was detected in Ni(*n*)ZSM-5, and these crystals occurred on the surface of zeolite crystals. It was found that the apparent density of a copper- or nickel-containing component arranged in the pore space of the zeolite was lower than the density of the bulk CuO and NiO phases by a factor of ~3 and 4, respectively, because of the size effect.

#### **BISMUTH MODIFIED Pd/C AS CATALYSTS FOR HYDROGEN RELATED REACTIONS**

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**M. Hantel\*\*\*\***, **M. Rzepka\*\***, **P. Dotzauer\*\***,  
**A.N. Popov**, **P.A. Simonov**, **V.N. Parmon**,  
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*Electrochem. Commun.*,  
12 (2010) pp. 1490–1492.

The electrocatalytic activity of bimetallic BiPd catalysts supported on Sibunit carbon towards hydrogen oxidation/evolution reactions (HOR/HER) was studied in a gas diffusion electrode (GDE) setup. Catalysts were synthesized by deposition of Pd on the carbon support, followed by impregnation of Pd/C precursor with Bi(NO<sub>3</sub>)<sub>3</sub> solution and reduction in hydrogen. Transmission electron microscopy and local EDX elemental analysis revealed that BiPd/C catalysts contain bimetallic particles with narrow size distribution with maxima at 3.2–4.1 nm. X-ray diffraction evidenced that bimetallic particles are constituted by Pd–Bi solid solution. It was shown that modification of Pd/C by

bismuth increases the specific activity of palladium towards HOR/HER by a factor of 3.

#### **GAS-CHROMATOGRAPHIC DETERMINATION OF THE COMPONENTS OF THE CATALYTIC OXIDATION OF ETHYLENE GLYCOL TO GLYOXAL**

**E.Yu. Yakovleva, V.Yu. Belotserkovskaya**

*J. Analyt. Chem.*,  
65(8) (2010) pp. 833-837.

The gas-chromatographic retention of formaldehyde, acetaldehyde, glyoxal, formic acid, and ethylene glycol on polar and nonpolar polymeric adsorbents was studied, and a procedure was developed for the determination of these components. The time of analysis was 12-14 min. The detection limits were 0.02 (acetaldehyde) to 0.7 (formic acid) ppm.

#### **CHROMATOGRAPHIC PROPERTIES AND POLARITY EVALUATION OF POLY(1-TRIMETHYLSILYL-1-PROPENE) AND POLY(1-PHENYL-1-PROPENE)**

**E.Yu. Yakovleva, V.Yu. Belotserkovskaya**

*J. Analyt. Chem.*,  
65(10) (2010) pp. 1014-1020.

The chromatographic properties of poly(1-trimethylsilyl-1-propene) (PTMSP) and poly(1-phenyl-1-propene) (PPP) were studied by gas chromatography using packed columns. The selectivity and efficiency of columns packed with PTMSP and PPP were compared to the data obtained for columns with other known adsorbents and stationary phases. The McReynolds and Rohrschneider constants, on the basis of which the polarity of the new phases was evaluated, were calculated. The results of the investigation of chromatographic properties allow PTMSP to be brought in line with the polymeric adsorbents Porapak Q, Porapak QS, and Chromosorb 106, while PPP, with the methyphenylsilicon phases SE-52 and OV-3.

#### **STUDY OF POLY(1-TRIMETHYLSILYL-1-PROPENE) AGING BY GAS CHROMATOGRAPHY**

**V.Yu. Belotserkovskaya, E.Yu. Yakovleva**

*J. Struct. Chem.*,  
51(suppl.) (2010) pp. S162-S166.

The possibilities of gas chromatography for determination of physicochemical properties of the surface are demonstrated. Changes in the surface

properties of poly(1-trimethylsilyl-1-propene) (PTMSP) with time were investigated. It was found that with the lapse of time the sorption-desorption processes proceed mainly in mesopores due to decreasing the polymer micropore volume. The formation of a chemically more uniform surface strongly increases the symmetry of chromatographic peaks and the column efficiency.

#### **CHROMATOGRAPHIC PROPERTIES OF POLY(1-PHENYL-1-PROPENE)**

**V.Yu. Belotserkovskaya, E.Yu. Yakovleva**

*Russ. J. Phys. Chem. A*,  
84(2) (2010) pp. 307-311.

The chromatographic properties of poly(1-phenyl-1-propene) (PPP) were studied by separating the C<sub>1</sub>-C<sub>10</sub> hydrocarbons, alcohols, aromatic, and sulfur-bearing compounds. The influence of the phase percentage for polymer adsorbent (Polysorb-1) on the process of component retention was investigated. A comparison of PPP and the nonpolar liquid phase SE-52 widely used in gas chromatography was performed.

#### **INVESTIGATION OF STRUCTURE AND PROPERTIES OF DIVINYLBENZENE-STYRENE COPOLYMER FILMS**

**O.A. Nikolaeva, Yu.V. Patrushev, V.N. Sidelnikov**

*J. Struct. Chem.*,  
51(suppl.) (2010) pp. S167-S170.

A method for preparation of divinylbenzene-styrene copolymer porous layer on the inner wall of a fused silica capillary was developed. Capillary gas chromatography was used to investigate the sorption capacity of copolymer film toward substances of various classes.

#### **STUDIES OF SPATIAL INHOMOGENEITY IN CHEMICAL COMPOSITION OF MULTIPHASE MULTICOMPONENT SOLID SUBSTANCES AND MATERIALS**

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4(60) (2010) pp. 147-151.

Under discussion are new stoichiographic methods for detection, identification and quantitative determination of phenomena of spatial inhomogeneity in chemical composition and structure of multielement substances and materials. Against the known chemical and physical methods, stoichiography allows

stoichiometric formulae and quantitative contents of solid inorganic compounds to be determined without reference samples simultaneously in their complex mixtures. Theoretical aspects of dynamic processes of solid dissolution are considered in terms of modern concepts of thermodynamics and kinetics of heterophase transformations and solid structure. The spatial distributions of components in products of doping, promoting and other processes of modifying chemical compositions of substances and materials are determined.

## **STOICHIOMETRY FOR INVESTIGATION OF THE FUNCTIONAL MATERIALS COMPOSITION, STRUCTURE, AND PROPERTIES**

**V.V. Malakhov**

*J. Struct. Chem.*,  
51(suppl.) (2010) pp. S155-S161.

Principles of the stoichiometry and differential dissolution method (DD) providing deep insight into the composition of mixtures containing unknown chemical compounds are considered. Compounds can be identified using their primary feature — the stoichiometry of elemental composition; reference samples of the compounds are not needed. The DD method makes it possible to analyze mixtures of crystalline and/or amorphous phases with constant and/or variable composition in the form of dispersed powders, ceramics, crystals, thin films and nanosize objects. Various aspects of the stoichiometry and DD method application to investigation of composition, structure and properties of the functional materials are discussed.

## **Fundamental and Practical Application to Catalyst Preparation. The Effects of Preparation Methods on Catalyst Structure and Performance**

### **TITANIUM, CERIUM, ZIRCONIUM, YTTRIUM AND ALUMINIUM OXIDES. PROPERTIES, APPLICATION AND SYNTHESIS**

**Z.R. Ismagilov, V.V. Kuznetsov, L.B. Okhlopkova, L.T. Tsykoza, S.A. Yashnik**

Novosibirsk, Publishing House SB RAS,  
Ed. V.N. Parmon, 2010, 244 pp.

The monograph summarizes and generalizes the published scientific and patent information on properties, application and preparation of oxides of titanium, cerium, zirconium, yttrium and aluminum. The levels of production and consumption of these oxides are among most important scientific, technological and economic characteristics of the world leading countries. The relevant characteristics reached in Russia are compared to the world levels. Of particular attention is analysis of the existent and search for new industrial technologies which will be more efficient, energetically profitable and ecologically sound and allow the said product qualities to be controlled under the customer's request. Recommendations on creation of small-scale production of these products using feedstocks available in Russia are provided.

### **EFFECT OF PREPARATION CONDITIONS ON THE PHASE COMPOSITION OF THE MoVTe(Nb) OXIDE CATALYST FOR THE OXIDATIVE CONVERSIONS OF PROPANE**

**E.V. Ishchenko, T.V. Andrushkevich, G.Ya. Popova, Yu.A. Chesalov, L.M. Plyasova, A.V. Ishchenko, T.Yu. Kardash, L.S. Dovlitova**

*Catal. Ind.*,  
2(4) (2010) pp. 291-298.

The replacement of expensive propylene by propane, which requires the development of catalysts for the direct oxidation of propane into acrylonitrile, is an important and insufficiently studied problem. Multicomponent  $\text{Mo}_m\text{V}_n\text{Te}_k\text{Nb}_x$  oxide systems are promising in one-stage ammoxidation of propane to acrylonitrile. Despite considerable attention of various authors to the preparation methods for these catalysts, the reproducibility of their physicochemical and catalytic properties is low. To optimize the technology of catalyst synthesis, the effect of drying method (evaporation or spray drying) for the aqueous suspension of the initial compounds on the formation of the  $\text{Mo}_1\text{V}_{0.3}\text{Te}_{0.23}(\text{Nb}_{0.12})$  oxide catalyst has been studied. It is shown that the method of drying

determines the chemical and phase composition of solid catalyst precursors and the phase composition of the final catalyst in high-temperature treatment. The use of spray drying provides the required physicochemical characteristics of the catalyst (the specific surface area and the phase composition) that determine the high activity and selectivity in the selective conversion of propane. These catalysts contain two crystalline phases: orthorhombic M1 and hexagonal M2 in an optimal ratio of 3 : 1.

#### **FORMATION OF ACTIVE COMPONENT OF MoVTaNb OXIDE CATALYST FOR SELECTIVE OXIDATION AND AMMOXIDATION OF PROPANE AND ETHANE**

**E.V. Ishchenko, T.V. Andrushkevich, G.Ya. Popova, V.M. Bondareva, Yu.A. Chesalov, T.Yu. Kardash, L.M. Plyasova, L.S. Dovitova, A.V. Ishchenko**

*Stud. Surf. Sci. Catal.*,  
175 (2010) pp. 479-482.

The effect of slurry pH on the formation of active component of MoVTaNbO catalyst for selective (amm)oxidation of ethane and propane has been studied. pH affects the nature and composition of the crude and dry precursors as well as chemical and phase composition of the final catalyst. The most effective catalyst is prepared at pH = 3.0, which is characterized by a maximum content of M1 phase.

#### **Mo<sub>5</sub>O<sub>14</sub>-LIKE V-Mo-Nb OXIDE CATALYSTS: STRUCTURE AND CATALYTIC PERFORMANCE**

**T.Yu. Kardash, L.M. Plyasova, V.M. Bondareva, T.V. Andrushkevich, L.S. Dovitova, A.V. Ishchenko, A.I. Nizovsky, A.V. Kalinkin**

*Appl. Catal., A*,  
375(1) (2010) pp. 26-36.

Within Mo<sub>5</sub>O<sub>14</sub>-like structural type for mixed V-Mo-Nb oxides, the effect of vanadium and niobium content and treatment temperature on the phase composition, structure and catalytic properties of the samples in oxidative dehydrogenation of ethane has been studied. The limits of V and Nb content in the ternary Mo<sub>5</sub>O<sub>14</sub>-like oxide are determined. V-Mo-Nb oxides are shown to be the substitution solid solutions of composition (Mo<sub>0.6</sub>V<sub>2</sub>Nb<sub>0.4-z</sub>)<sub>5</sub>O<sub>14</sub> (0 < z < 0.2) based on binary oxide (Mo<sub>0.6</sub>Nb<sub>0.4</sub>)<sub>5</sub>O<sub>14</sub>. The structure of crystalline V-Mo-Nb oxides is refined. Crystallization of Mo<sub>5</sub>O<sub>14</sub>-like structure occurs at temperatures >550°C. Below 550°C, V-Mo-Nb oxides

are nanocrystalline materials having a disordered layered structure with the interlayer distance of ca. 4 Å. The structural model of nanocrystalline oxides is discussed. The role of Nb in the formation of ternary V-Mo-Nb oxide compounds is revealed, and the surface state of these oxides at different temperatures is analyzed. The low-temperature V-Mo-Nb oxides are the most active catalysts in the reaction of ethane oxidative dehydrogenation. They are characterized by the ratio V/Nb ≈ 1 in the Mo<sub>5</sub>O<sub>14</sub>-like structure.

#### **PREPARATION, ACTIVE COMPONENT AND CATALYTIC PROPERTIES OF SUPPORTED VANADIUM CATALYSTS IN THE REACTION OF FORMALDEHYDE OXIDATION TO FORMIC ACID**

**E.V. Danilevich, G.Ya. Popova, T.V. Andrushkevich, Yu.A. Chesalov, V.V. Kaichev, A.A. Saraev, L.M. Plyasova**

*Stud. Surf. Sci. Catal.*,  
175 (2010) pp. 463-466.

The influence of the support nature was investigated with supported vanadium catalysts prepared by a wet impregnation method. SiO<sub>2</sub>, γ-Al<sub>2</sub>O<sub>3</sub>, ZrO<sub>2</sub> and TiO<sub>2</sub> (anatase) were used as supports. Two series of catalysts were prepared, the first one consisting of catalysts of composition ca. 20% wt. V<sub>2</sub>O<sub>5</sub>/80% wt. support (series 1) and the second one prepared by washing the series 1 samples with nitric acid (series 2). In the catalysts of series 1 (except 20% V<sub>2</sub>O<sub>5</sub>/80% SiO<sub>2</sub>), vanadium is represented by both monolayer species (monomeric and polymeric VO<sub>x</sub>) and crystalline V<sub>2</sub>O<sub>5</sub> phase. When vanadium is supported on SiO<sub>2</sub>, only the crystalline V<sub>2</sub>O<sub>5</sub> is formed. Washing the samples of series 1 with nitric acid removes crystalline V<sub>2</sub>O<sub>5</sub> phase. Monomeric and polymeric vanadia species are more active in the reaction of formaldehyde oxidation to formic acid as compared to V<sub>2</sub>O<sub>5</sub>.

#### **CATALYTIC COMBUSTION OF METHANE ON FERRITES**

**M.V. Bukhtiyarova, A.S. Ivanova, E.M. Slavinskaya, L.M. Plyasova, V.A. Rogov, V.V. Kaichev**

*Stud. Surf. Sci. Catal.*,  
175 (2010) pp. 355-359.

Ferrites with the components ratio typical for hexaferrite prepared by co-precipitation and calcined at 700–1000°C were characterized by different methods. It has been shown that hexaferrite phase

formed at 700°C is amorphous and its crystallization occurs at 800°C.

### SELECTIVE CATALYTIC REDUCTION OF NITROGEN OXIDE BY AMMONIA ON SUBSTITUTED STRONTIUM FERRITES

**M.V. Bukhtiyarova, A.S. Ivanova,  
E.M. Slavinskaya, L.M. Plyasova, V.V. Kaichev,  
P.A. Kuznetsov**

*Appl. Catal., A,*  
384(1-2) (2010) pp. 230-240.

Substituted Sr-ferrites  $\text{Sr}_{1-x}\text{Ce}_x\text{Mn}_{6-y}\text{W}_y\text{Fe}_4\text{Al}_2\text{O}_{19}$  ( $x = 0; 0.2; y = 0; 0.28; 0.56; 0.84$ ) with the components ratio typical for Sr-hexaferrite obtained by co-precipitation and calcined at 700°C have been characterized by thermal analysis (TG-DTA), X-ray diffraction (XRD), Fourier transformed infrared spectroscopy (FTIR), X-ray photoelectron spectroscopy (XPS) and adsorption method. Ferrites have been studied in the selective catalytic reduction of NO by ammonia (SCR) using temperature-programmed desorption of ammonia ( $\text{NH}_3$ -TPD) and temperature-programmed reaction of ammonia ( $\text{NH}_3$ -TPR) techniques. It was shown that the substituted Sr-ferrites are multiphase. Its specific surface area is 27–59  $\text{m}^2/\text{g}$ . Surface concentrations of elements in the samples differ from the bulk chemical contents. According to XPS data, the surface of the samples is enriched by strontium. The main components on the surface are in oxidized states:  $\text{Sr}^{2+}$ ,  $\text{Mn}^{3+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Al}^{3+}$ ,  $\text{Ce}^{4+}$  and  $\text{W}^{6+}$ . The surface acidity of the samples determined by  $\text{NH}_3$ -TPD increases from 0.023 to 0.071–0.082 mmol/g when Ce and W ions are introduced in  $\text{SrMn}_6\text{Fe}_4\text{Al}_2\text{O}_{19}$ . The most active ( $X_{\text{NO}} \approx 100\%$ ) and selective with respect to  $\text{N}_2$  catalyst is the  $\text{Sr}_{0.8}\text{Ce}_{0.2}\text{Mn}_{5.16}\text{W}_{0.84}\text{Fe}_4\text{Al}_2\text{O}_{19}$  ferrite obtained by the precipitation of the soluble nitrates of Sr, Fe, Mn, Al, Ce using ammonium para-tungstate and ammonium hydrocarbonate as precipitating agents. This sample is characterized by the highest surface acidity, the highest atomic ratio of Mn/Fe and the lowest oxygen reactivity.

### EFFECT OF PREPARATION PROCEDURE ON THE PROPERTIES OF $\text{CeO}_2$

**I.G. Danilova, E.M. Slavinskaya, V.I. Zaikovskiy,  
A.S. Ivanova, A.I. Boronin, R.V. Gulyaev,  
Yu.I. Amosov**

*Kinet. Catal.,*  
51(1) (2010) pp. 143-148.

The effect of preparation procedure on the physicochemical and catalytic properties of  $\text{CeO}_2$  was studied. Differences in the electronic and structural

characteristics of  $\text{CeO}_2$  depending on preparation procedure and treatment temperature were found using X-ray diffraction analysis, transmission electron microscopy, UV–visible electronic spectroscopy, and X-ray photoelectron spectroscopy. With the use of the temperature-programmed reaction with CO, it was demonstrated that  $\text{CeO}_2$  samples with a high concentration of point defects - oxygen vacancies caused by the presence of  $\text{Ce}^{3+}$  - were characterized by an increased mobility of bulk oxygen. The samples of  $\text{CeO}_2$  with a high concentration of structural defects - micropores of size 1–2 nm and stepwise vicinal faces in crystallites – exhibited a high catalytic activity in the reaction of CO oxidation.

### METAL-SUPPORT INTERACTIONS IN $\text{Pt}/\text{Al}_2\text{O}_3$ AND $\text{Pd}/\text{Al}_2\text{O}_3$ CATALYSTS FOR CO OXIDATION

**A.S. Ivanova, E.M. Slavinskaya, R.V. Gulyaev,  
V.I. Zaikovskiy, O.A. Stonkus, I.G. Danilova,  
L.M. Plyasova, I.A. Polukhina, A.I. Boronin**

*Appl. Catal., B,*  
97(1-2) (2010) pp. 57-71.

Platinum and palladium catalysts supported on  $\gamma\text{-Al}_2\text{O}_3$  were studied by XRD, UV–vis DRS, HRTEM, TPR- $\text{H}_2$ , XPS together with measurements of their catalytic properties. The properties of the catalysts denoted as  $\text{Pt(Pd)}/\text{Al}_2\text{O}_3(\text{X})\text{-Y}$  (X—the calcination temperature of support, °C; Y—the calcination temperature of catalyst, °C) were studied as a function of the temperatures used for calcination of the support and/or the catalyst in oxygen or in a reaction mixture of  $\text{CO} + \text{O}_2$ . It was found that the deposition of Pt or Pd on  $\gamma\text{-Al}_2\text{O}_3$  did not alter the structure of the support. Two types of the Pt and Pd particles were typically present on the  $\gamma\text{-Al}_2\text{O}_3$  surface: individual particles with dimensions of 1.5–3 nm and agglomerates about 100 nm in size. In the catalysts calcined at relatively low temperatures ( $\text{Pt}/\text{Al}_2\text{O}_3(550)\text{-}450$ ), platinum was present in the form of metal clusters. However, in the  $\text{Pd}/\text{Al}_2\text{O}_3(550)\text{-}450$  catalyst, the palladium particles were almost completely decorated with a thin layer of an aluminate phase. These structures are not reduced in hydrogen in the temperature range of –15 to 450°C, and are stable to treatment in a reaction mixture of  $\text{CO} + \text{O}_2$ . Pd deposition on the  $\gamma\text{-Al}_2\text{O}_3\text{-}800$  support was found to result in stabilization of the active component in two main forms,  $\text{Pd}^0$  and  $\text{PdO}$ , with varying degrees of interaction due to the decoration effect. Calcination at the low temperature of 550°C led to the formation of a

so-called “core–shell structure”, where a palladium metal core is covered with a thin shell of an aluminate phase. Depending on the calcination temperature of the catalyst in the range of 450–1000°C, the morphological form of the active component was converted from the “core-shell” state to a state consisting of two phases, Pd<sup>0</sup> and PdO, with a gradual decrease of the Pd<sup>0</sup>/PdO ratio, weakening the interaction with the support and the growth of palladium particles. Under the action of the reaction mixture, the Pd/Al<sub>2</sub>O<sub>3</sub>(800)-(450,600,800,1000) catalysts underwent changes in the Pd<sup>0</sup>/PdO ratio, which regulates the light-off temperature. After catalyst calcination at the highest temperature used in this study, 1200°C, the palladium particles became much larger due to the loss of the palladium interaction with the support. Only the metal phase of palladium was observed in these catalysts, and their catalytic activity decreases substantially.

#### **STRUCTURAL FEATURES OF THE FORMATION OF La<sub>1-x</sub>Ca<sub>x</sub>FeO<sub>3-δ</sub> (0≤x≤0,7) HETEROVALENT SOLID SOLUTIONS**

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*J. Struct. Chem.*,  
51(5) (2010) pp. 891-897.

A synthesis method with the use of polymer-salt compositions (calcination temperature 800°C) provides the preparation of various solid solutions of a La<sub>1-x</sub>Ca<sub>x</sub>FeO<sub>3-δ</sub> series in the 0≤x≤0,7 range, which belong to the perovskite structure type. A morphotropic phase transition occurs from the orthorhombic perovskite modification (0≤x≤0.4) to the cubic one (0.5≤x≤0.7). A growing number of microdistortions in the perovskite structure and the formation of a microblock structure in the morphotropic phase transition region are observed with increasing degree of calcium substitution for lanthanum. Calcination of solid solutions with x = 0.6 and 0.7 at temperatures above 1000°C in the air or under conditions of reduced oxygen partial pressure (laboratory vacuum of 10<sup>-3</sup> Torr) results in the formation of a nanostructured state with coherently grown blocks of perovskite and Grenier phase, which is due to irreversible oxygen loss.

#### **BIMETALLIC Co-Mo-COMPLEXES WITH OPTIMAL LOCALIZATION ON THE SUPPORT SURFACE: A WAY FOR HIGHLY ACTIVE HYDRODESULFURIZATION CATALYSTS PREPARATION FOR DIFFERENT PETROLEUM DISTILLATES**

**O.V. Klimov, A.V. Pashigreva, K.A. Leonova, G.A. Bukhtiyarova, S.V. Budukva, A.S. Noskov**

*Stud. Surf. Sci. Catal.*,  
175 (2010) pp. 509-512.

The preparation method of the catalyst for the deep hydrotreatment of vacuum gas oil and gasoline is described. The method is based on vacuum impregnation of the carrier with required average pore diameter with the solution of bimetallic CoMo complexes. It was shown that the use of Co-Mo complexes, containing chelating ligands and having different molecule size, allows to obtain catalysts with the uniform distribution of the surface species, containing supported metals only in the form of Co-Mo-S phase type II that is located inside of the pores exposed to all reacting heteroatomic molecules of the feedstock.

#### **Co–Mo CATALYSTS FOR ULTRA-DEEP HDS OF DIESEL FUELS PREPARED VIA SYNTHESIS OF BIMETALLIC SURFACE COMPOUNDS**

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*J. Mol. Catal. A: Chem.*,  
322(1-2) (2010) pp. 80-89.

The synthesis of bimetallic Co–Mo compounds from ammonium heptamolybdate, citric acid and cobalt acetate for the preparation of catalysts for the ultra-deep hydrodesulfurization (HDS) of diesel fuel is reported. The structure of the Co–Mo compounds formed in solution and on alumina surfaces was studied by <sup>95</sup>Mo, <sup>17</sup>O, <sup>13</sup>C, and <sup>27</sup>Al NMR, FTIR, Raman and XAS spectroscopy. It was found that the oxidic precursor of the catalyst consists of tetrameric molybdenum (VI) citrate anions with Co<sup>2+</sup> cations coordinated to the carboxyl groups and terminal oxygen atoms. After sulfidation, the prepared catalyst was tested in the HDS of straight run gas oil, demonstrating high activity in the production of ultra-clean diesel fuel.

**BIMETALLIC Co–Mo COMPLEXES:  
A STARTING MATERIAL FOR HIGH ACTIVE  
HYDRODESULFURIZATION CATALYSTS**

**O.V. Klimov, A.V. Pashigreva, G.A. Bukhtiyarova,  
S.V. Budukva, M.A. Fedotov, D.I. Kochubey,  
Yu.A. Chesalov, V.I. Zaikovsky, A.S. Noskov**

*Catal. Today,*  
150(3-4) (2010) pp. 196-206.

The preparation of high active hydrodesulfurization catalysts is described. The procedure is based on the synthesis of labile bimetallic Co–Mo complexes in aqueous solution followed by the deposition on alumina surface at the conditions providing the structure of precursor remains unaltered after the drying. The prepared oxide surface compounds contain cobalt in the immediate vicinity of molybdenum that provides the selective formation of bimetallic sulfide compounds during the sulfidation. These compounds reveal the high activity in hydrotreatment of diesel fuels.

**HIGH-ACTIVE HYDROTREATING  
CATALYSTS FOR HEAVY PETROLEUM  
FEEDS: INTENTIONAL SYNTHESIS OF CoMo  
SULFIDE PARTICLES WITH OPTIMAL  
LOCALIZATION ON THE SUPPORT SURFACE**

**A.V. Pashigreva, O.V. Klimov, G.A. Bukhtiyarova,  
D.I. Kochubey, I.P. Prosvirin, Yu.A. Chesalov,  
V.I. Zaikovsky, A.S. Noskov**

*Catal. Today,*  
150(3-4) (2010) pp. 164-170.

The method for preparation of high-active catalysts for hydrotreating of vacuum gas oil is described. The method is based on selective incorporation of bimetal Co–Mo complexes into pores of Al<sub>2</sub>O<sub>3</sub> exposed to all molecules of the feedstock. The composition, structure and morphology of the resulting surface bimetal sulfide particles are very close to the particles characteristic of the best modern hydrotreating catalysts. The obtained catalyst is very active to hydrotreating of vacuum gas oil and superior to the best of Russian industrial catalysts.

**THE SUPERIOR ACTIVITY OF THE CoMo  
HYDROTREATING CATALYSTS, PREPARED  
USING CITRIC ACID: WHAT'S THE REASON?**

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M.A. Fedotov, D.I. Kochubey, Yu.A. Chesalov,  
V.I. Zaikovsky, I.P. Prosvirin, A.S. Noskov**

*Stud. Surf. Sci. Catal.,*  
175 (2010) pp. 109-116.

It was demonstrated, that the main positive role of citric acid during the hydrotreating catalysts preparation is consist in the formation of bimetallic complex Co<sub>2</sub>[Mo<sub>4</sub>(C<sub>6</sub>H<sub>5</sub>O<sub>7</sub>)<sub>2</sub>O<sub>11</sub>]•nH<sub>2</sub>O, that is a good precursor for selective formation of catalyst active phase, so called Co-Mo-S phase type II. The preparation method for this bimetallic complex using different precursor is described. The catalysts prepared by the complex deposition onto alumina support were studied during the different stages of the catalyst genesis. Applicability of these catalysts for ultra low sulfur diesel production was shown.

**ACTIVITY AND SULFIDATION BEHAVIOR OF  
THE CoMo/Al<sub>2</sub>O<sub>3</sub> HYDROTREATING  
CATALYST: THE EFFECT OF DRYING  
CONDITIONS**

**A.V. Pashigreva, G.A. Bukhtiyarova, O.V. Klimov,  
Yu.A. Chesalov, G.S. Litvak, A.S. Noskov**

*Catal. Today,*  
149(1-2) (2010) pp. 19-27.

The influence of drying condition of the CoMo/Al<sub>2</sub>O<sub>3</sub> catalyst prepared using citric acid as a chelating agent on the sulfidation behavior and on the catalytic activity to hydrodesulfurization of straight-run gas oil (SRGO) was investigated. The catalysts dried at 110, 220, 300 and 400°C were studied using Raman, IRS and DTG techniques. The sulfidation behavior with straight-run gas oil spiked with dimethyldisulfide (DMDS) was investigated using gas chromatography (GC) with a thermal conductivity detector and GC with an atomic-emission detector for analysis of gas and the liquid phases, respectively. It was shown that the sulfidation behavior of the catalysts prepared using the chelating agents depends on the drying condition: the lower drying temperature, the later DMDS conversion and oxide precursor sulfidation starts. A higher activity in SRGO desulfurization was obtained with catalysts dried at 110 and 220°C. This phenomenon was accounted for by the stabilization effect of chelating agent that retards precursor sulfiding but provides favorably the formation of active CoMoS phase and achieving the highest activity.

**NANOSIZED Au/C CATALYST OBTAINED FROM A TETRAAMMINEGOLD(III) PRECURSOR: SYNTHESIS, CHARACTERIZATION, AND CATALYTIC ACTIVITY IN LOW-TEMPERATURE CO OXIDATION**

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*Kinet. Catal.*,  
51(6) (2010) pp. 885–892.

A method in which the water-soluble complex  $[\text{Au}(\text{NH}_3)_4](\text{NO}_3)_3$  is used as the active-component precursor is suggested for preparing nanosized Au/C catalysts (C = Sibunit, a mesoporous carbon material). The complex is unready reducible by the carbon matrix and can be involved in cation exchange with proton-containing groups of the support. This method is referred to as cationic adsorption. It has been demonstrated by X-ray diffraction, transmission electron microscopy, and X-ray photoelectron spectroscopy that the catalyst prepared in this way and treated with  $\text{H}_2$  at  $400^\circ\text{C}$  contains size-uniform gold metal particles with a dominant diameter of  $<5$  nm. The greater part of the gold particles is located on the outer surface of the Sibunit granules; that is, an egg shell type distribution of the active component takes place. The catalyst containing 1.3 wt % Au shows high activity in CO oxidation with excess humid air at  $40^\circ\text{C}$ . In this respect, it is far superior to the Au/C catalysts prepared by conventional methods (deposition–precipitation and impregnation), in which the typical gold particle size is several tens of nanometers.

**EFFECT OF THE PREPARATION PROCEDURE ON THE STRUCTURAL PECULIARITIES AND CATALYTIC PROPERTIES OF Pt/(CeO<sub>2</sub>-TiO<sub>2</sub>) CATALYSTS IN CO OXIDATION**

**A.A. Shutilov, G.A. Zenkovets**

*Stud. Surf. Sci. Catal.*,  
175 (2010) pp. 369-372.

The procedure of the Pt/(CeO<sub>2</sub>-TiO<sub>2</sub>) catalyst preparation is crucial for the microstructure and catalytic properties in CO oxidation. Impregnation of ceria doped TiO<sub>2</sub> with platinum nitrate solution followed heating in air at  $500^\circ\text{C}$  leads to the formation of ultra fine platinum particles 0.5-0.6 nm in size stabilized at interblock boundaries of the support formed by irregularly intergrown anatase particles. Calcination of the catalyst in hydrogen at  $250^\circ\text{C}$  leads

to the formation of the platinum particles with 2-5 nm in size. The catalyst containing ultra fine platinum particles is much more active than the catalyst with particles of 2-5 nm in size. Infrared spectra of CO adsorbed on Pt revealed that high CO oxidation activity is exhibited by ultra fine Pt particles due to the high concentration of weakly bonded Pt<sup>0</sup>-CO complexes.

**CHARACTERIZATION OF ALUMINA-SUPPORTED URANIUM OXIDE CATALYSTS IN METHANE OXIDATION**

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*Catal. Today.*,  
157(1-4) (2010) pp. 217-222.

Synthesis of alumina-supported uranium oxide catalysts and their characterization in methane oxidation have been performed. The catalysts containing 5% U were prepared by impregnation, solid-phase synthesis and mechanical mixing techniques. Samples were studied using BET, XRD, HRTEM and XPS methods. It was shown that physicochemical properties and activity of the catalysts depend on the preparation procedure and calcination temperature and are determined by the extent of the interaction between active component and support. Most active are the catalysts prepared by impregnation of alumina with an aqueous solution of uranyl nitrate. It was found that the 5%U/Al<sub>2</sub>O<sub>3</sub> catalyst calcined at  $1000^\circ\text{C}$  has the highest catalytic activity, and it is explained by the formation of highly active nanodispersed state of uranium on the surface of the support.

**MECHANOCHEMICAL ACTIVATION FOR RESOLVING THE PROBLEMS OF CATALYSIS**

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*Kona Powder Part. J.*,  
27(2009) pp. 38-54.

The role of structure-energy properties of the mechanically induced defects of the crystal structure of heterogeneous catalysts is considered here. The main concepts of the effect of mechanochemical activation on the activity and selectivity of catalysts are theoretically stated. The most promising research trends are presented.



## THE DEVELOPMENT OF CATALYSTS FOR DEHYDROGENATION OF LOWER C<sub>3</sub>-C<sub>4</sub> PARAFFINS USING THE PRODUCTS OF GIBBSITE THERMAL ACTIVATION

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*Catal. Ind.*,  
2(2) (2010) pp. 145-151.

A standardized approach to the preparation of alumina-chromia catalysts for dehydrogenation of lower C<sub>3</sub>-C<sub>4</sub> paraffins is discussed. The approach is based on the use of initial products prepared by thermochemical activation (TCA) of gibbsite in an industrial flow-type flash reactor and the centrifugal thermal activation (CTA) on the CEFLAR™ unit. Catalytic characteristics in reactions of dehydrogenation of propane and butanes in fluidized and fixed beds of microspherical and granulated catalyst samples are better than those of domestic commercial catalysts and are highly competitive with the best imported catalysts.

## PREPARATION AND CHARACTERIZATION OF MODIFIED ALUMINUM OXIDE CATALYSTS

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*Kinet. Catal.*,  
51(1) (2010) pp. 139-142.

The formation of individual and modified high-purity aluminum oxides ( $\gamma$ -Al<sub>2</sub>O<sub>3</sub>) prepared from aluminum alcoholates was studied. In the study of the hydrolysis of aluminum alcoholates and modified (Zr, Ti, and Si) aluminum alcoholates, it was found that an increase in the chain length of the alkoxy group and an increase in the aging temperature or aging time in mother liquor resulted in a decrease in the concentration of an amorphous phase, an increase in the concentration of a pseudoboehmite phase, and an improvement in its crystal structure. Hydrolysis in alkaline (a 0.5 wt % solution of ammonia) or neutral solutions made it possible to obtain samples with an almost 100% pseudoboehmite content. At the same time, the samples prepared by hydrolysis in an acidic solution (a 0.1 M solution of HCl) contained a considerable amount of an amorphous phase. It was

found that the specific rate of dehydration of *n*-butanol on the modified aluminum oxide samples linearly decreased with the concentration of donor sites and linearly increased with the concentration of acceptor sites, whose concentration was measured using the spin probe method.

## PHYSICOCHEMICAL PROPERTIES OF TiO<sub>2</sub> (ANATASE) PREPARED BY THE CENTRIFUGAL THERMAL ACTIVATION OF HYDRATED TITANIUM DIOXIDE

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*Kinet. Catal.*,  
51(3) (2010) pp. 444-448.

The physicochemical properties of titania (anatase) prepared from hydrated titanium dioxide by centrifugal thermal activation (CTA) at 140–700°C were studied. It was found that the microstructure and the texture parameters of anatase prepared by the above method were considerably different from those of the samples prepared by the traditional thermal decomposition of titanium hydroxide. The conditions of centrifugal activation exerted a considerable effect on the structure and the texture parameters of the resulting anatase. The crystal structure of anatase prepared at a temperature lower than 650°C was imperfect, and it approached a regular structure only at a temperature of >650°C. At temperatures higher than 300°C, the samples of TiO<sub>2</sub> prepared using CTA were characterized by higher specific surface areas, fine pore structures, and comparable mesopore volumes, as compared with the samples prepared by commonly used synthetic methods.

## EFFECT OF THERMAL TREATMENT CONDITIONS ON THE ACID PROPERTIES OF ZEOLITE BETA

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*Kinet. Catal.*,  
51(2) (2010) pp. 318-324.

The acid properties of zeolite Beta were studied by IR spectroscopy with the use of adsorbed NH<sub>3</sub> and CO probe molecules. It was found that the strength of the Brønsted acid sites (BASs) of zeolite Beta was the same as that of zeolite ZSM-5. Approximately a third of the total amount of BASs participated in the formation of hydrogen bonds at interdomain boundaries; thus, they were inaccessible to molecules larger than the ammonia molecule. The qualitative and

quantitative compositions of acid sites in zeolite Beta can be regulated by changing the conditions of calcination of the initial zeolite form (temperature and/or gas atmosphere). Calcination under vacuum conditions afforded the highest acidity of the zeolite with respect to all types of acid sites. Calcination in a flow of air resulted in the lowest acidity of the sample, especially, with respect to the concentration of strong Lewis acid sites, because of the formation of an alumina phase. Calcination in an atmosphere of helium resulted in a decrease (as compared with a vacuum) in the rate of removal of the organic template decomposition products of the initial zeolite form from the channel volume. The resulting ethylene was mainly adsorbed at strong Lewis sites and converted into undesorbed condensation products.

#### **EFFECT OF THERMAL TREATMENT CONDITIONS ON THE PROPERTIES OF THE IC-42-1 MONOLITH HONEYCOMB CATALYST**

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*Catal. Ind.*,  
2(2) (2010) pp. 152-157.

The method of thermal analysis is used to study the effect of starting material, commercial hematite prepared by sulfate or chloride technology, on the processes that occur in the course of thermal treatment of the IC-42-1 monolith honeycomb catalyst for ammonia oxidation. It is shown that the strength of monoliths depends on the nature of starting material and the quality of monoliths (the number of monoliths with cracks and their stability in thermal cycling). It is shown that the conditions of thermal treatment (continuous or stepwise temperature increase) affect the quality of monoliths. A stepwise increase in temperature during thermal treatment of the catalyst (irrespective of the starting material), especially at the stage of preliminary calcination, makes it possible to obtain a high yield (up to 90%) of monoliths without cracks that stand at least 10 thermal cycles. The optimal regime for the thermal treatment of the monolith honeycomb catalyst is determined.

#### **TRANSPORT PROPERTIES OF MODIFIED NAFION MEMBRANES: EFFECT OF ZEOLITE AND PRECURSORS**

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*Solid State Ionics*,  
180(40) (2010) pp. 1694-1701.

Fe-silicalite/Nafion composite membranes with high relative selectivity (as defined by the proton conductivity to methanol permeability ratio) of 5.4 and proton conductivity of 11 mS cm<sup>-1</sup> were prepared by *in situ* hydrothermal synthesis of the zeolite within the pores of Nafion membranes. The effects of the zeolite structure and precursor structure were evaluated in terms of transport properties and acidity levels for a series of Nafion membranes modified with silica and tetrapropylammonium (TPA) and tetrabutylammonium (TBA) cations. Introduction of up to 40% (w/w) of silica vs. pure Nafion shows little effect on the transport and acidity properties of the composite membranes. Introduction of tetraalkylammonium (TAA) cations reduces water uptake of the membranes, and results in the appearance of protons that are inaccessible for titration in water media. The selectivity of the composite membranes increases in the order: SiO<sub>2</sub>/Nafion < TAA/Nafion < Fe-silicalite/Nafion.

#### **HYDROTHERMAL SYNTHESIS IS A PROMISING METHOD FOR PRODUCTION OF PERMEABLE POROUS MATERIALS FROM DISPERSE ALUMINIUM**

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*50 Years of Powder Metallurgy in Belarus. History, Achievements, Prospects*,  
Minsk, 2010, p. 328-352 (632 p.).

Mechanically strong porous materials prepared by hydrothermal synthesis (HTS) are studied. The method is shown to provide the formation of porous solids with an extensive nanoporous structure and a large enough proportion of macropores. Variation of the powder particles in shape allows the oxide/aluminium ratio, as well as permeability of the porous solids, to be varied. The proportion of the oxide component can be increased considerably through encapsulation of precursors of the nanoporous materials in the macroporous system. In general, the

method of hydration hardening under hydrothermal conditions makes it possible to 'construct' porous materials over wide range for various practical applications.

#### **SYNTHESIS OF THE INDIUM-CONTAINING ZEOLITES AND STUDYING THEIR PROPERTIES**

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**L.M. Velichkina\***, **E.V. Urzhumova\***,  
**A.V. Vosmerikov\*** (*\*Institute of Petroleum Chemistry, Tomsk, Russia*)

*Neftepererabotka i Neftekhimiya*,  
2 (2010) pp. 17-21.

The indium containing zeolites were prepared by the methods of hydrothermal synthesis and dry mechanical mixing of high-silica zeolite with nanosized indium powder. Their physico-chemical and catalytic properties were studied in the course of methanol conversion into hydrocarbons and upgrading of straight-run gasoline oil fraction. An introduction of indium into the zeolite decreases concentration and strength of its acid sites but increases its selectivity with respect to alkene and isoalkane formation. The zeolite containing indium nanopowder was found to be most efficient for methanol and straight-run gasoline conversion.

#### **SYNTHESIS AND PROPERTIES OF HIGH-MODULAR ZEOLITES**

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**A.V. Vosmerikov\***, **L.M. Velichkina\***,  
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*Chem. Eng.*,  
1 (2010) pp. 15-20.

Zeolites with high silicate modules have been synthesized using hexamethylenediamine as structure-forming additives. Their physicochemical and acidic characteristics have been investigated. It has been shown that produced zeolites features high activity and stability in process of methanol conversion.

#### **BIMETALLIC Rh-Co/ZrO<sub>2</sub> CATALYSTS FOR ETHANOL STEAM REFORMING INTO HYDROGEN-CONTAINING GAS**

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**A.I. Gubanov\***, **E.Yu. Filatov\***, **P.E. Plyusnin\***,  
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*Kinet. Catal.*,  
51(6) (2010) pp. 893–897.

The main goal was to develop the target-oriented synthesis of bimetallic Rh–Co particles on the support surface.

The properties of supported bimetallic Rh–Co/ZrO<sub>2</sub> catalysts in ethanol steam reforming into hydrogen-containing gas were studied. The particles of Rh–Co solid solutions on the catalyst surface were prepared by the thermal decomposition of the double complex salt [Co(NH<sub>3</sub>)<sub>6</sub>][Rh(NO<sub>2</sub>)<sub>6</sub>] and the solid solution Na<sub>3</sub>[RhCo(NO<sub>2</sub>)<sub>6</sub>]. It was found that the bimetallic Rh–Co/ZrO<sub>2</sub> catalysts exhibited high activity in the reaction of ethanol steam reforming. The equilibrium composition of reaction products was attained at 500–700°C and a reaction mixture space velocity of 10 000 h<sup>-1</sup>.

#### **EFFECTS OF IRON, BISMUTH, AND VANADIUM OXIDES ON THE PROPERTIES OF CORDIERITE CERAMICS**

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**L.M. Plyasova**, **N.A. Rudina**

*Kinet. Catal.*,  
51(1) (2010) pp. 131-134.

The effects of iron oxide (10%), bismuth oxide (2.2%), and vanadium oxide (1.6%) admixtures on the physicochemical properties of cordierite ceramics are reported, including the phase composition, surface features, porous structure, and activity in ammonia oxidation. The formation of the cordierite phase is favored by introducing the oxide modifiers, by raising the heat treatment temperature, and by extending the heating time. The introduction of V<sub>2</sub>O<sub>5</sub> affords well-crystallized cordierite with a small specific surface area and a large proportion of macropores. The oxide modifiers markedly enhance the ammonia oxidation activity and nitrogen oxide selectivity of the cordierite ceramics.

### THE EFFECT OF 3d CATION MODIFICATION ON THE PROPERTIES OF CORDIERITE-LIKE CATALYSTS

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*Stud. Surf. Sci. Catal.*,  
175 (2010) pp. 343-346.

The MnO<sub>x</sub>-modified cordierite-like honeycomb catalysts 2(Mg<sub>1-x</sub>Mn<sub>x</sub>)O·2Al<sub>2</sub>O<sub>3</sub>·5SiO<sub>2</sub> (x=0-1) were prepared by mechanochemical method from clay, talk, alumina and MnO<sub>2</sub>. Catalysts were characterized by BET, XRD, XPS, and H<sub>2</sub>-TPR. Catalytic activity was studied in ammonia oxidation to NO. Phase composition, texture and catalytic activity of prepared monoliths were shown to depend on the MnO<sub>x</sub> content and preparation conditions (preliminary mechanical treatment, time and calcination temperature). The fully substituted catalyst (2MnO·2Al<sub>2</sub>O<sub>3</sub>·5SiO<sub>2</sub>) was shown to have the highest selectivity to NO at 900°C, atmospheric pressure and millisecond contact time.

### SYNTHESIS OF AN EFFICIENT CATALYST BASED ON NICKEL AND CHROMIUM OXIDES FOR HYDROGENATION REACTIONS

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*Kinet. Catal.*,  
51(1) (2010) pp. 135-138.

The dissolution of nickel metal in nitric acid in the presence of the dichromate anion as the oxidizer is reported. The formation of Ni(II) and Cr(III) nitrates takes place in two steps with the intermediate formation of nitrous acid. A new method to synthesize the nickel–chromium oxide catalyst from nickel and chromium nitrate solutions is suggested, in which the solutions are obtained by an environmentally friendly technology from nickel metal, chromium(VI) oxide, and nitric acid. The catalyst is highly active and selective in benzene hydrogenation and in CO preferential hydrogenation in the presence of CO<sub>2</sub>

### CATALYSTS BASED ON THE NANODISPERSED METASTABLE IRON OXYHYDROXIDE, 2-LINE FERRIHYDRITE

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In “*Advances in Nanotechnology*”, Nova Science Publishers, Eds. Z. Bartul, J. Trenor, 2010, vol. 2, pp. 347-363 (470 pp.).

The chapter reviews the data on the 2-line ferrihydrite structure and texture, methods for its preparation, and catalytic properties of 2LFH-based catalysts. Since 2-line ferrihydrite is metastable, the necessity and possibility of inhibiting its crystallization to hematite is discussed and new data on the kinetics of crystallization process are reported, including the effect of Al<sup>3+</sup>, Cr<sup>3+</sup>, and Cu<sup>2+</sup> promotion. During discussion, particular attention is paid to the potential of 2LFH in catalytic application.

### EFFECT OF ANIONIC ADMIXTURES ON THE COPPER–MAGNESIUM MIXED OXIDE REDUCTION

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*React. Kinet. Mech. Catal.*,  
101(1) (2010) pp. 73-83.

The anionic composition, structural parameters, optical properties and reduction behavior of Cu–MgO solid solution in hydrogen dramatically change after exposure to air. The air-exposed Cu–Mg oxide contains a lot of CO<sub>3</sub><sup>2-</sup> and OH<sup>-</sup> anions. Its reduction proceeds via two stages: (1) diffusion of Cu<sup>2+</sup> to the surface and (2) chemical interaction of Cu<sup>2+</sup> with hydrogen. The effective activation energy gradually increases from that of the chemical step (65 kJ/mol) to that of the transport step of Cu<sup>2+</sup> diffusion (130 kJ/mol). This behavior follows the “compensation effect”, which is close to those reported earlier for CuO reduction. On the contrary, reduction of Cu<sup>2+</sup> from the Cu–Mg oxide sample, which was not exposed to air after thermal pretreatment in the inert gas, proceeds in one step at 120–160°C with the effective activation energy of 19 kJ/mol, which is manifold less than the reported effective activation energies for various Cu-oxide systems. Water molecules eliminate from the sample slowly along with further heating up to 450°C.

**INFLUENCE OF CRYSTALLINITY ON THE PHYSICO-CHEMICAL PROPERTIES OF SAPO-31 AND HYDROCONVERSION OF n-OCTANE OVER Pt LOADED CATALYSTS**

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*Appl. Catal., A*,  
378(1) (2010) pp. 96-106.

SAPO-31 samples of different crystallinity were prepared at various stages of hydrothermal synthesis and characterized by XRD, SEM, chemical analysis, TGA/DTA, N<sub>2</sub> adsorption, IR spectra of adsorbed CO, and solid state NMR. The obtained data showed that the adsorptive and acidic properties of SAPO-31 are related to the crystallinity of the material. The concentration of strong acid sites increased simultaneously with the growth of the content of the microporous phase and attained a maximum value at a crystallinity of 80% with no further change. During earlier stages of crystallization, a microporous material deficient in silica is formed, and Si atoms are predominantly introduced into the framework by the substitution of P by Si according to a SM2 mechanism. At later stages, silica domains are formed in SAPO-31 due to the combined SM2 + SM3 substitution mechanisms. NMR data showed that the environment of the Si atoms changes after the maximum crystallinity is attained, which points to the continuous modification of the acidic properties of SAPO-31. The hydroconversion of n-octane was performed over Pt-containing silicoaluminophosphates with different crystallinity. Catalytic experiments showed that an increased time of hydrothermal synthesis of the silicoaluminophosphate gives rise to a further increase of both the catalytic activity and isomerization selectivity of Pt loaded catalysts. It was supposed that the acidic sites located in the near-surface layer of the SAPO-31 crystals are responsible for the improved catalytic properties of Pt/SAPO-31.

**METHYLATION OF NAPHTHALENE ON MTW-TYPE ZEOLITES. INFLUENCE OF TEMPLATE ORIGIN AND SUBSTITUTION OF Al BY Ga**

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*Appl. Catal., A*,  
375(2) (2010) pp. 279-288.

Two templates, methyltriethylammonium bromide (MTEA) and tetraethylammonium bromide (TEA) were used to synthesize aluminosilicate ZSM-12 zeolites. Additionally, zeolites isomorphously substituted (partially or totally) by gallium were prepared with MTEA. Various techniques including XRD, scanning electron microscopy (SEM), N<sub>2</sub> adsorption, NH<sub>3</sub>-TPD, Fourier transform infrared (FTIR) spectroscopy and MAS NMR were used to monitor the physico-chemical properties of these solids. Catalytic properties of the prepared zeolites were studied in the methylation of naphthalene. Higher conversion of naphthalene, higher content of 2,6-DMN among total DMNs and higher 2,6-/2,7-DMN ratio were observed on Al-TEA sample and on Ca-containing zeolites compared to Al-MTEA. The specific morphology of zeolite crystals and the presence of secondary mesopores were identified to be responsible for remarkable catalytic performance of MTW-type zeolites.

**EVALUATION OF SYNTHETIC CONDITIONS FOR H<sub>3</sub>PO<sub>4</sub> CHEMICALLY ACTIVATED RICE HUSK AND PREPARATION OF HONEYCOMB MONOLITHS**

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*Eurasian Chem.-Techn. J.*,  
11(3) (2009) pp. 245-252.

Activated carbons in this work were prepared from rice husk by phosphoric acid activation followed by alkaline desilication. Pseudo-random selection of 16 rice husk samples was subjected to carbonization at the following conditions: 0.5 to 2 h of activation time, 300-600°C and H<sub>3</sub>O<sub>4</sub>/precursor (wt/wt) impregnation ratio of 0.5 to 2. Concentration of NaOH desilication solution varied from 0.5 to 2M. It was found that out of the four factors impregnation ratio is clearly the strongest and at the impregnation ratio of 2 for 1 h at 500°C N<sub>2</sub> BET-surface area reaches 1690 m<sup>2</sup>/g (S<sub>BET</sub> (Ar)=2492 m<sup>2</sup>/g) while pore volume becomes 1.95 cm<sup>3</sup>/g. Elemental analysis showed highest carbon

content for this sample (87.96%). All samples have insignificant amount of Si and traces of metals, but considerable amount of phosphorus. Blocks of honeycomb structure prepared from Ca-montmorillonite and desiccated carbonized rice husk (impregnation ratio is 1.5, 1 h at 600°C) have BET-surface area obtained by thermal desorption of argon up to 856 m<sup>2</sup>/g.

#### **CHEMICAL AND PHASE TRANSFORMATIONS OF NANOCRYSTALLINE OXIDES IN A CARBON NANOREACTOR**

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In “*Nanotechnology 2010: Advanced Materials, CNTs, Particles, Films and Composites*”, NSTI, 2010, vol. 1, ch. 5, pp. 701-704.

The application of nanocrystalline oxides as precursors for synthesis of new nanomaterials with the preservation of the dimensions of the initial nanoparticles is of considerable interest. Its main problem is the sintering and growth of the nanoparticles during the solid-state chemical transformations at elevated temperatures. A promising approach to the solution of this problem is the deposition of a coating on the surface of the precursor nanoparticles that would prevent their sintering. Current study was devoted to the investigation of the solid-state phase and chemical transformations of nanocrystalline TiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> coated with carbon. In all cases the carbon coating acting as a carbon nanoreactor prevented the sintering of the nanoparticles. Meanwhile, such coating appeared to be penetrable by the molecules present in the gas phase. The anatase phase in all synthesized C/TiO<sub>2</sub> nanocomposites could be completely converted to TiOF<sub>2</sub> in the reaction with halocarbons. Intriguingly, this reaction was phase-selective: only anatase nanoparticles were converted. So, the carbon coating deposited on the oxide nanoparticles prevents their sintering and makes it possible to synthesize new nanomaterials. Small size of the oxide nanoparticles inside such carbon nanoreactor is the key factor preventing the phase transformations in the studied C/TiO<sub>2</sub> and C/Al<sub>2</sub>O<sub>3</sub> nanocomposites.

#### **SYNTHESIS OF SULFATED AND CARBON-COATED Al<sub>2</sub>O<sub>3</sub> AEROGELS AND THEIR ACTIVITY IN DEHYDROCHLORINATION OF 1-CHLOROBUTANE AND (2-CHLOROETHYL)ETHYL SULFIDE**

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In “*Nanotechnology 2010: Advanced Materials, CNTs, Particles, Films and Composites*”, NSTI, CRC Press, 2010, vol. 1, pp. 364-367.

Alumina is widely used as a sorbent and catalyst support. These applications typically require high surface area, stability in the used environment and often the presence of specific groups on the surface. Many of these goals can be achieved by the synthesis of alumina aerogels with specific modifying agents added to the alumina precursor before gelation. In this work it was studied how the gel modification with organic and inorganic acids, and beta-diketones affects the textural and adsorption properties of nanocrystalline alumina synthesized by the modified aerogel procedure. The highest specific surface areas obtained exceeded 1000 m<sup>2</sup>/g, being 4-5 times higher than those of typically used alumina materials. The activity of high surface area nanocrystalline alumina materials in dehydrochlorination of (2-chloroethyl)ethyl sulfide (2-CEES) and 1-chlorobutane compared favorably with that of other nanocrystalline metal oxides. The temperature of 1-chlorobutane decomposition could be decreased by ca. 100°C in comparison with MgO aerogel extensively studied earlier while maintaining 98% selectivity to butenes. Sulfated alumina aerogels had high surface areas ca. 600 m<sup>2</sup>/g after calcination at 600°C and showed excellent acidic properties typical for sulfated alumina. Overall, the developed nanoparticulate modified alumina aerogels appear to have excellent adsorption properties with enhanced stability and reactivity.

## **SYNTHESIS OF NANOCRYSTALLINE VO<sub>x</sub>/MgO AEROGELS AND THEIR APPLICATION FOR DESTRUCTIVE ADSORPTION OF CF<sub>2</sub>Cl<sub>2</sub>**

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In “*Nanotechnology 2010: Advanced Materials, CNTs, Particles, Films and Composites*”, NSTI, CRC Press, 2010, vol. 1, ch. 3, pp. 452-455 (976 pp.).

Nanocrystalline VO<sub>x</sub>/MgO is extensively studied as a potential catalyst and adsorbent. In this commutation further advances are reported to the recently developed method for synthesis of binary VO<sub>x</sub>/MgO aerogels by hydrolysis of the V and Mg alkoxides mixed together followed by gelation and supercritical drying. The suggested method of synthesis makes it possible to obtain aerogels with

exceptionally high surface areas (~ 1200 m<sup>2</sup>/g) and uniform vanadium distribution. Their dehydration yields nanocrystalline VO<sub>x</sub>/MgO with nanoparticle size ca. 5 nm. The aerogel method for synthesis of the binary VO<sub>x</sub>/MgO oxide allows for obtaining the nanocrystalline materials in a wide range of vanadium concentrations. The synthesized nanocrystalline VO<sub>x</sub>/MgO oxides were used as destructive sorbents for decomposition of halocarbons. In this case, the doping of MgO aerogels with a small amount of VO<sub>x</sub> (1-2%) results in significant acceleration of the reaction causing deeper MgO transformation to MgF<sub>2</sub> and shortening the induction period. The latter was found to shorten by as much as an order of magnitude as the vanadium concentration in the aerogel was increased. So, a method was developed for synthesis of binary VO<sub>x</sub>/MgO aerogels with high surface areas and uniform vanadium distribution that are potentially promising catalysts and destructive sorbents.

## **Carbon and Carbon Related Materials**

### **CATALYST AND TECHNOLOGY FOR PRODUCTION OF CARBON NANOTUBES**

**V.V. Chesnokov, R.A. Buyanov, A.S. Chichkan**

*Kinet. Catal.*,  
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Modifying the iron-aluminum catalyst with molybdenum oxide affords a marked increase in the yield of carbon nanotubes from 1,3-butadiene diluted with hydrogen. The optimum catalyst composition is 6.5% MoO<sub>3</sub>-52% Fe<sub>2</sub>O<sub>3</sub>-Al<sub>2</sub>O<sub>3</sub>. With this catalyst, it is possible to obtain over 100 g of carbon nanotubes per gram of catalyst in a reactor fitted with a McBain balance. Replacing expensive 1,3-butadiene with the cheaper commercial propane-butane mixture (80 mol % propane + 20 mol % butane) leads to a sharp decrease in the nanotube yield because of the lower reactivity of the latter. Based on the concept of the catalytic decomposition of hydrocarbons and the formation of nanosized carbon materials via the carbide cycle mechanism, a new, efficient, CoO-MoO<sub>3</sub>-Fe<sub>2</sub>O<sub>3</sub>-Al<sub>2</sub>O<sub>3</sub> catalyst has been developed. The enhancement of the activity of the MoO<sub>3</sub>-Fe<sub>2</sub>O<sub>3</sub>-Al<sub>2</sub>O<sub>3</sub> catalyst by promoting it with cobalt oxide is achieved without a change in the rate-limiting step of the process. The design of a continuous reactor for carbon nanotube synthesis is suggested. The characteristics of the resulting nanotubes are presented.

### **GROWTH OF CARBON NANOTUBES FROM BUTADIENE ON A Fe-Mo-Al<sub>2</sub>O<sub>3</sub> CATALYST**

**V.V. Chesnokov, V.I. Zaikovskiy, A.S. Chichkan, R.A. Buyanov**

*Kinet. Catal.*,  
51(2) (2010) pp. 293-298.

The MoO<sub>3</sub>-Fe<sub>2</sub>O<sub>3</sub>-Al<sub>2</sub>O<sub>3</sub> catalysts were prepared from metal nitrates using a coprecipitation method. It was found that the modification of an alumina-iron catalyst with molybdenum oxide resulted in the formation of a solid solution based on hematite, in which a portion of iron ions was replaced by aluminum and molybdenum ions. The MoO<sub>3</sub>-Fe<sub>2</sub>O<sub>3</sub>-Al<sub>2</sub>O<sub>3</sub> catalyst was reduced with a reaction mixture at 700°C. Under the action of 1,3-butadiene diluted with hydrogen, the solid solution based on hematite was initially converted into magnetite and then into an Fe-Mo alloy. The modification of an alumina-iron catalyst with molybdenum oxide considerably changed its properties in the course of carbon nanotube formation. As the Mo content was increased, the yield of carbon nanotubes passed through a maximum. The optimum catalyst was 6.5% MoO<sub>3</sub>-55% Fe<sub>2</sub>O<sub>3</sub>-Al<sub>2</sub>O<sub>3</sub>. The addition of small amounts of MoO<sub>3</sub> (to 6.5 wt %) to the alumina-iron catalyst increased the dispersity and modified the properties of active metal particles: because of the formation of an Fe-Mo alloy, the rate of growth decreased but the stability of carbon

nanotube growth and the yield of the nanotubes increased. A further increase in the molybdenum content decreased the yield because molybdenum is inactive in the test process.

#### **SYNTHESIS OF CARBON NANOFIBERS BY CATALYTIC METHANE PYROLYSIS OVER NICKEL-BASED CATALYSTS**

**A.S. Chichkan, V.V. Chesnokov**

*Izvestiya VUZov, Fizika,*  
12(2) (2009) pp. 130-132.

The existing methods for synthesis of carbon nanostructures are considered, advantages of catalytic methods highlighted. Carbon nanofibers are suggested to synthesized by methane pyrolysis over nickel-based catalysts. The operational stability of the Ni-Cu catalyst is shown to be improved by modifying it with iron.

#### **SYNTHESIS AND PROPERTIES OF A MICROPOROUS CARBON MATERIAL AS A CATALYST SUPPORT FOR FUEL CELLS**

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*Kinet. Catal.,*  
51(2) (2010) pp. 312-317.

It is stated that one-dimensional conductivity in amorphous microporous carbon material (AMCM) samples is associated with the considerable imperfection of graphene fragments in the carbon material rather than the presence of unshared electrons. It is likely that the graphene fragments are formed upon the carbonization of a carbon precursor accompanied by the partial or complete removal of precursor heteroatoms. It is hypothesized that the presence of localized unpaired electrons, which give EPR spectra, is due to the formation of local defects in carbene fragments. Thus, the effects of the value of conductivity and the concentration of unpaired electrons on the power output of a fuel cell cannot be distinguished based on the experimental data with the use of an AMCM as a catalyst support. The interaction of localized paramagnetic centers with electron gas can be interpreted in terms of the C-S relaxation model.

#### **MULTI-WALLED CARBON NANOTUBES WITH ppm LEVEL OF IMPURITIES**

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S.I. Moseenkov, L.M. Plyasova, I.Yu. Molina,  
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*Phys. Status Solidi B,*  
247(11-12) (2010) pp. 2695-2699.

Here multi-walled carbon nanotubes (MWNT) of different average diameters with ppm level purity were produced by a low temperature C<sub>2</sub>H<sub>4</sub> pyrolysis on Fe-Co type catalysts combined with forthcoming graphitization at 2200–2800°C in argon flow. Annealed nanotubes were characterized with X-ray fluorescent analysis, BET surface measurements, HRTEM, X-ray diffraction, DTA analysis, and measurements of temperature and magnetic field dependences of conductivity. The graphitization of MWNT results in removal of residual catalyst metal impurities, reduction of the wall defects, and closure of nanotube tips. It was found that extent of these effects depends on MWNT diameters. It was proposed that the graphitization is caused by a significant thermal displacement and diffusion of carbon atoms at temperatures higher the Debye temperature.

#### **RAMAN DIAGNOSTICS OF MULTI-WALL CARBON NANOTUBES WITH A SMALL WALL NUMBER**

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*Phys. Status Solidi B,*  
247(11-12) (2010) pp. 2827-2830.

Raman spectroscopy study of multi-wall carbon nanotubes (MWNTs) of different diameters (with a small controllable number of walls) produced using Fe-Co catalyst with a variable size of the same active component has been performed. As produced MWNTs with different diameters (series 1) and two types of tubes with fixed mean diameters (~10 and 20 nm) heated in a flow of pure argon at various temperatures (2200, 2600 and 2800°C – series 2) have been characterized. The Raman spectra of MWNTs have been registered in three spectral regions, corresponding to D (disorder-induced), G (graphite) and 2D (two-phonon scattering) bands.



A ratio of intensities  $I_{2D}/I_D$  for tubes of series 1 has demonstrated almost a linear dependence on the nanotube diameter. After heating (series 2), D (disorder-induced) Raman band has shown a substantial decrease in intensity. The variation of the Raman spectra parameters is discussed in terms of defectiveness of nanotubes.

#### **ELECTROPHYSICAL AND ELECTROMAGNETIC PROPERTIES OF PURE MWNTS AND MWNT/PMMA COMPOSITE MATERIALS DEPENDING ON THEIR STRUCTURE**

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*Fullerenes Nanotubes Carbon Nanostruct.*, 18(4-6) (2010) pp. 505–515.

Three types of carbon multi-walled nanotubes (MWNT) with different morphology and mean diameter (7.5, 10.5, 22 nm) were used to produce polymethylmetacrylate-based composites (MWNT/PMMA) with variable MWNT loading (0.5-5 wt%). The electrophysical properties of produced composites and electromagnetic response in frequency range of 2-12 GHz were investigated. Electrical conductivity of composites depends on the MWNT type increasing with lowering of the nanotube diameter and loading in composite. Reflectance and transmittance coefficients were calculated from dielectric permittivity data and found to correlate with electrical conductivity. The highest level of EM reflection was obtained for 7.5 nm MWNT-based composites and makes the value of 0.65-0.7, whereas 22 nm MWNT/PMMA composites are more transparent for EM radiation with reflection coefficient 0.3-0.4 for the highest MWNT loading. Thus MWNT/PMMA composites demonstrate high EM shielding properties.

#### **THE ELECTROMAGNETIC RESPONSE OF COMPOSITE MATERIAL MADE ON THE BASIS OF MULTI-WALLED CARBON NANOTUBES**

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*Doklady TUSUR*, 2(22) part 1 (2010) pp. 56-58.

The investigation results of the passage and reflection factors of composite materials made on the basis of carbon nanotubes and their mixtures with hexaferrite nanopowders are presented.

#### **INFLUENCE OF SURFACE LAYER CONDITIONS OF MULTIWALL CARBON NANOTUBES ON THEIR ELECTROPHYSICAL PROPERTIES**

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*Diamond Relat. Mater.*, 19(7-9 sp. issue) (2010) pp. 964-967.

The authors have investigated temperature  $\sigma(T)$  and magnetic field  $\sigma(B)$  dependences of the conductivity of multiwalled carbon nanotubes (MWNTs) with different average outer diameter  $d$  and composite materials on the bases of these MWNTs in dielectric polymethyl methacrylate matrix (PMMA). It was shown that the percolation threshold of the electrical conductance for MWNT/PMMA composites lies in the range of concentration of MWNTs lower than 0.5 wt.%. From the joint analysis of  $\sigma(T)$  and  $\sigma(H)$  it has been found that quantum corrections to the conductivity for interaction electrons is dominated in MWNTs with average outer diameters  $d \sim 23$  nm and MWNT/PMMA composite materials. The constant of electron–electron interaction  $\lambda$  from quantum correction to  $\sigma(T)$  and  $\sigma(H)$  were determined. In the result it has been found that  $\lambda$  depends on the concentration of MWNTs in MWNT/PMMA materials. This fact demonstrates that the interaction of the dielectric material of PMMA with surface atoms of MWNTs leads to the change of the  $\lambda$  in MWNTs. For MWNTs with average outer diameters  $d \leq 13$  nm and for composite on the base of these MWNTs the domination of one dimensional variable range hopping conductivity (VRHC) at a temperature below 20 K have been observed. From the data of VRHC the authors estimated the density of localized

states at the Fermi level  $N(E_F)$  and demonstrated that  $N(E_F)$  is decreasing in composite materials.

#### **NEW CARBON NANOMATERIALS: ELECTRON TRANSPORT IN COMPOSITES BASED ON CARBON NANOSTRUCTURES**

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*Thermophys. Aeromech.*,  
16(sp. issue) (2009) pp. 783–787.

Electron transport properties were studied using composites based on carbon multiwall nanotubes in a dielectric polymethylmethacrylate matrix. Composites containing 0.25 to 10 wt % of nanotubes were studied to determine temperature dependencies of their electric conductivity  $\sigma(T)$  at the temperature range  $T$  4.2–300 K, and magnetic conductivity  $\sigma(B)$  at the magnetic field range  $B$  0–6 T (at temperature 4.2 K of liquid helium). It was established that the percolation threshold of electric conductivity falls into the range of MW nanotube concentrations below 1 wt %. Combined analysis of  $\sigma(T)$  and  $\sigma(B)$  revealed changes in constant  $\lambda$  of electron-electron interaction at variations in the MW nanotube concentration in the nanocomposites to lead to conclude about interaction of the dielectric matrix material and surface atoms of the carbon MW nanotube.

#### **NEW CARBON NANOMATERIALS: CORRELATION BETWEEN ELECTROCONDUCTING PROPERTIES AND STRUCTURAL CHARACTERISTICS OF CARBON MULTIWALL NANOTUBES**

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*Thermophys. Aeromech.*,  
16(sp. issue) (2009) pp. 789–793.

Under discussion is the correlation of electron conducting properties of carbon multiwall nanotubes and their structural parameters such as diameter, wall number and defects. It is shown experimentally that an increase in diameter and wall number leads to an increase in the concentration of current carriers in the multiwall nanotubes. The metal-Anderson dielectric transition that is observed on variations in the nanotube structural parameters is considered.

#### **NEW CARBON NANOMATERIALS: MAGNETIC RESISTANCE AND TEMPERATURE DEPENDENCIES OF ELECTRIC RESISTANCE OF ONION-LIKE CARBON**

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**T.I. Buryakov\***, **K.R. Zhdanov\***, **V.L. Kuznetsov**,  
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*Thermophys. Aeromech.*,  
16(sp. issue) (2009) pp. 795–800.

Electrophysical properties of onion-like carbon are studied; the carbon particles are 4–8 nm in size and built-up by 5–10 embedded fullerene-like spheres which are linked to one another through 1 to 3 common curved graphene envelopes to form aggregates of 50–300 nm in size. Temperature dependence of electric resistance is discussed in terms of the Mott one-dimensional conductivity theory with a variable jump length, square-increasing positive magnetic resistance up to 6 T – shrinkage of wave functions of conductivity electrons. The negative magnetic resistance at magnetic fields below 1–2 T, which is observed with several samples of onion-like carbon, also is under consideration. The results obtained are used to estimate the current carrier concentration, state density of conductivity electrons at the Fermi level and to calculate the localization radius which appears comparable to the particle size of the carbon nanoion.

#### **NEW CARBON NANOMATERIALS: THE INFLUENCE OF GAS PHYSADSORPTION ON ELECTRIC CONDUCTIVITY OF THE NEW CARBON NANOMATERIALS**

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*Thermophys. Aeromech.*,  
16(sp. issue) (2009) pp. 801-807.

Experimental results of studying the influence of air, oxygen, hydrogen, and methane on electric conductivity of carbon multiwall nanotubes and onion-like carbon are presented. From the results obtained, the said gases physisorbed on the surface of the carbon materials. Depending on the chemical nature of the gas, the adsorption causes changes in the electric conductivity by different quantities but no more than 10%.

## EFFECT OF THERMAL TREATMENT CONDITIONS ON THE PROPERTIES OF ONION-LIKE CARBON BASED POLYMER COMPOSITE

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*Compos. Sci. Technol.*,  
70(16) (2010) pp. 2298-2303.

Dielectric properties of onion-like carbon (OLC) and polyurethane composite prepared using different procedures were investigated in the frequency range up to 1 MHz. It was shown that broadband dielectric spectroscopy is powerful tool to determine technological fingerprints in the studied materials. It is demonstrated that cured samples annealed at temperature close to the melting point (450 K) exhibit substantially higher dielectric permittivity and electrical conductivity in comparison with untreated samples. With the increase of temperature of an untreated sample, its dielectric permittivity, electric conductivity and critical frequency increase, while Maxwell–Wagner mean relaxation time and aggregate sizes of OLC decreases. Annealing of the composite at temperature close to the melting temperature produce sample with more homogeneous distribution of OLC. The temperature dependence of conductivity of the homogeneous sample is mainly caused by a weak positive temperature coefficient effect.

## DIELECTRIC PROPERTIES OF A NOVEL HIGH ABSORBING ONION-LIKE-CARBON BASED POLYMER COMPOSITE

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**G. Valusis\***, **J. Banys\*\*\***, **A. Paddubskaya\*\***,  
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**V.L. Kuznetsov**, **S.I. Moseenkov**,  
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*Diamond Relat. Mater.*,  
19(1) (2010) pp. 91-99.

A novel lightweight onion-like carbon (OLC) based poly(methyl metacrylate) (PMMA) composite with high electromagnetic (EM) absorption capability determined earlier in microwave range is studied in

low frequencies from 20 Hz to 1 MHz by dielectric spectroscopy. The investigation of dielectric properties of PMMA films with incorporated OLC in small concentration (0.5-2 wt.%) have been carried out by varying the temperature between 240 and 520 K. The temperature behavior of the complex permittivity at a given frequency is found to be strongly dependent on the OLC concentration. The experimental data are in quantitative agreement with the results of modified effective medium calculations of OLC-based polymer film's permittivity. Both the experimental observations and theoretical simulation demonstrate that small additions of onion-like carbon particles to a polymer matrix can noticeably modify the composite response to low-frequency EM radiation as well as improve its thermal stability (increased glass transition and melting temperatures, correspondingly).

## DIELECTRIC PROPERTIES OF POLYSTYRENE/ONION-LIKE CARBON COMPOSITES IN FREQUENCY RANGE OF 0.5-500 kHz

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**O.V. Sedelnikova\***, **I.V. Yushina\***, **V.L. Kuznetsov**  
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*Compos. Sci. Technol.*,  
70(5) 2010) pp. 719-724.

Composites of polystyrene and onion-like carbon (OLC), produced from detonation nanodiamonds (NDs), have been fabricated by forge-rolling method. Optical microscopy and optical absorption spectroscopy were used for control of dispersion of OLC in polymer matrix after repeating rolling cycles. The impedance measurements of composites with different filler loading indicated the percolation threshold at similar to 20-25 wt.% OLC concentration. The permittivity of the polystyrene/OLC composites varies from 2 to 10 with changing the OLC loading from 5 to 35 wt%. Fitting the experimental data with model based on the Maxwell-Garnett approximation lead to conclusion that the shear deformation applied under the composite preparing disintegrates OLC agglomerates to the size similar to 40-50 nm equal to the medium size of OLC in dimethylformamide slurry.

## ELECTROCORROSION PROPERTIES OF MULTIWALL CARBON NANOTUBES

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A.N. Simonov, I.N. Mazov, K.V. Elumeeva,  
N.S. Moseva

*Phys. Status Solidi B*,  
247(11-12) (2010) pp. 2738-2742.

The electrochemical corrosion properties of a set of multiwalled carbon nanotubes (MWCNT) with different average diameters ( $d = 7-22$  nm) and conventional carbon black Vulcan XC-72 were studied potentiostatically in 2 M H<sub>2</sub>SO<sub>4</sub> at 80°C. Effects of grinding and high-temperature treatment (2800°C) in inert gas on the corrosion stability of MWCNT were also examined. Excitation of MWCNTs with large  $d$  in activator mill results in local disruption of the fine structure of the NTs and increase of the corrosion current densities. No rupture and changes of the corrosion behaviour occur for MWCNTs with lower  $d$  after grinding. High-temperature treatment of MWCNTs leads to removal of amorphous carbon and defects from the surface of the materials and results in drastic decrease of the reactivity of the surface of the materials towards electrocorrosion. The A<sub>BET</sub>-weighted corrosion current densities of the MWCNTs are significantly lower than that of conventional XC-72 material evidencing of their higher stability towards electrocorrosion.

## INFLUENCE OF NAFION® IONOMER ON CARBON CORROSION

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*J. Appl. Electrochem.*,  
40(11) (2010) pp. 1933-1939.

Corrosion of the three carbon black materials which differ in their textural, sub-structural, and morphological characteristics is studied in H<sub>2</sub>SO<sub>4</sub> electrolyte at 80°C at positive polarization. Impregnation of carbon blacks with Nafion® ionomer is found to strongly affect their susceptibility to corrosion, which varies non-monotonically as a function of the Nafion® loading. Low temperature nitrogen adsorption is applied to study the distribution of the ionomer molecules in the carbon matrix.

## MICROSTRUCTURE EFFECTS ON THE ELECTROCHEMICAL CORROSION OF CARBON MATERIALS AND CARBON-SUPPORTED Pt CATALYSTS

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V.I. Zaikovsky, E.R. Savinova\* (\*Universite' de  
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*Electrochim. Acta*,  
55(1) (2010) pp. 8453-8460.

The electrochemical corrosion behavior of a set of porous carbonaceous materials of interest as catalyst supports for polymer electrolyte membrane fuel cells was examined in 2 M H<sub>2</sub>SO<sub>4</sub> at 80°C at constant electrode potential of 1.2 V vs. RHE. Correlations have been observed between the specific rates of corrosion of carbon materials and carbon-supported Pt catalysts on the one hand and their substructural characteristics derived from X-ray diffraction analysis on the other hand. Carbon supports of the Sibunit family and catalytic filamentous carbons possess lower specific (i.e., surface area normalized) corrosion currents compared to conventional furnace black Vulcan XC-72 and better stabilize Pt nanoparticles.

## INFLUENCE OF THE MORPHOLOGY AND THE SURFACE CHEMISTRY OF CARBONS ON THEIR CATALYTIC PERFORMANCES IN THE CATALYTIC WET PEROXIDE OXIDATION OF ORGANIC CONTAMINANTS

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V.N. Parmon, M. Besson\*, C. Descorme\* (\*Institut  
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Lyon, Villeurbanne, France)

*Appl. Catal., A*,  
387(1-2) (2010) pp. 55-66.

The catalytic behavior of five different carbon materials (mesoporous graphite-like carbon Sibunit, catalytic filamentous carbon (CFC), nano-diamonds (ND) and onion-like carbon (OLC)) in the catalytic wet peroxide oxidation of model organic compounds with different sorption capacities and stability towards oxidation (phenol, ethanol and formic acid) has been examined, in the absence and in the presence of Fe<sup>3+</sup> ions. All the bare carbons showed extremely low catalytic activity in the oxidation of phenol, ethanol and formic acid in the absence of iron. The homogeneous Fe-catalyzed oxidation of all substrates was much faster. Furthermore, in the Fe-promoted catalytic oxidation over carbons, the presence of ND inhibited the oxidation of the model

substrates compared to the homogeneous Fe<sup>3+</sup>-catalyzed oxidation. On the contrary, the other carbon samples (OLC, CFC, Sibunit) accelerated the oxidation of ethanol and formic acid under the same reaction conditions. More complex effects were observed for OLC, CFC and Sibunit in the oxidation of phenol. A tentative mechanism is discussed.

#### **CARBON CATALYSTS FOR CATALYTIC WET AIR OXIDATION OF ORGANIC CONTAMINANTS**

**O.P. Taran, E.M. Polyanskaya, C. Descorme\*, O.L. Ogorodnikova, M. Besson\*** (*\*Institut de Recherches sur la Catalyse et l'Environnement de Lyon, Villeurbanne, France*)

*J. Siberian Federal Univ., Chem.,*  
3(3) (2010) pp. 234-244.

Catalytic behavior of pure carbon materials with different morphology, structure and surface chemistry (catalytic filamentous carbons, nano-diamonds, onion-like carbon, mesoporous graphite-like carbon Sibunit and oxidized samples of Sibunit) towards the catalytic wet air oxidation of phenol were examined. Perceptible but low catalytic activity was registered for all graphite-like carbons, while no activity was exhibited by nano-diamonds. The correlations between catalytic performance of oxidized Sibunit catalysts and methods of oxidative pretreatment and amount of different oxygen-containing species on the carbon surface were evaluated. The conclusion about the key role of transition metal impurities in carbon catalysts was made.

#### **RUTHENIUM CARBON-BASED CATALYSTS FOR CATALYTIC WET AIR OXIDATION OF PHENOL**

**O.P. Taran, E.M. Polyanskaya, O.L. Ogorodnikova, C. Descorme\*, M. Besson\*** (*\*Institut de Recherches sur la Catalyse et l'Environnement de Lyon, Villeurbanne, France*)

*J. Siberian Federal Univ., Chem.,*  
3(3) (2010) pp. 245-252.

The correlations between catalytic performance in the phenol wet air oxidation (WAO) of ruthenium catalysts (3 % Ru/C) based on oxidized samples of carbon material Sibunit-4 and methods of support oxidative pretreatment and amount of different oxygen-containing species on the carbon surface were evaluated. The optimal method of carbon support pretreatment providing the most stable and active Ru/Sibunit catalyst for catalytic wet air oxidation of phenols was chosen.

#### **SIBUNIT-BASED CATALYTIC MATERIALS FOR THE DEEP OXIDATION OF ORGANIC ECOTOXICANTS IN AQUEOUS SOLUTION: I. SURFACE PROPERTIES OF THE OXIDIZED SIBUNIT SAMPLES**

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*Catal. Ind.,*  
2(4) (2010) pp. 381-386.

This article presents the first systematic study of the effect of oxidation under various conditions on the surface composition and catalytic properties of a carbon material of the Sibunit family (which is promising for oxidative treatment of industrial wastewater) in deep oxidation of organic ecotoxics in aqueous solution. Modification of the surface properties and texture of Sibunit-4 by various oxidative treatment procedures using nitric acid, sodium hypochlorite, hydrogen peroxide, or oxygen as the oxidant is reported. The chemical state of the surface of oxidized Sibunit and its texture have been investigated by a combination of physical and chemical methods (XPS, acid-base titration with bases differing in strength, pH slurry measurements, determination of pH at the point of zero charge, and low-temperature N<sub>2</sub> adsorption). By using different oxidation procedures, it is possible to obtain oxidized Sibunit samples with variable concentrations and natures of oxygen-containing surface groups.

#### **FORMATION OF A SUPPORTED COBALT CATALYST FOR THE SYNTHESIS OF CARBON NANOFIBERS ON ALUMINUM OXIDE**

**G.A. Kovalenko, N.A. Rudina, L.V. Perminova, O.V. Skrypnik**

*Kinet. Catal.,*  
51(3) (2010) pp. 449-455.

Comparative studies of the effect of the physicochemical characteristics of a support (aluminum oxide) on the formation of a supported Co catalyst and its activity in the pyrolysis of alkanes (propane-butane) were performed. The effect of the crystalline modification of alumina on the yield of catalytic filamentous carbon (CFC) ( $g_{CFC}/g_{Co}$ ) was studied. The surface morphologies of Co-containing catalysts and synthesized carbon deposits were studied by scanning electron microscopy. It was found that carbon deposits with a well-defined nanofiber structure were synthesized by

the pyrolysis of a propane–butane mixture in the presence of hydrogen at 600°C on supported Co catalysts prepared by homogeneous precipitation on macroporous corundum ( $\alpha$ -Al<sub>2</sub>O<sub>3</sub>). The yield of CFC was no higher than 4 g<sub>CFC</sub>/g<sub>Co</sub>. On the Co catalyst prepared by homogeneous precipitation on mesoporous Al<sub>2</sub>O<sub>3</sub>, the intense carbonization of the initial support; the formation of cobalt aluminates; and, as a consequence, the deactivation of Co<sup>0</sup> as a catalyst of FC synthesis occurred. The dependence of the yield of CFC on the preheating temperature (from 200 to 800°C) of Co catalysts before pyrolysis was studied. It was found that, as the preheating temperature of supported Co/Al<sub>2</sub>O<sub>3</sub> catalysts was increased, the amount of synthesized carbon, including CFC, decreased because of Co<sup>0</sup> deactivation due to the interaction with the support and coke formation.

#### **CORUNDUM IMPREGNATION CONDITIONS FOR PREPARING SUPPORTED Ni CATALYSTS FOR THE SYNTHESIS OF A UNIFORM LAYER OF CARBON NANOFIBERS**

**G.A. Kovalenko, N.A. Rudina, L.V. Perminova, O.V. Skrypnik**

*Kinet. Catal.*,  
51(5) (2010) pp. 762-770.

Impregnation techniques for corundum ( $S_{\text{BET}} = 0.5 \text{ m}^2/\text{g}$ ) as a support for Ni catalysts for C<sub>3</sub>-C<sub>4</sub> alkane pyrolysis into catalytic filamentous carbon (CFC) are compared. The effects of the following factors on the uniformity of the active component (Ni) deposition on the inert support and on the CFC yield (g<sub>CFC</sub>/g<sub>Ni</sub>) are reported: (1) pH of the nickel nitrate solution, (2) presence of aluminum(III) nitrate in the solution, (3) addition of viscosifying agents (glycerol, glucose, sucrose) to the solution, (4) catalyst calcination conditions before pyrolysis, and (5) catalyst drying technique. The surface morphology of the Ni catalysts and of the carbon deposits resulting from the catalytic pyrolysis of C<sub>3</sub>-C<sub>4</sub> alkanes in the presence of hydrogen has been investigated by scanning electron microscopy. The optimum way of preparing the supported Ni catalysts is by carrying out the incipient wetness impregnation of corundum with a nickel nitrate solution (0.05-0.1 mol/l) containing glycerol (20-25 vol %), drying the product in a microwave oven, and burning away the glycerol before alkane pyrolysis.

#### **CARBON DIOXIDE ADSORPTION ON CARBON NANOMATERIALS**

**V.Yu. Gavrilov, R.S. Zakharov**

*Kinet. Catal.*,  
51(4) (2010) pp. 609-614.

The adsorption of CO<sub>2</sub> on a number of activated carbons, thermal carbon black, and oxide materials at 195 K was studied using static and dynamic techniques. The landing surface areas  $\omega(\text{CO}_2) \approx 0.19 \text{ nm}^2$  on thermal carbon black and the absolute values of sorption for  $P/P_0 < 0.4$  were determined. The density of adsorbed CO<sub>2</sub> in the micropore volume was estimated at  $\rho(\text{CO}_2) = 0.91 \text{ g}/\text{cm}^3$ . It was demonstrated that the previously found effect of a weakening of the sorption interaction of nitrogen molecules with thin-walled materials (which manifested itself in an analysis of sorption isotherms by a comparative method) was pronounced to a lesser degree for the sorption of CO<sub>2</sub>. At the same time, the presence of supermicropores in activated carbon samples resulted in overestimated values of surface areas. A dynamic method was proposed to measure the spectra of CO<sub>2</sub> desorption at 195–260 K using a SORBI-MS system for evaluating the binding energy of sorbate molecules with the surface.

#### **CONTROL OF THE ADSORPTION CAPACITY OF A CARBON SORBENT WITH RESPECT TO PROTEIN MOLECULES BY SURFACE MODIFICATION WITH AMINOCAPROIC ACID**

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*Russ. J. Appl. Chem.*,  
83(3) (2010) pp. 406-413.

The carbon sorbent surface was modified with carboxy and amino groups. As modifying agent served aminocaproic acid combining the hydrophilic terminal groups with the hydrophobic hydrocarbon chain, which affords a balance between the groups when contacting proteins. The conditions and parameters of aminocaproic acid polycondensation were elucidated that allows the polymeric modifier to be strongly fixed on the carbon surface.

**STRUCTURAL CHANGES IN A NICKEL-COPPER CATALYST DURING GROWTH OF NITROGEN-CONTAINING CARBON NANOFIBERS BY ETHYLENE/AMMONIA DECOMPOSITION**

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*Carbon*,  
48(10) (2010) pp. 2792-2801.

Structural changes in a 65Ni-25Cu-Al<sub>2</sub>O<sub>3</sub> catalyst during the decomposition of a C<sub>2</sub>H<sub>4</sub>/NH<sub>3</sub> mixture at different stages of nitrogen-containing carbon nanofiber (N-CNF) synthesis were studied by X-ray diffraction (XRD) analysis, X-ray diffraction using anomalous scattering effect and extended X-ray absorption fine structure (EXAFS) spectroscopy. The N-CNF growth over a catalyst particle was supposed to proceed via the formation of an oversaturated solid solution of carbon and nitrogen in a nickel-enriched alloy "NiCu<sub>x</sub>C<sub>y</sub>N<sub>z</sub>". The latter resulted in an increase of the alloy lattice parameter to abnormally large values  $\alpha = 3.616-3.706 \text{ \AA}$  without destruction of its cubic structure. The formation of the "NiCu<sub>x</sub>C<sub>y</sub>N<sub>z</sub>" phase and its presence in the system coincide with the optimum time for the synthesis of N-CNFs with the highest nitrogen concentration and maximum values of specific surface area and total pore volume.

**NANOFIBROUS CARBON WITH HERRINGBONE STRUCTURE AS AN EFFECTIVE CATALYST OF THE H<sub>2</sub>S SELECTIVE OXIDATION**

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*Carbon*,  
48(7) (2010) pp. 2004-2012.

Granular nanofibrous carbons (NFCs) with herringbone structure were synthesised by the decomposition of natural gas over Ni/Al<sub>2</sub>O<sub>3</sub> catalysts, and their performance in the selective oxidation of hydrogen sulphide was studied. Samples obtained over pure Ni catalysts are the strongest mechanically and easiest to produce. However, they show low selectivity for sulphur and are unstable during operation. Boiling in nitric acid followed by annealing led to improvements in catalytic stability and a significant increase in the selectivity for sulphur in the direct oxidation of hydrogen sulphide. The addition of large amounts of water vapour to the reaction mixture dramatically improved the selectivity and stability of the NFCs.

**CARBON XEROGELS: NANO- AND ADSORPTION TEXTURES, CHEMICAL NATURE OF THE SURFACE AND GAS CHROMATOGRAPHY PROPERTIES**

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*Microporous Mesoporous Mater.*,  
130(1-3) (2010) pp. 7-13.

Nano- and adsorption textures, gas chromatography features of carbon xerogels obtained by carbonization of phenol-formaldehyde resins have been studied. The properties of carbon xerogels were compared with those of carbon-containing and silica-alumina adsorbents. Depending on carbonization, they have properties typical of carbons or carbons combined with silica-alumina materials. The application of carbon xerogels in practical gas chromatography has been exemplified.

**TREATMENT OF NANODIAMONDS IN SUPERCRITICAL WATER**

**V.I. Anikeev, An. Yermakova**

In "*Environanotechnology*", Elsevier B.V., Eds. M. Fan, Ch.-P. Huang, A.E. Bland, Zh. Wang, R. Slimane, I. Wright, 2010, ch. 8, pp. 165-176 (310 pp.).

Authors of the present work carried out a study aimed at the treatment of the detonation carbon mix produced at the Federal Scientific and Production Center "ALTAI," Biysk, Russia, both in supercritical water (SCW) and with the addition of hydrogen peroxide. The goal of this work was to study the morphological and structural changes of carbon species and to choose the composition of supercritical fluid and the process conditions.

The study showed that the interaction of detonation carbon with SCW is accompanied by the formation of large amounts of gaseous products, mainly CO and CO<sub>2</sub>, which is evidence of a deep oxidation of the carbon phase. The transformation of nondiamond carbon phase is most pronounced when the detonation carbon is treated in SCW that contains oxygen formed at decomposition of hydrogen peroxide. The SCW treatment of detonation carbon black was shown to increase considerably the weight fraction of diamond phase. It was found that oxidation of the diamond nucleus does not occur when detonation carbon is treated in SCW.

## TREATMENT OF DETONATION CARBON IN SUPERCRITICAL WATER

V.I. Anikeev, V.I. Zaikovsky

*Russ. J. Appl. Chem.*,  
83(7) (2010) pp. 1202-1208.

Experimental data on the treatment of detonation carbon in supercritical water are given.

## THE SYNTHESIS AND STUDY OF SILICON-CONTAINING NANODISPERSED CARBON

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*Russ. J. Appl. Chem.*,  
83(4) (2010) pp. 588-592.

Samples of silicon dioxide-modified nanospherical carbon were synthesized by thermooxidative pyrolysis of highly aromatized liquid hydrocarbons with a varied content of organosilicon polymer (polyorganosiloxane). The physicochemical properties of the product synthesized were studied.

## In-situ BULK ELECTROPHORETIC SEPARATION OF SINGLE-WALLED CARBON NANOTUBES GROWN BY GAS-PHASE CATALYTIC HYDROCARBON DECOMPOSITION

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Edinburgh, Midlothian Scotland)

*Chem. Vap. Deposition*,  
16(7-9) (2010) pp. 225-230.

Electrophoresis is used to separate carbon nanotubes from other by-products during CVD growth from iron catalyst particles, using  $C_2H_2$  as the carbon feedstock. Carbon nanotubes are trapped by electric fields with higher efficiency than other carbon-containing products. The structure and yield of the carbon nanotubes depends critically on the gas parameters and applied electric field used in the novel reaction chamber. A higher yield of single-walled carbon nanotubes (SWNTs) can be obtained on electrodes in comparison to exhaust filters. The results indicate that the NTs produced by thermal CVD in the gas phase are negatively charged.

## Composite Sorbents

### NOVEL AMMONIA SORBENTS "POROUS MATRIX MODIFIED BY ACTIVE SALT" FOR ADSORPTIVE HEAT TRANSFORMATION: I. BARIUM CHLORIDE IN VARIOUS MATRICES

J.V. Veselovskaya, M.M. Tokarev, Yu.I. Aristov

*Appl. Therm. Eng.*,  
30(6-7) (2010) pp. 584-589.

Novel composite materials  $BaCl_2$ /vermiculite,  $BaCl_2/\gamma-Al_2O_3$  and  $BaCl_2$ /(carbon Sibunit) have been synthesised by impregnation of the three porous matrices with barium chloride and studied as ammonia sorbents. Isosters of ammonia sorption are measured at  $T = 15-80^\circ C$  and  $P = 0.7-9$  bar. It was found that a modification of the host matrices by the salt increases the ammonia uptake due to a chemical reaction between ammonia and barium chloride. The enthalpy and entropy of ammonia sorption by the salt confined to the mesopores of alumina and Sibunit are significantly smaller with regard to those for the bulk salt. This indicates that the salt confinement to the small pores allows tuning its sorption properties. In the large vermiculite pores the properties remain unchanged. Based on the experimental isosteric charts, the feasibility

of the new materials for adsorptive heat transformation has been analyzed. For composite  $BaCl_2$ /vermiculite the coefficient of performance (COP) for ice making and air conditioning cycles calculated from the ammonia sorption data can reach 0.6 which seems quite encouraging from a practical point of view.

### NOVEL AMMONIA SORBENTS "POROUS MATRIX MODIFIED BY ACTIVE SALT" FOR ADSORPTIVE HEAT TRANSFORMATION: II. CALCIUM CHLORIDE IN ACF FELT

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*Appl. Therm. Eng.*,  
30(8-9) (2010) pp. 845-849.

Novel composite sorbent "31 wt.%  $CaCl_2$ /ACF" was synthesised by dry impregnation of an ACF felt with a  $CaCl_2$  aqueous solution. Apparent isosters of ammonia sorption were measured at  $T=20-90^\circ C$  and  $P=0.1-9.0$  bar by an isosteric adsorption method. The method is based on the sorbent heating in a closed volume of an adsorber with registration of the ammonia equilibrium pressure  $P$  as a function of



temperature  $T$ . The uptake covered the range of 5.7-36.9 wt.%. The apparent enthalpy and entropy of the ammonia sorption as a function of ammonia uptake were obtained directly from the slope of isosteric lines  $\ln P(1/T)$ . Appropriate correction to obtain the true sorption enthalpy was done by taking into account the “dead” volume of the adsorber. The new adsorbent was found to be an advanced material for adsorptive chilling.

**NOVEL AMMONIA SORBENTS “POROUS MATRIX MODIFIED BY ACTIVE SALT” FOR ADSORPTIVE HEAT TRANSFORMATION: III. TESTING OF “ $\text{BaCl}_2$ /VERMICULITE” COMPOSITE IN A LAB-SCALE ADSORPTION CHILLER**

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*Appl. Therm. Eng.*,  
30(10) (2010) pp. 1188-1192.

A composite adsorbent composed of  $\text{BaCl}_2$  impregnated into expanded vermiculite has been synthesized and tested in a laboratory scale adsorption chiller. Previous work has established the promising theoretical performance of this adsorbent with ammonia as a refrigerant, in terms of equilibrium uptake, suitable equilibrium temperatures for use in air conditioning applications and good reaction dynamics. Analysis of the adsorption phase revealed a simple exponential approach to equilibrium uptake which was not previously observed in larger scale experiments.

It was demonstrated that this material can provide effective operation of the chiller using a low potential heat source (80–90°C) giving  $COP$  as high as  $0.54 \pm 0.01$  and  $SCP$  ranging from 300 to 680 W/kg. The specific cooling power depends strongly on the driving temperature difference and the cycle duration.

**NOVEL SORBENTS OF ETHANOL “SALT CONFINED TO POROUS MATRIX” FOR ADSORPTIVE COOLING**

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*Energy*,  
35(6) (2010) pp. 2703-2708.

In this paper a new family of sorbents, specifically designed for ethanol sorption, is presented. The composites were synthesized by a dry impregnation of matrices with an aqueous solution of various salts. The ethanol sorption capacity of the composites, under conditions typical for adsorptive air conditioning cycle, has been measured by using an express method based on the Polanyi principle of temperature

invariance. Results obtained show that the best novel composites have the ethanol sorption ability which is higher than that of known ethanol sorbents. The composite  $\text{LiBr}(30 \text{ wt.}\%)/\text{SiO}_2$  appears to show the highest sorption capacity and an uptake variation  $\Delta w = 0.56$  and  $0.40 \text{ g/g}$  for air conditioning and ice making cycles, respectively. They are much larger than those obtained for conventional adsorbents. The correspondent cooling coefficient of performance ( $COP$ ) was estimated to be 0.66 and 0.61, which is comparable with the  $COP$  of the best water sorbents.

**EFFECT OF RESIDUAL GAS ON WATER ADSORPTION DYNAMICS UNDER TYPICAL CONDITIONS OF AN ADSORPTION CHILLER**

**I.S. Glaznev, D.I. Ovoshchnikov, Yu.I. Aristov**

*Heat Transfer Eng.*,  
31(11) (2010) pp. 924-930.

In this article the effect of a non-adsorbable gas (air) on kinetics of water adsorption on loose grains was studied for three adsorbents promising for adsorption chilling: SWS-1L (silica KSK modified by calcium chloride), silica Fuji type RD, and FAM-Z02. The experimental conditions were fixed similar to real operating conditions during an isobaric adsorption stage of the basic cycle of an adsorption chiller (AC). Reduction of the adsorption rate was revealed even at a partial pressure of residual air as low as 0.06 mbar. Dependence of the characteristic adsorption time on air partial pressure was found to be linear for partial pressures greater than 0.4 mbar, with the slope depending on the adsorbent nature. Desorption stage was less affected by the residual air. Specific power released in an AC evaporator during the adsorption process was estimated as a function of the partial pressure of residual air, and recommendations how to improve cooling performance are made.

**A COMPACT LAYER OF ALUMINA MODIFIED BY  $\text{CaCl}_2$ : THE INFLUENCE OF COMPOSITION AND POROUS STRUCTURE ON WATER TRANSPORT**

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*Microporous Mesoporous Mater.*,  
131(1-3) (2010) pp. 358-365.

This paper reports a target-oriented synthesis of the compact layer of alumina and its modification by a calcium chloride. The layer preparation procedure with the use of binder has been developed which

makes it possible to widely vary the layer porous structure. The effect of the layer structure and composition on water vapor transport in it has been studied by NMR imaging. The effects of the size of the primary alumina grains, the amounts of the binder and the guest salt on the porous structure of the layer and the mechanism of vapor transport in the layer have been investigated. Variation of these parameters has been shown to alter the relative contributions of the diffusional resistances in the intergrain macropores and in the intragrain mesopores and to permit an intentional transition between the different diffusional regimes of water sorption. If the sorption rate is limited by the diffusion of water in the intergrain voids, an adsorption front is formed in the sample and is propagating into the layer. The penetration depth depends on time as  $t_{0.5}$ . If the water sorption in the primary grains is the slowest process, the water uptake uniformly increases over the entire layer.

#### THE EFFECT OF CYCLE BOUNDARY CONDITIONS AND ADSORBENT GRAIN SIZE ON THE WATER SORPTION DYNAMICS IN ADSORPTION CHILLERS

I.S. Glaznev, Yu.I. Aristov

*Int. J. Heat Mass Transfer*,  
53(9-10) (2010) pp. 1893-1898.

The aim of this work was an experimental study of the temporal evolution of isobaric adsorption uptake (release) for simplest configuration of an adsorbent-heat exchanger unit, namely, a monolayer of loose adsorbent grains located on a metal plate. The study was performed by a large temperature jump method at four various boundary conditions of an adsorptive heat transformation cycle typical for air-conditioning application driven by low temperature heat:  $T_c = 5$  and  $10^\circ\text{C}$ ,  $T_c = 30$  and  $35^\circ\text{C}$  and  $T_{HS} = 80^\circ\text{C}$ . The size of the Fuji silica grains was varied from 0.2 to 1.8 mm to investigate its effect on water sorption dynamics. For each boundary set and grain size the experimental kinetic curve could be described by an exponential function up to 80–90% of the equilibrium conversion. Desorption runs are found to be faster than appropriate adsorption runs by a factor of 2.2–3.5, hence, for optimal durations of the isobaric ad- and desorption phases of the chilling cycle should be selected accordingly. The size  $R$  of the adsorbent grains was found to be a powerful tool to manage the dynamics of isobaric water ad-/desorption. For large grains the characteristic time was strongly dependent on the grain size and

proportional to  $R^2$ . Much less important appeared to be an impact of the boundary conditions which variation just weakly affected the dimensionless kinetic curves for the four tested cycles. The maximal specific cooling/heating power was proportional to the maximal temperature difference  $\Delta T$  and the contact area  $S$  between the layer and the metal plate, and can exceed 10 kW/kg. The heat transfer coefficient  $\alpha$  estimated from this power was as large as 100–250 W/(m<sup>2</sup> K) that much exceeds the value commonly used to describe the cycle dynamics.

#### SYNTHESIS AND WATER SORPTION PROPERTIES OF A NEW COMPOSITE “CaCl<sub>2</sub> CONFINED INTO SBA-15 PORES”

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*Microporous Mesoporous Mater.*,  
129(1-2) (2010) pp. 243-250.

The composite CaCl<sub>2</sub>@SBA-15 with the mass ratio Ca/Si=0.58 has been synthesized via impregnation of nano-structured mesoporous silica SBA-15 with saturated aqueous solution of calcium chloride. The material was studied by methods of X-ray diffraction, differential scanning calorimetry, infrared spectroscopy, nitrogen adsorption and thermal analysis. It was shown that calcium chloride is located basically in the pores of silica. The isotherms and kinetics curves of water ad-/desorption were measured at temperature 50°C and vapor pressure range from 0 to 50 mbar. A small hysteresis was observed for the formation di- and tetra-hydrates of CaCl<sub>2</sub> in the pores. The equilibrium pressure over the dispersed CaCl<sub>2</sub> dihydrate was found to be 2.5–4 times lower relative to the bulk one, allowing a significant improvement of the salt desiccative properties. The possible reasons of this pressure decrease might be the higher mobility of water in the dispersed dihydrate and/or the increase in the surface energy of the salt (hydrate) confined to the SBA pores. The latter effect is a reason of the melting point depression for dispersed hydrates CaCl<sub>2</sub>·nH<sub>2</sub>O with  $n = 2, 4$  and  $6$  that was revealed by DSC tests. The effective water diffusivity in SBA pores has been determined. The Knudsen diffusion in the SBA mesopores was found to be a rate-limiting stage of water sorption on CaCl<sub>2</sub>@SBA-15. The equilibrium sorption curves for the new composite were compared with other composite sorbents “CaCl<sub>2</sub> in porous matrix”. Several possible applications of the new sorbent, among which are the gas

drying, heat storage and adsorptive heat pumps, were preliminary evaluated.

#### **EFFECT OF RESIDUAL GAS ON THE DYNAMICS OF WATER ADSORPTION UNDER ISOBARIC STAGES OF ADSORPTION HEAT PUMPS: MATHEMATICAL MODELLING**

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*Int. J. Heat Mass Transfer*,  
53(7-8) (2010) pp. 1283-1289.

A mathematical model of the coupled heat and mass transfer in an adsorbent layer was developed to study the effect of a non-adsorbable gas (air, hydrogen) on kinetics of water adsorption on loose grains of the composite adsorbent SWS-1L (silica modified by calcium chloride). The adsorbent monolayer was placed on the surface of an isothermal metal plate at  $T = 60^\circ\text{C}$  and equilibrated with the mixture of water vapor at constant  $P = 10.3$  mbar in the presence of the non-adsorbable gas at a variable partial pressure  $P_A = 0.06$ -14.3 mbar. After that the metal plate is subjected to a temperature drop down to  $35^\circ\text{C}$  that initiates water adsorption. It is shown that

the adsorption of water causes effective gas sweeping to the surface where it was accumulated as a gas-rich layer. This results in dramatic slowing down of the adsorption and heat transfer processes.

#### **MESOPOROUS “CORE-SHELL” ADSORBENTS AND CATALYSTS WITH CONTROLLABLE MORPHOLOGY**

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*Appl. Surf. Sci.*,  
256(17) (2010) pp. 5513-5519.

A simple geometrical model is applied to predict the thickness of mesoporous shells over monodisperse spherical particles. As an example, mesoporous Ti-silicate nearly monodisperse particles with the “core-shell” structure, synthesized via the one-pot procedure are considered. The unique features of the materials are orientation of mesopores perpendicularly to the surface of non-porous cores and uniformity of mesoporous shells structure and thickness. This allows considering these materials as interesting catalysts for partial oxidation of bulk organic molecules with hydrogen peroxide.

## **Polyoxometalate Catalysis**

#### **POLYOXOMETALATE-BASED HETEROGENEOUS CATALYSTS FOR LIQUID PHASE SELECTIVE OXIDATIONS: COMPARISON OF DIFFERENT STRATEGIES**

**O.A. Kholdeeva**, **N.V. Maksimchuk**, **G.M. Maksimov**

*Catal. Today*,  
157(1-4) (2010) pp. 107-113.

Recent achievements in the preparation of polyoxometalate-based heterogeneous catalysts using different immobilization techniques, such as embedding into a silica matrix by sol-gel method, irreversible adsorption on active carbon, electrostatic attachment to  $\text{NH}_2$ -modified mesoporous silica, and incorporation within nanocages of the metal-organic framework MIL-101 are surveyed. The catalyst characterization is reported, and their catalytic performances in liquid phase selective oxidations are compared, with special attention being paid to the questions of catalyst stability to leaching, nature of catalysis and catalytic properties after recycling. The scope and limitations of the POM-based heterogeneous catalysts are discussed.

#### **MECHANISTIC INSIGHTS INTO THE ALKENE EPOXIDATION WITH $\text{H}_2\text{O}_2$ BY Ti- AND OTHER TM-CONTAINING POLYOXOMETALATES: ROLE OF THE METAL NATURE AND COORDINATION ENVIRONMENT**

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*J. Am. Chem. Soc.*,  
132 (2010) pp. 7488-7497.

The oxidation of alkenes by  $\text{H}_2\text{O}_2$  catalyzed by Ti(IV)-containing polyoxometalates (POMs, as models of Ti-single site catalysts) has been investigated at DFT level and has been compared with other early transition metal-substituted polyoxometalates. The authors have studied in detail the reaction mechanism of the  $\text{C}_2\text{H}_4$  epoxidation with  $\text{H}_2\text{O}_2$  mediated by two different POMs, the Ti-monosubstituted Keggin-type POM  $[\text{PTi}(\text{OH})\text{W}_{11}\text{O}_{39}]^{4-}$  (**1A**) and the Ti-disubstituted sandwich-type POM  $[\text{Ti}_2(\text{OH})_2\text{As}_2\text{W}_{19}\text{O}_{67}(\text{H}_2\text{O})]^{8-}$

(1B). These species exhibit well-defined 6- and 5-coordinated titanium environments. For both species, the reaction proceeds through a two-steps mechanism: (i) the Ti-OH groups activate H<sub>2</sub>O<sub>2</sub> with a moderate energy barrier yielding either Ti-hydroperoxo (Ti-OOH) or Ti-peroxo (Ti-OO) intermediate, and (ii) the less stable but more reactive Ti-hydroperoxo species transfers oxygen to alkene to form epoxide, being this latter step the rate-determining step. The higher activity of the sandwich anion 1B was attributed to the absence of dimer formation, and its higher selectivity to the larger energy cost of homolytic O-O bond breaking in hydroperoxo intermediate. Several requisites to improve the efficiency of Ti-containing catalysts have been proposed, including flexible and 5-fold (or lower) coordinated Ti environments, as well as reagent accessible Ti sites. Calculations on other TM-containing Keggin-type POMs [PTM(OH)W<sub>11</sub>O<sub>39</sub>]<sup>4-</sup> (TM = Zr(IV), V(V), Nb(V), Mo(VI), W(VI) and Re(VII)) showed that when the authors move from the left to the right in the periodic table the formation of the epoxide via peroxo intermediate becomes competitive because of the higher mixing between the orbitals of the TM and the O-O unit.

#### HEXA-ZIRCONIUM/HAFNIUM CONTAINING TUNGSTOARSENATES(III) AND THEIR OXIDATION CATALYSIS PROPERTIES

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16 (2010) pp. 11797-11800.

Two novel Zr<sub>6</sub>- and Hf<sub>6</sub>-containing tungstoarsenates(III) [M<sub>6</sub>O<sub>4</sub>(OH)<sub>4</sub>(H<sub>2</sub>O)<sub>2</sub>(CH<sub>3</sub>COO)<sub>5</sub>(AsW<sub>9</sub>O<sub>33</sub>)<sub>2</sub>]<sup>11-</sup> (M = Zr, Hf) have been synthesized and structurally characterized in solution and in the solid state. Both polyanions contain an unprecedented octahedral Zr<sub>6</sub>/Hf<sub>6</sub> assembly which is stabilized by two (*B-α-AsW<sub>9</sub>O<sub>33</sub>*) groups and five terminal acetate ligands. The TBA-salts of both compounds revealed high catalytic activity and selectivity in the liquid-phase oxidation of organic compounds with aqueous H<sub>2</sub>O<sub>2</sub>. A heterolytic oxidation mechanism is manifested by the high yields of epoxide and diol in the oxidation of cyclohexene.

#### EPOXIDATION OF ALKENES WITH H<sub>2</sub>O<sub>2</sub> CATALYZED BY DITITANIUM-CONTAINING 19-TUNGSTODIARSENATE(III): EXPERIMENTAL AND THEORETICAL STUDIES

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*Eur. J. Inorg. Chem.*,  
2010(33) (2010) pp. 5312–5317.

Epoxidation of a range of alkenes with aqueous H<sub>2</sub>O<sub>2</sub> easily proceeds in the presence of the dititanium-containing 19-tungstodiarсенate(III) [Ti<sub>2</sub>(OH)<sub>2</sub>As<sub>2</sub>W<sub>19</sub>O<sub>67</sub>(H<sub>2</sub>O)]<sup>8-</sup> (**1**), which contains five-coordinate Ti atoms. The results of product and kinetics studies support a mechanism that involves a reversible interaction between H<sub>2</sub>O<sub>2</sub> and the Ti-OH group of **1** to produce a titanium hydroperoxo complex followed by electrophilic oxygen atom transfer from the hydroperoxo ligand to the alkene substrate in the rate-limiting step. The effect of the alkene substrate nature has been investigated at the DFT level. A clear correlation between the energy of π<sub>C=C</sub> orbitals in the alkene and the turnover frequency values has been found, thereby indicating that the higher nucleophilicity of the alkene, the higher the reactivity. ONIOM (ONIOM = our own *n*-layered integrated molecular orbital and molecular mechanics) calculations have been employed to evaluate the role of steric effects of alkene substituents. The calculations show that the steric bulk can play a secondary role and tunes the activity in specific cases such as *trans*-stilbene.

#### CATALYTIC PROPERTIES OF THE POLYOXOMETALATE [Ti<sub>2</sub>(OH)<sub>2</sub>As<sub>2</sub>W<sub>19</sub>O<sub>67</sub>(H<sub>2</sub>O)]<sup>8-</sup> IN SELECTIVE OXIDATIONS WITH HYDROGEN PEROXIDE

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*Kinet. Catal.*,  
51(6) (2010) pp. 816-822.

The catalytic properties of the sandwich polyoxometalate [Ti<sub>2</sub>(OH)<sub>2</sub>As<sub>2</sub>W<sub>19</sub>O<sub>67</sub>(H<sub>2</sub>O)]<sup>8-</sup>, which contains two (*B-α-As<sup>III</sup>W<sub>9</sub>O<sub>33</sub>*) fragments linked together by a “belt” consisting of one octahedral WO(H<sub>2</sub>O)<sup>4+</sup> and two square-pyramidal Ti(OH)<sup>3+</sup> groups, have been investigated in the selective liquid-

phase oxidation of organic compounds by aqueous hydrogen peroxide. The polyoxometalate shows high catalytic activity and selectivity in the oxidation of alkenes, alcohols, diols, and thioethers. The composition of the reaction products indicates that hydrogen peroxide is activated via a heterolytic mechanism.

#### **BI-COMPONENT REDOX SYSTEMS OBTAINED FROM IRIDIUM OR RHODIUM CHLORIDES AND MOLYBDOPHOSPHORIC HETEROPOLY ACID**

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*React. Kinet. Mech. Catal.*,  
101(1) (2010) pp. 1-12.

An XP, IR and UV–VIS spectroscopy study was carried out to characterize the compositions of molybdophosphate heteropoly anions with Ir(III) or Rh(III) hydroxo–chloro complexes, such as  $\text{Ir}_{1.1}\text{Cl}_{2.4}\text{O}_x\text{H}_y \cdot (\text{PMo}_{12}\text{O}_{40}) \cdot 16.0 \text{ H}_2\text{O}$ , where  $x$  is the number of oxygen atoms in the coordination sphere of iridium, and also mixed-valent species containing partially reduced and dehydrated heteropoly acid of suggested composition  $(\text{Ir}^0, \text{Ir}^{\text{III}})_n\text{Cl}_m\text{O}_x\text{H}_y \cdot (\text{PMo}_{12-p}^{\text{VI}}\text{Mo}_p^{\text{V}}\text{O}_{40-p/2})$  (on the average,  $n \approx 1$ ,  $m < 1$ ,  $p \geq 4$ ). The mixed-valent Ir containing species showed stable activity in the gas-phase oxidation of benzene by  $\text{O}_2$ – $\text{H}_2$  mixture.

#### **HYBRID POLYOXOTUNGSTATE/MIL-101 MATERIALS: SYNTHESIS, CHARACTERIZATION, AND CATALYSIS OF $\text{H}_2\text{O}_2$ -BASED ALKENE EPOXIDATION**

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*Inorg. Chem.*,  
49(6) (2010) pp. 2920-2930.

Polyoxotungstates  $[\text{PW}_4\text{O}_{24}]^{3-}$  ( $\text{PW}_4$ ) and  $[\text{PW}_{12}\text{O}_{40}]^{3-}$  ( $\text{PW}_{12}$ ) have been inserted into nanocages of the metal organic framework MIL-101. The hybrid materials  $\text{PW}_x/\text{MIL-101}$  ( $x = 4$  or  $12$ ) containing 5–14 wt % of polyoxotungstate have been obtained and characterized by elemental analysis,  $\text{N}_2$  adsorption, FT-IR, Raman, and  $^{31}\text{P}$  NMR MAS spectroscopic techniques. Their catalytic performance was assessed in the selective oxidation of alkenes with aqueous hydrogen peroxide under mild reaction conditions ( $[\text{H}_2\text{O}_2] = 0.1\text{--}0.2 \text{ M}$ ,  $50^\circ\text{C}$ , MeCN).

$\text{PW}_x/\text{MIL-101}$  enclosing 5 wt % of polyoxotungstate demonstrated fairly good catalytic activities in the epoxidation of various alkenes (3-carene, limonene,  $\alpha$ -pinene, cyclohexene, cyclooctene, 1-octene), the turnover frequencies (TOF) and alkene conversions were close to the corresponding parameters achieved with homogeneous  $\text{PW}_x$ . For the oxidation of substrates with aromatic groups (styrene, *cis*- and *trans*-stilbenes), a higher level of olefin conversion was attained using  $\text{PW}_{12}/\text{MIL-101}$ . Moreover, confinement of  $\text{PW}_{12}$  within MIL-101 nanocages allowed to reach higher epoxide selectivities at higher alkene conversions. The hybrid  $\text{PW}_x/\text{MIL-101}$  materials were stable to leaching, behaved as true heterogeneous catalysts, were easily recovered by filtration, and reused several times with the maintenance of the catalytic performance.

#### **AMINE-MODIFIED SILICA $\text{NH}_2\text{-(CH}_2\text{)}_x\text{-SiO}_2$ ( $x=0, 2, 3$ ) AS SUPPORT FOR COBALT-SUBSTITUTED POLYOXOMETALATE $\text{TBA}_4\text{HPW}_{11}\text{CoO}_{39}$ : EFFECT OF THE NATURE OF THE SUPPORT ON THE OXIDATION ACTIVITY**

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*J. Mol. Catal. A: Chem.*,  
319(1-2) (2010) pp. 119-125.

Acidic tetra-*n*-butylammonium (TBA) salts of Keggin type transition-metal-monosubstituted polyoxometalate  $\text{TBA}_4\text{HPW}_{11}\text{CoO}_{39}$  (Co-POM) have been chemically anchored to the amino-modified silica  $\text{NH}_2\text{-(CH}_2\text{)}_x\text{-SiO}_2$ ,  $x=0, 2, 3$  containing 40-710  $\mu\text{mol}$   $\text{NH}_2$ -groups per 1 g of support. The interaction of Co-POM with  $\text{NH}_2\text{-(CH}_2\text{)}_x\text{-SiO}_2$  was studied by DR-UV-vis spectroscopy. Spectroscopic data revealed that the type of interaction between Co-POM and support depends on the amount of anchored functional  $\text{NH}_2$ -groups. It was found that Co-POM was attached to  $\text{NH}_2\text{-(CH}_2\text{)}_x\text{-SiO}_2$  support both by the electrostatic  $\text{NH}_3^+\cdots\text{Co-POM}^-$  and dative ( $\text{Si-OH}\cdots\text{Co-POM}$ ) bonding. Both the amount of anchored  $\text{NH}_2$ -groups and the distance between  $\text{NH}_2$ -group and surface of silica affect the nature of the interaction between Co-POM and surface functional groups. The increase of the amount of anchored  $\text{NH}_2\text{-(CH}_2\text{)}_3$ -groups leads to decrease in the contribution of dative bonding  $\text{Si-OH}\cdots\text{Co-POM}$  and an increase in the contribution of electrostatic bonding  $\text{NH}_3^+\cdots\text{Co-POM}^-$ . The activity of the immobilized

Co-POM was tested in benzyl alcohol oxidation with oxygen in CH<sub>3</sub>CN medium. It was found that catalytic activity and stability of Co-POM depend on the nature of interaction between Co-POM and surface groups of support that is adjustable both by the amount of anchored functional NH<sub>2</sub>-groups on silica support and the distance between NH<sub>2</sub>-group and surface of silica.

#### **PRECIPITATION OF PHOSPHORUS AS NH<sub>4</sub>[MgPO<sub>4</sub>] · xH<sub>2</sub>O IN THE PRESENCE OF CITRATES, *d,l*-TARTARIC ACID, AND THEIR COMPLEXES WITH VANADIUM AND MOLYBDENUM**

**V.F. Odyakov, E.G. Zhizhina**

*Russ. J. Inorg. Chem.*,  
55(3) (2010) pp. 472-478.

For determination of phosphorus in Mo-V-phosphoric heteropoly acid solutions, it must be separated from considerable amounts of vanadium and molybdenum by precipitation as NH<sub>4</sub>[MgPO<sub>4</sub>] · xH<sub>2</sub>O. It is shown that the known method of its precipitation in citrate medium is too slow because of low reactivity of Mg(II)-citrate complexes. The authors propose to precipitate NH<sub>4</sub>[MgPO<sub>4</sub>] · xH<sub>2</sub>O in the presence of *d,l*-tartaric acid and next to purify the prepared precipitate. This method permits to shorten preparation of the pure precipitate in several times.

#### **TITRIMETRIC DETERMINATION OF PHOSPHORUS IN AQUEOUS SOLUTIONS OF Mo-V-PHOSPHORIC HETEROPOLY ACIDS AND CATALYSTS BASED ON THEM**

**V.F. Odyakov, E.G. Zhizhina**

*J. Siberian Federal Univ., Chem.*,  
3(1) (2010) pp. 20-26.

The analyzed aqueous solution of Mo-V-phosphoric heteropoly acid is reduced by *d,l*-tartaric acid (*d,l*-H<sub>2</sub>Tart) at room temperature. *d,l*-H<sub>2</sub>Tart reduces only V(V) to V(IV). The obtained solution is neutralized by ammonia, then the Mg(NO<sub>3</sub>)<sub>2</sub> solution is added to it giving the NH<sub>4</sub>[MgPO<sub>4</sub>]·xH<sub>2</sub>O precipitate. This precipitate is washed by the triple buffer NH<sub>4</sub>NO<sub>3</sub> + NH<sub>3</sub> + (NH<sub>4</sub>)<sub>3</sub>HCit solution containing citrate, then by the buffer NH<sub>4</sub>NO<sub>3</sub> + NH<sub>3</sub> solution without citrate. Next the precipitate is dissolved in 1.0 M HNO<sub>3</sub>. The obtained solution is titrated by 0.100 M Pb(II) nitrate solution in the acetate buffer in the presence of Cl<sup>-</sup> ions using the Pb-selective electrode. In the course of the titration, the Pb<sub>5</sub>Cl(PO<sub>4</sub>)<sub>3</sub> precipitate is formed.

#### **PHYSICO-CHEMICAL AND CORROSIVE PROPERTIES OF OXIDATION CATALYSTS BASED ON SOLUTIONS OF Mo-V-PHOSPHORIC HETEROPOLYACIDS**

**E.G. Zhizhina, V.F. Odyakov**

*Catal. Ind.*,  
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Modified aqueous solutions of Mo-V-P heteropoly acids (HPA) are used as high-performance catalysts in two-stage processes of oxidation of various classes of organic compounds with O<sub>2</sub>. In the 1st stage, HPA oxidizes the substrate. In the 2nd stage, the catalyst is regenerated with dioxygen of air, closing the catalytic cycle. In such two-stage redox processes the HPA solutions are reversible oxidants, and their physicochemical properties are continuously varied. The modified HPA solutions are attractive because of their thermal stability and increased rate of regeneration. However, there is no information about their physicochemical and corrosive properties as yet. Doubtlessly, it hardly retards commercial development of these processes. It has been shown that pH, density (ρ), and viscosity (η) of this solution increase in the 1st stage and reach their maxima, but its redox potential (E) comes up to its minimum. In the 2nd stage, the E value increases, but the pH, ρ, and η values decrease and reach their initial values. Thus, in the processes involving alternate reduction and oxidation of the catalyst based on the HPA solution, all alterations of its physicochemical properties are completely reversible.

#### **STRUCTURE AND PROPERTIES OF TUNGSTEN PEROXOPOLYOXO COMPLEXES – PROMISING CATALYSTS FOR ORGANICS OXIDATION: I. STRUCTURE OF PEROXOCOMPLEXES STUDIED DURING THE STEPWISE SYNTHESIS OF TETRA(DIPEROXOTUNGSTEN)PHOSPHATE-TETRA-*n*-BUTYL AMMONIUM**

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*J. Mol. Catal. A: Chemical*,  
332(1-2) (2010) pp. 122-127.

Catalysis via metal complexes has been studied using EXAFS and Raman spectroscopy. Main polyoxo and peroxopolyoxocomplexes of tungsten, resulting from the interaction of phosphorus tungsten heteropolyacid with hydrogen peroxide in water solutions, were investigated with regard to their form and structure. Interaction was shown to reduce the

nuclearity of forming complexes with respect to their precursor –  $\text{H}_3\text{PW}_{12}\text{O}_{40}$ . The symmetry of oxygen environment of tungsten was found to change, distances W–W remaining the same. After complex  $[\text{Bu}^n_4\text{N}]_3\{\text{PO}_4[\text{WO}(\text{O}_2)_2]_4\}$  is isolated, binuclear anion  $[\text{W}_2\text{O}_3(\text{O}_2)_4(\text{H}_2\text{O})_2]^{2-}$  stays in the solution. Raman scattering, EXAFS and IR spectroscopy show that isolated peroxocomplex in its crystal state has four

nuclei, and most probably retains this structure being dissolved in acetonitrile. Synthesized catalytic complex  $[\text{Bu}^n_4\text{N}]_3\{\text{PO}_4[\text{WO}(\text{O}_2)_2]_4\}$  was tested in oxidation of cyclic alkenes and alcohols with hydrogen peroxide. Complex  $[\text{Bu}^n_4\text{N}]_3\{\text{PO}_4[\text{WO}(\text{O}_2)_2]_4\}$  was proved to provide higher yields of mono- and dicarbonic acids in comparison to *in situ* synthesized catalyst.

## Synthesis, Properties and Application of Nanocomposite/Nanostructured Materials

### PEROVSKITES AND THEIR NANOCOMPOSITES WITH FLUORITE-LIKE OXIDES AS MATERIALS FOR SOLID OXIDE FUEL CELLS CATHODES AND OXYGEN-CONDUCTING MEMBRANES: MOBILITY AND REACTIVITY OF THE SURFACE/BULK OXYGEN AS A KEY FACTOR OF THEIR PERFORMANCE

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In “*Perovskites: Structure, Properties and Uses*”,  
Nova Science Publishers, Ed. M. Borowski, 2010,  
pp. 67-178 (571 pp.).

This paper presents results of research aimed at elucidating factors controlling mobility and reactivity of the surface and bulk oxygen of complex perovskite-like  $\text{A}_n\text{B}_m\text{O}_z$  oxides (manganites, ferrites, cobaltites, nickelates of La/Sr) and their nanocomposites with fluorite-like oxides (doped ceria or zirconia) as related to their performance in the intermediate temperature solid oxide fuel cells (IT SOFC) cathodes and oxygen separation membranes. Real/defect structure and surface properties of these materials as a function of both composition, preparation route and sintering temperature were studied by combination of TEM with EDX, XRD, EXAFS, UV-Vis and XPS methods. Lattice oxygen mobility, strength of oxygen species bonding with the surface sites and their reactivity for

both dispersed and dense materials were estimated by oxygen isotope exchange, temperature-programmed desorption and reduction by  $\text{H}_2$  and  $\text{CH}_4$ . Coefficients of oxygen diffusion in dense materials were estimated by analysis of dynamics of the oxygen loss monitored by measurements of the weight loss or conductivity relaxations. For analysis of atomic-scale factors controlling oxygen bonding strength and mobility, quantum-chemical approaches including the Density Functional Theory (DFT) in spin-polarized SLDA approximation and semiempirical Interacting Bonds Method were applied. For simple perovskites, along with well-known effects of anion vacancies/interstitial oxygen atoms and high-charge transition metal cations generated due to the oxygen nonstoichiometry, pronounced effects of the surface faces termination, ordering of oxygen vacancies and appearance of extended defects were found to affect the oxygen mobility and reactivity as well. From the microscopic point of view, decreasing the Me-O bonding strength and increasing the distance between A and B cations due to the lattice parameter increase helps to decrease the activation barriers for the oxygen diffusion in the perovskite-like lattice. For perovskite-fluorite nanocomposites, interfaces were shown to be paths for fast oxygen diffusion due to redistribution of cations between contacting phases. The most promising combinations of perovskites and perovskite-fluorite nanocomposites possessing a high lattice oxygen mobility and conductivity were used for synthesis of functionally graded cathodes of IT SOFC with thin layers of doped zirconia electrolytes as well as for manufacturing asymmetric Ni-Al compressed foam supported membranes for oxygen separation and syngas generation. High and stable performance of these materials promising for the practical application was demonstrated.

**DOPED LANTHANUM SILICATES WITH THE APATITE STRUCTURE AS OXIDE-ION CONDUCTING ELECTROLYTES: SYNTHESIS, CHARACTERIZATION AND APPLICATION FOR DESIGN OF INTERMEDIATE TEMPERATURE SOLID OXIDE FUEL CELL**

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In "*Lanthanum: Compounds, Production and Applications*", Nova Science Publishers, Ed. R.J. Moore, 2010, pp. 1-126.

The research is aimed at development of efficient synthesis procedures and characterization of oxide-ion conducting electrolytes based on doped lanthanum silicate with the apatite structure promising for design of solid oxide fuel cells operating at intermediate temperatures. Phase-pure nanocrystalline powders of doped lanthanum silicate  $\text{La}_{10-y}(\text{Sr, Mg})_y\text{Si}_{6-x}(\text{Fe, Al})_x\text{O}_{26+z}$  were successfully prepared via the mechanical activation of a mixture of solid reagents in high-power planetary ball mills followed by annealing at 700-1000°C provided reagents nature and activation parameters are properly optimized. Real/defect structure and surface properties of these materials as a function of both composition, preparation route and sintering temperature were studied by combination of TEM with EDX, XRD, EXAFS, Mossbauer spectroscopy (for Fe-doped samples), Si and Al MAS NMR (for Al-doped samples), UV-Vis, XPS and SIMS methods. Redistribution of  $\text{Me}^{3+}$  dopants between tetrahedral and octahedral positions (including those in the perovskite-like microinclusions) and condensation of  $\text{SiO}_4$  tetrahedra was revealed and analyzed. Effects of samples chemical composition and real structure on their transport properties characterized by the oxygen isotope heteroexchange for powders and impedance spectroscopy for dense materials were analyzed. For best compositions, intermediate temperature conductivity is close to that of Gd-doped ceria. Isotope exchange data support a model in which the oxygen mobility is determined by the non-linear

cooperative movement of oxide ions between interstitial and tetrahedral positions.

Approaches to design of cathode and anode materials for solid oxide fuel cells with the apatite-type electrolyte compatible by thermal expansion and chemical tolerance were elaborated. For cathodes, nanocomposites comprised of complex Ni-containing perovskites and Fe-doped La silicate were found to be promising materials due to chemical compatibility and non-additive increase of both reactivity in  $\text{O}_2$  activation and oxygen mobility due to positive role of interfaces and moderate redistribution of elements between domains of different phases. For anode materials operating in the mode of internal steam reforming of methane, the highest activity and coking stability were demonstrated by nanocomposites comprised of Ni, Sr-doped apatite and Sr-doped La titanate.

**MECHANOCHEMICAL SYNTHESIS OF Fe-DOPED APATITE-TYPE LANTHANUM SILICATES**

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*Eur. J. Inorg. Chem.*,  
4 (2010) pp. 589-601.

Apatite-type lanthanum silicates (ATLS) doped by Fe were prepared by using mechanical activation, and the effect of the nature of the dopant raw materials on the formation of ATLS was considered. Genesis of the formation of apatite was studied by XRD, IR, HRTEM, and Mössbauer and UV/Vis electron spectroscopy. The possibility of a partial substitution of Si in the apatite with a dopant and the mechanism of formation of ATLS in the course of mechanical activation depend on the dopant parent compound. The structural compatibility of  $\text{La}_2\text{O}_3$ ,  $\text{La}(\text{OH})_3$ , and apatite was shown to favor the rapid formation of apatite-type silicate by a cluster-topotactic mechanism during mechanical activation, and the formation of an amorphous ferrosilicate intermediate in the case of the  $\text{La}(\text{NO}_3)_3/\text{SiO}_2$  precursor favors dopant incorporation into the apatite. However, dopant incorporation into the apatite structure was shown to be partially or fully hampered through milling when  $\text{FeO}(\text{OH})$  and  $\alpha\text{-Fe}_2\text{O}_3$  are used as reagents. Using crystalline hydrates as dopant precursors was shown to result in the formation of apatite through a dissociative mechanism.



## TRANSFORMATION OF CH<sub>4</sub> AND LIQUID FUELS INTO SYNGAS ON MONOLITHIC CATALYSTS

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*Fuel*,  
89(6) (2010) pp. 1230-1240.

Active components comprised of fluorite-like Ln<sub>x</sub>(Ce<sub>0.5</sub>Zr<sub>0.5</sub>)<sub>1-x</sub>O<sub>2-y</sub> (Ln = La, Pr, Sm) and perovskite-like La<sub>0.8</sub>Pr<sub>0.2</sub>Mn<sub>0.2</sub>Cr<sub>0.8</sub>O<sub>3</sub> mixed oxides and their composites with yttria-doped zirconia (YSZ) promoted by precious metals (Pt, Ru) and/or Ni were supported on several types of heat-conducting substrates (compressed Ni–Al foam, Fecralloy foil or gauze protected by corundum layer, Cr–Al–O microchannel cermets, titanium platelets protected by oxidic layer) as well as on honeycomb corundum monolithic substrate. These structured catalysts were tested in pilot-scale reactors in the reactions of steam reforming of methane, selective oxidation of decane and gasoline and steam/autothermal reforming of biofuels (ethanol, acetone, anisole, sunflower oil). Applied procedures of supporting nanocomposite active components on monolithic/structured substrates did not deteriorate their coking stability in real feeds with a small excess of oxidants, which was reflected in good middle-term (up to 200 h) performance stability promising for further up-scaling and long-term tests. Equilibrium yield of syngas at short contact times was achieved by partial oxidation of decane and gasoline without addition of steam usually required to prevent coking. For the first time possibility of successive transformation of biofuels (ethanol, acetone, anisole, sunflower oil) into syngas at short contact times on monolithic catalysts was demonstrated. This was provided by a proper combination of active component, thermal conducting monolithic substrates and unique evaporation/mixing unit used in this research.

## METAL SUPPORTED SOFC ON THE GRADIENT PERMEABLE METAL FOAM SUBSTRATE

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*Adv. Mater. Res.*,  
123-125 (2010) pp. 1083-1086.

Gradient permeable metallic substrate material consisting of two layers of NiAl alloy was developed for the SOFC design. The open-cell foam layer (thickness 1-2 mm, cell density 60 ppi) provides the structure robustness, while a thin (100-200 μm) mesoporous layer facilitates supporting functional layers. Cathode layers (LSM, LSFN and their nanocomposites with GDC or YSZ) and anode layers (NiO/YSZ, NiO/YSZ + Ru/Ln-Sr-Mn-Cr-O nanocomposite catalyst) were deposited by slip casting, electrophoretic deposition or air brushing. Thin (5-10 μm) YSZ layer was deposited by MO CVD. Power density up to 550 mW/cm<sup>2</sup> at 700°C was obtained on button-size cells using wet H<sub>2</sub>-air feeds.

## STEAM REFORMING OF METHANE ON Ru AND Pt PROMOTED NANOCOMPOSITES FOR SOFC ANODES

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*Mater. Res. Soc. Symp. Proc.*,  
1217 (2010) Y03-11.1-6.

Nanocomposite cermet materials comprised of NiO/YSZ (20-90 wt. %) co-promoted with SmPrCeZrO or LaPrMnCrO complex oxides and Pt, Pd or Ru were synthesized by Pechini method. These materials were characterized by BET, TEM with EDX, and CH<sub>4</sub> TPR. The catalytic properties were studied for the steam reforming (SR) of CH<sub>4</sub> at short contact times. Factors controlling performance of these composites in CH<sub>4</sub> SR (Ni content, interaction between components in composites as dependent upon their chemical composition) were determined. Ru-promoted composite supported on Ni-Al foam demonstrated a high (up to 75%) methane conversion at 650°C in the feed containing 20% CH<sub>4</sub> and 40% H<sub>2</sub>O in Ar.

**DESIGN OF ANODE MATERIALS FOR IT SOFC: EFFECT OF COMPLEX OXIDE PROMOTERS AND PT GROUP METALS ON ACTIVITY AND STABILITY IN METHANE STEAM REFORMING OF Ni/YSZ (ScSZ) CERMETS**

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*J. Fuel Cell Sci. Technol.*,  
7(1) (2010) 011005 (6 pp.).

Ni/YSZ or Ni/ScCeSZ cermets were promoted by up to 10 wt % of fluoritelike (Pr-Ce-Zr-O, La-Ce-Zr-O, and Ce-Zr-O) or perovskitelike (La-Pr-Mn-Cr-O) oxides and small (up to 1.4 wt %) amounts of Pt group metals (Pd, Pt, or Ru). Reactivity of samples, their lattice oxygen mobility, and their ability to activate methane were characterized by temperature-programmed reduction by CH<sub>4</sub>. The catalytic properties of these samples in methane steam reforming were studied at 500-850°C and short contact times (10 ms) in feeds with 8 mol % of CH<sub>4</sub> and steam/methane ratio of 1: 3. Oxide promoters ensure stable performance of cermets in stoichiometric feeds at T > 650°C by suppressing carbon deposition. Copromotion with precious metals enhances performance in the intermediate temperature (450-600°C) range due to more efficient activation of methane. Factors determining specificity of these cermet materials' performance (chemical composition, microstructure, oxygen mobility in oxides, interaction between components, and reaction media effect) are considered. The most promising systems for practical application are Pt/Pr-Ce-Zr-O/Ni/YSZ and Ru/La-Pr-Mn-Cr-O/Ni/YSZ cermets demonstrating a high performance in the intermediate temperature range under broad variation in steam/CH<sub>4</sub> ratio.

**OXYGEN MOBILITY OF Pt-PROMOTED DOPED CeO<sub>2</sub>-ZrO<sub>2</sub> SOLID SOLUTIONS: CHARACTERIZATION AND EFFECT ON CATALYTIC PERFORMANCE IN SYNGAS GENERATION BY FUELS OXIDATION/REFORMING**

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*Catal. Today*,  
157(1-4) (2010) pp. 55-60.

For nanocrystalline ceria-zirconia samples doped with rare-earth (Gd, Pr, La) cations prepared via modified Pechini route and promoted by Pt supported by wet impregnation, parameters characterizing their oxygen mobility and reactivity were estimated from results of oxygen isotope exchange experiments in both isothermal and temperature-programmed modes using different procedures. Observed trends in variation of oxygen exchange parameters with the type and content of a dopant were explained with a due regard for the real structure and surface properties of samples earlier characterized in detail. For the reaction of partial oxidation and dry reforming of methane in diluted feeds, catalytic activity correlates both with Pt dispersion and oxygen mobility. For the reactions of acetone autothermal reforming and CH<sub>4</sub> dry reforming in realistic feeds, catalytic activity is mainly determined by the surface/bulk oxygen mobility responsible for coking suppression.

**DESIGN OF ASYMMETRIC MULTILAYER MEMBRANES BASED ON MIXED IONIC-ELECTRONIC CONDUCTING COMPOSITES SUPPORTED ON Ni-Al FOAM SUBSTRATE**

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*Catal. Today*,  
156(3-4) (2010) pp. 173-180.

This paper presents the results of research aimed at design of multilayer asymmetric oxygen separation membranes comprised of functionally graded by composition and porosity nanocomposite layers with mixed ionic-electronic conductivity (MIEC) and a high oxygen mobility supported on the compressed

Ni–Al alloy foam substrate. Complex oxides with fluorite-like structure ( $\text{Ce}_{0.9}\text{Gd}_{0.1}\text{O}_{2-\delta}$ ), perovskite-like structure  $\text{La}_{0.8}\text{Sr}_{0.2}\text{Fe}_{1-x}\text{Ni}_x\text{O}_{3-\delta}$  ( $x = 0.3\text{--}0.4$ ) and spinel structure  $\text{MnFe}_2\text{O}_4$  synthesized via polymerized precursors (Pechini) route were used for the preparation of these nanocomposites by ultrasonic dispersion of their mixtures in isopropanol with addition of polyvinyl butyral. Parameters characterizing their oxygen mobility and reactivity were estimated by oxygen isotope heteroexchange, weight loss transients, temperature-programmed reduction by  $\text{CH}_4$  and reoxidation by  $\text{CO}_2$ . Membranes were prepared by successively supporting on one side of substrate macroporous–mesoporous–microporous–dense layers of MIEC nanocomposites finally covered by a porous layer of La–Ni–Pt/ $\text{Pr}_{0.3}\text{Ce}_{0.35}\text{Zr}_{0.35}\text{O}_{2-x}$  catalyst. Preliminary tests of this membrane in the lab-scale reactor in the process of methane selective oxidation/oxi-dry reforming into syngas demonstrated their oxygen permeability and performance promising for the practical application.

#### SYNGAS GENERATION FROM HYDROCARBONS AND OXYGENATES WITH STRUCTURED CATALYSTS

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In “*Syngas: Production Methods, Post Treatment and Economics*”, Nova Science Publishers, Eds. A. Kurucz, I. Bencik, 2009, pp. 53-140 (454 pp.).

Syngas generation by oxidative or steam reforming of hydrocarbons and oxygenates now attracts a lot of attention of researchers in the fields of heterogeneous catalysis and chemical engineering due to its tremendous importance for energy generation and synfuels production. This book reviews results of the long-term research of the international team of scientists aimed at development of efficient processes of syngas generation in structured catalytic reactors. Multiscale integrated optimisation approach is applied throughout of this work including design of nanocomposite active components stable to coking and sintering; developing heat-conducting monolithic substrates comprised of refractory alloys and cermets (honeycomb and microchannel structures, gauzes etc) and procedures of their loading with active

components; design and manufacturing of several types of pilot-scale reactors (with the radial or the axial flow direction) equipped with unique liquid fuel evaporation and mixing units and internal heat exchangers. Extended tests of these reactors fed by fuels from C1 to gasoline, mineral and sunflower oil have been carried out with a broad variation of experimental parameters including stability tests up to 1000 h. Performance analysis has been made with a due regard for equilibrium restrictions on the operational parameters. Transient behaviour of the monolith reactor during start-up (ignition) of the methane partial oxidation to synthesis gas was studied and analysed via mathematical modelling based upon detailed elementary step mechanism. This provides required bases for theoretical optimisation of the catalyst bed configuration and process parameters.

#### Pd/ $\gamma$ - $\text{Al}_2\text{O}_3$ CATALYSTS ON CELLULAR SUPPORTS FOR VOC VAPOR NEUTRALIZATION

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2(4) (2010) pp. 387-392.

The results from investigating the influence of temperature, concentration, and flow rate on the catalytic oxidation of vapors of volatile organic compounds (VOCs) in the presence of Pd/ $\gamma$ - $\text{Al}_2\text{O}_3$  catalyst on cellular supports are presented. The activity of Pd/ $\gamma$ - $\text{Al}_2\text{O}_3$  catalysts on ceramic and metal monolith supports with a cellular structure during the catalytic neutralization of VOC (ethanol, ethyl acetate) vapors under laboratory conditions was determined, and the most stable catalyst for the preliminary study of a large batch was chosen. A pilot unit was created to test a large batch of cellular monolith catalyst in neutralizing VOC vapors under conditions of flexographic production. It was established that a high rate of conversion (> 99 %) was achieved for VOC concentrations of  $0.5 \text{ g/m}^3$  at space velocities of up to  $\sim 10^4 \text{ h}^{-1}$ , and for VOC concentrations of  $5.0 \text{ g/m}^3$  at space velocities of up to  $\sim 5 \times 10^5 \text{ h}^{-1}$ . The change in the activity of the catalysts on metal (nickel alloyed by aluminum) and ceramic cellular supports in service was investigated. After 300–500 min of operation, virtually complete deactivation of catalyst on a metal

support was observed, accompanied by the formation of nickel oxide and acetate. Pilot unit tests with catalyst on cellular supports having a volume of 14.5 l in neutralizing the ventilation exhausts of flexographic production confirmed the possibility of more than 90% conversion at VOC concentrations of  $\sim 0.1 \text{ g/m}^3$  and more than 97% at VOC concentrations of over  $1 \text{ g/m}^3$ . A consistently high conversion of VOC was observed during a 100 h test of the pilot unit. A system for recovering the heat released during VOC oxidation lowers the operating costs of the pilot unit.

#### **DESIGN OF COMPOSITE POROUS CERMETS SYNTHESIZED BY HYDROTHERMAL TREATMENT OF CrAl POWDER FOLLOWED BY CALCINATION**

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*J. Mater. Sci.*,  
45(12) (2010) pp. 3160-3168.

The microstructure of the porous Cr-Al metal-oxide cermet was studied by means of XRD, SEM,

EDX as well as IR and Raman spectroscopy. This cermet was synthesized by mechanical alloying of Cr-Al powders in an AGO-2 planetary ball mill followed by hydrothermal treatment in a special stainless steel die and calcination in air. As a result, a highly porous monolith comprised of metal-like particles randomly distributed in the oxide matrix ( $\text{Cr}_2\text{O}_3$  and  $\text{Al}_2\text{O}_3$ ) was formed. Two types of the composite cores were found in cermets. The first one consisted of chromium phase containing nanoparticles sized from 50 to 140 nm and Al-enriched phase at the interfaces. The second one consisted of new chromium oxide phases with hexagonal  $\text{Cr}_2\text{N}$ -like and fcc CrN-like structures probably with  $\text{Cr}_2\text{O}$  and CrO stoichiometry. These new phases were stabilized within aggregates of the nanocomposite particles containing inclusions of alumina. The relations between different preparation stages and the cermet microstructure are discussed.

## **Photocatalytic and Related Processes**

#### **PHOTOCATALYTIC TRANSFORMATIONS OF SULFUR-BASED ORGANIC COMPOUNDS**

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In "Environmentally Benign Photocatalysts. Applications of Titanium Oxide-Based Materials", Springer, Eds. M. Anpo, P.V. Kamat, 2010, ch. 25, pp. 579-621 (757 pp.)

Organic sulfur compounds play significant roles in the life of modern humankind. There are many instances of the need to destroy or convert sulfur compounds into useful products. Photocatalytic reactions are capable of removing and transforming these compounds. High reactivity of sulfur compounds allows utilization of a variety of photocatalysts – heterogeneous like  $\text{TiO}_2$  or other species, homogeneous, heterogeneous, or heterogenized. The transformations are mediated by electron transfer with generation of sulfur radical cations, energy transfer, or hydrogen atom transfer. Further reactions majorly comprise S-oxidation,  $\alpha\text{-C-H}$  deprotonation, C-S bond cleavage, and polymerization. Reactions with photogenerated OH radicals are also possible.

#### **PHOTOCATALYSIS: LIGHT ENERGY CONVERSION FOR THE OXIDATION, DISINFECTION, AND DECOMPOSITION OF WATER**

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*Kinet. Catal.*,  
51(6) (2010) pp. 801-808.

The results of many-year studies of the relationship between the physical properties and photocatalytic activity of  $\text{TiO}_2$  and  $\text{Pt/TiO}_2$  in photocatalytic purification and disinfection of air and water and water photodecomposition with oxygen evolution are presented. Recommendations are given as to finding the optimal method for platinum supporting on  $\text{TiO}_2$  to achieve the highest possible catalytic activity. Multisite kinetic models of the gas-phase oxidation of simple organic substances are considered. Methods for regenerating the photocatalyst after its deactivation in the oxidation of sulfur-containing organic substances are suggested. New data are discussed on the acceleration of air purification by the combination of photocatalytic oxidation with atmospheric electric discharges, the

addition of gaseous hydrogen peroxide, and oxidation on photocatalysts existing in the aerosol state. As compared to pure TiO<sub>2</sub>, platinated titanium dioxide has a higher capability for disinfection and complete mineralization of microorganisms. Two promising methods for production of hydrogen from water using solar light are presented.

**ADSORBENT AS AN ESSENTIAL PARTICIPANT IN PHOTOCATALYTIC PROCESSES OF WATER AND AIR PURIFICATION: COMPUTER SIMULATION STUDY**

**D.S. Selishchev, P.A. Kolinko, D.V. Kozlov**

*Appl. Catal., A*,  
377(1-2) (2010) pp. 140-149.

The influence of adsorbent on the kinetics of (photo)catalytic oxidation processes was studied using the simple single and double stage reaction mechanisms with the adsorption stages corresponding to Langmuir model. It was found that in the static reactor in all cases the usage of adsorbent leads to the prolongation of substrate (*A*) removal and product (*P*) accumulation kinetic curves but the substrate concentration becomes lower during almost all (photo)catalytic process. Fitting of experimental data points of acetone photocatalytic oxidation reaction demonstrated a good correlation with proposed reaction mechanism. Calculated adsorption constants values are close to those reported in literature.

The using of adsorbent in flowing conditions leads to the decrease of maximum outlet substrate concentration in the case if the inlet substrate concentration is too high to be oxidized completely for one run.

Kinetics simulation in the case of (photo)catalytic reaction with intermediate demonstrates that adsorbent could accumulate additional intermediate quantity thus keeping catalyst surface more active and contributing to faster substrate (*A*) removal.

**INCREASING THE EFFICIENCY OF DEEP PHOTOCATALYTIC OXIDATION OF ORGANIC VAPORS UNDER THE ACTION OF NEGATIVE ATMOSPHERIC CORONA DISCHARGE**

**A.S. Besov, D.A. Trubitsyn, A.V. Vorontsov**

*Catal. Ind.*,  
2(2) (2010) pp. 118-125.

The influence of atmospheric corona discharge on the photocatalytic oxidation of acetone (83 ppm), acetaldehyde (108 ppm), and toluene

(12–46 ppm) vapors on TiO<sub>2</sub> under the action of soft UV light in a closed chamber has been studied. Corona discharge was created around the wire of the charging unit of an electric filter at the inlet of a photocatalytic reactor. The rate of the removal and deep oxidation of substances to CO<sub>2</sub> increased considerably and reached maximum for acetaldehyde. The concentrations of intermediates in the course of experiments did not exceed 1 ppm. In the presence of corona discharge, the rate of deep oxidation per unit of consumed electric power increased by a factor of 1.2–3.6. The results show that combined photocatalytic and plasmocatalytic oxidation is promising for the purification of released gases.

**PHOTOCATALYTIC HYDROGEN EVOLUTION FROM AQUEOUS SOLUTIONS OF ORGANOPHOSPHOROUS COMPOUNDS**

**E.A. Kozlova, A.V. Vorontsov**

*Int. J. Hydrogen Energy*,  
35(14) (2009) pp. 7337-7343.

Photocatalytic hydrogen production from water solutions of dimethyl methylphosphonate (DMMP), trimethyl phosphate (TMP), triethyl phosphate (TEP), and radiation protective amine WR 2721, that imitate nerve chemical warfare agents was studied for the first time. Platinized titanium dioxide Degussa P25 was used as catalyst. No significant hydrogen evolution was detected without organic electron donors - sacrificial agents.

The hydrogen evolution rate was shown to grow slowly with the rise of initial DMMP concentration. The initial rate vs. DMMP concentration curve is well fitted by Langmuir-Hinshelwood (L-H) equation. The DMMP adsorption constant obtained from the L-H equation fit is markedly higher than that obtained from the Langmuir adsorption isotherm. Reactions of full destruction into inorganic products of the four organophosphorous compounds were conducted. Amounts of evolved hydrogen and carbon dioxide were completely consistent with stoichiometry of proposed reaction. There were no initial compounds and only trace of total organic carbon after the end of the reaction. Complete water purification and production of an amount of valuable hydrogen was achieved.

Intermediates of DMMP oxygen-free destruction were identified by means of GC/MS. They were the same as those in the case of DMMP photocatalytic oxidation. A mechanism of hydrogen evolution that explains smaller rates of mineralization compared to photocatalytic oxidation by oxygen was proposed.

## AIR DETOXIFICATION WITH NANOSIZE TiO<sub>2</sub> AEROSOL TESTED ON MICE

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*J. Hazard. Mater.*,  
173(1-3) (2010) pp. 40-46.

A method for fast air purification using high concentration aerosol of TiO<sub>2</sub> nanoparticles is evaluated in a model chemical catastrophe involving toxic vapors of diisopropyl fluorophosphate (DFP). Mice are used as human model in a closed 100 dm<sup>3</sup> chamber. Exposure of mice to 37 ppm of DFP vapor for 15 min resulted in acute poisoning. Spraying TiO<sub>2</sub> aerosol in 2 min after the start of exposure to DFP vapors resulted in quick removal of DFP vapors from the chamber's air. Animals did not show signs of poisoning after the decontamination experiment and exposure to TiO<sub>2</sub> aerosol alone. Reactive oxygen species (ROS) and antioxidant activity (AOA) of mice blood plasma were measured for animals exposed to sound of aerosol generator, DFP vapors, TiO<sub>2</sub> aerosol and DFP vapors + TiO<sub>2</sub> aerosol. Reduced ROS and increased AOA were found for mice exposure to sound, DFP and TiO<sub>2</sub> aerosol. Exposure to DFP and decontamination with TiO<sub>2</sub> nanoparticles resulted in decreased AOA in 48 h following the exposure. The results suggest that application of TiO<sub>2</sub> aerosol is a powerful method of air purification from toxic hydrolysable compounds with moderate health aftermaths and requires further study and optimization.

## INACTIVATION AND MINERALIZATION OF AEROSOL DEPOSITED MODEL PATHOGENIC MICROORGANISMS OVER TiO<sub>2</sub> AND Pt/TiO<sub>2</sub>

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*Environ. Sci. Technol.*,  
44(13) (2010) pp. 5121-5126.

Air disinfection from bacteria and viruses by means of photocatalytic oxidation is investigated with microorganisms loaded over photocatalysts' films from aerosols. Deposition method and equipment have been developed to load *Mycobacterium smegmatis*, *Bacillus thuringiensis*, vaccinia virus, and influenza A (H3N2) virus on slides with undoped TiO<sub>2</sub> and platinumized sulfated TiO<sub>2</sub> (Pt/TiO<sub>2</sub>). Inactivation dynamics was measured under UVA irradiation and in the dark. About 90% inactivation is reached in 30 min irradiation on TiO<sub>2</sub> and from 90 to 99.8% on Pt/TiO<sub>2</sub>. The first-order inactivation rate coefficient ranged from 0.18 to 0.03 min<sup>-1</sup>, over Pt/TiO<sub>2</sub> being higher than on TiO<sub>2</sub> for all microorganisms except *Bacillus thuringiensis*. The photocatalytic mineralization of *Bacillus thuringiensis* was performed on TiO<sub>2</sub> and Pt/TiO<sub>2</sub> with different photocatalyst and microorganism loadings. Completeness of mineralization depended on the TiO<sub>2</sub> to bacteria mass ratio. The rate of the photocatalytic carbon dioxide production grows with both the cell mass increase and the photocatalyst mass increase. Pt/TiO<sub>2</sub> showed increased rate of mineralization as well as of the inactivation likely due to a better charge carrier separation in the doped semiconductor photocatalyst. The results demonstrate that photocatalytic filters with deposited TiO<sub>2</sub> or Pt/TiO<sub>2</sub> are able to inactivate aerosol microorganisms and completely decompose them into inorganic products and Pt/TiO<sub>2</sub> provides higher disinfection and mineralization rates.

**PHOTOCHEMICAL PROPERTIES OF  
PRECIPITATED SOLID AEROSOL  
PRODUCED BY BURNING OF TITANIUM  
MICROPARTICLES UNDER AMBIENT AIR**

**V.S. Zakharenko, S.A. Khromova**

*Mater. Sci. Appl.*,  
1(2) (2010) pp. 97-102.

In order that to neutralize a drastic pollution of the environment (technogenic catastrophe) it is suggested to use technogenic technologies of chemical compound decontamination. One in such technologies can be the technology using metal oxide solid aerosols which are active in removal of pollutant compounds and obtainable by combustion under ambient air of appropriate metal particles, for example, aluminum, magnesium, titanium and etc. It is shown that the titanium dioxide out of an solid aerosol, obtained by pyrotechnic mixture combustion containing titanium microparticles has optic, chemical and photocatalytic properties close to properties of titanium dioxide produced by a different way. The production of such aerosol in direct place of a technogenic catastrophe can be made for the cleaning of atmosphere near a pollution source.

**PHOTOCATALYTIC PROPERTIES OF  
PRECIPITATED TITANIUM OXIDE AEROSOL  
PRODUCED FROM RUTILE CRYSTAL UNDER  
AMBIENT AIR**

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University, Tomsk, Russia)

*Atmosph. Oceanic Optics*,  
23(6) (2010) pp. 515-518.

A simple method for modulation of aerosol particle formation in Earth's atmosphere is suggested. In this way a crystal of natural mineral is dispersed under ambient air. Interactions between the surface of such obtained TiO<sub>2</sub> and halogen-containing organic compounds, acetone and CO, enriched by isotope <sup>13</sup>C, under darkness and irradiation were investigated. The mechanisms of investigation processes are suggested.

## Polymerization Catalysts and Polymer Materials

### DEVELOPMENT OF TECHNOLOGY FOR PRODUCING MODERN EFFECTIVE CATALYSTS FOR THE INDUSTRY OF HIGH DENSITY POLYETHYLENE AND POLYPROPYLENE

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*Russ. J. Appl. Chem.*,  
82(12) (2009) pp. 2238-2241.

The results of development and testing catalysts for producing polypropylene are presented. The innovative project is based on the development of highly efficient catalysts of a propylene polymerization carried out at the Boreskov Institute of Catalysis, and on the successful cooperation of Tomskneftechim and the Boreskov Institute Catalysis on testing these catalysts in the industrial production of PP.

### NEW GENERATION OF SUPPORTED ZIEGLER-TYPE CATALYSTS FOR POLYETHYLENE PRODUCTION

T.B. Mikenas, V.A. Zakharov, V.E. Nikitin, L.G. Echevskaya, M.A. Matsko

*Russ. J. Appl. Chem.*,  
83(12) (2010) pp. 2210-2219.

On the basis of new methods of synthesis of magnesium-containing carriers with the use of soluble organomagnesium compounds a wide spectrum of supported titanium-magnesium and vanadium-magnesium catalysts of various composition with controllable optimal morphology for obtaining versatile brands of polyethylene by suspension and gas-phase methods was elaborated. The usage of various modifications of these catalysts allows production of high amount (up to 1000 kg/g Ti) of polyethylene of high, medium, and low density with narrow and wide molecular weight distribution (including bimodal) as well as different brands of polyethylene ultrahigh molecular weight.

### HETEROGENEITY OF ACTIVE SITES OF ZIEGLER-NATTA CATALYSTS: THE EFFECT OF CATALYST COMPOSITION ON THE MWD OF POLYETHYLENE

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*J. Appl. Polym. Sci.*,  
115(4) (2010) pp. 2432-2439.

Experimental data on the molecular weight distribution (MWD) of polyethylene (PE) produced over a broad number of Ziegler-Natta catalysts differing in composition and preparation procedure are presented. These catalysts include nonsupported  $\text{TiCl}_3$  catalyst, four types of supported titanium-magnesium catalysts (TMC) differing in the content of titanium and the presence of various modifiers in the composition of the support, and a supported catalyst containing  $\text{VCl}_4$  as an active component instead of  $\text{TiCl}_4$ . The studied catalysts produce PE with different molecular weights within a broad range of polydispersity ( $M_w/M_n = 2.8-16$ ) under the same polymerization conditions. The heterogeneity of active sites of these catalysts was studied by deconvolution of experimental MWD curves into Flory components assuming a correlation between the number of Flory components and the number of active site types. Five Flory components were found for PE produced over nonsupported  $\text{TiCl}_3$  catalysts ( $M_w/M_n = 6.8$ ), and three-four Flory components were found for PE produced over TMC of different composition. A minimal number of Flory components (three) was found for PE samples ( $M_w/M_n$  values from 2.8 to 3.3) produced over TMC with a very low titanium content (0.07 wt %) and TMC modified with dibutylphthalate. It was shown that five Flory components are sufficient to fit the experimental MWD curve for bimodal PE ( $M_w/M_n = 16$ ) produced over VMC.



### CHANGE OF THE WEAR RESISTANCE OF A SUPERHIGH-MOLECULAR-WEIGHT POLYETHYLENE PLATE UPON ITS MODIFICATION BY A MECHANICALLY ACTIVATED CERAMIC NANOPOWDER

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*Theor. Found. Chem. Eng.*, 44(4) (2010) pp. 521-524.

The object of investigation is superhigh-molecular-weight polyethylene (SMWP) with a molecular weight of up to  $8 \times 10^6$  g/mole and a particle size from 63 to 315  $\mu\text{m}$  obtained by the technique developed at the Boreskov Institute of Catalysis.

The purpose of this work is to create structural polymer materials with high physical and technical characteristics and to extend the scope of the application of this polymer in industry. The problem is solved by the modification of SMWP powder particles via the introduction of nanodispersed ceramic materials.

Based on the data obtained, the methods of the production of the article samples from nanocomposite materials based on SMWP via the hot-pressing technique were developed and the influence of the nanomodifiers on the properties of the composite material was studied.

### COMPOSITE MATERIALS BASED ON ULTRAHIGH MOLECULAR WEIGHT POLYETHYLENE: PROPERTIES, POTENTIAL APPLICATION AREAS

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*Chem. Sustain. Devel.*, 18(3) (2010) pp. 375-388.

Methods for synthesis and properties of ultrahigh molecular polyethylene (UHMPE) – a material that features a high resistance under severe operation conditions, in comparison to traditional polymer types – are discussed. The UHMPE modifying with ultradisperse inorganic particles is shown to improve service properties of UHMPE products. Results of studies on development technologies for production of specials from composite materials based on the modified UHMPE are generalized. New plastic polymer materials

are synthesized on the basis of modified UHMPE, butadiene-nitril, cis-isoprene and divinyl rubbers. The obtained composite materials can have numerous applications due to the exceptionally high attrition resistance and improved lifetime under extreme conditions.

### THE KINETIC EVIDENCE FOR THE FORMATION OF MULTIPLE ACTIVE SPECIES IN A BIS(PHENOXY-IMINE) ZIRCONIUM DICHLORIDE/MAO CATALYST DURING ETHYLENE POLYMERIZATION

A.A. Barabanov, N.V. Semikolenova, M.A. Matsko, L.G. Echevskaya, V.A. Zakharov

*Polym.*, 51(15) (2010) pp. 3354-3359.

The effects of polymerization time and temperature on the molecular weight and molecular weight distribution of polyethylene, produced over homogeneous catalyst bis[*N*-(3-*tert*-butyl salicylidene)anilinato]zirconium(IV) dichloride 'Bu-L<sub>2</sub>ZrCl<sub>2</sub>/MAO have been studied. The data on the number of active centers ( $C_p$ ) and propagation rate constants ( $k_p$ ) at different polymerization time have been obtained as well. It was found that at a short polymerization time two types of active centers, producing low molecular weight PE ( $M_w = 4-10 \times 10^3$  g mol<sup>-1</sup>) are formed. The number of these centers was estimated to be 11% of total zirconium complex and their reactivity is very high (the  $k_p$  value was found to be  $54 \times 10^3$  L mol<sup>-1</sup> s<sup>-1</sup> at 35°C). High initial activity of the catalyst fell with the increase in polymerization time, whereas the polydispersity values of the resulting PE increase due to formation of new centers, producing high molecular weight PE ( $M_w = (30-1300) \times 10^3$  g mol<sup>-1</sup>). It was found that the decrease in activity is caused by reducing the initial active centers number and lower reactivity of the new-formed centers ( $k_p = 17 \times 10^3$  L mol<sup>-1</sup> s<sup>-1</sup>).

### DRIFTS STUDY OF THE INTERACTION OF THE INTERNAL DONOR IN TiCl<sub>4</sub>/di-*n*-BUTYL PHTHALATE/MgCl<sub>2</sub> CATALYSTS WITH AlEt<sub>3</sub> COCATALYST

A.G. Potapov, G.D. Bukatov, V.A. Zakharov

*J. Mol. Catal. A: Chem.*, 316(1-2) (2010) pp. 95-99.

The interaction of di-*n*-butyl phthalate (DBP as the internal donor) with AlEt<sub>3</sub> cocatalyst was studied by DRIFT spectroscopy for the DBP/MgCl<sub>2</sub> sample and supported TiCl<sub>4</sub>/DBP/MgCl<sub>2</sub> catalysts. For

DBP/MgCl<sub>2</sub> sample, it was shown that AlEt<sub>3</sub> partially removes all types of DBP complexes from the MgCl<sub>2</sub> surface and adsorbs on surface sites released by the removal of DBP. In the case of supported catalysts, the cocatalyst predominantly removes more weak DBP complexes. In addition, the cocatalyst completely removes loosely coordinated DBP and surface complexes of phthaloyl chlorides formed at the catalyst preparation. A part of removed DBP can adsorb again on the catalyst surface as complexes with surface alkylaluminum chloride compounds. The external donor alkylalkoxysilane does not markedly influence the removal of DBP complexes from the catalyst surface.

### BIODEGRADABLE POLYMERS: TOWARDS THE FUTURE

A.G. Potapov, V.N. Parmon

*Ekologiya i Promyshlennost' Rossii,*  
Sp. issue (2010) pp. 4-8.

The major factors determining ability of polymers for biodegradation are considered. The basic types of industrially assimilated biodegradable polymers, the methods of their manufacture and improvement of their properties as well as approaches to reduction in value of their manufacture are viewed. The special attention is given to biodegradable polymers obtained from renewable vegetative raw materials and to the ways of their modification. The ways of materials modification on the basis of polyolefines with the purpose of giving them the ability of biodegradation are discussed.

### *In situ* EPR MONITORING OF CHROMIUM SPECIES FORMED DURING Cr-PYRROLYL ETHYLENE TRIMERIZATION CATALYST FORMATION

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K.P. Bryliakov, V.A. Zakharov, E.P. Talsi

*Organomet.,*  
29(13) (2010) pp. 2943-2950.

The catalyst systems Cr(acac)<sub>3</sub>/pyrrole/AlEt<sub>3</sub>/AlEt<sub>2</sub>Cl and Cr(EH)<sub>3</sub>/pyrrole/AlEt<sub>3</sub>/AlEt<sub>2</sub>Cl in cyclohexane (EH = 2-ethylhexanoate), modeling the Phillips ethylene trimerization catalyst, have been studied by EPR spectroscopy. The effect of various components of these catalyst systems on the concentration of Cr(III) and Cr(I) species and ethylene trimerization activity has been investigated. The relationship between the trimerization data and the nature of the chromium species present in the reaction solution has been studied.

### NONCOVALENT INTERACTIONS IN o-FLUORINATED POST-TITANOCENE LIVING ETHYLENE POLYMERIZATION CATALYST

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*Organomet.,*  
29(20) (2010) pp. 4428-4430.

Multinuclear NMR spectroscopic studies of polymerization-active species generated by activation of o-F-substituted and nonsubstituted bis(enolatoimine) titanium complexes with MAO evidence a noncovalent interaction of the o-F moieties with the metal center, which is likely to facilitate living ethylene polymerization via suppression of chain transfer and of β-hydrogen transfer processes.

### REACTIVE INTERMEDIATES FORMED DURING OLEFIN POLYMERIZATION BY METHYLALUMOXANE-ACTIVATED ANSA-ZIRCONOCENE CATALYSTS: IDENTIFICATION OF A CHAIN-CARRYING INTERMEDIATE BY NMR METHODS

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*J. Am. Chem. Soc.,*  
132(2) (2010) pp. 452-453.

Addition of 1-hexene to methylalumoxane-activated catalyst systems based on *rac*-Me<sub>2</sub>Si(ind)<sub>2</sub>ZrMe<sub>2</sub> causes, concurrent with polyhexene formation and diminution of the otherwise prevalent cation [*rac*-Me<sub>2</sub>Si(ind)<sub>2</sub>Zr(μ-Me)<sub>2</sub>AlMe<sub>2</sub>]<sup>+</sup>, formation of a hitherto unobserved species [*rac*-Me<sub>2</sub>Si(ind)<sub>2</sub>Zr(μ-R)(μ-Me)AlMe<sub>2</sub>]<sup>+</sup>, where R is a Zr-bound polyhexyl chain. As hexene is increasingly consumed, this cation decays, mainly back to [*rac*-Me<sub>2</sub>Si(ind)<sub>2</sub>Zr(μ-Me)<sub>2</sub>AlMe<sub>2</sub>]<sup>+</sup> and, in part, to some species containing a Zr-bound allylic chain end.

### POLYMER MEMBRANES FOR FUEL CELLS: MANUFACTURE, STRUCTURE, MODIFICATION, PROPERTIES

S.S. Ivanchev, S.V. Myakin

*Russ. Chem. Rev.,*  
79(2) (2010) pp. 101-117.

The state of the art in the field of synthesis, structural modification and practical use of polymer membranes for fuel cells is analyzed. Synthetic methods, physicochemical characteristics and specific features of their composition, microphase separation and the structure of water ionic channels are considered for different types of proton-conducting

materials together with the approaches to enhancement of their operational characteristics. Attention is focused on Nafion and other fluorinated polymers as the materials that occupy the leading position as regards the design level and the broad use in small sized fuel cells. The newest developments in the field of alternative non-fluorinated membrane materials are also discussed, particularly, polycondensation polymer systems and hybrid organic-inorganic polymers.

#### **POLYMERIZATION OF ETHYLENE IN THE PRESENCE OF BIS(PHENOXYIMINE) COMPLEXES OF TITANIUM CHLORIDE THAT CONTAIN VARIOUS SUBSTITUENTS IN A PHENOXY GROUP**

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*Polym. Sci., ser. B,*  
52(7-8) (2010) pp. 443-449.

The kinetic features of ethylene polymerization with six methylaluminumoxane-activated

bis(phenoxyimine) complexes of titanium chloride that are distinguished by the electronic properties of substituents in the phenoxy group are studied in the temperature range 30–80°C and at an ethylene pressure of 0.3 MPa. It is shown that, in the presence of an electro-donor or electron-acceptor substituent in the phenoxy group, the catalytic systems under study exhibit high activity (up to  $\sim 700 \text{ t}_{PE} \text{ mol}_{cat}^{-1} \text{ mol}_{ethylene}^{-1} \text{ h}^{-1} \text{ h}^{-1}$ ) and form high-molecular-mass PE samples ( $M_n = (500-900) \times 10^3$ ) with different molecular-mass distributions. In the case of titanium bis(phenoxyimine) complexes containing donor substituents at the para position of the phenoxy group, the polymerization of ethylene follows the living-chain mechanism, while the introduction of acceptor substituents diminishes the contribution of this mechanism to the reaction.

## **Biocatalysts, Biochemical Phenomena, Biochemical Ecology**

#### **INVESTIGATION ON MACROKINETICS OF HETEROGENEOUS PROCESS OF MONOSACCHARIDE ISOMERIZATION USING NON-GROWING CELLS OF A GLUCOISOMERASE PRODUCER *ARTHROBACTER NICOTIANAE* IMMOBILIZED INSIDE SiO<sub>2</sub>-XEROGEL**

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*Appl. Biochem. Microbiol.,*  
46(7) (2010) pp. 697-705.

Macrokinetic peculiarities of heterogeneous process of monosaccharide (glucose/fructose) isomerization using biocatalysts prepared by incorporation of non-growing cells of a glucose isomerase-producing strain *Arthrobacter nicotianae* inside SiO<sub>2</sub>-xerogel have been investigated. It was shown that the process proceeds in kinetic regime without diffusion limitation and biocatalyst activities at 60 and 75°C were 19 and 32 U/g, respectively. Time of equilibrium in the reaction of monosaccharide isomerization was a function of starting (“triggering”) glucose isomerase activity in a unit of reaction volume. When the activity exceeds 10 U/ml, equilibrium equimolar mixture of glucose and fructose was produced within a few hours. It was established that a continuous process carried out in a plug-flow

packed-bed reactor is more efficient than a batch process accompanied with recycling, first of all, due to significant improvement of operation stability of the designed biocatalysts. Under model conditions of industrial heterogeneous process of producing glucose-fructose syrups, the half-life time of inactivation of the biocatalysts was more than 500 h at (65 ± 5)°C.

#### **HETEROGENEOUS BIOCATALYSTS FOR PRODUCTION OF SWEETENERS - STARCH TREACLE AND SYRUPS OF DIFFERENT CARBOHYDRATE COMPOSITION**

**G.A. Kovalenko, L.V. Perminova, L.I. Sapunova**

*Catal. Ind.,*  
2(2) (2010) pp. 180-185.

The biocatalytic heterogeneous processes are the basis for the most up-to-date biotechnologies of starch renewable resources processing into called-for sweeteners, such as treacle and syrups. Heterogeneous biocatalysts prepared by immobilization of enzymatically active substances, in particular whole non-growing microorganisms on/inside inorganic supports were developed for the different stages of the starch processing. For starch saccharification the biocatalyst with glucoamylase activity was prepared by adsorption of the enzyme GlucoLux on the nanoporous carbon support Sibunit. The biocatalyst

possessed remarkably high activity (ca. 700 U/g) and stability (the half-inactivation time  $t_{1/2} > 350$  h) at 60°C. For glucose isomerisation the biocatalyst with glucose isomerase activity was prepared by inclusion of biomass of bacterial strains produced intracellular enzyme and insoluble hydroxo compounds of Co(II), which are required for enzyme stabilization, inside the silica xerogel. The stability of the biocatalyst in continuous process of glucose isomerisation was sufficiently high ( $t_{1/2} > 500$  h) at 65°C. For sucrose inversion the biocatalyst with invertase activity was prepared by inclusion of biomass of baker's yeast inside the silica xerogel. At optimal ratio of biomass to SiO<sub>2</sub> the invertase activity of the biocatalyst reached up to ca. 600 U/g, and  $t_{1/2}$  was more than 200 h at 50°C.

#### **A STUDY OF THE CATALYTIC PROPERTIES OF THE NITRILE HYDRATASE IMMOBILIZED ON ALUMINUM OXIDES AND CARBON-CONTAINING ADSORBENTS**

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*Appl. Biochem. Microbiol.*,  
46(4) (2010) pp. 379-384.

The nitrile hydratase isolated from *Rhodococcus ruber* strain gtl, displaying a high nitrile hydratase activity, was immobilized on unmodified aluminum oxides and carbon-containing adsorbents, including the carbon support Sibunit. The activity and operational stability of the immobilized nitrile hydratase were studied in the reaction of acrylonitrile transformation into acrylamide. It was demonstrated that an increase in the carbon content in the support led to an increase in the amount of adsorbed enzyme and, concurrently, to a decrease in its activity. The nitrile hydratase immobilized on Sibunit and carbon-containing aluminum  $\alpha$ -oxide having a "crust" structure displayed the highest operational stability in acrylonitrile hydration. It was shown that the thermostability of adsorbed nitrile hydratase increased by one order of magnitude.

#### **STRUCTURAL FEATURES OF TiO<sub>2</sub> NANOPARTICLES INTERACTION WITH A CELL**

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*ASA Newslett.*,  
10-3(138) (2010) pp. 20-23.

The interaction of amorphous, anatase, brookite and rutile TiO<sub>2</sub> nanoparticles (4-5 nm) with MDCK cells was examined. The cells were incubated in presence of 100  $\mu$ g/mL TiO<sub>2</sub> nanoparticles during 1, 3 and 5 h, and then fixed for electron microscopy. The number of living MDCK cells was counted, using trypan blue assay, after 5 and 15 h treatment with 100  $\mu$ g/mL TiO<sub>2</sub> nanoparticles at 37°C. All of the TiO<sub>2</sub> nanoparticles were toxic to the MDCK cells. Different modes of cell-nanoparticle interaction were observed with the four types of TiO<sub>2</sub>. The interaction of amorphous, anatase and brookite TiO<sub>2</sub> nanoparticles involve intimate cellular mechanisms. This calls for careful examination of nanoparticle-cellular interaction before introducing such particles into living tissues or the environment.

#### **INTERACTION OF TITANIUM DIOXIDE NANOPARTICLES WITH INFLUENZA VIRUS**

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*Nanotechn. Russ.*,  
5(5-6) (2010) pp. 417-420.

Titanium dioxide (TiO<sub>2</sub>) in a suspension or absorbed on a film possesses bactericidal and virucidal properties caused by photocatalytic reactions. Electron microscopic examinations showed that titanium dioxide nanoparticles destroyed the influenza virus after 30 min incubation. Virological studies revealed the inactivation of the influenza virus by TiO<sub>2</sub>, depending on the concentration of nanoparticles and the incubation period. The inactivation effect was observed when the incubation was performed in darkness, unlike the TiO<sub>2</sub> suspension. It was proposed that the virus-inactivation properties of TiO<sub>2</sub> are mainly based on the direct contact between nanoparticles and virus particles.

## SELECTIVE VAPOUR-PHASE $\alpha$ -PINENE ISOMERIZATION TO CAMPHENE OVER GOLD-ON-ALUMINA CATALYST

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*Appl. Catal., A*,  
385(1-2) (2010) pp. 136-143.

The vapour-phase isomerization of  $\alpha$ -pinene for the first time was studied over a supported Au catalyst.  $\alpha$ -Pinene was isomerized to camphene over the 2.2% Au/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub> catalyst at 463-483 K using a solution of the reagent in n-octane as the initial reaction mixture and H<sub>2</sub> or N<sub>2</sub> as a carrier gas. Under these conditions, the selectivity to camphene reaches 60-80% at 99.9% conversion of  $\alpha$ -pinene. The reaction is found to be first-order with respect to  $\alpha$ -pinene, the apparent activation energy being similar to that observed with the conventional TiO<sub>2</sub> catalyst. The prominent catalyst deactivation has been observed at increased  $\alpha$ -pinene concentrations in the inlet reaction mixture ( $\geq 4$  vol% in n-octane solution). According to HRTEM and TPO results, the deactivated catalyst contains the carbonaceous deposits that may block the catalyst surface. Almost complete regeneration was done in flowing O<sub>2</sub> at temperature up to 923 K required to totally eliminate the coke deposits.

## LINOLEIC ACID ISOMERIZATION OVER MESOPOROUS CARBON SUPPORTED GOLD CATALYSTS

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*Catal. Today*,  
150(1-2, sp. issue) (2010) pp. 32-36.

Gold catalyst supported on mesoporous carbon and silica were synthesized, characterized by TEM, XRD, XPS and tested in linoleic acid isomerization. Nature of the support affects the selectivity towards isomerization in relation to unwanted hydrogenation. In particular carbon support allowed much higher selectivity in double bond migration compared to silica. Effect of carbon surface oxidative pre-treatment on selectivity of catalyst was investigated.

## SELECTIVE OXIDATION OF GLUCOSE OVER CARBON-SUPPORTED Pd AND Pt CATALYSTS

I.V. Delidovich, O.P. Taran, L.G. Matvienko, A.N. Simonov, I.L. Simakova, A.N. Bobrovskaya, V.N. Parmon

*Catal. Lett.*,  
140(1) (2010) pp. 14-21.

Selective oxidation of glucose into gluconic acid by molecular oxygen over carbon-supported Pt and Pd catalysts was studied. Under examination were kinetic regularities of the process in respect of the electronic state of the noble metal surface, dispersion of the active component particles as well as substrate:Pt(Pd) ratio. Catalytic activity of the Pt/C catalysts being normalized to the dispersion of the platinum particles appeared independent of the particles mean diameter in the 1-5 nm range. A negative particle size effect for the Pd/C catalysts, caused by feasibility of oxidation of the surface of noble metal particles with the size less than 3 nm, was observed. Pt/C catalysts exhibited lower specific activity and provided poor selectivity of the glucose oxidation in comparison with Pd/C. Deactivation of Pd/C catalysts arising from the formation of surface Pd(II) oxides was retarded when the reaction was carried out under an oxygen-diffusion control.

## INVESTIGATION OF BIOLOGICAL SAFETY OF NANOSUBSTANCES AND NANOMATERIALS

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*Chem. Sustain. Devel.*,  
18(4) (2010) pp. 527-534.

Results of the toxicological investigation *in vivo* and *in vitro* of nanosubstances and nanomaterials obtained at the Institutes of the Siberian Branch of RAS are presented. It was established that, on the basis of acute toxicity value, the nanopowders of the oxides of aluminium, zinc, titanium, and carbon materials may be related to the 3<sup>rd</sup> class (moderate toxicity) or the 4<sup>th</sup> class (low toxicity), while nanopowders of copper correspond to the 2<sup>nd</sup> class of toxicity. It is demonstrated through the

*in vitro* investigation involving titanium nanopowder as an example that metal nanopowders can be highly toxic for various primary (healthy) cells but cause no death of cancer cells.

#### SEARCH FOR RENEWABLE SOURCES OF MULTI-PURPOSE CELLULOSE

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*Informatsionnyi Vestnik VOGiS,*  
14(3) (2010) pp. 569-578.

Conditions of miscanthus delignification in the course of cellulose production were studied. The yield and parameters of the product, as well as the properties of bleached pulp and carboxymethylcellulose, were determined. Search for new cellulose sources reproducible by seeds and adapted to various environmental settings was conducted.

#### OBTAINING MANNANOLIGOSACCHARIDE PREPARATIONS BY MEANS OF THE MECHANOENZYMATIC HYDROLYSIS OF YEAST BIOMASS

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*Appl. Biochem. Biotechnol.,*  
162(7) (2010) pp. 2008-2014.

A new method of obtaining biologically active mannanoligosaccharides is proposed. It involves mechanical activation of the enzymatic hydrolysis of components forming the supramolecular structure of the cell wall. Processes that take place during mechanical treatment and enzymatic hydrolysis of yeast biomass and lead to an increase in the

availability of mannanoligosaccharides of the yeast cell walls are investigated. The efficiency of the use of mechanoenzymatic approach to obtaining mannanoligosaccharide preparations is evaluated.

#### ASTROCATALYSIS — ABIOGENIC SYNTHESIS AND CHEMICAL EVOLUTION AT PREGEOLOGICAL STAGES OF THE EARTH'S FORMATION

V.N. Snytnikov

*Paleontological J.,*  
44(7) (2010) pp. 761-777.

The Earth's biosphere appeared in a self-organization process along with the appearance of the Solar System. It is shown, based on the methods of self-organization examination and existing knowledge, that major stages of the chemical evolution in the early development of the biosphere include the "cold prehistory of life" in dense molecular nebulae, "pre-planetary chemocoenosis," "RNA-world" in a circumsolar nebula, and primary biocoenoses of protocells (life) on the planetary bodies. Estimates for carbon in the primordial biosphere on the young Earth's surface give  $2.4 \times 10^{19}$  kg. The decay of the primordial Earth's biomass and biogeochemical cycles in 2.5 Myr led to the "planet of bacteria" with  $2.0 \times 10^{15}$  kg of biota in the Proterozoic (at the time of an oxygenated atmosphere). The main parameters (pressure, temperature, and state of catalytic solid phase) are estimated for these stages of the early evolution of life. It is shown that the abiogenic synthesis of the primordial matter was preformed in the Solar System on a grand scale with practically every atom in nanoparticles-catalysts participating. Selection among catalytically active nanoparticles worked towards the ability to synthesize high molecular compounds in a protoplanetary disk. Autocatalysts participated in the preplanetary chemical evolution, beginning from such simple substances as ethylene or glycolaldehyde. Primary synthesis of autocatalysts depended on external sources of energy, e.g., on ultraviolet radiation.

## Catalysis in Organic Synthesis

### ACTIVITY OF ZEOLITES IN DIMETHYL SULFIDE SYNTHESIS

A.V. Mashkina, L.N. Khairulina

*Kinet. Catal.*,  
51(4) (2010) pp. 579-583.

Dimethyl disulfide conversion into dimethyl sulfide over various zeolites in an inert medium at atmospheric pressure and  $T = 190\text{--}330^\circ\text{C}$  is reported. A significant activity in dimethyl sulfide formation is shown by the decationized zeolites HNaY and HZSM-5, whose surface has strong protonic and nonprotonic acid sites. Cobalt-containing faujasite is more active than HNaY, and the activity of CoHZSM-5 is comparable with the activity of its decationized counterpart.

### DIMETHYL DISULFIDE CONVERSION INTO DIMETHYL SULFIDE IN THE PRESENCE OF SULFIDIZED CATALYSTS

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*Kinet. Catal.*,  
51(5) (2010) pp. 724-730.

The conversion of dimethyl disulfide in the presence of various supported sulfidized metal-containing catalysts at atmospheric pressure and  $T=150\text{--}350^\circ\text{C}$  was studied. Sulfidized transition metals supported onto aluminum oxide were more active than catalysts based on a carbon support, silicon dioxide, amorphous aluminosilicate, and zeolite ZSM-5. The most active catalyst was 10% Co/Al<sub>2</sub>O<sub>3</sub> prepared with the use of cobalt acetate as an active component precursor and treated with a mixture of hydrogen sulfide with hydrogen at  $T=400^\circ\text{C}$ . From kinetic data, it follows that all of the reaction products were formed simultaneously at a temperature of  $< 200^\circ\text{C}$ , whereas a consecutive reaction scheme took place at higher temperatures. In the presence of a sulfidized alumina-cobalt catalyst, the output of dimethyl sulfide was higher than that reached with the use of other well-known catalysts.

### THE IONIC LIQUID [bmim]Br AS AN ALTERNATIVE MEDIUM FOR THE CATALYTIC CLEAVAGE OF AROMATIC C-F AND C-Cl BONDS

S.A. Prikhodko, N.Yu. Adonin, V.N. Parmon

*Tetrahedron Lett.*,  
51(17) (2010) pp. 2265-2268.

The potential of [bmim]Br as an alternative to aprotic dipolar solvents in nickel-catalyzed hydrodehalogenation reactions is demonstrated. Hydrodechlorination of pentafluorochlorobenzene proceeds under the action of zinc in aqueous [bmim]Br. Under the above conditions aromatic C-F bonds also undergo slow cleavage. The reaction is significantly accelerated in the presence of nickel complexes with 2,2'-bipyridine or 1,10-phenanthroline. In the case of pentafluoroacetanilide highly regioselective ortho-hydrodefluorination leading to the formation of 3,4,5-trifluoroacetanilide is observed.

### SYNTHESIS OF PERFLUOROALKYL(METHOXY)BORATES AND -BORANES

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*Main Group Met. Chem.*,  
32(3) (2009) pp. 109-114.

Interaction of lithium perfluoroalkyltrimethoxyborates, Li[C<sub>n</sub>F<sub>2n+1</sub>B(OMe)<sub>3</sub>] ( $n = 3, 4, 6$ ), with ClSiMe<sub>3</sub> results in formation of the corresponding esters C<sub>n</sub>F<sub>2n+1</sub>B(OMe)<sub>2</sub> in 93-95 % yields. They are sufficiently stable both in CH<sub>2</sub>Cl<sub>2</sub> and etheral solvents (ether, THF) and react with C<sub>2</sub>F<sub>5</sub>Li to give Li[(C<sub>n</sub>F<sub>2n+1</sub>)(C<sub>2</sub>F<sub>5</sub>)B(OMe)<sub>2</sub>]. Metathesis of the latter with KF leads to corresponding potassium salts.

### SYNTHESIS AND PROPERTIES OF PERFLUOROALKENYLBORONIC ACIDS AND SOME OF THEIR DERIVATIVES

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Duisburg-Essen, Duisburg, Germany)

*Main Group Met. Chem.*,  
32(3) (2009) pp. 153-159.

Lithium perfluoroalkenyltrimethoxyborates were prepared and characterised by NMR spectroscopy.

These compounds possess the properties inherent in both organic and inorganic boron-containing compounds.

$\text{Li}[\text{XCF}=\text{CFB}(\text{OMe})_3]$  ( $\text{X} = \text{F}, \text{cis-CF}_3, \text{trans-CF}_3$ ) react with  $\text{I}_2$  to give corresponding 1-iodopolyfluoroalkenes in 30-50 % yields. Interaction of  $\text{Li}[\text{XCF}=\text{CFB}(\text{OMe})_3]$  with  $\text{ClSiMe}_3$ , followed by working up with  $\text{XCF}=\text{CFLi}$  leads to formation of lithium bis(perfluoroalkenyl)dimethoxyborates. Reaction of  $\text{Li}[(\text{XCF}=\text{CF})_n\text{B}(\text{OMe})_{4-n}]$  with  $\text{K}[\text{HF}_2]$  in aqueous HF gives corresponding potassium perfluoroalkenylfluoroborates,  $\text{K}[(\text{XCF}=\text{CF})_n\text{BF}_{4-n}]$ .

### POLYFLUORINATED ORGANIC COMPOUNDS OF BORON

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*Russ. Chem. Rev.*,  
79(9) (2010) pp. 757-785.

Data on polyfluorinated organic compounds of boron published in the last decade are generalized. Methods for the synthesis, NMR spectra, and the reactivities of organoboron compounds containing one or several polyfluorinated organic groups at the boron atom are considered.

### CLEAN CATALYTIC OXIDATION OF 8-HYDROXYQUINOLINE TO QUINOLINE-5,8-DIONE WITH (BuOOH)-Bu-t IN THE PRESENCE OF COVALENTLY BOUND FePcS-SiO<sub>2</sub> CATALYSTS

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*Green Chem.*,  
12(6) (2010) pp. 1076-1082.

The clean catalytic oxidation of 8-hydroxyquinoline (8-HQ) with tert-butyl hydroperoxide to quinoline-5,8-dione (QD), a molecular framework fragment of antitumor compounds, over silica-supported iron tetrasulfophthalocyanine catalysts (FePcS) is reported. The pronounced influence of the FePcS state (monomer vs. dimer) and the support (amorphous SiO<sub>2</sub> vs. mesoporous MCM-41) on the catalytic activity and selectivity is revealed. Depending on the catalyst structure, turnover frequency values determined from the initial rates of 8-HQ consumption varied from 215 to 3570 h<sup>-1</sup>. The effects of solvent,

temperature, reagent concentrations and catalyst amounts on the substrate conversion and QD selectivity were studied to optimize the reaction conditions. With an optimal catalyst, the yield of the target product reached 66%. The truly heterogeneous nature of the catalysis was also demonstrated.

### HETEROGENEOUS SELECTIVE OXIDATION OF ALKENES TO $\alpha,\beta$ -UNSATURATED KETONES OVER COORDINATION POLYMER MIL-101

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*Adv. Synth. Catal.*,  
352(17) (2010) pp. 2943-2948.

The mesoporous metal-organic framework MIL-101 is an efficient heterogeneous catalyst for the selective allylic oxidation of alkenes with tert-butyl hydroperoxide. The selectivity towards  $\alpha,\beta$ -unsaturated ketones reaches 86–93%. The temperature of the catalyst activation strongly affects the ketone yield. MIL-101 is stable to chromium leaching, behaves as a true heterogeneous catalyst, can be easily recovered by filtration, and can be reused several times without loss of the catalytic performance.

### $\gamma$ -HYDROXYPROPYLATION OF 2,6-DIALKYL(ARYL)PHENOLS WITH ALLYL ALCOHOL AND ITS DERIVATIVES

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*Russ. J. General Chem.*,  
80(11) (2010) pp. 2290-2297.

The composition of the products of the reaction of 2,6-disubstituted phenols with allyl alcohol and its derivatives in an alkaline medium was investigated, the conditions for carrying out the reaction with the predominant formation of 4-(3-hydroxypropyl)-2,6-dialkyl(aryl)phenols were found, and its mechanism was suggested. The reaction was examined on an industrial scale. An important result is the practical demonstration of alkaline catalysis performed under homogeneous conditions with participation of phenols, when the used alkaline catalyst is recovered in the process without the formation of waste waters.



## Mathematical Simulation, Calculating Models

### MATHEMATICAL MODELING OF TOLUENE OXIDATION WITH A PERIODIC REACTOR OPERATION

S.I. Reshetnikov

*React. Kinet. Mech. Catal.*,  
99(1) (2010) pp. 23-28.

Mathematical modeling of toluene oxidation under unsteady state catalyst created by forced oscillations of concentration of toluene in the reactor inlet was carried out. It is shown that, under periodic reactor operation, the benzaldehyde yield was up to several times higher than the steady state value. This was explained by the participation of different oxygen species in the partial and total oxidation on the catalyst surface.

### A STUDY OF NICOTINIC ACID SYNTHESIS ON A PILOT INSTALLATION AND ITS SIMULATION

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N.V. Vernikovskaya, V.N. Kashkin,  
T.V. Andrushkevich

*Russ. J. Appl. Chem.*,  
83(5) (2010) pp. 846-853.

Technological process parameters of the nicotinic acid synthesis by oxidation of  $\beta$ -picoline on a vanadium-titanium catalyst in a unit tube of a pilot installation were determined: conversion of  $\beta$ -picoline, yield and selectivity for products, and parametric sensitivity of the "hot point" temperature to variation of parameters at the reactor inlet. A mathematical simulation of the process was carried using the model of heat-and-mass transfer in a bed of a tubular reactor and the kinetic model of oxidation of  $\beta$ -picoline.

### OPTIMAL CONDITIONS FOR CATALYTIC COMBUSTION OF WEAK METHANE-AIR MIXTURES IN THE SYSTEMS WITH REGENERATIVE HEAT EXCHANGE

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O.P. Klenov

*Khimicheskaya Promyshlennost' Segodnya*,  
10 (2010) pp. 34-42.

Ventilating emissions of collieries in which the maintenance of methane does not exceed 1,5 %, are offered to utilise by deep catalytic methane oxidations in devices with regenerative heat exchange. A mathematical modeling of the process is executed at concentration of methane 0,2-0,7 %, technological

parameters of a reactor for methane burning are found in an artificially created non-stationary conditions and optimum modes of its start-up.

### STABILITY PROBLEM OF CANARD-CYCLES ON A FINITE INTERVAL

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*AIP Conf. Proceed.*,  
1281(3) (2010) pp. 2181-2183.

A detailed study of two-variable mathematical model of a heterogeneous catalytic reaction is presented with special attention to the stability problem of canard-cycles on a finite interval. Analysis of the global error behavior in a long-time numerical integration shows that a high sensitive dependence on the initial conditions appears due to the existence of a shower-type bundle of trajectories which is formed by stable and unstable canard solutions.

### THERMAL TRANSFORMATIONS OF SOME MONOTERPENE COMPOUNDS IN SUPERCRITICAL LOWER ALCOHOLS

V.I. Anikeev

*Flavour Fragrance J.*,  
25(6) (2010) pp. 443-455.

This review systematizes the results of basic studies on thermal transformations of phytogetic terpene compounds ( $\alpha$ - and  $\beta$ -pinenes, turpentine, *cis*-verbenol) performed in supercritical lower alcohols. The main result is the demonstration of a considerable increase in the rate of thermal isomerization of monoterpene compounds in supercritical lower alcohols, with the retained selectivity toward target products of the reaction as compared to the rate of similar reactions conducted in the gas or liquid phase. The kinetic studies provided a deep insight into the isomerization mechanism of these compounds and elucidated the role of supercritical solvent in a manifold increase of the reaction rate compared to isomerization of the same compounds in the gas or liquid phase. The pressure of a supercritical solvent was shown to strongly facilitate the acceleration of the reactions under study. Chemical transformations of the considered terpene compounds in supercritical fluid solvents can be used as a basis of modern highly efficient technologies for

the production of a wide range of practically important organic compounds, medicinal and perfume substances.

#### THE THERMODYNAMIC CHARACTERISTICS OF MULTICOMPONENT REACTION MIXTURES IN THE SUB- AND SUPERCRITICAL STATES

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*Russ. J. Phys. Chem. B*,  
3(7) (2009) pp. 1044-1061.

The paper presents mathematical models and calculation methods for solving particular research problems related to the thermodynamic characteristics of multicomponent and multiphase mixtures. The special features of chemical and phase equilibria in such mixtures are considered in the ideal gas approximation and taking nonideality into account. The conditions of equilibrium phase stability are studied for multiphase systems. The results of calculations of characteristic phase diagrams and binodal and spinodal are given for model systems with a fixed chemical composition, and a new interpretation of the mathematical model for localizing the critical point of a multicomponent mixture with a given composition is presented. A new interpretation of the well-known classic homotopy method is suggested for solving complex nonlinear systems of equations. Some anomalies of phase portraits and critical curves that are necessary to take into account in selecting (planning) experimental conditions and calculating chemical processes and reaction parameters are considered separately. The possibility of calculating thermodynamic and thermophysical properties (entropy, enthalpy, heat capacity, heat effects of reactions, and adiabatic heating) is demonstrated for the example of particular multicomponent nonideal mixtures. The conclusion is drawn that cubic equations of state can be used for predicting the deviations of these properties from the ideal gas state and their anomalies in the vicinity of the critical points of mixtures.

#### KINETICS OF THERMAL CONVERSIONS OF MONOTERPENIC COMPOUNDS IN SUPERCRITICAL LOWER ALCOHOLS

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*Kinet. Catal.*,  
51(2) (2010) pp. 162-193.

The most important information concerning thermal conversions of vegetable terpenes ( $\alpha$ -pinene,  $\beta$ -pinene, turpentine, and *cis*-verbenol) in supercritical lower alcohols is systematized. The kinetics of selected reactions is reported and is compared with the kinetics of the same reactions in the gas and liquid phases. Thermodynamic calculations of the phase states and kinetic parameters are presented for a number of multicomponent multiphase systems containing terpenes and lower alcohols. The effect of the supercritical solvent pressure on the rate and selectivity of the selected reactions is reported.

#### CHEMICAL AND PHASE EQUILIBRIA CALCULATION OF $\alpha$ -PINENE HYDRATION IN CO<sub>2</sub>-EXPANDED LIQUID

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I.V. Kozhevnikov (\*Novosibirsk Institute of Organic  
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*J. Supercrit. Fluids*,  
51(3) (2010) pp. 295-305.

Modeling of chemical and phase equilibria of  $\alpha$ -pinene hydration in subcritical CO<sub>2</sub> was carried out by calculation. A simplified scheme of the conversion includes 6 reaction routes. Thermochemical and physicochemical parameters of individual compounds ( $T_{cr}$ ,  $P_{cr}$ ,  $T_b$ ,  $\Delta H_f^\circ$  (298.15K),  $\Delta S_f^\circ$  (298.15 K),  $C_p$  (T),  $\omega$ ) were preliminary estimated. Phase diagrams of the model reaction mixtures were calculated, coordinates of the critical point were found, and the region of subcritical parameters T and P, where the initial mixture divides into the gas phase and CO<sub>2</sub>-expanded liquid, was located. Dependence of the products distribution and yield of CO<sub>2</sub>-expanded liquid on the reaction temperature and pressure was studied. The reaction equilibrium as a function of temperature and pressure was determined, and heat effects of all reactions under consideration were calculated. The drift of critical parameters versus reaction mixture composition was examined. It was shown that during the hydration and alcohols accumulation the critical pressure of reaction mixture increases continuously, the critical temperature at first elevates and then

begins to decrease, and the phase diagrams starts to degenerate. If amount of alcohols becomes more than 80 mol%, the mixture has no critical point and cannot pass to a supercritical state.

#### KINETICS OF LIMONENE HYDROGENATION IN HIGH-PRESSURE CO<sub>2</sub> AT VARIATION OF HYDROGEN PRESSURE

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*Ind. Eng. Chem. Res.*,  
49(5) (2010) pp. 2084-2090.

A mechanism of limonene hydrogenation in high-pressure CO<sub>2</sub> has been suggested using the experimental data. The experimental data on limonene hydrogenation in high-pressure CO<sub>2</sub> were processed to elucidate the reaction mechanism and calculate the kinetic constants in rate equations for the proposed reaction scheme. The relationship between the total rate constant of limonene reduction and hydrogen pressure has been found.

#### THE THERMOLYSIS OF $\alpha$ -PINENE AND VERBENONE EPOXIDES IN SUPERCRITICAL SOLVENTS

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*Russ. J. Phys. Chem. A*,  
84(7) (2010) pp. 1112-1117.

Thermal transformations of  $\alpha$ -pinene and verbenone epoxides were studied in supercritical solvents with complex compositions, including CO<sub>2</sub>, lower alcohols (ethanol and isopropanol), and water, over the temperature and pressure ranges 387-575 K and 135-215 atm. The main product from  $\alpha$ -pinene epoxide in a supercritical solvent containing water was campholenic aldehyde and pinocamphone; the total content of these products in the reaction mixture was 80%. Suggestions concerning the mechanism of the thermal isomerization of  $\alpha$ -pinene epoxide depending on the acidity of supercritical solvents were made. The direction of verbenone epoxide transformations was independent of the presence of water in the mixture. The main identified products were ketoalcohols with para-menthane and camphane frameworks.

#### REACTIVITY OF $\alpha$ -PINENE EPOXIDE IN SUPERCRITICAL SOLVENTS

V.I. Anikeev, I.V. Il'ina\*, K.P. Volcho\*, An. Yermakova, N.F. Salakhutdinov\* (\*Novosibirsk Institute of Organic Chemistry, Novosibirsk, Russia)

*J. Supercrit. Fluids*,  
52(1) (2010) pp. 71-75.

Thermal transformations of  $\alpha$ -pinene epoxide in composite supercritical solvents that contain CO<sub>2</sub>, lower alcohols (ethanol, isopropyl alcohol) and water were studied in the temperature range of 387–575 K at pressure 13.5–21.5 MPa. Campholenic aldehyde and carveol were shown to be the main products of  $\alpha$ -pinene epoxide reactions in supercritical solvents containing water. In the absence of water, thermolysis of  $\alpha$ -pinene epoxide in supercritical solvent yields campholenic aldehyde and pinocamphone, with their total amount in the reaction mixture attaining 80%. Suggestions were made on the mechanism of  $\alpha$ -pinene epoxide thermal isomerization depending on acidity of supercritical solvent.

#### A PARALLEL ALGORITHM FOR SOLVING THE GRAVITATIONAL PHYSICS PROBLEMS BASED ON DOMAIN DECOMPOSITION

E.A. Kuksheva, V.N. Snytnikov

*Numer. Meth. Programm.*,  
11(1) (2010) pp. 168-175.

A parallel algorithm for solving the unsteady 3D problems of collisionless dynamics in the case of more than 10<sup>9</sup> bodies in a self-consistent gravitational field in Cartesian coordinates is proposed. The algorithm is based on the computational domain decomposition method. The parallel code that implements this algorithm can be used to perform computations on grids of more than 1000<sup>3</sup> nodes. Some results of supercomputer simulation obtained by the code are discussed.

#### AN EXPLICIT MULTI-STEP ALGORITHM FOR THE SIMULATION OF SELF-GRAVITATING GAS DYNAMICS

O.A. Stadnichenko, V.N. Snytnikov

*Numer. Meth. Programm.*,  
11(1) (2010) pp. 53-67.

For the self-gravitating gas dynamics simulation, an explicit multi-step algorithm is proposed. A spatial three-dimensional programming code on a Cartesian grid in the Euler variables was developed using the Fluid-in-Cell method. The numerical algorithm has the first-order approximation. The corresponding grid

viscosity provides the stability of numerical solutions. Computations for a sequence of grid refinements show the convergence of the algorithm. Results of isothermal collapse simulation, isentropic self-gravitating gas rotations, and its self-similar expansion are discussed. The computations were performed at the Siberian Supercomputer Center using a common memory computer SMP16x256.

### **INSTABILITY OF A NONUNIFORM COMPRESSIBLE GAS WITH SELF-GRAVITY**

**V.N. Snytnikov, O.A. Stadnichenko**

*AIP Conference Proceedings*,  
1242 (2010) pp. 355-362.

For the case of nonuniform gas with viscosity and gravity, a dispersion relation was obtained for the perturbations whose wave vector is directed along the density gradient. The gravitational-convective perturbations were shown to be locally unstable in a non-viscous isothermal gas over the entire spectrum of wave vectors. This leads to instability of the gas stationary distributions. Solving the initial-boundary value problems of non-stationary dynamics of isothermal gas requires regularization by, e.g., viscosity. Viscosity does not hinder the growth of the Jeans part of gravitational-convective instability. Numerical experiments based on a completely three-dimensional transient code demonstrated the development of collapse in isothermal cloud.

### **FEATURES OF SPH GAS DYNAMICS FOR MODELING OF NONLINEAR GRAVITATIONAL WAVES IN MULTIPHASE MEDIUM**

**O.A. Stoyanovskaya, V.N. Snytnikov**

*Matem. Mod.*,  
22(5) (2010) pp. 29–44.

SPH is widely adopted to treat gas dynamics in astrophysical calculations. One of the most state-of-art problems is connected with testing codes based on this method and verification of nonstationary solutions provided by the codes. Description of developed SPH-code Sombrero and results of modeling of axisymmetrical and radial-azimuth perturbations spreading in multiphase gravitational medium are presented in the article. By means of comparison of numerical results obtained using SPH and FLIC methods, the ability of SPH to reproduce nonlinear waves in gas-collisionless bodies' medium was demonstrated even if shear and cross-current flows take place in the system.

### **COMPUTER SIMULATION OF CHEMICAL PROCESSES AND FLUID FLOWS IN CHEMICAL REACTORS**

**I.G. Chernykh, T.I. Mishchenko, V.I.N. Snytnikov, V.N. Snytnikov**

*AIP Conf. Proceed.*,  
1233(2) (2010) pp. 1418-1422.

The optimization of kinetic scheme of chemical process is the important stage of computer simulation of chemical processes. This iterative method consists from the next steps: proposition of scheme from theory, translation scheme to the system of ordinary differential equation (ODE), finding solution of this system, comparison results with experimental data, modification of kinetics scheme. ChemPAK software package was created for automation of this process. ChemPAK consists of network database, computational modules and visual interface. User can create and edit systems of chemical equations with unlimited number of equations with easy-to-use interface, translate systems of chemical equations to systems of ODE, add some new equations to the translated system and solve this system by using one of ChemPAK computational modules. Optimized kinetics scheme can be used in FLUENT software for modelling fluid flows, heat transfer and chemical reactions. ChemPAK creates FLUENT database file with chemical reactions kinetics scheme.

The results of 3D modelling of gas-dynamics reactant flows with heat transfer from laser energy are presented by example of the C<sub>2</sub>-hydrocarbons conversion task. Kinetics scheme was optimized with using ChemPAK software package. CFD with chemical reactions effect modelling was produced with using FLUENT software.

### **AUTOCATALYTIC TRANSFORMATION OF ETHANE TO ETHYLENE USING CO<sub>2</sub>-LASERS**

**V.N. Snytnikov, T.I. Mishchenko, V.I.N. Snytnikov, O.P. Stoyanovskaya, V.N. Parmon**

*Problems and Achievements of Applied Mathematics and Mechanics: on occasion of 70th anniversary of Academician V.M. Fomin*, Novosibirsk, Parallel, 2010, pp. 564-574 (650 pp.).

Gas-phase thermal pyrolysis of ethane was studied at volume heating of the reaction mixture using radiation of a continuous CO<sub>2</sub>-laser with the energy absorbed by unsaturated hydrocarbons. Experimental data on the composition of ethane dehydrogenation products differs noticeably from the composition calculated using the HYSYS program package for conditions of

thermodynamic equilibrium. To describe the pyrolysis process, the ethylene-autocatalytic mechanism is suggested for ethane dehydrogenation. The mechanism involves the reaction of ethane with ethylene to give methyl and propyl radicals.

#### **AUTOCATALYTIC GAS-PHASE ETHANE DEHYDROGENATION IN A WALL-LESS REACTOR**

**V.N. Snytnikov, T.I. Mishchenko,  
V.I. Snytnikov, O.P. Stoyanovskaya,  
V.N. Parmon**

*Kinet. Catal.*, 51(1) (2010) pp. 10-17.

The thermal gas-phase pyrolysis of ethane was studied under conditions of the bulk heating of the

reaction mixture with IR-laser radiation. The concentrations of ethane pyrolysis products as functions of reaction time were calculated in accordance with standard kinetic schemes; they showed that a classical radical chain mechanism corresponded to only highly dilute mixtures of ethane with an inert gas. As found by calculations, the experimental data on the kinetics of consumption of the initial substance and on the kinetics of buildup of pyrolysis products in undiluted mixtures of ethane and its conversion products were adequately described by an autocatalytic (with respect to ethylene) mechanism of ethane dehydrogenation. This mechanism involved the step of ethane interaction with ethylene to form methyl and propyl radicals.

## **Fuel Chemistry and Technology**

#### **HYDROCONVERSION OF SUNFLOWER OIL ON Pd/SAPO-31 CATALYST**

**O.V. Kikhtyanin, A.E. Rubanov, A.B. Ayupov,  
G.V. Echevsky**

*Fuel*,  
89(10) (2010) pp. 3085-3092.

This work presents results from the hydroconversion of sunflower oil on the bifunctional Pd/SAPO-31 catalyst as a perspective technological way for single-stage production of hydrocarbons in the diesel fuel range that have improved low-temperature properties. Transformation of sunflower oil was performed at temperatures of 310–360°C and  $WHSV = 0.9\text{--}1.6\text{ h}^{-1}$ , under a pressure of 2.0 MPa in a laboratory flow reactor. Gaseous and liquid reaction products were analyzed by GC using an internal standard method as well as by  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectroscopy. At temperatures 320–350°C, liquid reaction product contained only hydrocarbons, the main components were identified as  $\text{C}_{17}$  and  $\text{C}_{18}$  *n*-alkanes and *i*-alkanes. Pd/SAPO-31 catalyst demonstrated high initial activity for the hydroconversion of the feed and good isomerizing properties, but its deactivation was followed after several hours of operation. Physico-chemical properties of both fresh and spent catalysts were compared. The influence of reaction conditions on the composition of the reaction products is also discussed.

#### **REMOVAL OF NITROGEN COMPOUNDS FROM LIQUID HYDROCARBON STREAMS BY SELECTIVE SORPTION ON METAL-ORGANIC FRAMEWORK MIL-101**

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*Mendeleev Commun.*,  
20(1) (2010) pp. 57-58.

High sorption capability and selectivity towards N-containing compounds were observed for metal-organic framework MIL-101 under the sorption from light cycle oil and model mixtures.

#### **MODERN CATALYSTS OF DEEP HYDROTREATMENT IN THE PRODUCTION OF LOW-SULFUR DIESEL FUELS AT RUSSIAN OIL REFINERIES ACCORDING TO EURO-3 AND EURO-4 STANDARDS**

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G.A. Bukhtiyarova, V.N. Kashkin, A.S. Noskov,  
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Moscow, Russia)**

*Catal. Ind.*,  
2(2) (2010) pp. 101-107.

The production of diesel fuels with sulfur contents according to Euro-3 and Euro-4 standards at Russian oil refineries requires the replacement of catalysts by more active ones without reconstructing the existing hydrotreating units. The majority of Russian catalysts can be used for the production of diesel fuels containing 350 ppm S and more. For the production of fuels with less than 50 and 10 ppm residual sulfur, however, last generation catalysts should be used,

whose surface compounds are highly active Co(Ni)-Mo-S phases of the second type. A typical representative of these catalysts is the Russian IC-GO-1 catalyst, which has been manufactured since 2007 by ZAO Industrial Catalysts under the license of Boreskov Institute of Catalysis. The characteristics of hydrotreatment ensured by the developer and producer were confirmed during the commercial use of the catalyst at OAO Saratov Oil Refinery. The production of IC-GO-1 can reach 600 t per year, which is sufficient for the re-equipment of 10 standard Russian hydrotreating units.

#### **COMPARATIVE RESEARCH OF NiMo/Al<sub>2</sub>O<sub>3</sub> AND CoMo/Al<sub>2</sub>O<sub>3</sub> NEW GENERATION CATALYSTS IN REACTIONS OF HYDRODESULPHURIZATION AND HYDRODENITROGENATION OF VACUUM GASOIL**

**P.V. Aleksandrov, V.N. Kashkin,  
G.A. Bukhtiyarova, A.L. Nuzhdin,  
A.V. Pashigreva, O.V. Klimov, A.S. Noskov**

*Neftepererabotka i Neftekhimiya,*  
9 (2010) pp. 3-9.

Novel CoMo/Al<sub>2</sub>O<sub>3</sub> and NiMo/Al<sub>2</sub>O<sub>3</sub> hydrotreating catalysts were compared in vacuum gasoil hydrotreating in a pilot plant over a wide range of operating conditions. The kinetic model for the hydrodesulfurization and hydrodenitrogenation reactions of VGO was developed using the obtained experimental data. The proposed kinetic model allows to forecast the catalysts performance in the whole range of working conditions and to select the optimal operating conditions of the process required to obtain products with a specified residual sulfur content. The obtained results suggest that the hydrotreating catalyst developed at the Institute of Catalysis is more active in vacuum gasoil hydrotreating comparing with the catalysts for this process manufacturing in Russia, and also is not inferior to the activity of foreign analogues.

#### **OPTIMIZATION OF A METHOD OF PREPARATION AND REGENERATION OF THE CATALYST IC-GO-1 FOR DEEP HYDROPURIFICATION OF DIESEL FUEL**

**O.V. Klimov, G.A. Bukhtiyarova,  
A.V. Pashigreva, S.V. Budukva, S.N. Kirichenko,  
A.S. Noskov**

*Neftepererabotka i Neftekhimiya,*  
3 (2010) pp. 33-37.

A preparation method of the Russian catalyst IC-GO-1 for deep hydrotreatment, based on the use of bimetallic complexes is described. The deposition of the complexes onto alumina supports, prepared by

different ways, allows to obtain the catalysts for the production of diesel fuel with sulfur content less than 50 ppm. Different version of the IC-GO-1 can be used for hydrotreatment of secondary middle distillates and heavier product — vacuum gasoil. Hydrotreating of vacuum gasoil using IC-GO-1 allows to obtain a product with a residual sulfur content less than 500 ppm and the hydrodenitrogenation degree exceeding 80%. The regeneration of the deactivated catalyst IC-GO-1 was studied as well. It was demonstrated that an oxidative regeneration does not allow to completely restore the catalytic activity. The treating of regenerated catalyst with chelating agents leads to restoration of the initial catalyst activity level by more than 99%.

#### **NICKEL CATALYSTS FOR THE HYDRODEOXYGENATION OF BIODIESEL**

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M.Yu. Lebedev, V.M. Novopashina, V.G. Sister\*,  
A.I. Yakimchuk\*\*, V.A. Yakovlev (\*Moscow State  
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Electrochemistry, Moscow, Russia)**

*Kinet. Catal.,*  
51(5) (2010) pp. 704-709.

A number of nickel and nickel-copper catalysts for the hydrodeoxygenation of fatty acid esters (biodiesel) were studied. The CeO<sub>2</sub> and ZrO<sub>2</sub> oxides and the CeO<sub>2</sub>-ZrO<sub>2</sub> binary system were used as supports. The Ni-Cu/CeO<sub>2</sub>-ZrO<sub>2</sub> catalyst exhibited the highest activity; it allowed to quantitatively convert biodiesel into linear alkanes under mild conditions (290-320°C, 1.0 MPa). It was found that the selectivity of the formation of the main product (heptadecane) was 70-80%. The main correlations between the nature of catalysts and their activity under conditions of the target reaction were determined using temperature-programmed reduction, X-ray diffraction analysis, and electron microscopy. It was hypothesized that the high activity of Ni-Cu/CeO<sub>2</sub>-ZrO<sub>2</sub> in the test reaction can be explained by the presence of a Ni<sub>1-x</sub>Cu<sub>x</sub> (x = 0.2-0.3) solid solution as a constituent of the active component of the catalyst.

## GUAIACOL HYDRODEOXYGENATION OVER Ni-CONTAINING CATALYSTS

M.V. Bykova, O.A. Bulavchenko, D.Yu. Ermakov, M.Yu. Lebedev, V.A. Yakovlev, V.N. Parmon

*Catal. Ind.*,  
2(4) (2010) pp. 44-51.

A series of Ni-containing catalysts were investigated in the reaction of guaiacol hydrodeoxygenation. Guaiacol is a model compound products of fast biomass pyrolysis. The reaction was carried out in an autoclave at temperature 320°C and hydrogen pressure 17 MPa. The main products of guaiacol hydrodeoxygenation were cyclohexane, 1-methylcyclohexanediol-1,2 and cyclohexanone (products of aromatic ring reduction). Scheme of transformations of guaiacol, which explains the formation of the main products, is offered. The most active are Ni-containing catalysts on SiO<sub>2</sub> and SiO<sub>2</sub>-ZrO<sub>2</sub>, prepared by the sol-gel method. According to XRD and EM, high activity of these catalysts is due to high content of active component (nickel) in the dispersed form of restored films on the surface of silicate structures. There is assumption about the prospects of the proposed catalysts in the refining process of fast pyrolysis of biomass products for fuel hydrocarbon.

## DEVELOPMENT OF A PROCESS FOR REFINING RAPE SEED OIL INTO BIODIESEL AND HIGH-CETANE COMPONENTS OF DIESEL FUEL

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*Chem. Technol. Fuels Oils*,  
46(1) (2010) pp. 1-8.

New technology for refining vegetable oil (in particular, rapeseed oil) is proposed; a distinctive feature is the use of one transesterification reactor with a stationary bed of heterogeneous catalyst and an additional stage of hydrotreating of part of the biodiesel to obtain green diesel (high-cetane hydrocarbons) that can be used as an ameliorant for traditional diesel fuels is proposed. The material and heat balances were calculated for the proposed technology.

## A STUDY OF CHEMICAL TRANSFORMATIONS OF ORGANIC COMPOUNDS UNDER THE ACTION OF CAVITATION

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*Russ. J. Phys. Chem. B*,  
4(2) (2010) pp. 227-234.

The action of ultrasonic and hydrodynamic cavitation on the cracking of organic compounds of various natures (linear alkanes (octane, undecane, and hexadecane), carboxylic acids (decanoic and oleic acids), alcohols (decanol), and esters (ethyl caprate)) and transesterification of rapeseed oil and methanol and ethanol was studied. An analysis of hydrocarbon transformation products by gas chromatography, high-performance liquid chromatography, NMR, and chromatography-mass spectrometry showed that cavitation treatment of any class of organic compounds of those mentioned above resulted in transformations no more than 1.1% deep. In experiments with transesterification of rapeseed oil and methanol and ethanol without a catalyst under the action of cavitation, the yield of fatty acid esters did not exceed 2.1 mol %. The energy efficiency of the cavitation apparatus was estimated at 0.002% for endothermic cracking of alkanes. The data obtained led to conclude that the use of cavitation directly for performing cracking of various organic compounds and noncatalytic transesterification of vegetable oils was ineffective.

## DEOXYGENATION OF DODECANOIC ACID UNDER INERT ATMOSPHERE

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*Fuel*,  
89(8) (2010) pp. 2033-2039.

A laboratory-scale fixed bed reactor (down-flow) was developed for the deoxygenation of dodecanoic (lauric) acid under inert atmosphere. This reaction can be utilized to produce renewable diesel components. The main products were undecane (decarboxylation) and undecene (decarbonylation). The catalysts were a

palladium catalyst supported on mesoporous carbon, Sibunit (egg-shell type with spherical granules of 1.6-2.0 mm in diameter), and a commercial Pd/C catalyst. The challenge was to obtain a high activity and selectivity to undecane, as pure dodecanoic acid without any solvents was used. Moreover, the complete reactor system consisting of a reactant feed vessel, a pump, lines, a sampling valve, and a collector of the residue had to be heated, since saturated fatty acids with more than nine carbon atoms have a melting point exceeding 20°C. The influence of reaction conditions, such as reaction temperature (300-360°C), pressure (5-20 bar), gas flow rate, reactant concentrations, was studied. The reactant conversion increased when an inert gas (argon) flow was applied co-currently with the reactant flow. The argon pressure did not have any significant impact on the performance. The reason for the catalyst deactivation was determined to be coking.

#### CONTINUOUS MODE LINOLEIC ACID HYDROGENATION ON Pd/SIBUNIT CATALYST

**An. Bernas\***, **I.L. Simakova**, **K. Eränen\***, **J. Myllyoja\*\***, **T. Salmi\***, **D.Yu. Murzin\***  
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 \*\*Neste Oil Oyj, Finland)

*Catal. Ind.*,  
 2(2) (2010) pp. 95-100.

Catalytic hydrogenation of linoleic acid and oleic acid to stearic acid over palladium on mesoporous carbon sibunit (Pd/sibunit) catalyst was studied in a continuous trickle-bed reactor with the weight hourly space velocity 5.4 h<sup>-1</sup> at 120°C and 30 bar using tall oil fatty acid (TOFA) as reactor feed. Stearic acid preparation using TOFA as a raw material is of industrial importance. Pd/Sibunit catalysts with spherical particle shape of the size 1.62 mm were synthesized with the palladium loadings 0.5, 1, and 2 wt %. The metal dispersion (%), metal particle size (nm), as well as metallic surface area (m<sup>2</sup>/g metal) of the three synthesized Pd/Sibunit samples were measured by CO chemisorption. The Pd/C catalysts were tested in linoleic acid hydrogenation, showing promising behavior in terms of activity, selectivity and stability to be used in fixed bed applications. The product stream from the fixed bed reactor was saved and analyzed by X-ray fluorescence (XRF) and direct current plasma (DCP) spectroscopy. The catalyst activity increased with the Pd loading. The lowest metal loading of 0.5 wt % gave the most stable catalyst. This catalyst can be recommended for further pilot testing.

#### CATALYTIC DEOXYGENATION OF TALL OIL FATTY ACIDS OVER A PALLADIUM-MESOPOROUS CARBON CATALYST: A NEW SOURCE OF BIOFUELS

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*Top. Catal.*,  
 53(15-18) (2010) pp. 1274-1277.

Catalytic deoxygenation of tall oil fatty acids (TOFA) was demonstrated over palladium catalysts supported on mesoporous carbon at 300°C using dodecane as a solvent. Maximally 95% selectivity to linear C<sup>17</sup> hydrocarbons was achieved. The effects of reaction atmosphere and initial TOFA concentration were investigated.

#### DECARBOXYLATION OF FATTY ACIDS OVER Pd SUPPORTED ON MESOPOROUS CARBON

**I.L. Simakova**, **O.A. Simakova**, **P. Maki-Arvela\***, **D.Yu. Murzin\*** (\*Åbo Akademy University, Åbo-Turku, Finland)

*Catal. Today*,  
 150(1-2, sp. issue) (2010) pp. 28-31.

Fatty acid decarboxylation was studied in a semibatch reactor over 1 wt.% Pd/C (Sibunit) using five different fatty acids, C<sub>17</sub>-C<sub>20</sub> and C<sub>22</sub>, as feeds. The same decarboxylation rates were obtained for pure fatty acids, whereas extensive catalyst poisoning and/or sintering and coking occurred with low purity fatty acids as reactants. One reason for catalyst poisoning using behenic acid (C<sub>22</sub>) as a feedstock was its high phosphorus content. The decarboxylation rate of fatty acids decreased also with increasing fatty acid to metal ratio.

#### LIQUID PRODUCTS OF THE HYDROLYSIS OF BROWN COAL FROM THE LENA BASIN

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*Solid Fuel Chem.*,  
 44(3) (2010) pp. 169-174.

Liquid hydrocarbon products were obtained by the hydrolysis of brown coal from a deposit in the northern Lena basin on an iron-containing catalyst. The individual and group compositions of gasoline and diesel fractions were determined with the use of



capillary chromatography and chromatography-mass spectrometry. The gasoline fraction with a boiling point to 180°C was characterized by a high octane number; it mainly contained monocyclic aromatic hydrocarbons and normal alkanes. The diesel fraction mainly consisted of bi- and tricyclic aromatic hydrocarbons and C<sub>13</sub>-C<sub>19</sub> n-alkanes.

## MODIFICATION OF THE ORGANIC MATTER OF BOGHEAD WITH NITROUS OXIDE

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*Solid Fuel Chem.*,  
44(6) (2010) pp. 414-418.

The modification of the organic matter of boghead with nitrous oxide at elevated temperature and pressure was studied. It was found that unselective carbonylation observed in reactions with synthetic polymers was due to the irregular structure of the polymer-like sapropelite kerogen.

## Chemical Engineering

### ELABORATION AND REALISATION OF INNOVATIVE PROJECTS BY "TOMSKNEFTEKHIM" Ltd. AND INSTITUTE OF CATALYSIS OF SB RAS IN FRAMES OF SPECIAL ECONOMIC ZONE

**E.A. Maier\***, **V.K. Dudchenko\***, **K.M. Kolkov\***, **O.L. Arkatov\***, **V.A. Zakharov**, **V.E. Nikitin**, **T.B. Mikenas** (\**Tomskneftekhim" Ltd., Tomsk, Russia*)

*Russ. J. Appl. Chem.*,  
83(3) (2010) pp. 579-582.

The innovative project on elaboration of modern Russian UHMPE production technology, which allows obtaining a wide brand assortment of this polymer, became the result of collaboration of "Tomskneftekhim" Ltd. and Institute of Catalysis. UHMPE testing lots are accumulated and by different consumers successfully tested. Obtained results are the basis for creation of UHMPE industrial production and widespread introduction of this new polymer on Russian market.

### PRECIOUS METALS IN CATALYST PRODUCTION

**V.N. Parmon**, **V.I. Simagina**, **L.P. Milova**

*Catal. Ind.*,  
2(3) (2010) pp. 199-205.

The present and prospective use of precious metals (platinum and palladium) in catalyst production is analyzed with allowance made for the appearance of new catalytic technologies. The production of precious metal catalysts in Russia is now being curtailed. Russian companies are reconstructing plants that employ precious metal catalysts and building new ones in cooperation with foreign partners who will provide advanced technologies and the necessary

catalysts. The proportion of imported palladium- and platinum-containing catalysts in Russia remains rather high (as much as 60%). The consumption of precious metals in organic synthesis and in the pharmaceutical industry remains at the level of the 1990s. In this overview, the possibilities are analyzed for the wider use of platinum in the design of novel catalysts that could compete with imported analogues, including those for petroleum refining, petroleum chemistry, organic synthesis, and environmental applications. New areas for Russia: nanocomposites for hydrogen energy, new drugs, and membrane technologies are also covered.

### PYROLYSIS OF LIQUID HEXADECANE WITH SELECTIVE MICROWAVE HEATING OF THE CATALYST

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*Theor. Exp. Chem.*,  
46(6) (2010) pp. 384-392.

The pyrolysis of liquid n-hexadecane was studied on various catalysts with selective microwave (MW) heating of a catalyst possessing much greater microwave absorption capacity than the long-chain hydrocarbon studied. This method permits rapid heating of the catalyst to temperatures much higher than 400°C, leading to reflux of the liquid substrate, movement of the catalyst granules within the substrate, and chemical transformations (cracking) of hexadecane. High pyrolysis selectivity relative to  $\alpha$ -olefins was found on various catalysts such as magnetic microspheres (coal combustion ash), Al<sub>2</sub>O<sub>3</sub>/Al, and Pd/KTP (glass fiber). This behavior

may be attributed to tempering of the primary products in the bulk of the liquid reagent. Furthermore, MW pyrolysis on magnetic microspheres was found to be accompanied by formation of rather thick carbon microfibers with diameter 300–500 nm.

#### **OXIDATIVE DESTRUCTION OF CHLORINATED HYDROCARBONS ON Pt-CONTAINING FIBER-GLASS CATALYSTS**

**E.A. Paukshtis, L.G. Simonova, A.N. Zagoruiko, B.S. Bal'zhinimaev**

*Chemosphere*,  
79(2) (2010) pp. 199-204.

Novel catalysts comprising noble metals (Pt), supported on fiber-glass woven materials demonstrated efficient oxidation of different chlorinated hydrocarbons (chlorobenzene, dichloroethane, dichloropropane, butyl chloride) and industrial mixed chlorinated organic wastes into HCl, CO<sub>2</sub> and H<sub>2</sub>O at moderate temperatures without formation of highly toxic by-products (dioxins, phosgene, polychlorinated hydrocarbons, elemental chlorine). The highest oxidation activity and selectivity was observed for the platinum catalysts produced from fiber-glass supports with increased acidity and with additional introduction of promoting elements (such as Co, Mn and Cu). Long-term tests (more than 100 h) have shown no deactivation of the said catalysts. In combination with competitive catalyst price (due to the extra-low content of Pt - below 0.05% mass) it opens the way for development of highly efficient and feasible technology for utilization and detoxication of various chloro-organic wastes.

#### **GLASS-FIBER CATALYSTS: NOVEL OXIDATION CATALYSTS, CATALYTIC TECHNOLOGIES FOR ENVIRONMENTAL PROTECTION**

**B.S. Bal'zhinimaev, E.A. Paukshtis, S.V. Vanag, A.P. Suknev, A.N. Zagoruiko**

*Catal. Today*,  
151(1-2) (2010) pp. 195-199.

Novel glass-fiber based catalysts containing extra-low amounts of noble metals (0.01–0.02 mass% of Pt or Pd) demonstrate unique performance in many catalytic reactions, resulted from the ability of the glass fibers to stabilize the transient metals in the glass bulk in a highly dispersed form. Moreover, specific heat/mass transfer properties,

original geometry, high flexibility and high mechanical strength of such catalysts give the way to develop really new catalytic processes and novel reactor designs. The paper is dedicated to the successful research and development (from fundamental research issues to pilot and semi-industrial tests) of different catalytic oxidation processes based on application of fiber-glass catalysts for solution of various environmental problems, namely: purification of automotive exhausts, incineration of VOCs (volatile organic compounds) in waste/vent gases, utilization of chlorinated VOCs and dioxins abatement in waste gases, sulphur dioxide oxidation for purification of waste gases and for conditioning of flue gases of coal-fired powerplants.

#### **GLASS FIBER MATERIALS AS A NEW GENERATION OF STRUCTURED CATALYSTS**

**B.S. Bal'zhinimaev, E.A. Paukshtis, O.B. Lapina, A.P. Suknev, P.E. Mikenin, A.N. Zagoruiko**

*Stud. Surf. Sci. Catal.*,  
175 (2010) pp. 43-50.

Molecular structure of Zr-silicate glass fiber materials was studied to evaluate their potentiality in catalysis. Basing on NMR and IRS data the framework structure where Zr(IV) cations serve as a connectors linked with a few SiO<sub>4</sub> tetrahedra was proposed. The effective ways of transition ions (Pt, Pd, Co) incorporation into the glassmatrix and their stabilization in highly dispersed state (clusters) were found. The obtained glass fiber based catalysts showed high activity and selectivity in oxidation of hydrocarbons and selective hydrogenation of acetylene-ethylene feedstock. The example of successful design of structured bed and commercialization of VOC removal process is presented.

#### **SELECTIVE METHANATION OF CO IN THE PRESENCE OF CO<sub>2</sub> IN HYDROGEN-CONTAINING MIXTURES ON NICKEL CATALYSTS**

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*Kinet. Catal.*,  
51(6) (2010) pp. 907-913.

The screening of commercial nickel catalysts for methanation and a series of nickel catalysts supported on CeO<sub>2</sub>,  $\gamma$ -Al<sub>2</sub>O<sub>3</sub>, and ZrO<sub>2</sub> in the reaction of

selective CO methanation in the presence of CO<sub>2</sub> in hydrogen-containing mixtures (1.5 vol % CO, 20 vol % CO<sub>2</sub>, 10 vol % H<sub>2</sub>O, and the balance H<sub>2</sub>) was performed at the flow rate WHSV = 26 000 cm<sup>3</sup> (g cat)<sup>-1</sup> h<sup>-1</sup>. It was found that commercial catalytic systems like NKM-2A and NKM-4A (NIAP-07-02) were insufficiently effective for the selective removal of CO to a level of <100 ppm. The most promising catalyst is 2 wt % Ni/CeO<sub>2</sub>. This catalyst decreased the concentration of CO from 1.5 vol % to 100 ppm in the presence of 20 vol % CO<sub>2</sub> in the temperature range of 280–360°C at a selectivity of >40%, and it retained its activity even after contact with air. The minimum outlet CO concentration of 10 ppm at 80% selectivity on a 2 wt % Ni/CeO<sub>2</sub> catalyst was reached at a temperature of 300°C.

#### DEVELOPMENT OF GRANULAR CATALYSTS AND NATURAL GAS COMBUSTION TECHNOLOGY FOR SMALL GAS TURBINE POWER PLANTS

**Z.R. Ismagilov, M.A. Kerzhentsev, S.A. Yashnik, N.V. Shikina, A.N. Zagoruiko, V.N. Parmon, V.M. Zakharov\*, B.I. Braynin\*, O.N. Favorski\***  
 (\*Central Institute of Aviation Motors, Moscow, Russia)

In “Gas Turbines”, Sciyo, Ed. I. Gurrappa, 2010, ch. 4, pp. 79-108 (364 pp.).

The results are presented on development and study of alternative granular catalysts with reduced Pd content for methane catalytic combustion in mini gas turbines of 400-500 kW power with regenerative cycle, intended for decentralized power supply. The small power of these turbines result in reduced catalyst loading and makes possible the use of granular catalysts which are less expensive and can be easily manufactured existing industrial facilities.

#### CONTROL OF THE THICKNESS OF MESOPOROUS TITANIA FILMS FOR APPLICATION IN MULTIPHASE CATALYTIC MICROREACTORS

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*J. Catal.*,  
 271(2) (2010) pp. 161-169.

A new method of sol-gel polymer template synthesis of mesoporous catalytic thin films has been

proposed which allows controlling the chemical nature of the film, the porosity, thickness and loading with an active species. The mesoporous films with a long-order structure can be obtained in a narrow range of surfactant-to-metal precursor molar ratios from 0.006 to 0.009. The catalytic film thickness was varied from 300 to 1000 nm while providing a uniform catalyst distribution with a desired catalyst loading (1 wt. % Au nanoparticles) throughout the film. The films were characterized by TEM, SEM, ethanol adsorption and contact angle measurements. The calcination of the as-synthesized films at 573 K reduced Ti<sup>4+</sup> sites to Ti<sup>3+</sup>. A 300 nm thick Au-containing film showed an initial TOF of 1.4 s<sup>-1</sup> and a selectivity towards unsaturated alcohols as high as 90% in the hydrogenation of citral. Thicker films demonstrated a high selectivity towards the saturated aldehyde (above 55%) and a lower intrinsic catalytic activity (initial TOF of 0.7–0.9 s<sup>-1</sup>) in the absence of internal diffusion limitations.

#### SYNTHESIS OF OZONE-SAFE FREONS AND METHODS FOR IMPROVING THE RUSSIAN INDUSTRIAL CATALYST FOR THEIR PRODUCTION

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*Catal. Ind.*,  
 2(4) (2010) pp. 307-304.

Industrially important reactions of gas-phase synthesis of ozone-safe freons of the ethane family - hydrofluorination of perchloroethylene into pentafluoroethane (Halocarbon-125) and trichloroethylene into 1,1,1,2-tetrafluoroethane (Refrigerant-134a) on an Cr-Mg catalyst were investigated to improve the Russian industrial Cr-Mg catalyst for freon production. The influence of reaction conditions — temperature, contact time, pressure, etc. — on the rate and selectivity of reactions was studied. The mechanism of reactions is discussed on the basis of the dependences obtained with regard to multistage nature of processes. The computational method for assessing the optimal content of the active component and specific surface were used. The optimal content of the active component and catalyst activity were shown to increase with increasing specific surface of the support, MgF<sub>2</sub>. Thus, an increase in the specific surface of MgF<sub>2</sub> from 100 to 200 m<sup>2</sup>/g may result in increased activity by a factor of almost 2. However, obtaining such highly disperse MgF<sub>2</sub> is difficult; moreover, the catalyst would be

poorly thermostable. Therefore, the possibilities of considerable enhancing the activity of industrial Cr-Mg catalysts are limited by the existing technology. Significant enhancement in the properties of the catalyst of gas-phase synthesis of freons is possible upon a change in the method of preparing the catalyst and the chemical composition of the support.

#### **CATALYTIC PROCESSES AND CATALYSTS FOR PRODUCTION OF ELEMENTAL SULFUR FROM SULFUR CONTAINING GASES**

**A.N. Zagoruiko, G.A. Bukhtiyarova,  
V.V. Shinkarev, S.V. Vanag**

*Catal. Ind.*,  
2(4) (2010) pp. 343-352.

The modern technologies for production of elemental sulfur are considered. It is demonstrated that along with the further wide application of the conventional Claus process with conventional alumina catalyst in the observable future some new trends which may significantly influence the technological picture of recovered sulfur manufacturing may be formulated: active development of Claus tail gas cleanup processes with the stress on replacement of subdewpoint Sulfreen-type processes by processes of hydrogen sulfide selective oxidation by oxygen; development of novel highly-efficient technologies for hydrogen sulfide decomposition to sulfur and hydrogen; application of new catalysts forms, first of all — at microfiber supports for Claus and H<sub>2</sub>S oxidation processes; wider application of titania and vanadia catalysts at the newly constructed Claus units; development of technologies and catalysts for direct purification of H<sub>2</sub>S-containing gases and for catalytic reduction of SO<sub>2</sub> for sulfur recovery from smelter gases. All these prospective routes are actively developed by Russian science and some of them are completely based on domestic developments in this area.

#### **THE PROCESS FOR CATALYTIC INCINERATION OF WASTE GAS ON IC-12-S102 PLATINUM GLASS FIBER CATALYST**

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*Catal. Ind.*,  
2(2) (2010) pp. 113-117.

The process for catalytic afterburning of volatile organic compounds (VOCs) in waste industrial gases was developed on the basis of a new platinum glass fiber catalyst (GFC) IC-12-S102 with low platinum

content (~0.02 wt %). The catalyst was shown to be more effective than the known industrial afterburning catalysts. The way of glass fiber catalyst loading to a reactor in the form of vertical spiral cartridges, structured with wire mesh of bulk weaving is described. The successful application of the IC-12-S102 catalyst was confirmed by its operation at OAO Nizhnekamskneftekhim in the process of waste gases afterburning in an industrial reactor with cleaned gases capacity up to 15000 m<sup>3</sup>/h. During the reactor operation in harsh conditions (low oxygen content, high content of water vapor), the degree of gas cleaning was 99.5–99.9% and the residual VOC content in the purified gases was no higher than 10–15 mg/m<sup>3</sup>. For more than 15 months of catalyst operation, the degree of gas purification was not reduced; thus, overall lifetime of the IC-12-S102 catalyst may be substantially longer than the life of well-known industrial afterburning catalysts.

#### **NOVEL CATALYTIC PROCESS FOR FLUE GAS CONDITIONING IN ELECTROSTATIC PRECIPITATORS OF COAL-FIRED POWER PLANTS**

**A.N. Zagoruiko, B.S. Bal'zhinimaev, S.V. Vanag,  
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Research Triangle Pk, USA)**

*J. Air Waste Manage. Assoc.*,  
60(8) (2010) pp. 1002-1008.

One of the most important environmental protection problems for coal-fired power plants is prevention of atmospheric pollution of flying ash. The ash particles are typically removed from flue gases by means of electrostatic precipitators, for which the efficiency may be significantly increased by lowering the resistance of fly ash, which may be achieved by controlled addition of micro-amounts of sulfur trioxide (SO<sub>3</sub>) into the flue gases. This paper describes the novel technology for production of SO<sub>3</sub> by sulfur dioxide (SO<sub>2</sub>) oxidation using the combined catalytic system consisting of conventional vanadium catalyst and novel platinum catalyst on the base of silica-zirconia glass-fiber supports. This combination provides highly efficient SO<sub>2</sub> oxidation in a wide temperature range with achievement of high SO<sub>2</sub> conversion. The performed pilot tests have demonstrated reliable and stable operation, excellent resistance of the novel catalytic system to

deactivation, and high overall efficiency of the proposed process. The scale of the plant was equivalent to the commercial prototype; therefore, no further scale-up of the oxidation process is required.

#### **THEORETICAL AND EXPERIMENTAL BASICS OF THE FORMATION OF OPTIMAL CATALYST LAYERS IN TUBULAR REACTORS**

**O.P. Klenov, V.M. Khanaev, E.S. Borisova, A.A. Sviridonov, A.S. Noskov**

*Catal. Ind.*,  
2(4) (2010) pp. 334-342.

The results from laboratory studies and pilot tests of the process of the catalytic reduction of sulfur dioxide are presented and discussed. The possibility of the low-temperature reduction of SO<sub>2</sub> to elemental sulfur by methane and synthesis gas at 230–250°C on the developed CuCr-AlO catalyst is shown. Pilot operational life tests confirm the high activity and stability of this catalyst. It is concluded that the low-temperature reduction of sulfur dioxide by synthesis gas has certain advantages over the homogeneous catalytic reduction of sulfur dioxide by methane.

Results from research on the structure of a catalyst's granular bed are presented. The influence on catalytic reactions is estimated and the industrial use of the obtained results in tubular reactors of natural gas steam conversion is demonstrated. The influence of the methods for loading catalyst particles on the spatial distribution of the porosity and filterable flow in a fixed bed of a tubular reactor tube is proved theoretically and experimentally. The possibility of the method of loading influencing the process technological parameters is demonstrated via the example of the commercially important reaction of natural gas conversion by steam. It was shown in particular that the correct loading of a bed can lower the temperature of the external walls of steam conversion reactor tubes by several tens of degrees. This ensures a longer service life for tubes operating at 700–1000°C. A loading device was designed on the basis of the obtained results and has been used to load Russia's largest reactors for the conversion of natural gas by steam in M-750 units of methanol production in Tomsk and Gubakha.

#### **TECHNOLOGY OF METHANE COMBUSTION ON GRANULATED CATALYSTS FOR ENVIRONMENTALLY FRIENDLY GAS TURBINE POWER PLANTS**

**Z.R. Ismagilov, N.V. Shikina, S.A. Yashnik, A.N. Zagoruiko, M.A. Kerzhentsev, V.A. Ushakov, V.A. Sazonov, V.N. Parmon, V.M. Zakharov\*, B.I. Braynin\*, O.N. Favorskii\*** (\*Central Institute of Aviation Motors, Moscow, Russia)

*Catal. Today*,  
155(1-2) (2010) pp. 35-44.

The technology of methane combustion in small gas turbine catalytic combustors on alternative granulated catalysts with a low content of noble metals has been developed and studied. The operations of catalyst packages for environmentally clean combustion consisting of one-, two- or three catalysts with different chemical composition, shape and size of granules have been analyzed. The optimized design of a three-stage catalyst package with total height of 340 mm includes 40 mm of highly active Pd-Ce-Al<sub>2</sub>O<sub>3</sub> at the entrance of the combustion chamber in the form of 7.5 mm x 7.5 mm x 2.5 mm rings; 240 mm of a basic catalyst based on Mn-hexaaluminate with the granules of the same form and size; and 60 mm of Pd-Mn-La-Al<sub>2</sub>O<sub>3</sub> catalyst as 4-5 mm spherical granules at the exit of the combustion chamber. Such optimal design yielded methane combustion over 99.97% at T-inlet = 470-580°C and oxygen excess coefficient 5.2-7.0. The emission levels were NO<sub>x</sub> < 1 ppm, CO < 10 ppm, HC < 10 ppm. The pressure drop across the catalyst package was less than 4% of the total pressure (1 bar).

#### **DESIGN, SCALE-OUT, AND OPERATION OF A MICROCHANNEL REACTOR WITH A Cu/CeO<sub>2-x</sub> CATALYTIC COATING FOR PREFERENTIAL CO OXIDATION**

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*Chem. Eng. J.*,  
160(3 sp. issue) (2010) pp. 923-929.

A preferential CO oxidation device consisting of an array of 26 parallel microchannel reactors with each a 5 wt% Cu/CeO<sub>2-x</sub> catalytic coating was designed for application with a 100 W<sub>e</sub> fuel cell system. The reactor device is able to reduce the carbon monoxide concentration in a realistic reformat gas from 1.5 vol.% to 10 ppm at an inlet oxygen to carbon

monoxide ratio of 1.5, a WHSV of  $240 \text{ Lg}^{-1}\text{h}^{-1}$ , and within the temperature range of 230–240°C. The device outperforms similar reactor designs as presented in the literature.

#### **ANALYSIS OF A POWER SYSTEM BASED ON LOW-TEMPERATURE FUEL CELLS AND A FUEL PROCESSOR WITH A MEMBRANE HYDROGEN SEPARATOR**

**V.A. Kirillov, V.D. Meshcheryakov, O.F. Brizitskii\*, V.Ya. Terent'ev\*** (\*Russian Federal Nuclear Center, All-Russian Research Institute of Technical Physics, Sarov, Nizhnii Novgorod oblast, Russia)

*Theor. Found. Chem. Eng.*,  
44(3) (2010) pp. 227-235.

An analysis of the schematic of a power system based on low-temperature proton transfer fuel cells and a fuel processor with a membrane reactor for the steam reforming of CO was carried out. A membrane reactor based on disc diffusion separators of hydrogen and a medium-temperature catalyst of the steam reforming of CO were shown to ensure the thermal efficiency of the fuel processor and the electrical efficiency of a 0.5- to 10-kW power system at a level of 80% and 50%, respectively.

#### **CATALYTIC SYSTEMS OF HYDROGEN PRODUCTION FOR FUEL ELEMENTS WITH SIMULTANEOUS REMOVAL OF CARBON DIOXIDE FROM THE REACTION MEDIUM**

**B.N. Lukyanov, A.I. Lysikov, A.G. Okunev**

*Chem. Sustain. Devel.*,  
18(2) (2010) pp. 115-128.

Basic catalytic processes and types of fuel for the production of hydrogen for low-temperature fuel elements with simultaneous removal of carbon dioxide from the reaction medium are described. Types of adsorption reactors used for absorption-catalytic conversion (ACC) with pressure or temperature shifts, as well as reactors with membrane separation of hydrogen are considered. Descriptions of the new regenerative membrane systems are presented: adsorbent-membrane reactor and space life support systems. Outlooks for the use of ACC in hydrogen power engineering are evaluated.

#### **ABSORPTION-CATALYTIC CONVERSION OF HYDROCARBONS: REACTORS, SORBENTS AND CATALYSTS**

**A.I. Lysikov, B.N. Lukyanov, A.G. Okunev**

*Chem. Sustain. Devel.*,  
18(6) (2010) pp. 691-704.

Absorption-catalytic conversion is considered as a method of obtaining pure hydrogen with a high yield. This process includes simultaneous catalytic transformation of hydrocarbon raw material into hydrogen and sorption-assisted removal of the side product - carbon dioxide. Different aspects of absorption-catalytic conversion are analyzed, in particular its technological implementation in flow reactors with boiling and static beds, and high-temperature CO<sub>2</sub> absorbers and catalysts necessary for the process. It is shown that the process flowsheet preferable for obtaining hydrogen from methane is that with two or more reactors with static bed loaded with a mixture of one of conventional nickel-containing catalyst of vapour conversion and a high-temperature recyclable absorber. Analysis of promising high-temperature recyclable CO<sub>2</sub> absorbers is carried out: supported and individual oxides of alkaline earth metals, mixed lithium salts, promoted hydrotalcites. It is shown that absorbers based on CaO possess sufficient steady dynamic capacity stable under the conditions of absorption-catalytic conversion, and optimal thermodynamic properties.

#### **ENHANCEMENT OF WATER GAS SHIFT REACTION EFFICIENCY: CATALYSTS AND VERSIONS OF THE CATALYST BED ARRANGEMENT**

**N.S. Baronskaya, T.P. Minyukova, A.A. Khassin, T.M. Yurieva, V.N. Parmon**

*Russ. Chem. Rev.*,  
79(11) (2010) pp. 1112-1133.

The results of studies devoted to the search for catalysts for the steam conversion of CO that are highly active over a broad temperature range are generalized. New compositions based on traditional and alternative, as regards composition, catalysts of medium- and low-temperature conversion of CO are considered in detail. The one-stage variants of steam conversion process of carbon monoxide ensuring low temperature gradients in the radial direction of the catalyst bed are discussed.

## **ON-BOARD SYNTHESIS GAS GENERATOR FOR SPARK VEHICLE APPLICATIONS**

**S.F. Peretrukhin\*, O.F. Brizitski\*, V.A. Kirillov, N.A. Kuzin, S.I. Kozlov** (\*Russian Federal Nuclear Center, All-Russian Research Institute of Technical Physics, Sarov, Nizhnii Novgorod oblast, Russia)

*Transport na Alternativnom Toplivo,*  
5(17) (2010) pp. 68-74.

An overview of the current status and perspectives of lean fuel-air mixtures enriched with hydrogen or synthesis gas additives for internal combustion engine (ICE) applications is presented. Comparative analysis of on-board hydrogen supply systems (cylinder, liquefied, generated from borohydrides or by water electrolysis) is performed. An approach based on air conversion of a part of primary fuel into synthesis gas and the use of the produced gas as an additive to primary fuel was proved most promising. For this purpose, a number of catalysts for natural gas (used as fuel) conversion to synthesis gas were developed. A compact under-cowling mounted synthesis gas generator was designed together with its control unit adjusted with ICE control system. A vehicle equipped with synthesis gas generator and fuelled by natural gas with synthesis gas additives was tested on bench, chassis dyno-rollers and road. Potentials of the proposed technology for the development of energy-efficient ecologically conscious engines are analyzed.

## **PROSPECTS FOR USE GAS CYLINDERS MOTOR CARS WITH ON-BOARD SYNTHESIS GAS GENERATOR**

**N.G. Pevnev\*, V.A. Kirillov, O.F. Brizitskiy\*\*, V.A. Burtsev\*\*\*** (\*Siberian State Auto-Road Academy, Omsk, Russia; \*\*Russian Federal Nuclear Center, All-Russian Research Institute of Technical Physics, Sarov, Nizhnii Novgorod oblast, Russia; \*\*\*LC "Gazomotor-R", Rybinsk, Russia)

*Transport na Alternativnom Toplivo,*  
3(15) (2010) pp. 40-45.

The article deals with the analysis of dynamics of variation of the requirements to composition of exhaust gas of internal combustion engines and the measures on an abatement of poisonous reductants in their composition. As a key problem of improving the ecological and economical indexes of internal combustion engines the ways of application of alternative fuel, in particular natural gas methane and addition of synthesis gas (hydrogen containing mixture) to the basic fuel are concerned. The results of automobile "Gazelle" trials on different fuels are given.

## **COMBUSTION OF COMMUNAL WASTEWATER SLUDGE IN THE FLUIDIZED BED OF A CATALYST**

**A.D. Simonov, N.A. Yazykov, A.V. Trachuk, V.A. Yakovlev**

*Int. J. Alternative Energy Ecol.,*  
86(6) (2010) pp. 61-66.

In the work the study of communal wastewater sludge of Moscow, Omsk and Novosibirsk was carried out. It was shown, that the composition of the sludge of different cities differs insignificantly. At the combustion of the sludge in the fluidized bed of a catalyst the burn off reaches 99.5%. The concentration of CO, NO<sub>x</sub>, SO<sub>x</sub>, dioxins in flue gases does not exceed sanitary regulations. Hazard class of the ash is 4 (lightly dangerous).

## **CATALYTIC COMBUSTION OF THE DEPOSITS OF WASTE WATER FROM COMMUNITY SERVICES**

**A.D. Simonov, O.V. Chub, N.A. Yazykov**

*Chem. Sustain. Devel.,*  
18(6) (2010) pp. 749-753.

Air-fluidized bed combustion of the deposits of communal waste water from the sludge beds of Gorvodokanal enterprise (Novosibirsk) was investigated. It was established that the deposit burnout degree at the process temperature of 700°C reaches 98 %. The concentrations of toxic substances (Hg, HCl, SO<sub>3</sub>, P<sub>2</sub>O<sub>5</sub>, CO, NO<sub>x</sub>) correspond to the sanitary standards. It was shown that combustion of deposits is possible after preliminary drying to the humidity of 54 %.

## **WATER DEPOLLUTION AND THE ODOR CONTROL BY WET AIR CATALYTIC OXIDATION OF AMMONIA, SULFIDES AND MERCAPTANS OF INDUSTRIAL WASTEWATERS**

**N.M. Dobrynkin, M.V. Batygina, A.S. Noskov**

*Chem. Eng. Trans.,*  
23 (2010) pp. 339-344.

Catalytic properties of the carbon material Sibunit (commercial samples) and ceria- and zirconia-promoted precious metals (Ru, Pt, Pd) supported on carbon were studied in the processes of the catalytic wet air oxidation (CWAO) of number toxic odour compounds at elevated pressures and temperatures (T=160 – 250°C, P<sub>O<sub>2</sub></sub>=0.3 - 1.0 MPa).

A possibility of the creation of effective ruthenium catalysts Ru-CeO<sub>2</sub>/Sibunit and Ru-ZrO<sub>2</sub>-CeO<sub>2</sub>/Sibunit with a low ruthenium content (~0.6% Ru) for the deep cleaning of the polluted waters at the moderate temperatures and the odor prevention is demonstrated.

The described catalysts and method are solving the problem of the development of the environmentally reliable method for the treatment of the fluids and allow carrying out the adsorption of the pollutants on the carbon catalyst with the following regeneration of the carbon catalyst without the loss of adsorptive qualities. The experiments have shown a principal capability simultaneously to use the carbon catalyst as the adsorbent and either as the catalyst, or as the catalyst support for oxidation of the odorous substances in water solutions.

The catalysts allow: a) to decrease the mineralization temperature for typical organic pollutants from 200–240°C to 160°C, and b) to increase the activity of the low ruthenium content catalyst. The full cleaning (more than 99,99 %) is possible at the continuous realization of the process in the unsteady conditions.

#### **DEVELOPING NEW CATALYSTS AND IMPROVING CATALYTIC METHODS FOR PURIFYING THE FLUE GASES OF VANUKOV AND FLASH SMELTING FURNACES**

**Z.R. Ismagilov, S.R. Khairulin, S.A. Yashnik, I.V. Ilyukhin, V.N. Parmon**

*Catal. Ind.*,  
2(4) (2010) pp. 353-359.

Results of laboratory investigations and pilot tests of the processes of catalytic reduction of sulfur dioxide to elementary sulfur using methane and synthesis gas as reducing agents are presented. It is concluded that low temperature process of catalytic reduction of sulfur dioxide by synthesis gas has several advantages in comparison with homogenous and catalytic processes of reduction of sulfur dioxide by methane.

#### **THE MECHANISM OF FORMATION OF CENOSPHERES AND OTHER FLY ASH PRODUCTS OF HIGH-TEMPERATURE COAL COMBUSTION ON THERMAL POWER PLANTS**

**V.B. Fenelonov, M.S. Melgunov, V.N. Parmon**

*Kona: Powder Part. J.*,  
28 (2010) pp. 180-208.

A systematic analysis of the probable scenario of the supramolecular structure (texture) formation of mineral products, formed during the high-temperature combustion of pulverized bituminous coals at thermal power plants (TPP) is provided. The general physico-chemical consideration of the processes that occur at different stages (heating, burning, viscoplastic state in the hot zone, and cooling) during the combustion of bituminous coal makes it possible to allocate the following systematic transformations: coal → char → network structures (including plerospheres) → cenospheres → fly ash.

#### **EXPRESS GAS CHROMATOGRAPHY ON MULTICAPILLARY COLUMNS AND ITS POTENTIAL**

**V.N. Sidelnikov, Yu.V. Patrushev, O.A. Nikolaeva**

*Catal. Ind.*,  
2(3) (2010) pp. 206-216.

Approaches to the acceleration of chromatographic processes are considered. The most radical way to reduce separation time is shown to be the use of short capillary columns with a small-diameter (10–50 μm) capillary. Limitations on the applications of such columns are discussed, a major one being the introduction of an extremely small amount of sample. This can be overcome by using a pack of capillaries with a small diameter. This works as a single chromatographic column and is called a multicapillary column (PAC). The amount of sample introduced in the column is greater, making it possible to work over a wide range of concentrations. Different types of PAC are considered. The mechanism of the broadening of the chromatographic peak on PAC and its properties are discussed. Examples of using the column for gas liquid and gas adsorption chromatography are given. Examples are also given of the use of PAC in catalytic studies.



**MULTICAPILLARY COLUMNS WITH A  
POROUS LAYER BASED ON THE  
DIVINYLBENZENE COPOLYMER**

**Yu.V. Patrushev, O.A. Nikolaeva, V.N. Sidelnikov**

*Russ. J. Phys. Chem. A,*  
84(5) (2010) pp. 871-875.

A method for preparing a multicapillary column with a porous layer based on the styrene-divinylbenzene copolymer has been developed. The column makes it possible to quickly separate C<sub>1</sub>-C<sub>4</sub> hydrocarbons and oxygen-containing compounds. The main chromatographic properties of the columns were studied.

**INVESTIGATION OF THE LOADING  
CAPACITY OF MULTICAPILLARY  
CHROMATOGRAPHIC COLUMNS**

**Yu.V. Patrushev, O.A. Nikolaeva, V.N. Sidelnikov**

*J. Analyt. Chem.,*  
65(11) (2010) pp. 1129-1131.

It was shown by the comparison of multicapillary and single-capillary columns for gas-liquid chromatography that an approximately 100-fold larger sample could be injected into multicapillary columns without substantial loss in efficiency, because the injected sample volume, causing no significant loss in efficiency, is two orders of magnitude higher for gas-liquid multicapillary columns than for gas-adsorption multicapillary columns.

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## PARTICIPATION IN CONFERENCES, SEMINARS

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Fluorinated Organic Boron Compounds: Synthesis, Reactivity and Utilization,  
*All-Russian Scientific Youth School-Conference "Chemistry under the Sigma Sign: Investigations, Innovations, Technologies"*, May 16-24, Omsk, Russia. Abstracts, pp. 24-25.
2. **N.Yu. Adonin, M.M. Bykov, (V.V. Bardin, H.-J. Frohn), V.N. Parmon**,  
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5. **D.S. Afanasiev, A.S. Lisitsyn, N.I. Kuznetsova**,  
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