ANNUAL REVIEW 2010

BORESKOV INSTITUTE OF CATALYSIS

SIBERIAN BRANCH OF THE RUSSIAN ACADEMY OF SCIENCES

ANNUAL REVIEW 2010

http://catalysis.ru

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of Activities in Basic Research Areas
2009

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

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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 2009 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 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 reflect Russia entering into the market economy. BIC is affiliated to the Siberian Branch of the Russian Academy of Sciences. However, according to new realities of Russia, a lot deal of activity of BIC 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, already for several years BIC is positioned as a leading Russian chemical institution from the point of view of citation. 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 website of BIC.

The BIC has a strategic collaboration with some both Russian and foreign industrial companies 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 an 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. The facilities of the UIC include three 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 are inviting the readers to visit the BIC’s website **www.catalysis.ru**.

Valentin N. Parmon

THE 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, the Institute has the highest total citation index among the Russian chemical institutions over the last 7 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 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 feedstocks 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 (BIC) 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 Center for Catalyst Characterization and Testing has a bank of catalyst samples, supports and adsorbents, a set of State Standard Samples to calibrate and certify instruments for measurements of specific surface areas of various dispersed and porous materials.

The main directions of its activity are:

- Complex investigation and characterization of the catalysts by a set of modern physicochemical methods.
- Joint scientific projects with foreign firms and organizations on studying certain catalytic processes and catalysts for the purpose of their improvement.
- Development of modern standard experimental methods and equipment for catalyst studies and tests. Design of catalysts and materials with certificated properties.

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:
V.N. Novikov, S.E. Glaznev,
I.A. Kamolkin, A.A. Vedyagin
A.S. Noskov, V.N. Parmon,
V.I. Bukhtiyarov, B.S. Bal'zhinimaev

DIRECTOR

Directorate

Scientific Council

Research Departments

DEPARTMENT OF PHYSICOCHEMICAL METHODS FOR CATALYST INVESTIGATION
Head: Corr. Member RAS Valerii I. Bukhtiyarov

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

DEPARTMENT OF HETEROGENEOUS CATALYSIS
Head: Prof. Aleksandr A. Khassin

DEPARTMENT OF EXPLORATORY AND APPLIED INVESTIGATIONS
Center for Catalyst Characterization and Testing
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: Prof. Sergey S. Ivanchev

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 Corr. Member RAS Valerii I. Bukhtiyarov

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

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

Laboratory of Investigation of the Mechanisms of Catalytic Reactions
Head: Prof. Eugenii 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: Corr. Member RAS Valerii I. Bukhtiyarov

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

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

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 Adsorption
Head: Prof. Oleg N. Martyanov

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

DEPARTMENT OF HETEROGENEOUS CATALYSIS

HEAD OF THE DEPARTMENT PROF. ALEKSANDR A. KHASSIN

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

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

Laboratory of Dehydrogenation
Head: Prof. Victor V. Molchanov

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

Laboratory of Oxidative Catalysis over Zeolites
Head: Prof. Gennadii I. Panov

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

Laboratory of Ecological Catalysis
Head: Prof. Zinfer R. Ismagilov

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

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

DEPARTMENT OF CATALYTIC PROCESS 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

Sector of Complex Engineering Projects
Head: Dr. Victor A. Chumachenko

Group of Dynamics of Catalytic Processes
Head: Prof. Andrey N. Zagoruiko

Group of Kinetics of Catalytic Processes
Head: Prof. Sergei I. Reshetnikov

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

Group of Development and Optimization of Catalytic Processes with Catalyst Deactivation
Head: Dr. Vitalii N. Kashkin

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

Group of Processes in Fixed Catalyst Layer
Head: Ilya A. Zolotarsky

Group of Membrane-Catalytic Processes
Head: Dr. Ilya V. Mishakov

DEPARTMENT OF EXPLORATORY AND APPLIED INVESTIGATIONS

Head of the Department Prof. Bair S. Bal'zhinimaev

Center for Catalysts Characterization and Testing
Head: Prof. Bair S. Bal'zhinimaev

Laboratory of Zeolites and Acid Base Catalysis
Head: Prof. Eugenii A. Paukshtis

Laboratory of Catalysts Activity Testing
Head: Dr. Nikolai N. Bobrov

Group of Catalytic Conversion of Sulfur-Containing Compounds
Head: Prof. Anna V. Mashkina

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

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

ST. PETERSBURG DEPARTMENT OF THE BORESKOV INSTITUTE OF CATALYSIS

Director and Scientific Manager
Corr. Member RAS Sergey S. Ivanchev

Vice-Director Dr. Valery P. Tulmankov

Laboratory of Micellar Catalysis, Emulsion Polymerization and Processes for Obtaining Polymeric Composites
Head: Prof. Valery N. Pavlyuchenko

Laboratory of Novel Catalytic Systems for Olefin Polymerization and Copolymerization
Head: Corr. Member RAS Sergey S. Ivanchev

VOLGOGRAD DEPARTMENT OF THE BORESKOV INSTITUTE OF CATALYSIS

Scientific Director
Dr. Aleksandr M. Beskopylnyi

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 OF THE INSTITUTE

Basic research

- *Determination of general regularities of catalysis*
- *Development of the scientific basis for molecular design the catalysts and materials*
- *Development of the theoretical basis to design the catalytic processes*
- *In-situ methods to study catalyst and mechanism of important catalytic reactions*
- *Methods and algorithms to predict the catalytic action*
- *Methods and facilities for the catalysts testing*
- *Nanotechnological approach to design the catalysts with predictable properties*

Applied catalysis

- *Design of highly efficient catalysts for production of key chemicals; oil & gas processing*
- *Design of the catalytic composites and technologies for applied purposes*
- *Catalysts and catalytic technologies for polymers production with specified properties*
- *Design of sorbents, catalysts and technologies for detoxication of hazardous man-caused wastes*
- *Selective oxidation of light* C_1*-*C_4 *paraffins by molecular oxygen*
- *Gas-phase propylene epoxidation by molecular oxygen*
- *Reactors and new processes based on membrane catalysts. Microchannel catalytic reactors*
- *Catalysts and processes related to hydrogen energy technologies*
- *Catalysis concerned to the environmental and energy problems*
- *New directions and opportunities of catalysis in utilization of renewable natural resources*

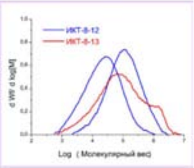
Some catalytic technologies owned by BIC

- **Reverse process** for catalytic gas purification
- **Zeoforming processes** for synthesis of gasoline
- **Alphox technology** for the one step phenol form benzene production
- **Adsoroption-catalytic purification** of industrial wastewaters
- **Nitric acid** production with the use of honeycomb catalysts
- **One-step** technology for the **nicotinic acid** production
- New technology of **loading the reactors** with catalysts
- **BIMT technology** for production of gasoline and diesel fuel without preliminary rectifying the hydrocarbon feedstock
- Reburning of Klaus **tail gases**
- The **BICYCLAR** technology for production of aromatic hydrocarbons with methane involvement
- Direct catalytic **oxidation of** H_2S to elemental sulfur

- Treating of mixed **radioactive organic wastes**
- Liquid-phase oxidative **treating of industrial wastewater**
- Technology for **formaldehyde** production
- Technology for **one step formic acid** production
- Technology for **reducing atmospheres** production
- Technique for preparation of **emulsifiers and water-bitumen emulsions**

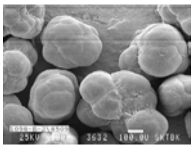
Some industrial catalysts by BIC

Vanadium pentoxide catalysts for sulfuric acid production



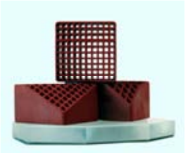
Supported catalysts ICT-8-12 and ICT-8-13 for polyethylene production

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



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

Microspherical zeolite-containing cracking catalysts

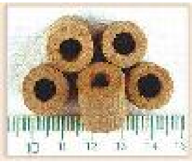


Non-platinum catalyst IC-42-1 for nitrogen acid production

Gasoline reforming catalysts of PR series

High silica zeolite catalysts

Hydrogenation catalysts of for manufacture of medical products and edible fats



Thermostable catalyst ICT-12-40 for gas purification

Catalyst for hydropurification of terephthalic acid

Some catalyst supports by BIC

Superfine silica gels IC-01-2

Composite selective water sorbents SWS

Mesoporous carbonaceous material SIBUNIT

Catalytic Processes from Molecular Level to Industrial Plants

2009 **Catalytic heating units (CHU) for autonomous heat power generation**

2008 **Family of fiberglass catalysts (GFC) for treatment of industrial waste gases**

2007 **Catalyst for fine hydrotreatment of diesel fractions (Euro-4 and Euro-5 standards for sulfur)**

2006 **Catalyst for synthesis ultrahigh molecular weight polyethylene (UHMWPE)**

2005
2003 **New generation of LUKS series catalysts for cracking and PR and RU series catalyst for reforming**
Developed in cooperation with the IAP, Omsk, Russia

2002 **Composite selective water sorbents**

2000 **New method for loading of catalysts into industrial reactors**

1997 **Non-platinum catalyst for production of nitric acid**

1996 **Alphox technology (benzene + $[N_2O]$ → phenol)**
Developed in cooperation with Solutia, USA

1995 **Catalyst for FCC (oil cracking)**

1993 **Carbonaceous material SIBUNIT**
Since 1994 is beaing supplied to USA, Europe, Japan

1992 **ZEOFORMING process for synthesis of gasoline**
Lurgi, 1997, Germany

1988 **Catalyst for propylene polymerization**
DSM, 1995, the Netherlands

1982 **REVERS-Process**
Monsanto Enviro Chem. Systems, 1992, USA

1978 **Vanadium catalysts for sulfuric acid production**

1958

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 2009

Austria	1	Finland	3	Netherlands	11
Azerbaijan	1	France	29	Saudi Arabia	4
Belarus	6	Germany	38	Singapore	1
Belgium	3	Greece	5	Spain	10
Brazil	4	Italy	23	Sweden	2
Bulgaria	2	Japan	3	Turkey	1
Canada	1	Kazakhstan	2	Ukraine	5
China	13	Korea	2	United Kingdom	2
Cyprus	2	Mexico	3	USA	8
Denmark	3	Poland	8	Venezuela	1
India	6				

Visits of foreign specialists to the Boreskov Institute of Catalysis in 2009

Australia	1	Israel	1	Republic of Korea	6
Belgium	1	Italy	5	Saudi Arabia	1
Bulgaria	2	Japan	2	Slovenia	1
China	12	Jordan	1	Switzerland	2
Croatia	1	Kazakhstan	1	Syria	1
Denmark	7	Lebanon	1	Tunisia	1
Finland	2	Morocco	2	Turkey	1
France	9	Netherlands	10	United Kingdom	2
Germany	16	Pakistan	1	USA	7
India	12	Poland	16		

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), Lyon in the frame of the Russian-French European associated Laboratory on Catalysis headed by Acad. 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: Selective Oxidation with Hydrogen Peroxide***
- ***Advanced Spectroscopic Methods: Mobility of Species in Porous Medium.***

INDIA

In the frame of Indo-Russian Integrated Long Term Programme of cooperation in science and technology (ILTP) BIC collaborates with *National Chemical Laboratory,* Pune on the Project ***"Design of Bifunctional Supported Non-Iron Catalysts for Low Temperature Ammonia Synthesis"***. Coordinators: **Dr. B. Moroz** (*BIC*) and **Dr. S.B. Halligudi** (*National Chemical Laboratory*).

Coordinators on the Program "Catalysis" are Prof. V. Parmon and Dr. S. Sivaram.

SWEDEN

Cooperation with *Chalmers University of Technology,* Göteborg on the Project ***"Modeling of Hydrogen Adsorption by Metal Nanoparticles"***. Coordinators: **Prof. V. Zhdanov** (BIC) and **Prof. B. Kasemo** (*Chalmers University of Technology*).

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*** $\boldsymbol{TiO_2}$***"***. 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*).

THE NETERLANDS

Cooperation with University of Technology, Eindhoven on the Project ***"Preparation of Mesoporous Titania Films on the Support"***. Coordinators: **Prof Z. Ismagilov** (*BIC*) and **Prof. J.C. Schouten** (*University of Technology*).

JAPAN

Bilateral agreement *BIC - Research Laboratory for Nuclear Reactors*, Tokyo Institute of Technology, on the Project ***"Adsorption and Chemical Reactions for Heat Transformation"***. Coordinators: **Prof. Yu. Aristov** (*BIC*), **Prof. Y. Kato** (*Research Laboratory for Nuclear Reactors*).

CHINA

The cooperation in the frame of *Associated Research Laboratory* which was established by an agreement signed December 4, 2004 by the Boreskov 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

INTAS - SB RAS Supported Project

Electromagnetic Response Properties of Carbon Onions and Carbon Onion-Based Composites

Project Coordinator:

Dr. Ph. Lambin, *Facultes Universitaires Notre-Dame de la Paix*, Namur, Belgium

Participants:

Belarus State University, Minsk, Belarus; *University of Joensuu*, Finland; *Institute for Technical Physics and Materials Science*, Budapest, Hungary; *The Boreskov Institute of Catalysis*, Novosibirsk, Russia (**Dr. V. Kuznetsov)**, *Nikolaev Institute of Inorganic Chemistry*, Novosibirsk, Russia.

CRDF Project

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

Project Coordinators:

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

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

EUROPEAN COMMUNITY SIXTH FRAMEWORK PROGRAM

I. International Partnership for a Hydrogen Economy for Generation of New Ionomer Membranes

Coordinator: **Dr. R. Mallant**, *Energy Research Centre of The Netherlands*, Petten, The Netherlands

Partners:

Daimler Chrysler; *FuMA-Tech GmbH*; *CNRS Montpellier*; *Dohgyue Chenzhou New Materials Company*; *Shanghai Jiao Tong University*, Shanghai, China; *The Boreskov Institute of Catalysis*, Novosibirsk, Russia (**Prof. V. Bukhtiyarov**).

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

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

Partners:

Rijksuniversiteit Groningen, The Netherlands; *The 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.

III. 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; *The Boreskov Institute of Catalysis*, Novosibirsk, Russia (**Acad. 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.

IV. Novel Materials for Silicate-Based Fuel Cells

Coordinator: **Dr. Ch. Arguirusis**, *Technische Universität Clausthal*, Clausthal, Germany

Partners:

University of Aveiro, Aveiro, Portugal; *Foundation of Research and Technology Hellas*, Greece; *Katholieke University of Leuven*, Belgium; *Max-Plank Institute of Colloids and Interfaces*, Munchen, Germany; *The Boreskov Institute of Catalysis*, Novosibirsk, Russia (**Prof. V. Sadykov**); *Ceramics and Refractories Technological Development Company*, Greece; *Ceramiques Techniques et Industrielles*, France.

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; *The 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; *The 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'Environment*, Lyon, France; *Ruhr-Universität Bochum*, TECHEM, Bochum, Germany; *Instituto de Technologia 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; *The Boreskov Institute of Catalysis*, Novosibirsk, Russia **(V.A. Sadykov)**; *INEOS*, Belgium; *Institut fur Mikrotechnik Mainz Gmbh*, Germany; *Eni SpA*, Italy.

NATO PROGRAMME: SCIENCE FOR PEACE AND SECURITY

Capture and Decontamination of Chemical & Biological Agents by Novel Catalysts and Millisecond Jet Reactors

Project Coordinator from a NATO Country:

Prof. P. Smirniotis, *University of Cincinnaty*, Cincinnaty, United States

Project Coordinator from a Partner Country:

Prof. A. Vorontsov, *The Boreskov Institute of Catalysis*, Novosibirsk, Russia.

INTERNATIONAL SCIENCE AND TECHNOLOGY CENTER (ISTC)

I. Small Capacity Fuel Cells

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

II. Environmentally Friendly Small Capacity Power Plants Based on Fuel Cells for Stationary Application

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

III. Development of High-Performance Oxygen-Containing Membranes and Compact Syn-Gas Generators on Their Base

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

IV. 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**.

V. Catalytic Production of SO_3 for Conditioning of Electrostatic Precipitators Using in Russia and the Newly Independent States (NIS)

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

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

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

VII. Synthesis, Formation and Modification of Organic Nanoparticles in Supercritical Fundamentals and Applications

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

VIII. Composite Nanocarbon Based Materials for Electromagnetic Applications
Project Manager from BIC **Dr. V. Kuznetsov**.

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 2009, the most important BIC's achievements in the area of applied catalysis were presented at the Exhibitions:

- *"High Technologies of XXI Century"*, April 21-24, Joint–Stock Company "All-Russian Exhibition Centre", Moscow, Russia
- *"Innovations. Investments. Progress"*, May 19-22, Kemerovo, Russia
- *"Belarussian Industrial Forum-2009"*, May 18-24, Minsk, Belarus
- *"Eighth Exhibition of Military Equipment, Technologies and Armament for Land Forces "VTTV-Omsk-2009"*, July 2-6, The Intersib International Exhibition Centre, Omsk, Russia
- *"XX China Harbin International Economic and Trade Fair"*, July 15-19, Harbin, China
- *"IX Moscow International Innovations' and Investments' Show"*, August 26-29, Moscow, Russia
  Golden Medal and Diploma for "*Vehicle Syngas Generator*"
- *"Chemistry-2009"*, September 28-October 2, Expocentre, Moscow, Russia
- *"II International Forum on Nanotechnologies"*, October 3-5, Expocentre, Moscow, Russia
- *"Oil. Gas. Chemistry"*, October 20-22, Expo Volga, Samara, Russia
- *"SibPolyTech-2009"*, October 27-30, Novosibirsk, Russia.
  Big Golden Medal and Diploma ITE for *"Fiber-Glass Based Catalysts – New Type of Catalytic Systems"*.

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 NIS, but foreign participants as well.

VIII INTERNATIONAL CONFERENCE "MECHANISMS OF CATALYTIC REACTIONS"

dedicated to the 70th anniversary of Professor Kirill I. Zamaraev

June 28-July 2, Novosibirsk, Russia

The MCR-Conference was organized under the auspices of the European Federation of Catalysis Societies (EFCATS).

Conference organizers:

- Siberian Branch of the Russian Academy of Sciences
- Scientific Council on Catalysis of the Russian Academy of Sciences
- Ministry of Education and Science of the Russian Federation, Moscow
- The Boreskov Institute of Catalysis, Novosibirsk
- Lomonosov Moscow State University, Moscow
- Russian Mendeleev Chemical Society, Novosibirsk Department.

The Program of the Conference consisted of 5 plenary and 18 keynote invited lectures, 32 oral presentations (20 min), 39 oral presentations (10 min), 38 oral presentations of young scientists (10 min) and 120 posters. The oral presentations were scheduled in parallel sections:

- Mechanisms of Homogeneous and Heterogeneous Catalysis at the Atomic and Molecular Scale
- Physical Methods for the Study of Mechanisms of Heterogeneous and Homogeneous Catalytic Reactions, Including *in situ* Techniques
- Theory and Quantum-Mechanical Approaches in Catalysis
- Catalysis in Nature, Biomimetic Catalysis and Photocatalysis.

Plenary lectures:

- **M. Bochmann** (*University of East Anglia, Norwich, UK*) – "Reactivity and Mechanism of Single-Site Olefin Polymerization Catalysts"
- **V.I. Bukhtiyarov** (*The Boreskov Institute of Catalysis, Novosibirsk, Russia*) – "Ambient Pressure X-Ray Photoelectron Spectroscopy: From *in situ* to *operando* Studies of Heterogeneous Catalytic Reactions"
- **H.-J. Freund, M. Sterrer** (*Fritz-Haber-Institut der Max-Planck-Gesellschaft, Berlin, Germany*) – "Surface Science Models in Catalysis: A View at the Atomic Level"
- **N. Rösch** (*Technische Universität München, Garching, Germany*) – "Hydrogen Activation by Transition Metals"
- **A.G. Stepanov** (*The Boreskov Institute of Catalysis, Novosibirsk, Russia*) – "What Did We Learn from NMR of Hydrocarbon Conversion on Solid Acid Catalysts? Advances for the Last 20 Years".

250 Scientists from 19 countries took part in the Conference.

3rd INTERNATIONAL SCHOOL-CONFERENCE ON CATALYSIS FOR YOUNG SCIENTISTS "CATALYST DESIGN"

July13-19, The tourist center "Chusovaya", Yekaterinburg region, Russia

This event continues the tradition established in Novosibirsk, Russia (2002) and Altay**,** Russia (2005).

Conference organizers:

- The Boreskov Institute of Catalysis, Novosibirsk
- Council of Young Scientists of the Boreskov Institute of Catalysis, Novosibirsk
- Institute of Solid State Chemistry, Yekaterinburg
- Council of Young Scientists of the Institute of Solid State Chemistry, Yekaterinburg
- Scientific Council on Catalysis of the Russian Academy of Sciences
- Russian Foundation for Basic Research.

The Program of the School-Conference consisted of 9 plenary lecturers, 42 oral presentations and 15 posters. The oral presentations were scheduled in 6 sessions:

- Preparation of Catalysts and Adsorbents
- Mechanisms of Heterogeneous Catalysis, Methods for Catalyst Characterization
- Kinetics and Modeling of Catalytic Reactions and Reactors
- Catalysis for Environmental Protection, Photocatalysis
- Catalysis for Fine Organic Synthesis, Natural Gas and Petroleum Chemistry
- Catalysis in Energy Production, Electrocatalysis.

Plenary lectures:

- **M.S. Melgunov** (*The Boreskov Institute of Catalysis, Novosibirsk, Russia*) – "Templating Mechanisms of Catalysts and Adsorbents Formation"
- **V.L. Kuznetsov** (*The Boreskov Institute of Catalysis, Novosibirsk, Russia*) – "Catalysts Design for Production of Carbon Nanotubes and Nanowhiskers"
- **V.V. Kaichev, V.I. Bukhtiyarov** (*The Boreskov Institute of Catalysis, Novosibirsk, Russia*) – "Application of Surface Science Method for *in situ* Studying of Heterogeneous Catalytic Reactions"
- **N.V. Vernikovskaya** (*The Boreskov Institute of Catalysis, Novosibirsk, Russia*) – "The Role of Mathematical Modeling in the Design of Catalytic Processes and Reactors"
- **S.A. Beloshapkin** (*Materials and Surface Science Institute, University of Limerick, Limerick, Ireland*) – "Application of Focused Ion Beam Technique for the Structured Catalysts Investigation. Cross-Sectional Study and Specimen Preparation for Transmission Electron Microscopy"
- **D.V. Kozlov** (*The Boreskov Institute of Catalysis, Novosibirsk, Russia*) – "Photocatalysis: Fundamentals and Applications"
- **V.I. Sobolev** (*The Boreskov Institute of Catalysis, Novosibirsk, Russia*) – "Fundamental and Technological Aspects of Heterogeneous Selective Oxidation of C1-C4 Organic Compounds"
- **A. Malyschew** (*SASOL Germany GmbH, Hamburg FR Germany*) – "Alumina and Mixed Oxide Catalyst Supports for Sophisticated Catalyst Design"
- **A.G. Okunev** (*The Boreskov Institute of Catalysis, Novosibirsk, Russia*) – "Catalysts and Catalytic Layers in Polymer Electrolyte Membrane Fuel Cells".

The whole number of participants was 66 specialists from 7 countries.

2nd RUSSIAN SCHOOL-CONFERENCE FOR YOUNG SCIENTISTS "FUNCTIONAL NANOMATERIAL IN CATALYSIS AND ENERGETICS"

July13-18, The tourist center "Chusovaya", Yekaterinburg region, Russia

Conference organizers:

- The Boreskov Institute of Catalysis, Novosibirsk
- Council of Young Scientists of the Boreskov Institute of Catalysis, Novosibirsk
- Institute of Solid State Chemistry, Yekaterinburg
- Council of Young Scientists of Institute of Solid State Chemistry, Yekaterinburg
- Scientific Council on Catalysis of the Russian Academy of Sciences
- Russian Foundation for Basic Research.

The Program of the School-Conference consisted of 9 plenary lecturers, 32 oral presentations and 19 posters. The oral presentations were scheduled in 5 sessions:

- Synthesis of Nanostructured Systems
- Methods of Investigation of Nanomaterials
- Mathematical Modeling and Computational Materials Science
- Novel Materials for Hydrogen Power Systems, Solid Polymer Electrolytes and Membranes
- Nanomaterials in Petrochemistry, Catalytic Production of Fuels and Energy from Renewable Sources.

Plenary lectures

- **V.L. Kozhevnikov** (*Institute of Solid State Chemistry, Yekaterinburg, Russia*) – “Prospect Materials for Membrane Reactors for Methane Oxidation”
- **A.L. Ivanovskii** (*Institute of Solid State Chemistry, Yekaterinburg, Russia*) – “Computational Modeling of Structural, Mechanical, Thermal and Magnetic Properties of Inorganic Nanostructures”
- **I.V. Mishakov, A.A. Vedyagin** (*The Boreskov Institute of Catalysis, Novosibirsk, Russia*) - “Carbon Nanotubes and Nanofibres: Synthesis, Structure, Application”
- **M.V. Kuznetsov** (*Institute of Solid State Chemistry, Yekaterinburg, Russia*) – “Electron Spectroscopy and Scanning Tunneling Microscopy: A Tools for Studying the Surface of Solids”
- **I.V. Melikhov** (*Moscow Lomonosov State University, Moscow, Russia*) – “Evolution of Catalysts Properties Upon Its Preparation and Use”
- **V.A. Sadykov** (*The Boreskov Institute of Catalysis, Novosibirsk, Russia*) – “Scientific Bases for Design of Nanocomposite Materials for Media-Temperature Solid Fuel Cells and Structured Catalysts of Biofuel Conversion to Syn-Gas and Hydrogen”
- **A.V. Lavrenov** (*Institute for Hydrocarbons Processing, Omsk, Russia*) – “Catalysts and Processes for Production of Ecologically Pure Free Gasoline Components”
- **O.V. Klimov** (*The Boreskov Institute of Catalysis, Novosibirsk, Russia*) – “Modern Catalysts for Hydropurification”.

RUSSIAN-INDIAN SYMPOSIUM ON CATALYSIS AND ENVIRONMENTAL ENGINEERING

September 13-14, Novosibirsk, Russia

Symposium organizers:

- Siberian Branch of Russian Academy of Sciences
- The Boreskov Institute of Catalysis, Novosibirsk
- Department of Science and Technology, Government of India, Delhi, India
- Scientific Council on Catalysis of the Russian Academy of Sciences.

The Scientific Program comprised 7 invited plenary, 19 oral lectures (20 min.), 8 oral lectures (10 min.) and 9 posters. It covered the following topics:

- Catalysis for Utilization of Non-Traditional Feedstock and Renewable Energy Sources
- Catalytic Environmental Technologies Including Industrial and Motor Waste Treatment
- Photocatalysis.

Plenary lectures

- **Raksh Vir Jasra** (*Reliance Industries Limited, Vadodara Manufacturing Division P.O. Petrochemicals, Vadodara, India*) – “Hydroformylation of Alkenes in Constrained Media”

- **M.A. Kerzhentsev, Z.R. Ismagilov, V.N. Parmon, O.N. Favorski*** (*The Boreskov Institute of Catalysis, Novosibirsk, Russia; *Central Institute of Aviation Motors, Moscow, Russia*) – "Development of Advanced Catalysts for Environmentally Friendly Methane Combustion in Gas Turbine Catalytic Combustors"
- **Sadhana S. Rayalu** (*Environmental Materials Unit, National Environmental Engineering Research Institute, Nehru Marg, Nagpur, India*) – "CO_2 Sequestration Paves New Way for Energy and Fuel"
- **Swaminathan Sivaram** (*National Chemical Laboratory, Pune, India*) – "Reactivity of Novel Early and Late Transition Metal Complexes in the Polymerization and Oligomerization of Ethylene"
- **A.Yu. Stakheev, G.O. Bragina, N.S. Telegina, G.N. Baeva, P.V. Pributkov** (*Zelinsky Institute of Organic Chemistry, Moscow, Russia*) – "Modern Trends in the Development of Catalytic Aftertreatment Systems for Lean-Burn and Diesel Engines"
- **A.V. Vorontsov, E.A. Kozlova, A.S. Besov, D. Tsydenov*, S. Kiselev*, A. Safatov*, D.V. Kozlov, V.N. Parmon** (*The Boreskov Institute of Catalysis, Novosibirsk, Russia; *Novosibirsk, State University, Novosibirsk, Russia*) – "TiO_2 Photocatalysis for Pollutants Abatement and Hydrogen Production"
- **V.A. Yakovlev, S.A. Khromova, V.O. Dundich, O.V. Sherstyuk, A.S. Ivanova, M.V. Bukhtiyarova, V.G. Sister*, V.N. Parmon** (*The Boreskov Institute of Catalysis, Novosibirsk, Russia; *Moscow State University of Engineering Ecology, Moscow, Russia*) – "Catalysts Design for Biofuels: Biodiesel, Green Diesel, Upgraded Biooil".

REGIONAL YOUTH SCIENTIFIC TECHNICAL FORUM “SIBERIA – CHEMISTRY, INNOVATIONS, TECHNOLOGIES”, SibHIT-2009”

September 14–16, Novosibirsk, Russia

Forum organizers:

- Department of Chemistry and Materials Science of the Russian Academy of Sciences
- The Boreskov Institute of Catalysis, Novosibirsk
- Council of Young Scientists of the Boreskov Institute of Catalysis, Novosibirsk.

The aim of the Forum was: Stimulating of the mass participation of the scientific youth in innovation organizational and financial support of the scientific technological and innovation projects; exchange of information and discussion of an opportunity of cooperation in the frames of youth scientific integration projects; demonstration of scientific achievements of the youth of the Siberia region.

Forum subject: Innovation developments, fundamental and applied investigations in chemistry, chemical technology, biochemistry and material science. The contest UMNIK of “The Foundation for Assistance to Small Innovative Enterprises” has been held in the frames of the Forum.

SEMINAR “RUSSIAN TECHNOLOGIES FOR OIL REFINING AND RENEWABLES PROCESSING”

September 23-25, Rio de Janeiro, Brazil

The purpose of the Seminar was to attract the attention of the researchers and the companies’ representatives from Mercosur countries to the modern Russian technologies as well as to join the efforts in the creation of the new developments in petrochemistry and renewable sources processing. The participants of the Seminar got acquainted with R&D activities of the Russian academic organizations and enterprises in the field of oil and renewables treatment.

The outstanding scientists, directors and presidents of the large companies and institutions, working in the field of petrochemistry and renewable sources processing, were invited to present their results. 60 representatives of scientific and business communities took part in the Seminar. The program included 6 presentations of the Russian Scientists:

V.N. Parmon (*The Boreskov Institute of Catalysis, Novosibirsk, Russia*) – “Catalysis in Russia: Oil Refining and Petrochemistry”

V.M. Kapustin (*General Director of the Engineering Company on Petrochemical and Chemical Industry VNIPINeft, Head of the Oil Refining Technology Department of the Gubkin Russian State Oil & Gas University, Moscow, Russia*) - “Modern Oil-Refining Enterprises”

A.S. Noskov (*The Boreskov Institute of Catalysis, Novosibirsk, Russia*) – “Catalytic Processing of Vegetable Oils in the Fat-and-Oil Industry”

B.N. Kuznetsov (*Institute of Chemistry and Chemical Technology, Krasnoyarsk, Russia*) – “Catalytic Methods in the Production of Valuable Chemicals and Biofuels from Plant Biomass”

V.A. Yakovlev (*The Boreskov Institute of Catalysis, Novosibirsk, Russia*) – “Development of Catalytic Processes for Production of the New Generation Biofuels”

I.D. Reznichenko (*General Director of the Plant for the Catalyst Production and Organic Synthesis (Angarsk, Irkutsk region*) – “Modern Catalysts for Oil Refining and Petrochemistry”.

1ST RUSSIAN CONFERENCE “METHODS OF INVESTIGATION OF THE FUNCTIONAL MATERIALS COMPOSITION AND STRUCTURE”

October 11-16, Novosibirsk, Russia

Conference organizers:

- The Boreskov Institute of Catalysis, Novosibirsk
- Scientific Council on Analytic Chemistry of the Russian Academy of Sciences and Its Siberian Branch
- Scientific Council on Physical Chemical Foundations of Semiconductor Material Sciences of the Russian Academy of Sciences

The Scientific program comprised 20 plenary lectures, 7 key and 7 presentation lectures, 76 oral sectional presentations and 130 posters on the following topics:

- General Problems of Composition and Structure Study
- Methods for Determination of the Chemical Composition of Inorganic and Organic Solid Functional Materials at Macro-, Micro- and Nanolevels
- Methods for Crystal Structure Determination
- Methods for Electron Properties Determination
- Methods for Determination of Dispersity and Texture
- Thermal Analytical Methods.

Presentation seminars of three companies - Nytek Instruments, Interactive Corporation and Netzsch Gerätebau GmbH - took place in the frames of the Conference.

6 leading scientists of the Institute presented plenary lectures – **V.V. Malakhov, S.V. Tsybulya, O.N. Martyanov, O.B. Lapina, D.I. Kochubey, V.I. Zaikovsky.**

The whole number of participants was 250.

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 within 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.

From the first years, the strategy of the training of students was built up by leading scientists of BIC and harmonically combines two aspects: on the one hand, curricula provide the students with deep theoretical and practical knowledge in catalysis, i.e. theory of the prevision of the catalytic action of substances, kinetics of catalytic reactions, scientific bases of the preparation of catalysts, bases of the technology of catalytic processes, application of modern physical and mathematical methods for investigation of catalysts and catalysis; on the other hand, the educational process arrangement allows to wide significantly the sphere of the postgraduate activity of the students. 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 of the chair of adsorption and catalysis are the principal source of recruitment of the personnel of the Institute.

BIC – NSU

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. Sobyanin) 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 deal 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

Novosibirsk State Technical University is one of the largest research and educational centers in Russia. The University trains and retrains 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 industry. The engineers will be able to successfully work 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 to increase quality of 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. Head of the ESC is **Prof. V. Bukhtiyarov** (BIC).

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 Nanomaterial
- Catalytic Reactors Modeling.

The Educational Scientific Center *"Catalytic Hydrocarbon Processing"* is organized jointly with Gubkin Russian State University of Oil and Gas, Moscow in 2009. Head of the ESC is **Prof. V. Kapustin** (Gubkin University).

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.

The Educational Scientific Center "*Chemical Material Science and Prospect Technologies*" jointly with Tomsk State University in 2007. Head of the ESC is **Prof. V. Kozik** (TSU).

Specialization:

- Chemistry, Chemical Material Science.

The Educational Scientific Center *"Chemical Technologies and Catalysis"* was made on the bases of Kazan State Technological University in 2008. Head of the ESC is **Prof. Kh. 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 "Novosibirsky VNIPIET" in 2008. Head of the ESC is **Prof. Z. Ismagilov** (BIC).

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.

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 *Boreskov Institute of Catalysis* and *Novosibirsk State Technical University*, created in 2002. Scientific Coordinators are **Prof. Aleksandr S. Noskov** (The Boreskov Institute of Catalysis) and **Prof. Gennadii G. Kuvshinov** (Novosibirsk State Technical University).

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 Apparatuses with Moving Bed.

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

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 Mechanism of 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 *Boreskov Institute of Catalysis* (Novosibirsk) and *Volgograd State Technical University* (Volgograd) created in 2004. Scientific Coordinator is **Prof. Yu. 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 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

Besides teaching of the students in many Russian universities, researchers of the Institute participate in the training of young scientists of the highest qualification capable to provide original basic and applied research in the field of catalysis. Their training has priority importance for the Institute and is directly related 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 specialties: 02.00.15 "Catalysis" within chemical sciences and 02.00.04 "Physical Chemistry" within chemical sciences. Head of the Dissertation Council is the director of the Institute – Academician V. Parmon.

1 Doctoral thesis and 19 Ph.D. theses have been defended in 2009.

Oleg N. Martyanov – "*Size Effect and Interparticle Interaction in Magnetic Resonance Spectroscopy of Dispersed Magnets and Catalytic Systems on Their Base*"

Ph.D. theses:

Artem A. Barabanov – "*Kinetics of Ethylene Polymerization in the Presence of Fe, Co and Cr Bis(imine)Pyridine Complexes According to Data on the Number of Active Sites and Growth Rate Constants*"

Alexandr G. Gribovskii – "*Studying the Peculiarities and Diagnostics of Methane Steam Conversion in Microchannel Reactors*"

Elena L. Gubanova – "*Experimental Study and Modeling of the Selective Methane Oxidation to Syn-Gas over Monolith Catalysts at Short Contact Times*"

Irina V. Deliy – "*Study the Reaction of Hydrogenation and Isomerization of C=C Double Bond of Fatty Acids Methyl Esters and Monoterpenes in the Presence of Pd, Rh, Ru, Pt and Ir Based Catalysts*"

Petr M. Eletskii – "*Synthesis and Investigation of Carbon-Silica Composites, Meso- and Microporous Carbon Materials from High Ash Biomass*"

Aigana P. Kagyrmanova – "*Optimization of the Shape and Size of Catalyst Grain in Tubular Reactors with Fixed Granular Layer*"

Ivan V. Kozhevnikov – "*Thermal Transformation of Some Monoterpenes in Supercritical Conditions*"

Pavel A. Kolinko – "*Regularities of Photocatalytic Oxidation of Nitrogen Containing Compounds on the Surface of Titania*"

Eugenii A. Kravtsov – "*Structure and Reactivity of Active Centers of Post-Metallocene Catalysts for Olefine Polymerization Based on Ti- and Zr-Complexes*"

Anton I. Lysikov – "*Study of the Adsorbents and Catalysts for Adsorption-Catalytic Methane Conversion in Fixed Layer*"

Maria M. Matrosova – "*Acidic and Catalytic Properties of Tungstate Heteropoly Acids of 5 Structural Types – Keggin ($H_nPW_{11}XO_{40}$, where X = W(VI), Ti(IV), Zr(IV)), Dawson (α-$H_6P_2W_{18}O_{62}$), $H_6P_2W_{21}O_{71}(H_2O)_3$, $H_6As_2W_{21}O_{69}(H_2O)$ and $H_{21}B_3W_{39}O_{132}$*"

Il'ya Yu. Pakharukov - "*Study of the Peculiarities of the Kinetics of Heterogeneous Catalytic Reactions by Flow Circulation Method with Improved Operation*"

Anastasiya V. Pashigreva – "*Co-Mo Catalysts for Deep Hydropurification of Diesel Fractions*"

Mikhail N. Simonov – "*Development of Ecologically-Sound Method of Propylene Glycol Synthesis by Lactic Acid and Its Ethers Hydrogenation over Reduced Cu-Containing Catalysts*"

Irina A. Simonova – "*Composite Water Sorbents "$Ca(NO_3)_2$/Silica" and "$LiNO_3$/Silica"*"

Sergey Yu. Troitskii – "*Polynuclear Palladium, Ruthenium and Platinum Hydroxocomplexes: Synthesis, Composition, Structure and Application in the Processes of Catalysts Preparation*"

Zalia A. Fedorova – "*Synthesis and Investigation of Nickel Catalysts Based on Metal Supports for Steam Methane Conversion to Syn-Gas*"

Olga V. Chub - "*Studying of the Mass Transfer Processes in Glass Fiber Systems*"

Irina S. Yakovleva – "*Peculiarities of the Structure of Substituted Perovskites ($La_{1-x}Ca_xFeO_{3-y}$, $La_{1-x}Ca_xMnO_{3-y}$, $La_{1-x}Sr_xCoO_{3-y}$) and Their Reactivity in RedOx Processes*".

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

SCIENTIFIC SOCIAL LIFE

- **Valentin Parmon** was awarded the very prestigious Russian Federation State Prize in 2009 for science and technology for contribution to the theory and practice of catalytic methods for hydrocarbon raw materials processing and use of renewable resources.

- On the eve of New 2009 Year the Boreskov Institute of Catalysis was visited by a representative of the embassy of Republic Korea in the Russian Federation, attaché for science, Director of the representative office of the Foundation “International Cooperation in Science and Engineering” of Republic Korea and head of the laboratory of the Korean Institute of Science and Technology (Seoul).

- **On May, 21** the delegation of the Russian Nanotechnologies Corporation headed by the director general, the chairman of the State corporation board **A.B. Chubais** on their visit to Novosibirsk and Tomsk visited also the Boreskov Institute of Catalysis.

- On March, **Governor of the Novosibirsk Region Viktor Tolokonskii** visited the Institute.
- The *Second Nanotechnology International Forum* was held in accordance with the Concept approved by Russian Corporation of Nanotechnologies Supervisory Board and the Russian Federation Government Decree on October 6–8, 2009 in Moscow Central Exhibition Complex “Expocentre”. Forum program was concluded by the summary of the

Second International Competition of Scientific Papers in Nanotechnology for Young Researchers. 505 reports of Russian contestants from 58 cities and 13 reports of foreign participants from 9 countries were presented at the competition. 51 young scientists became laureates of the competition. The 1st Prize was given to **Dr. Ilya Mazov**, who participated in the section “*Carbon Nanomaterials: Nanodiamonds, Carbon Nanotubes, Fullerenes, Grapheme*”. He suggested novel composite materials, containing multilayer carbon nanotubes.

- **Best Paper** for the 1st WSEAS International Conference NANOTECHNOLOGY'09, University of Cambridge, UK, February 21-23, 2009 was evaluated to be *“Multi-Wall Carbon Nanotubes in Microwaves”* by **S.I. Moseenkov, V.L. Kuznetsov, A.N. Usoltseva, I.N. Mazov, A.V. Ishchenko** (T. Buryakov, O. Anikeeva, A. Romanenko, P. Kuzhir, D. Bychenok, K. Batrakov, S. Maksimenko, co-athors).

- **Prize-winners** of the Scholarship Student Competition-2009 became the following participants form BIC of the “*XLVII International Student Scientific Conference*” in sections “Catalysis and adsorption” and “Physical methods to study functional materials and nanosystems”: **Dmitrii A. Svintsitskii** (4th year student), **Stanislav S. Yakushkin** (1st year undergraduate), **Baira G. Donoeva** (5th year student), **Anna M. Ozerova** (5th year student) and **Alexey S. Zyryanov** (5th year student).

- **Invention** (Patent RF № 2314870) “*Catalyst for Steam CO Conversion, Method of Its Preparation and Principles of Use*” by **T.M. Yurieva, M.P. Demeshkina, A.A. Khassin, T.P. Minyukova, L.M. Plyasova, N.A. Baronskaya, M.V. Lebedeva,** (I.D. Reznichenko, L.G. Volchatov, A.P. Bocharov, M.I. Tselyutina, O.M. Posokhova, T.I. Andreeva, co-authors from Angarsk Catalyst and Organic Synthesis Plant Co.) was elected in nominations “**100 Best Inventions of Russia**” and awarded by Rospatent Diploma.

ФЕДЕРАЛЬНАЯ СЛУЖБА
ПО ИНТЕЛЛЕКТУАЛЬНОЙ
СОБСТВЕННОСТИ,
ПАТЕНТАМ И ТОВАРНЫМ
ЗНАКАМ

FEDERAL SERVICE
FOR INTELLEKTUAL
PROPERTY, PATENTS
AND TRADEMARKS

Диплом

НАГРАЖДАЕТСЯ

В номинации «100 лучших изобретений России»
Институт катализа им. Г.К. Борескова
Сибирского отделения Российской Академии наук,
ОАО Ангарский завод катализаторов и органического синтеза
за высоко-активный катализатор паровой конверсии
(патент РФ № 2314870)

Руководитель
Б.П. Симонов

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

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

- The Boreskov 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.

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.

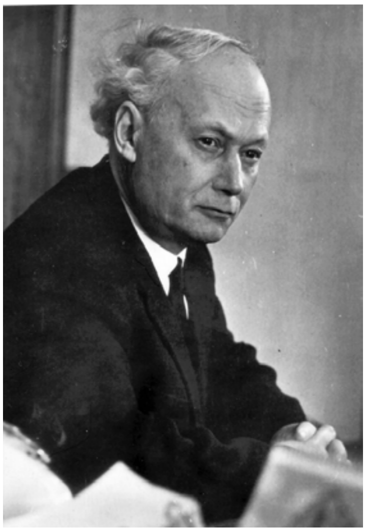
The main aim of the Foundation is to support gifted Russian scientific youth working in the field of chemical catalysis, physical methods of its research and chemical physics.

Major part of Foundation means is spent on Zamaraev post-graduate scholarships, prizes and grants. Foundation plans to support the 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 2009 **T. Kardash** and **I. Soshnikov** have got the post-graduate scholarship; **M. Ivanov, D. Kolokolov, Zh. Veselovskaya** and **A. Gabrienko** - incentive post-graduate scholarships.

- 17 Ph.D. students and scientific employees of the Institute form the personnel of the **Council of Scientific Youth.** The main principle of the Council is submission of the interests of the youth in the Institute. It includes 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 directions of activity connected with the improving of a position of the young employees in the Institute.

Memorial Rooms of Academician Georgii K. Boreskov and Academician Kirill I. Zamaraev

The **Memorial Room of Academician Georgii K. Boreskov**, the founder and first director of the Institute of Catalysis, was opened in June 1987, on the occasion of his 80th 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. Boreskov.



There is a small exhibition based on the documents, awards, books, Boreskov'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. Boreskov 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 Boreskov 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.

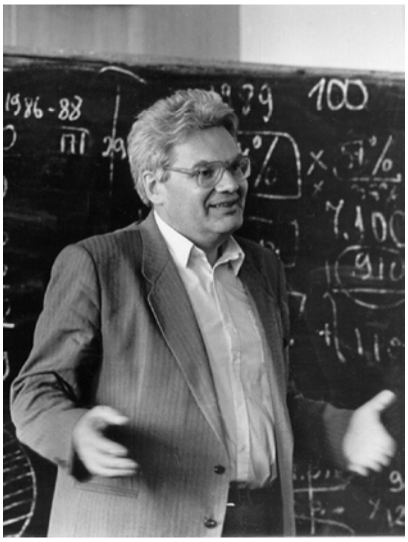
Boreskov's activity in the area of catalysis was recognized worldwide. He was a foreign member of the National Academy of Sciences of German Democratic Republic, honorary member of the New York Academy of Sciences, Doctor HONORIS CAUSA of the Wroclaw Polytechnic Institute (Poland) and of the Poitiers University (France). Government of Bulgarian Republic awarded the Order of Kirill and Mephodium to G.K. Boreskov. Visitors can see an unusual exposition displaying the full attributes of ceremonies of awarding the HONORIS CAUSA titles to Georgii Boreskov 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 Boreskov'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 20th of May 1999, the day of 60th 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 of the Memorial Room.

IMPORTANT RESULTS
OF THE R&D ACTIVITY

RESEARCH ACTIVITY

Quantum-Chemical Investigations

ON THE NATURE OF RADICALS FORMED IN METHANOL CATALYTIC OXIDATION

I.V. Malakhova, A.A. Shubin

Kinet. Catal.,
50(4) (2009) pp. 583-586.

The quantum-chemical calculations of the hydroxymethyl radical •CH_2OH were performed for the first time and a theoretical EPR spectrum of this radical was constructed. The formation of the hydroxymethyl radical in the reaction of methanol oxidation is thermodynamically favourable. The shape and parameters of the constructed spectrum differed from those for radicals experimentally detected in the catalytic oxidation of methanol using the matrix isolation method. However, they are consistent with the spectrum ascribed to the EPR spectrum of •CH_2OH observed in the direct photolysis of methanol. This result allows one to refine the identification of the nature of radicals formed in the catalytic reaction of methanol oxidation.

NITROSONIUM NITRITE ISOMER OF N_2O_3: QUANTUM-CHEMICAL DATA

I.I. Zakharov*,, O.I. Zakharova****
(**The Boreskov Institute of Catalysis, Novosibirsk, Russia; **Severodonetsk Technological Institute, V. Dal West Ukrainian National University, Severodonetsk, Ukraine*)

J. Struct. Chem.,
50(2) (2009) pp. 212-218.

The geometrical, electronic, and thermodynamic parameters of three known isomers of dinitrogen trioxide N_2O_3 were calculated by the density functional theory DFT/B3LYP method using the 6-311++G(3*df*) basis. The structure of the new isomer, $NONO_2$, was calculated. From the calculation of vibrational frequencies it follows that the structure of $NONO_2$ has a local potential energy minimum and corresponds to the stationary state of the N_2O_3 isomer. The molecular structure of $NONO_2$ is characterized by a substantial negative charge on the NO_2 fragment and positive charge on the NO fragment. The electronic structure of the $NO^+NO_2^-$ isomer can be characterized as *nitrosonium nitrite*, which can be oxidized to nitrite and participate in nitrosylation in accordance with the biogenic characteristics of the NO_x intermediate, assumed to be formed in biological systems during the oxidation of NO.

EFFECT OF MASS TRANSFER ON THE REACTION RATE IN A MONOLITHIC CATALYST WITH POROUS WALLS

O.P. Klenov, S.A. Pokrovskaya, N.A. Chumakova, S.N. Pavlova, V.A. Sadykov, A.S. Noskov

Catal. Today,
144(3-4) (2009) pp. 258-264.

The influence of reacting gas flow on the heterogeneous reaction in catalyst volume of honeycomb porous monolith with triangular channels was studied. The spatial distributions of the reacting gas flow, the rates of local mass transfer between channel walls and gas flow, as well as the interaction of transfer processes and a catalytic reaction were determined on the basis of the computational fluid dynamics. The reactions of deep oxidation and steam reforming of methane were considered as model reactions.

It was shown that there is no stabilization of the reacting flow over the whole channel length under studied conditions. The most intensive changing of the gas streams appears near the channel inlet, which causes the highest local rates of interphase exchange processes, the rate difference is up to two orders of magnitude. A higher reaction rate exists in the initial part due to penetration of feed components into the catalyst volume through the frontal surface. This leads to increasing the effectiveness factor whose value is above unity near the channel inlet. The reaction rate limitation by the transport of reagents inside porous wall was observed along the monolith length.

OXIDATION OF FORMALDEHYDE TO FORMIC ACID OVER V_2O_5/TiO_2 CATALYSTS: A DFT ANALYSIS OF THE MOLECULAR REACTION MECHANISMS

V.I. Avdeev, V.N. Parmon

J. Phys. Chem.,
113(7) (2009) pp. 2873-2880.

Oxidation of formaldehyde to formic acid on the surface of titania-supported vanadia is studied with density functional theory (DFT) and a dioxo-vanadyl (O=V-O-V=O) catalyst model. Two probable mechanisms (a redox Mars-van Krevelen mechanism and an associative mechanism) are considered and transition states and intermediates along these reaction pathways are investigated. The key intermediate of the redox mechanism is a dioxymethylene complex. Its successive transformation leads to a stable surface formate complex. The subsequent transformation of formate to formic acid can occur only in the presence of dioxygen. The key intermediate of the associative mechanism is a peroxo-oxo-methylene complex. Its successive transformation leads to the adsorbed formic acid. On the basis of the calculated activation barriers, the associative mechanism appears to be more probable.

ELECTRONIC STRUCTURE AND STABILITY OF PEROXIDE DIVANADATE SPECIES V(O−O) ON THE TiO_2(001)

V.I. Avdeev, V.M. Tapilin

J. Phys. Chem. C,
113(33) (2009) pp. 14941-14945.

Electronic structure and stability of the VO_x species forming on the anatase-TiO_2(001)-(4×1) reconstructed surface at redox cycle V^{5+}−O−V^{5+} ↔ V^{4+}−O−V^4 have been investigated on the basis of periodic density functional theory calculations. It is shown that oxidizing of the key intermediate of Mars–van Krevelen mechanism [O−V^{4+}−O−V^{4+}] by the gas phase dioxygen leads to the substantial structure relaxation of the surface VO_x species within a monolayer. As a result, the binding energy of the vanadyl bond V═O decreased and the stable peroxide complex V(O−O) can be formed on the fully oxidized VO_x/TiO_2 surfaces. This complex manifests itself as a peak near Fermi level (−0.23 eV) in the total density of states (DOS) and O-projected density of states (PDOS).

QUANTUM CHEMICAL CALCULATION OF THE CATALYTIC REACTION OF ETHANE DEHYDROGENATION ON GALLIUM OXIDE-HYDROXIDE BINUCLEAR CLUSTERS IN OXIDIZED GaO/ZSM-5 ZEOLITE

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Kinet. Catal.,
50(5) (2009) pp. 752-759.

The catalytic activity of oxidized GaO/HZSM-5 in the reaction of alkane dehydrogenation can be due to hydrogenated gallium oxide clusters stabilized in the cationic positions of the zeolite. The binuclear gallium oxide clusters $[Ga_2O_2]^{2+}$ in oxidized gallium-substituted high-silica zeolite HZSM-5, which are isomeric to two gallyl ions $[GaO]^+$ stabilized on two spatially separated lattice aluminum ions, were considered using the DFT method within the framework of a cluster approach. It was found that, even in the case of a relatively large distance between these aluminum ions, gallium oxide particles in oxidized GaO/HZSM-5 can occur as charged planar $[Ga_2O_2]^{2+}$ four-membered rings. These cluster particles exhibited a high affinity to hydrogen, and they were readily hydrogenated with the retention of their structural integrity. It was demonstrated that this partially hydrogenated cluster could be responsible for the catalytic process of ethane dehydrogenation. In the first step, ethane dissociatively added to the $[Ga_2O_2H_2]^{2+}$ cluster. Then, the ethylene molecule was eliminated from the resulting intermediate to leave the $[Ga_2O_2H_4]^{2+}$ cluster. The cycle was closed by the elimination of a hydrogen molecule with the formation of the initial structure of $[Ga_2O_2H_2]^{2+}$.

ON THE STABILITY OF COPPER(II) ORGANIC COMPOUNDS WITH THE σ BOND Cu-C: A QUANTUM-CHEMICAL STUDY

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Doklady Chem.,
426(2) (2009) pp. 143-145.

In this paper, the results of quantum chemical calculations of the structure and properties of copper(II) organic compounds are reported with the aim to demonstrate the possibility of their existence by the example of $Cl_nCu(II)R_{1-n}$, where R is C and C_3, and determine their geometric parameters and the Cu–C bond energy. In addition, it was important to calculate the energies of possible mono- and bimolecular reactions involving $Cl_nCu(II)R_{1-n}$ and justify the most probable mechanism of $Cl_nCu(II)R_{1-n}$ transformations.

DFT ANALYSIS OF PROPANE CYCLIZATION OVER BINUCLEAR Ga-CLUSTERS IN MORDENITE

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J. Mol. Catal. A: Chem.,
305(1-2) (2009) pp. 90-94.

Molecular and dissociative adsorption of propane on binuclear $(Ga_2O_2)^{2+}$ cluster located at the cation positions of Ga-exchanged mordenite zeolite are modelled using DFT calculations via the isolated cluster approach. Relative energies of the dissociative intermediates show the most stable bidentate complexes, i.e., via both primary C atoms of propane to both Ga atoms. This structure really is the suitable precursor of cyclo-propane formation. The hypothesis of cyclo-propane intermediate was proposed by Derouane et al. to explain the $^{13}C/^{12}C$ exchange migration within propane molecules in Ga-exchanged zeolites.

ION-EXCHANGED BINUCLEAR CLUSTERS AS ACTIVE SITES OF SELECTIVE OXIDATION OVER ZEOLITES

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J. Phys. Chem., C,
113(19) (2009) pp. 8258-8265.

A new catalytic oxidation cycle over binuclear Zn- and Ca-cation clusters in zeolites is proposed. Intermediate active clusters appear due to trapping of dioxygen. CO oxidation is considered as a model reaction over a cluster located in an 8-membered (8R) ring. Geometries of active clusters involved in the catalytic cycle vary depending on the nature and size of the cation. Reagents, transition states, and products have been optimized at the isolated cluster level considering DFT functionals (B3LYP, B3P86, and B3PW91) with the 6-31G* or 6-311++G** basis sets. Moderate activation energies of 23.0 and 35.7 kcal/mol at the B3LYP/6-31G* level are obtained for CO oxidation over Zn- and Ca-clusters, respectively.

GENERATION OF O¯ RADICAL ANIONS ON MgO SURFACE: LONG-DISTANCE CHARGE SEPARATION OR HOMOLYTIC DISSOCIATION OF CHEMISORBED WATER?

S.E. Malykhin, A.M. Volodin, A.F. Bedilo, G.M. Zhidomirov

J. Phys. Chem., C,
113(24) (2009) pp. 10350-10353.

O^- radical anions were observed over a partially hydroxylated MgO surface after illumination by monochromatic light with λ = 280 and 303 nm. As all comer oxygen atoms are covered with adsorbed hydroxyl groups after activation at 450°C, this process is initiated by selective excitation of 3-coordinated complexes $[Mg^{2+}—O^{2-}]_{3C}$ containing chemisorbed water. A new mechanism for generation of the O^- radical anions is suggested. It is based on homolytic dissociation of chemisorbed water followed by migration of a hydrogen atom, probably, to a different nanoparticle rather than on long-distance separation of charges. The surface structure remaining after its detachment consists of an •OH radical stabilized near the corner oxygen atom. The imminent charge transfer generates a hole at the corner oxygen atom stabilized by a hydroxyl group $[OH—\cdots Mg^{2+}—O^-]_{3C}$. DFT simulation of this structure showed that it reproduces the main characteristics of the radical anions $O^-{}_{3C}$. The overall structure is electrically neutral. The charge of the hole is compensated by the nearby hydroxyl group. So, no long-distance charge separation is required.

INTERACTION OF A SINGLE WATER MOLECULE WITH A SINGLE GRAPHITE LAYER: AN INTEGRATED ONIOM STUDY

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J. Phys. Chem., C,
113(15) (2009) pp. 6118-6123.

Integrated ONIOM calculations using the two- and/or three-layer model chemistries were applied to mimic the interaction of water with the basal graphitic layer of HOPG material. The high-level layer in the ONIOM approach is treated at the electron correlated second-order Moller-Plesset perturbation (MP2/6-31G*) level. On the basis of the results obtained, the formation of structural defect sites, molecular and dissociative adsorption complexes of water on a regular and on structural defect sites, the discrepancy in the nature of the surface O-H and C-H groups appeared on SWNT and HOPG materials has been briefly discussed. It was shown that the dissociation of water is highly endothermic on regular sites of HOPG while the energy needed for dissociation decreases substantially if it proceeds on structural defect sites. This finding points out indirectly that the dissociation probability of water increases on SWNT materials as compared to HOPG because of the presence of a larger number of structural defect sites.

MP2 STUDY ON DECOMPOSITION OF NITROUS OXIDE ON THE Ga-ZSM-5

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Int. J. Quantum Chem.,
108(14) (2008) pp. 2732-2743.

The reaction mechanism for nitrous oxide decomposition has been studied on gallium site in Ga-ZSM-5 using the MP2/6-31+G(d) method. The active centers were taken to be mononuclear $[Ga]^+$, $[Ga{=}O]^+$, and $[GaO_2]^+$ and the surrounding portion of the zeolite was represented by a 3T cluster, namely $[AlSi_2O_4H_8]^-$. The first elementary step of N_2O decomposition involves the formation of $[GaO]^+$ and the release of N_2. The metal-oxo species produced in this step then reacts with N_2O again, to release N_2 and form $[GaO_2]^+$. The calculated activation energies at MP2 level for N_2O dissociation on Ga-ZSM-5 and GaO-ZSM-5 are 15.7 and 26.5 kcal/mol at 298 K, respectively. The third elementary step of N_2O decomposition on GaO_2-ZSM-5 involves the formation of $[GaO_3]^+$ and the release of N_2. The calculated activation energy at MP2 level for N_2O dissociation on GaO_2-ZSM-5 is 43.7 kcal/mol. Four-order perturbation theory (MP4//MP2) predicts that the activation barriers for nitrous oxide dissociation at 298 K on Ga-ZSM-5, GaO-ZSM-5, and GaO_2-ZSM-5 are 13.9, 13.0, and 34.4 kcal/mol, respectively. The calculated energy for desorption of singlet O_2 from the $3T^-[Ga(O)_3]^+$ cluster at MP2 level is 33.4 kcal/mol. When one takes into account the entropy gained on desorption of singlet O_2, the contribution of entropy to the free energy of desorption is $T\Delta S$ = 12.3 kcal/mol at 298 K. The calculated energy of the singlet oxygen desorption from $3T^-[OGaO_2]^+$ cluster ΔH (298 K) = +43.57 kcal/mol and ΔG(298 K) = 30.13 kcal/mol is significantly higher than the barriers of the singlet molecular oxygen desorption from $3T^-[Ga(O)_3]^+$ cluster.

SURFACE SPECIES OF TITANIUM(IV) AND TITANIUM(III) IN $MgCl_2$-SUPPORTED ZIEGLER-NATTA CATALYSTS. A PERIODIC DENSITY FUNCTIONAL THEORY STUDY

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

Macromol.,
42(21) (2009) pp. 8165-8171.

A systematic consideration of different Ti(IV) and Ti(III) species on the (104) and (110) $MgCl_2$ surfaces has been implemented within DFT using cyclic boundary conditions. Some new mononuclear and dinuclear surface complexes of Ti(IV) and Ti(III) were obtained due to implication of zip coordination mode. A possible spin state of dinuclear Ti(III) species was thoroughly studied: antiferromagnetic (ESR silent) state proved to be the most preferable in a number of cases. The zip antiferromagnetic Ti_2Cl_6 complexes residing on the dominant (104) $MgCl_2$ surface make it possible to rationalize the fact that the most part of Ti(III) incorporated in activated $MgCl_2$ is ESR silent. Besides, these species produce aspecific active sites, thus explaining that aspecific centers significantly prevail over stereospecific one according to kinetic data on the simplest $TiCl_4/MgCl_2$ + AlR_3 system.

THEORETICAL STUDY OF CARBON SPECIES ON Pd(111): COMPETITION BETWEEN MIGRATION OF C ATOMS TO THE SUBSURFACE INTERLAYER AND FORMATION OF C_N CLUSTERS ON THE SURFACE

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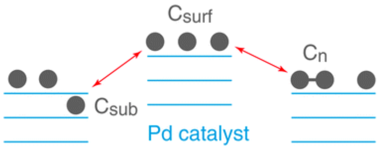
***University of Sofia, Sofia, Bulgaria*)

Phys. Chem. Chem. Phys.,
11(46) (2009) pp. 10955-10963.

Subsurface carbon species of Pd catalysts recently attracted considerable attention because they affect the selectivity of hydrogenation reactions. The migration of C atoms from the Pd(111) surface to interstitial subsurface sites was calculated to be energetically favorable. Yet, thermodynamically more stable is a graphene-like phase on the Pd surface. Applying a density functional method on periodic models, the formation of C_n (n = 2–4) clusters on Pd(111) was explored. At low coverage, carbon monomers on the surface and at octahedral subsurface sites were calculated to be more stable than dimer species, C_2, on the surface. However, at a C coverage of about half a monolayer, the formation of C_2 and C_3 species, precursors of a graphene phase, becomes competitive with migration of C monomers to octahedral subsurface sites. While discussing these findings, the authors also addressed the problem of C_1 formation on Pd catalysts from simple organics.



MECHANISM OF SELECTIVE HYDROGENATION OF α,β-UNSATURATED ALDEHYDES ON SILVER CATALYSTS: A DENSITY FUNCTIONAL STUDY

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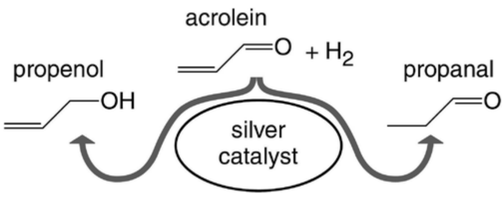
J. Phys. Chem. C,
113(30) (2009) pp 13231-13240.

Supported silver catalysts exhibit a remarkably high selectivity in the industrially important hydrogenation of α,β unsaturated aldehydes to unsaturated alcohols. Density functional calculations have been carried out to clarify factors that affect the catalytic function of silver in hydrogenating unsaturated aldehydes. The activity and the selectivity of model silver catalysts for acrolein, the simplest, yet most difficult unsaturated aldehyde to be selectively hydrogenated was examined. The focus has been made on describing bulky catalyst particles, represented by sites on extended silver surfaces, on the regular clean Ag(110) surface and the surface O_{sub}/Ag(111) with subsurface oxygen centers. On Ag(110) the results imply propanal, the undesired saturated aldehyde, to be the main product. In contrast, the calculations suggest a very high selectivity of O_{sub}/Ag(111) for the corresponding unsaturated alcohol, allyl alcohol, although the activity of this system is lower than that of clean silver. At variance with Pt(111), where the selectivity to allyl alcohol is strongly reduced by the hindered desorption of the latter, allyl alcohol and propanal products are predicted to desorb easily from both Ag(110) and O_{sub}/Ag(111) at common reaction temperatures. Inherent limitations for an accurate description of the chemical regioselectivity by contemporary computational methods have been also analyzed.



KOOPMANS’ THEOREM IN THE RESTRICTED OPEN-SHELL HARTREE−FOCK METHOD: I. A VARIATIONAL APPROACH

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J. Phys. Chem. A,
113(45) (2009) pp 12386-12395.

A general formulation of Koopmans’ theorem is derived for high-spin half-filled open shells in the restricted open-shell Hartree−Fock (ROHF) method based on a variational treatment of *both* the initial (nonionized) open-shell system under study, e.g., X, and the corresponding high-spin ions X_k^+, X_m^+, and X_v^- having a hole or an extra electron in the closed, open, and virtual shell, respectively. The ions are treated within a FCI-RAS (full CI in the restricted active space) method with a use of arbitrary ROHF orbitals optimal for the initial system. It was shown that the desired *canonical* ROHF orbitals and orbital energies satisfying Koopmans’ theorem, first defined within the canonical ROHF treatment [Plakhutin; et al. *J. Chem. Phys.* 2006, *125*, 204110], generally appear as the natural CI orbitals and the eigenvalues of CI matrices for the respective ions $X^{\pm}$. A comparison is performed between the results derived with the present CI approach and the canonical ROHF method for the specific case where the canonical orbital energies satisfying Koopmans’ theorem do not satisfy the Aufbau principle.

PAIRED ORBITALS FOR DIFFERENT SPINS EQUATIONS

I.L. Zilberberg, S.F. Ruzankin

J. Comput. Chem.,
31(1) (2009) pp. 84-89.

Eigenvalue-type equations for Löwdin-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 eigenvectors 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.

PROTON ENVIRONMENT OF REDUCED RIESKE IRON−SULFUR CLUSTER PROBED BY TWO-DIMENSIONAL ESEEM SPECTROSCOPY

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J. Phys. Chem. A,
113(4) (2009) pp 653-667.

The proton environment of the reduced [2Fe-2S] cluster in the water-soluble head domain of the Rieske iron−sulfur protein (ISF) from the cytochrome bc_1 complex of *Rhodobacter sphaeroides* has been studied by orientation-selected X-band 2D ESEEM. The 2D spectra show multiple cross-peaks from protons, with considerable overlap. Samples in which ^1H_2O water was replaced by ^2H_2O were used to determine which of the observed peaks belong to exchangeable protons, likely involved in hydrogen bonds in the neighborhood of the cluster. By correlating the cross-peaks from 2D spectra recorded at different parts of the EPR spectrum, lines from nine distinct proton signals were identified. Assignment of the proton signals was based on a point-dipole model for interaction with electrons of Fe(III) and Fe(II) ions, using the high-resolution structure of ISF from *Rb. sphaeroides*. Analysis of experimental and calculated tensors has led to conclude that even 2D spectra do not completely resolve all contributions from nearby protons. Particularly, the seven resolved signals from nonexchangeable protons could be produced by at least 13 protons. The contributions from exchangeable protons were resolved by difference spectra (^1H_2O minus ^2H_2O), and assigned to two groups of protons with distinct anisotropic hyperfine values. The largest measured coupling exceeded any calculated value. This discrepancy could result from limitations of the point dipole approximation in dealing with the distribution of spin density over the sulfur atoms of the cluster and the cysteine ligands, or from differences between the structure in solution and the crystallographic structure. The approach demonstrated here provides a paradigm for a wide range of studies in which hydrogen-bonding interactions with metallic centers has a crucial role in understanding the function.

Monte-Carlo Simulation to Study Physico-Chemical Processes

SELF-OSCILLATIONS AND CHEMICAL WAVES IN CO OXIDATION ON Pt AND Pd: KINETIC MONTE CARLO MODELS

V.I. Elokhin, A.V. Matveev, V.V. Gorodetsky

Kinet. Catal.,
50(1) (2009) pp. 40-47.

Theoretical studies of the spatiotemporal dynamics of CO oxidation on Pt(100) and Pd(110) single crystal surfaces have been carried out by the kinetic Monte Carlo method. For both surfaces, Monte Carlo simulation has revealed oscillations of the CO_2 formation rate and of the concentrations of adsorbed species. The oscillations are accompanied by wave processes on the model surface. Simulations have demonstrated that there is a narrow reaction zone when an oxygen wave propagates over the surface. The existence of this zone has been confirmed by experimental studies. Taking into account the anisotropy of the Pd(110) crystal has no effect on the oscillation period and amplitude, but leads to the formation of elliptic oxygen patterns on the surface. It is possible to obtain a wide variety of chemical waves (cellular and turbulent structures, spirals, rings, and strips) by varying the parameters of the computational experiment.

SIMULATION OF PHYSICOCHEMICAL PROCESSES PROCEEDING OVER CATALYTIC NANOPARTICLES

V.I. Elokhin, E.V. Kovalev

Chem. Ind. Today,
9 (2009) pp. 23-26.

The situation in the field of simulation of kinetics of catalytic reactions on the supported metals is considered and an approach is proposed (at qualitative level) which permits to take into account the change in the shape and surface morphology of nanoparticles under the influence of the reaction media to interpret reactions kinetics.

SIMULATION OF ADSORPTION PROCESSES PERFORMANCE OVER SUPPORTED METAL NANOPARTICLES

E.V. Kovalev, V.I. Elokhin

Chem. Engn. J.,
154(1-3) (2009) pp. 88-93.

The statistical lattice model has been proposed which permits one to take into account the change in the shape and surface morphology of the nanoparticle under the influence of the reaction media. The influence of monomolecular and dissociative adsorption on the particles equilibrium shape and surface morphology has been studied. It has been shown that by taking into account attraction “adsorbate-metal” the reshaping of the initial hemispheric particle into the truncated pyramidal one occurs induced by adsorption, similar to the experimentally observed reversible reshaping of active nanoparticles.

FROM SINGLE CRYSTALS TO SUPPORTED NANOPARTICLES IN OSCILLATORY BEHAVIOR OF CO+O$_2$ REACTION ON PLATINUM AND PALLADIUM SURFACES: EXPERIMENT AND STOCHASTIC MODELS

V.I. Elokhin, A.V. Matveev, E.V. Kovalev, V.V. Gorodetsky

Chem. Engn. J.,
154(1-3) (2009) pp. 94-106.

The dynamic behavior of the catalytic CO oxidation reaction on the metal surfaces (Pt, Pd) of different structures: single crystals Pt(100), Pd(110); microcrystals (Pt, Pd tips); nanoparticles (Pd/support)) has been studied experimentally by FEM, HREELS, TPR, TDS, MB techniques and kinetic Monte Carlo modeling. The CO, O$_2$ adsorption and CO + O$_2$ reaction was studied by HREELS, TPR and TDS. Sharp tips of Pt and Pd, several hundreds angstroms in radius, were used to perform *in situ* investigations of the dynamic surface processes by FEM. HREELS studies on the Pt(100) surface demonstrate that the self-oscillations and waves propagation are connected with periodic changes in the surface structure of nanoplane (100)–(hex) ↔ (1x1), varying the catalytic property of metal. The fundamentally different feedback mechanism generating the kinetic oscillations has been identified on the Pd surfaces: the reversible subsurface oxygen formation, which modifies the adsorption and catalytic properties of the surface. Based on experimental results, two models for the CO+O$_2$ reaction on Pt(100) and Pd(110) surfaces are proposed for stochastic Monte Carlo modeling. Carbon monoxide spillover was taken into account in the stochastic model of oscillatory CO oxidation reaction over model Pd supported nanoparticles.

CATALYTIC PROCESSES: NANOSCALE SIMULATIONS

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In *“Dekker Encyclopedia of Nanoscience and Nanotechnology”*, Eds. C.I. Contescu, K. Putyera,
Taylor & Francis: New York, 2009,
vol. II., pp. 782-793.

In heterogeneous catalysis, adsorption and reaction processes usually occur on supported metal nanoparticles. To study such processes experimentally, a vast variety of surface-science techniques came into the use in the last decades. Along with the appearance of high-precision techniques for surface characterization, considerable progress in manufacturing of model catalysts with well-defined properties like particle size, shape and separation has been achieved. But the cognitive potential of the experimental studies in itself remain still limited because the measurements are usually indirect and the information derived demands quantitative processing and interpretation. This can be done only with the use of mathematical models. Successful interpretation of the results requires constructing mathematical models providing accurate description of systems under consideration. This entry is focused on theoretical studies (simulation) of the catalytic reaction kinetics on the active supported nanoparticles. The goal of this contribution is to describe the main achievements of the corresponding theoretical works.

KINETIC AND THERMODYNAMIC CHARACTERIZATION OF SINGLE-MISMATCH DISCRIMINATION USING SINGLE-MOLECULE IMAGING

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Nucleic Acids Res.,
37(14) (2009) e99.

A single-molecule detection setup based on total internal reflection fluorescence (TIRF) microscopy has been used to investigate association and dissociation kinetics of unlabeled 30mer DNA strands. Single-molecule sensitivity was accomplished by letting unlabeled DNA target strands mediate the binding of DNA-modified and fluorescently labelled liposomes to a DNA-modified surface. The liposomes, acting as signal enhancer elements, enabled the number of binding events as well as the residence time for high affinity binders $K_d < 1$ nM, $k_{off} < 0.01$ s^{-1}) to be collected under equilibrium conditions at low pM concentrations. The mismatch discrimination obtained from the residence time data was shown to be concentration and temperature independent in intervals of 1-100 pM and 23-46°C, respectively. This suggests the method as a robust means for detection of point mutations at low target concentrations in, for example, single nucleotide polymorphism (SNP) analysis.

SIGNAL PROPAGATION IN STEM-CELL NICHES

V.P. Zhdanov

Physica A,
388(18) (2009) pp. 3791-3797.

Stem cells, maintaining tissue homeostasis, are nurtured in microscopic niches formed of so-called environmental cells. The kinetics of proliferation and differentiation of stem cells in such niches depend on their interaction with the messenger proteins secreted by environmental cells. A generic mean-field kinetic model of the propagation of such signals is proposed. To motivate the study, the authors briefly describe a stem-cell niche in the Drosophila ovary. The model is however applicable to other niches as well. In particular, it helps one to understand the necessary conditions for the niche function. For example, the model predicts that in the case of the Drosophila ovary each germline stem cell should have in the external membrane at least 700 receptors interacting with the signalling Dpp and Gpp proteins emanating from the cap cells.

KINETICS OF THE FORMATION OF CANCER METASTASES VIA INDUCED PREMETASTATIC CANCER-STEM-CELL NICHES

V.P. Zhdanov

Phys. Rev. E,
79(6) (2009) 061913 (4 pp.)

Kinetic model describing the formation of new metastases by cancer stem cells in premetastatic stem-cell niches induced by the factors produced by a primary tumor and already formed metastases is presented. The corresponding kinetics is analyzed by employing mean-field kinetic equations and Monte Carlo simulations. In agreement with observations, the model predicts a long latent period with low rate of the metastase growth followed by explosive increase in the number of metastases. The duration of the latent period is found to depend on a multitude of rate constants characterizing various processes.

INTRACELLULAR VIRAL KINETICS LIMITED BY THE SUPPLY OF AMINO ACIDS FOR SYNTHESIS OF VIRAL PROTEINS

V.P. Zhdanov

Biosyst.,
97(2) (2009) pp. 117-120.

A minimal model proposed by the author [Zhdanov, V.P., 2004. Stochastic kinetics of reproduction of virions inside a cell. Biosystems 77, 143-150] to describe intracellular viral kinetics includes genome replication, mRNA and protein synthesis and degradation, capsid assembly, and virion release from a cell. Here, this model is complemented by the terms describing the balance of the amino acid determining the rate of the synthesis of viral capsid protein. If the effect of virions on this balance is negligible, the model predicts either a steady state or unlimited growth of the virion population. In the latter case, the cell eventually reaches the situation when the amino-acid concentration is reduced due to the synthesis of viral protein. For this stage, the viral-genome replication is asymptotically predicted to be unlimited while the virion population is limited. The unlimited viral-genome replication practically means that the cell will either die or the kinetics will be limited by additional feedbacks which were not taken into account in the model. All these findings, illustrating the use of the methods of integrative biology of biosystems, help to understand the role of the amino-acid supply in intracellular viral kinetics.

INTERPLAY OF BISTABLE KINETICS OF GENE EXPRESSION DURING CELLULAR GROWTH

V.P. Zhdanov

J. Phys. A: Math. Theor.,
42(6) (2009) 065102 (8 pp).

In cells, the bistable kinetics of gene expression can be observed on the level of (i) one gene with positive feedback between protein and mRNA production, (ii) two genes with negative mutual feedback between protein and mRNA production, or (iii) in more complex cases. The interplay of two genes of type (ii) governed by a gene of type (i) during cellular growth was analysed. In particular, using kinetic Monte Carlo simulations, it was shown that in the case where gene 1, operating in the bistable regime, regulates mutually inhibiting genes 2 and 3, also operating in the bistable regime, the latter genes may eventually be trapped either to the state with high transcriptional activity of gene 2 and low activity of gene 3 or to the state with high transcriptional activity of gene 3 and low activity of gene 2. The probability to get to one of these states depends on the values of the model parameters. If genes 2 and 3 are kinetically equivalent, the probability is equal to 0.5. Thus, the model illustrates how different intracellular states can be chosen at random with predetermined probabilities. This type of kinetics of gene expression may be behind complex processes occurring in cells, e.g., behind the choice of the fate by stem cells.

OSCILLATORY KINETICS OF GENE EXPRESSION: PROTEIN CONVERSION AND SLOW mRNA TRANSPORT

V.P. Zhdanov

J. Exp. Theor. Phys.,
108(6) (2009) pp. 1050-1053.

The negative feedback between mRNA and regulatory-protein production may result in oscillations in the kinetics of gene expression if the mRNA-protein interplay includes protein conversion. Using a mean-field kinetic model, it is shown that such oscillations can be amplified due to limitations of the mRNA transport between the nucleus and cytoplasm. This effect may be dramatic for the mRNA population in the nucleus.

CONDITIONS OF APPRECIABLE INFLUENCE OF microRNA ON A LARGE NUMBER OF TARGET mRNAs

V.P. Zhdanov

Mol. Biosyst.,
5(6) (2009) pp. 638-643.

In eukaryotic cells, messenger RNAs (mRNAs) can be regulated by microRNAs (miRNAs) via association and subsequent degradation. Each miRNA is now believed to potentially have hundreds of target mRNAs. Employing a generic kinetic model with physically reasonable parameters, the author has quantified the mutual influence of miRNA and mRNAs in the case when the number of target mRNAs is large (e. g., 100). The decrease in the population of mRNAs due to interaction with miRNA is found to be appreciable (about 1.5-2-fold) only if the rate of the miRNA synthesis is very high. In the absence of the miRNA-mRNA interaction, it should be sufficient to maintain the miRNA population in the order of 10^4 per cell. In addition, the average mRNA population should not be too high (lower than or comparable to 100 for each kind of mRNA). For lower miRNA synthesis rates, the significant influence of miRNA on mRNAs is only possible provided that the average mRNA population is very low (of the order of 10). These general findings are complemented by a brief discussion of some relevant recent experimental results.

BISTABILITY IN GENE TRANSCRIPTION: INTERPLAY OF MESSENGER RNA

V.P. Zhdanov

Biosyst.,
95(1) (2009) pp. 75-81.

The author proposes a kinetic model describing the interplay of messenger ribonucleic acid (mRNA), protein, produced via translation of this RNA, and nonprotein coding RNA (ncRNA). The model includes association of mRNA and ncRNA and regulation of the ncRNA production by protein. In the case of positive feedback between the production of protein and ncRNA, the steady state of the system is found to be unique. For negative feedback, the model predicts in the mean-field case either unique steady state or bistable kinetics. With incorporation of fluctuations, the bistability is manifested in the form of kinetic bursts provided that the number of reactants is low. Basically, the model describes the simplest biological switch operating with participation of ncRNA. Although the results obtained are applicable to ncRNSs in general, the presentation is focused primarily on microRNAs (miRNAs) which form a large important subclass of ncRNAs and are thought to regulate up to one third of all human genes.

FEASIBILITY OF THE NON-CODING RNA GRADIENTS IN CELLS

V.P. Zhdanov

Biophys. Rev. Lett.,
4(3) (2009) pp. 267-272.

The author proposes a coarse-grained kinetic model of the supply of oil during the next 50-year period. The oil supply, characterized by amount of oil extracted per day, is considered to depend on the oil price, existing capacities of oil extraction, the ability to find and develop new oil reserves, and also on the delay between investments and oil production. With reasonable parameters, the model predicts what may happen in realistic, optimistic, and pessimistic scenarios.

MODEL OF GENE TRANSCRIPTION INCLUDING THE RETURN OF A RNA POLYMERASE TO THE BEGINNING OF A TRANSCRIPTIONAL CYCLE

V.P. Zhdanov

Phys. Rev. E,
80(5) (2009) 051925 (6 pp.).

The gene transcription occurs via the RNA polymerase (RNAP) recruitment on the DNA promoter sequence, formation of a locally open DNA chain, promoter escape, steps of the RNA synthesis, and RNA and RNAP release after reading the final DNA base. Just after the end of the RNA synthesis, RNAP surrounds the closed DNA chain and may diffuse along DNA, desorb, or reach the promoter and start the RNA-synthesis cycle again. A generic kinetic model is presented taking the latter steps into account and show analytically and by Monte Carlo simulations that it predicts transcriptional bursts even in the absence of explicit regulation of the transcription by master proteins.

COARSE-GRAINED MODEL OF LONG-TERM SUPPLY OF OIL

V.P. Zhdanov

Eur. Phys. J. B,
71(2) (2009) pp. 289-292.

The author proposes a coarse-grained kinetic model of the supply of oil during the next 50-year period. The oil supply, characterized by amount of oil extracted per day, is considered to depend on the oil price, existing capacities of oil extraction, the ability to find and develop new oil reserves, and also on the delay between investments and oil production. With reasonable parameters, the model predicts what may happen in realistic, optimistic, and pessimistic scenarios.

MASS TRANSPORT ON COMPOSITE CATALYTIC SURFACES

V.P. Zhdanov

J. Catal.,
266(2) (2009) pp. 391-392.

Mattos and Aarao Reis [T.G. Mattos, F.D.A. Aarao Reis, J. Catal. 263 (2009) 67] have proposed a kinetic model of catalytic reaction including mass transport on a composite catalytic surface. The authors show that the boundary conditions for the reaction–diffusion equation they use contradict the prescription of the detailed balance principle for the relation between the rate constants for adsorbate diffusion jumps via the boundary and desorption from different areas, and, for certain parameters, the predictions of their model are qualitatively inconsistent. Quantitatively, their results are inconsistent in the situations where the role of the diffusion jumps from the catalytically active regions to the inactive regions is significant.

KINETICS OF CATALYTIC REACTIONS ON A UNIFORM SURFACE AND SINGLE SITES: SIMILARITY AND DIFFERENCE

V.P. Zhdanov

Surf. Rev. Lett.,
16(5) (2009) pp. 757-760.

To illustrate typical similarities and differences in the reaction kinetics occurring on a uniform surface and single sites (e.g. on single metal atoms incorporated into the inner walls of zeolite), a generic reaction scheme of N_2O decomposition running in the presence of oxygen was analyzed. In both cases, there are three reaction regimes, (i) controlled exclusively by the N_2O-decomposition step, (ii) partly inhibited by oxygen, or (iii) limited by oxygen desorption. Regime (i) is kinetically similar (first order in N_2O) in the two cases. The states of the catalytic sites are however different. Regime (ii) exhibits different reaction orders in oxygen (-0.5 for the surface and -1 for single sites). Regime (iii) is fully identical in both the cases.

ON THE FEASIBILITY OF STRAIN-INDUCED FORMATION OF HOLLOWS DURING HYDRIDING OR OXIDATION OF METAL NANOPARTICLES

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

Nano Lett.,
9(5) (2009) pp. 2172-2176.

Hydriding or oxidation of a metal nanoparticle starts on the surface and results in the formation of a hydride or oxide shell. Due to expansion of the shell, the strain in the metal core is tensile, and in principle it may induce the formation of a hollow structure (this hollow-formation mechanism is alternative to that based on the Kirkendall effect). Using the theory of elasticity, it is shown that this is feasible for oxides but not feasible for hydrides.

EFFECT OF LATTICE STRAIN ON THE DEHYDRIDING KINETICS IN NANOPARTICLES

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

J. Phys. Chem., C,
113(17) (2009) pp. 6894-6897.

Hydriding or dehydriding of metal nanoparticles is accompanied by the lattice strain. Using an exactly solvable model describing the strain distribution, the authors show the effect of strain on the dehydriding kinetics controlled by hydrogen desorption from the surface layer. The strain is found to suppress the desorption rate primarily during the initial stage of the kinetics, and this stage may be relatively slow. The corresponding nonlinear kinetic features are observed in experiments.

THE FORMATION OF A NEW PHASE IN NANOPARTICLES

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

Physica E,
41(5) (2009) pp. 775-778.

First-order phase transitions in nanoparticles (e.g., metal hydride formation) occur via absorption of species from or release to the gas or liquid phase. If the lattice strain is negligible, the uptake corresponding to a new phase exhibits a stepwise rise or drop with increasing or decreasing pressure or chemical potential of the absorbed species. With decreasing nanoparticle size, L, and increasing role of the surface and subsurface layers, this feature becomes less manifested because the system is less ideal. 3D lattice Monte Carlo simulations is presented, explicitly illustrating this effect for L in the range from 100 down to 10 (L is measured in units of lattice spacing).

Studying of Active Sites, Mechanism and Reaction Kinetics

KINETICS OF OXIDATION OF β-PICOLINE TO NICOTINIC ACID OVER VANADIA-TITANIA CATALYST: IV. KINETIC MODEL

E.V. Ovchinnikova, G.Ya. Popova, T.V. Andrushkevich

React. Kinet. Catal. Lett.,
96(1) (2009) pp. 91-100.

A kinetic model for the β-picoline oxidation over vanadia-titania catalyst is presented. The model covers the reaction mechanism, temperature dependences and a system of kinetic equations. The activation energies and constants in these equations are calculated.

OXIDATION OF β-PICOLINE TO NICOTINIC ACID OVER V_2O_5-TiO_2 CATALYST: KINETIC STUDIES AND REACTION MECHANISM

E.V. Ovchinnikova, T.V. Andrushkevich, G.Ya. Popova, V.D. Meshcheryakov, V.A. Chumachenko

Chem. Eng. J.,
154(1-3) (2009) pp. 60-68.

Kinetic model of β-picoline oxidation to nicotinic acid over V_2O_5-TiO_2 catalyst in the temperature range 270–300°C was elaborated on the base of the experimental data obtained in a differential reactor. Reaction mechanism was studied by *in situ* FTIR method. Kinetic parameters of the reaction rates were evaluated. The model gives satisfactory agreement between predicted and experimental results over a wide range of experimental conditions.

In situ FTIR STUDY OF THE KINETICS OF FORMIC ACID DECOMPOSITION ON V-Ti OXIDE CATALYST UNDER STATIONARY AND NON-STATIONARY CONDITIONS. DETERMINATION OF KINETIC CONSTANTS

E.A. Ivanov, G.Ya. Popova, Yu.A. Chesalov, T.V. Andrushkevich

J. Mol. Catal.,
312(1-2) (2009) pp. 92-96.

Decomposition of formic acid over V-Ti oxide catalyst under stationary and non-stationary conditions was studied by *in situ* IR spectroscopy at temperature range 50-150^{o}C. The kinetic scheme was shown to include the steps of reverse adsorption of formic acid and formate decomposition to CO. The rate constants and activation energies were estimated according the proposed kinetic model. The kinetic model satisfactorily describes the experimental data under stationary and non-stationary conditions.

In situ FTIR STUDY OF TRANSFORMATION OF 3-CYANOPYRIDINE ON V–Ti–O CATALYST

Yu.A. Chesalov, G.Ya. Popova, G.B. Chernobai, T.V. Andrushkevich

React. Kinet. Catal. Lett.,
98(1) (2009) pp. 43-50.

Surface complexes of cyanopyridine on a V–Ti–O catalyst were studied by *in situ* FTIR spectroscopy. In the temperature range of

130–290°C, two types of adsorbed cyanopyridine bound to different sites (Lewis acid sites or Brönsted acid sites) were found to transform into surface nicotinamide. At 220–290°C, surface heterocyclic complexes decompose to form carboxylates and ammonium ions.

TAP STUDY OF THE IMPACT OF THE OXIDATION STATE OF Pt/PrCeZrO AND Pt/GdCeZrO CATALYSTS ON THEIR REACTIVITY IN THE PARTIAL/DEEP OXIDATION OF METHANE

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React. Kinet. Catal. Lett.,
97(2) (2009) pp. 349-354.

The temporal analysis of products (TAP) technique coupled with the oxygen TPD was used to elucidate the effects of the Pt-supported fluorite-like doped ceria–zirconia oxide chemical composition and the type of pretreatment on their oxygen bonding strength, mobility, and reactivity as related to catalytic properties in the partial oxidation of methane into synthesis gas. A rapid evolution of hydrogen under CH_4 pulse observed for oxidized catalysts agrees with the direct route of the methane selective oxidation into syngas. This route is favoured by the Pt-support interaction and a moderate bonding strength of surface oxygen species along with a high lattice oxygen mobility.

INFLUENCE OF THE MOBILITY OF OXYGEN IN A COMPLEX OXIDE CARRIER ON THE MECHANISM OF PARTIAL OXIDATION OF METHANE

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Russ. J. General Chem.,
78(11) (2009) pp. 2191-2202.

The catalytic partial oxidation of methane was studied over single channels of monolith catalysts Pt/PrCeZrO/α-Al_2O_3 and Pt/GdCeZrO/α-Al_2O_3 using the temporal analysis of products (TAP) and kinetic transients. Effects of catalyst composition, oxidation state, time offset between O_2 and CH_4 pulses on activity, selectivity and dynamics of product formation were elucidated. Realization of the direct pyrolysis-CH_4 partial oxidation route was reliably established. This route is favored by optimum lattice/surface oxygen mobility and reactivity controlled by the dopant type (Gd, Pr) and oxidation state of the complex cerium/zirconium oxide.

EVALUATION OF KINETIC MODELS FOR THE PARTIAL OXIDATION OF METHANE TO SYNTHESIS GAS OVER A Pt/PrCeZrO$_x$ CATALYST COATED ON A TRIANGULAR MONOLITH

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Chem. Eng. J.,
154(1-3) (2009) pp. 174-184.

The evaluation of kinetic models for the partial oxidation of methane to synthesis gas over a 1.4-wt% $Pt/Pr_{0.3}Ce_{0.35}Zr_{0.35}O_x$ catalyst coated on the surface of a triangular corundum channel is presented. The mathematical form of the tested models accounts for global surface steps. The formulation of reaction steps for two cases proposed in the literature contained lumped formulations to restrict reasonably the number of parameters to estimate. One mechanism considering an oxygen assisted methane activation and another considering methane dissociation without oxygen involvement were tested. In both cases a satisfying description of the experimental data was possible, suggesting that the initial activation of methane is of less importance for the overall progress of the partial oxidation than the oxidation of carbonaceous intermediates to adsorbed carbon monoxide assumed in both cases. Comparing the kinetics over unsupported platinum extensively reported in literature to the data over the 1.4-wt% $Pt/Pr_{0.3}Ce_{0.35}Zr_{0.35}O_x$ catalyst in this work reveals significantly different rates for oxygen adsorption and carbon monoxide oxidation. These differences are explained by considering the active role the ceria plays in the catalyst performance.

PARTIAL OXIDATION OF METHANE INTO SYNTHESIS GAS OVER A Pt-SUPPORTED COMPLEX FLUORITE-LIKE OXIDE: ONE-CHANNEL STUDIES IN REALISTIC FEEDS

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React. Kinet. Catal. Lett.,
98(1) (2009) pp. 19–26.

Partial oxidation of methane (POM) into synthesis gas over a single channel of monolithic catalyst 1.4 wt% Pt/$Gd_{0.3}Ce_{0.35}Zr_{0.35}O_x$/α-$Al_2O_3$ was investigated. The effect of the catalyst pretreatment and of the process parameters (temperature, contact time, feed composition) on the catalytic activity and synthesis gas (syngas) selectivity was studied at conditions close to realistic ones with a minimum impact of the mass and heat transfer phenomena. Experimental data are in favor of the direct route of the syngas formation via the methane pyrolysis—selective oxidation sequence.

PARTIAL OXIDATION OF METHANE INTO SYNGAS ON Pt-SUPPORTED MIXED OXIDES: EFFECT OF SURFACE/LATTICE OXYGEN MOBILITY IN COMPLEX OXIDES ON THE KINETIC FEATURES OF THE REACTION AND PERFORMANCE STABILITY

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React. Kinet. Catal. Lett.,
98(1) (2009) pp. 27-33.

Partial oxidation of methane (POM) into syngas at short contact times on two catalysts containing Pt/PrCeZrO and Pt/LaCeZrO deposited on single channels of a corundum monolithic substrate was investigated. The effects of the catalyst composition, pretreatment and process parameters on activity and selectivity of the syngas formation were studied. The oxygen mobility in complex oxides was shown to determine both their stability to coking and realization of a direct selective oxidation route of the syngas generation.

DRY REFORMING OF METHANE OVER FLUORITE-LIKE MIXED OXIDES PROMOTED BY Pt

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React. Kinet. Catal. Lett.,
98(1) (2009) pp. 35-41.

Dry reforming of methane into syngas was studied over complex Pt-promoted fluorite-like oxides (PrCeZrO, GdCeZrO and LaCeZrO) supported on a corundum substrate. Activity and syngas selectivity of these catalysts were found to depend strongly on their composition, pretreatment and reaction conditions. At low temperatures and short contact times, the H_2/CO ratio in the syngas is below 1 due to a fast reverse water–gas shift reaction. The catalyst performance was found to correlate with the bulk oxygen mobility in complex fluorite-like oxides, which prevents the surface coking.

MECHANISM OF HIGH-TEMPERATURE STEAM CONVERSION OF CO ON Ce-Zr-(La)-O AND Pt/Ce-Zr-(La)-O

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Chem. Sustain. Devel.,
17(4) (2009) pp. 371-377.

Dynamics of transition modes in the reaction of steam conversion of CO on $Ce_{0.5-x}Zr_{0.5-x}La_{2x}O_2$ (x = 0, 0.1) and platinum catalysts on this basis at 650°C is investigated. Numerical modeling of the response curves showed that all the transformations on the support run within the oxidation-reduction mechanism. At the catalyst under the steady conditions, the major contribution into the activity is made by the transformations according to the association mechanism. The dynamics of transient modes is determined by the oxidation-reduction mechanism.

INFLUENCE OF OXYGEN MOBILITY ON CATALYTIC ACTIVITY OF La–Sr–Mn–O COMPOSITES IN THE REACTION OF HIGH TEMPERATURE N_2O DECOMPOSITION

D.V. Ivanov, E.M. Sadovskaya, L.G. Pinaeva, L.A. Isupova

J. Catal.,
267(1) (2009) pp. 5-13.

Previous study suggested that either high oxygen mobility in layer-structured $(La_{1-y}Sr_y)_2MnO_4$ perovskite or high oxygen diffusion through intergrain boundaries is the reason why multiphase $La_{1-x}Sr_xMnO_3$ samples exhibit a high catalytic activity in high temperature N_2O decomposition. The absence of inhibiting effect of oxygen on the reaction rate for these samples allows us to suppose that surface segregation of layered perovskite increases oxygen mobility and facilitates oxygen desorption from the surface. In this paper, the authors aimed at determining the influence of surface composition on oxygen mobility and catalytic activity in high temperature N_2O decomposition. By means of steady-state isotopic transient kinetic analysis (SSITKA) the mechanism and kinetics of oxygen exchange were elucidated for three $La_{1-x}Sr_xMnO_3$ ($x = 0$, 0.3, and 0.5) samples considerably differing in phase composition and catalytic activity. The results obtained indicate that inactive single-phase $LaMnO_3$ exhibits both the lowest rate of oxygen exchange on the surface and the lowest rate of oxygen diffusion in the bulk. For $La_{0.3}Sr_{0.7}MnO_3$, the increased values of both rates as compared with $LaMnO_3$ can be interpreted as the appearance of a fast pathway of oxygen transfer through vacancies formed in the perovskite lattice to compensate the reduced cation charge. The highest values of the content of fast-exchangeable oxygen and oxygen diffusion coefficient were found for a multiphase sample containing layered perovskite, thus providing a strong correlation between oxygen mobility and catalytic activity in the reaction of nitrous oxide decomposition.

TRANSIENT ISOTOPIC KINETICS STUDY TO INVESTIGATE REACTION MECHANISMS

B.S. Bal'zhinimaev, E.M. Sadovskaya, A.P. Suknev

Chem. Eng. J.,
154(1-3) (2009) pp. 2-8.

This overview includes discussion of the principles, experimental and theoretical features of the isotopic transient kinetics for the study of reaction mechanism and evaluation of kinetic parameters. The results of the mechanistic study of ethylene epoxidation over silver, selective NO reduction with methane over Co-ZSM-5 and fiberglass based catalysts are presented. Investigation of ^{18}O isotope transfer dynamics allowed to reveal the reaction pathways towards ethylene oxide and CO_2 formation as well as to estimate the concentrations of active oxygen species and reaction rate coefficients of key steps. It was found that both ethylene epoxidation and deep oxidation proceed very fast in the microsecond range. A detailed SSITKA study of the NO reduction with methane using ^{15}N, ^{13}C and ^{18}O labels showed that reaction occurred via two parallel pathways involving nitrite-nitrate and $NO_2^{\delta+}$ species, the last one being 20 times more reactive. For the first time a transient isotopic kinetic study was used for evaluation of mass transfer parameters by an example of NO and water diffusion into the bulk of Pt-containing fiberglass catalyst.

FROM SINGLE CRYSTALS TO SUPPORTED NANOPARTICLES IN EXPERIMENTAL AND THEORETICAL STUDIES OF H_2 OXIDATION OVER PLATINUM METALS (Pt, Pd): INTERMEDIATES, SURFACE WAVES AND SPILLOVER

V.V. Gorodetsky, A.A. Sametova, A.V. Matveev, V.M. Tapilin

Catal. Today,
144(3-4) (2009) pp. 219-234.

The present paper reviews investigations concerning the mechanism of $H_2 + O_2$ reaction on the metal surfaces (Pt, Pd) at different structures: single crystals (Pt(1 1 1), Pt(1 0 0), Pd(1 1 0)); microcrystals (Pt tips); and nanoparticles ($Pd–Ti^{3+}/TiO_2$). Field electron microscopy (FEM), field ion microscopy (FIM), high-resolution electron energy loss spectroscopy (HREELS), XPS, UPS, work function (WF), TDS and temperature-programmed reaction (TPR) methods have been applied to study the kinetics of H_2 oxidation on a nanolevel. The adsorption of both O_2 and H_2 and several dissociative products (H_{ads}, O_{ads}, OH_{ads}) was studied by HREELS. Using the DFT technique the equilibrium states and stretching vibrations of H, O, OH, H_2O, adsorbed on the Pt(1 1 1) surface, have been calculated depending on the surrounding of the metal atoms. Sharp tips of Pt, several hundreds angstroms in radius, were used to perform *in situ* investigations of the dynamic surface processes. The FEM and FIM studies on the Pt-tip surface demonstrate that the self-oscillations and waves propagations are connected with periodic changes in the surface structure of nanoplane (1 0 0)-(hex) ↔ (1 × 1), varying the catalytic property of metal. The role of defects (Ti^{3+}-$\square_O$) in the adsorption centers formation, their stabilization by the palladium nanoparticles, and then the defects participation in $H_2 + O_2$ steady-state reaction over $Pd–Ti^{3+}/TiO_2$ surface have been studied by XPS, UPS and photodesorption techniques (PhDS). This reaction seems to involve the “protonate” hydrogen atoms (H^+/TiO_x) as a result of spillover effect: diffusion of H_{ads} atoms from Pd particles on a TiO_x surface. The comprehensive study of H_2, O_2 adsorption and $H_2 + O_2$ reaction in a row: single crystals → tips → nanoparticles has shown the same nature of active centers over these metal surfaces.

EXPERIMENTAL EVIDENCE OF PROPAGATING WAVES IN HYDROGEN OXIDATION ON PLATINUM-GROUP METALS (Pt, Rh, Ir)

V.V. Gorodetsky

Kinet. Catal.,
50(2) (2009) pp. 304-313.

The $H_{2(g)} + O_{ads}$ reaction on nanoplanes of Pt-, Rh- and Ir-tips with a radius of ~10^3 Å was studied by FEM with a resolution of ~20 Å, and the critical conditions (P_i , T) under which this reaction is accompanied by the formation of a propagating wave front with a sharp boundary between the H_{ads}- and O_{ads}-layers were determined. The $H_2 + O_2$ reaction on a Pt-tip under steady-state conditions was studied by FIM with a lateral atomic resolution of ~5 Å, and images of H_2O molecules (H_2O^+ ions) visualizing the spatial distribution of catalytically active sites (platinum atoms) were obtained for the first time. The origin of the kinetic self-oscillations in isothermal H_2 oxidation *in situ* was investigated in detail. The regular chemical waves were found to be initiated by the reversible phase transition (Pt(100)-*hex* ↔ 1 × 1) of the Pt(100) nanoplane surface. A stepwise H_2O formation mechanism involving OH_{ads}-groups was deduced from HREELS and TDS for the $H_{ads} + O_{ads}$ reaction on the Pt(111) and Pt(100) single crystal surfaces.

OXIDATION OF ALKANES AND OLEFINS WITH HYDROGEN PEROXIDE IN ACETONITRILE SOLUTION CATALYZED BY A MESOPOROUS TITANIUM-SILICATE Ti-MMM-2

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Appl. Catal., A,
365(1) (2009) pp. 96-104.

Mesoporous titanium-silicate Ti-MMM-2 catalyzes oxidation of alkanes (cyclooctane, *n*-heptane, *n*-octane, isooctane, methylcyclohexane, *cis*- and *trans*-1,2-dimethylcyclohexane) and olefins (cyclooctene, 1-decene, (*S*)-limonene) by H_2O_2 in acetonitrile solution at 60°C is described. The catalytic reaction is truly heterogeneous in nature. The oxidation occurs *via* the formation of a ‘Ti–OOH’ species on the catalyst surface which either epoxidizes a nucleophilic double bond or generates, after O–O bond splitting, hydroxyl radical. The HO• radical attacks an alkane or olefin C–H bond producing alkyl radical. The reaction $R\bullet + O_2 \rightarrow ROO\bullet$ leads to the formation of alkyl hydroperoxide as the main product of the alkane oxidation. In the case of alkenes this reaction leads to allylic oxidation products. The composition of products of the olefin oxygenation (the epoxide/alkyl hydroperoxide or epoxide/diol ratios, etc.) strongly depends on the nature of the substrate. In the oxidation of (*S*)-limonene, isomeric diepoxides are formed along with monoepoxides even at the early stage of the reaction and (*SRR*)-diepoxide predominates among other products. This can be rationalized by suggesting the epoxidation of the two double bonds to occur simultaneously on two adjacent ‘Ti–O–OH’ centers on the catalyst surface.

HIGHLY SELECTIVE OXIDATION OF ALKYLPHENOLS TO BENZOQUINONES WITH HYDROGEN PEROXIDE OVER SILICA-SUPPORTED TITANIUM CATALYSTS: TITANIUM CLUSTER SITE *VERSUS* TITANIUM SINGLE SITE

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Adv. Synth. Catal.,
351(11-12) (2009) pp. 1877-1889.

Titanium-silica catalysts have been prepared by supporting titanium(IV) precursors with different nuclearity {mononuclear titanocene dichloride $Ti(Cp)_2Cl_2$, dinuclear titanium diethyl tartrate and the tetranuclear titanium peroxo complex $(NH_4)_8[Ti_4(C_6H_4O_7)_4(O_2)_4]\cdot 8\ H_2O$} onto the surface of silica materials with different textural characteristics. The supported catalysts have been explored as highly active and reusable catalysts for the oxidation of 2,3,6-trimethylphenol (TMP) and 2,6-dimethylphenol (DMP) to 2,3,5-trimethyl-1,4-benzoquinone (TMBQ, vitamin E key intermediate) and 2,6-dimethyl-1,4-benzoquinone (DMBQ), respectively, using aqueous hydrogen peroxide as green oxidant. Catalysts prepared by grafting mononuclear $Ti(Cp)_2Cl_2$ revealed a strong dependence of the product selectivity on the surface concentration of titanium active centers. Mesoporous materials with titanium surface concentration in the range of 0.6-1.0 Ti/nm^2 were identified as optimal catalysts for the transformation of alkylphenols to benzoquinones. Catalysts having <0.6 Ti/nm^2 produced a mixture of benzoquinones and dimeric by-products. Conversely, when di-/tetranuclear titanium precursors were employed for the catalyst preparation, a diminution of the titanium surface concentration had no impact on the benzoquinone selectivity, which was typically as high as 96-99%. DR-UV spectroscopic studies revealed that the catalysts capable of producing alkylbenzoquinones with nearly quantitative yields possess titanium dimers and/or subnanometer-size clusters homogeneously distributed on a silica surface. On the contrary, catalysts with isolated titanium sites give a considerable amount of dimeric by-products. This is the first example which clearly demonstrates the advantages of titanium cluster-site catalysts over titanium single-site catalysts in hydrogen peroxide-based selective oxidation reaction.

HOW TO REACH 100% SELECTIVITY IN H_2O_2-BASED OXIDATION OF 2,3,6-TRIMETHYLPHENOL TO TRIMETHYL-*p*-BENZOQUINONE OVER Ti,Si-CATALYSTS

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A comprehensive study of structure/activity/selectivity relationships and mechanistic aspects of the oxidation of 2,3,6-trimethylphenol (TMP) with aqueous H_2O_2 over a wide variety of titanium-silicate catalysts allowed to infer requirements to an optimal catalyst and optimal reaction conditions for this reaction and to produce 2,3,5-trimethyl-1,4-benzoquinone (TMBQ, Vitamin E precursor) with nearly 100% selectivity at 100% substrate conversion. The main by-products in the TMP oxidation are C–C and C–O dimers, formed by coupling of intermediate phenoxyl radicals. The formation of TMBQ is favoured by (1) a poor coordinating solvent (MeCN), (2) elevated temperature (80°C), (3) low TMP concentration (not higher than 0.1 M), (4) high H_2O_2/TMP molar ratio (ca. 3.5), and (5) low TMP/Ti ratio (<10–20). The crucial factors which determine the selectivity of Ti,Si-catalysts in TMP oxidation to TMBQ are mesoporosity and an optimal surface concentration (ca. 0.7–1.0 Ti atoms/nm^2) of accessible highly dispersed, probably, dimeric Ti(IV) species. The catalysts prepared by a simple, affordable and cheap synthesis methodology *via* grafting titanium(IV) precursors onto the surface of commercial mesoporous silica completely fulfil these requirements and thus can be viewed as promising catalysts for environmentally benign TMBQ production.

UNIQUE CATALYTIC PERFORMANCE OF THE POLYOXOMETALATE $[Ti_2(OH)_2As_2W_{19}O_{67}(H_2O)]^{8-}$: THE ROLE OF 5-COORDINATED TITANIUM IN H_2O_2 ACTIVATION

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Eur. J. Inorg. Chem.,
2009(34) (2009) pp. 5134-5141.

Tetra-*n*-butylammonium (TBA) salts of the dititanium sandwich-type 19-tungstodiarsenate(III) $[Ti_2(OH)_2As_2W_{19}O_{67}(H_2O)]^{8-}$ (**1**) revealed a unique catalytic performance in the selective oxidation of organic compounds with aqueous hydrogen peroxide. The selectivity of alkene oxidation strongly depends on the amount of acidic protons in the cationic part of **1**, which can be controlled by pH of precipitation of the TBA salt. Selectivities of almost 100 % were achieved for cyclohexene epoxidation by using $TBA_{5.5}Na_{1.5}K_{0.5}H_{0.5}$-**1**. In the presence of $TBA_{5.5}K_{0.5}H_2$-**1**, cyclohexene epoxide readily transformed into *trans*-1,2-cyclohexanediol, 2-ydroxycyclohexanone, and C-C bond-cleavage products. No allylic oxidation products were found. Vicinal diols yielded α-hydroxyketones and (di)carboxylic acids. Ketonization of cyclohexanol proceeded with selectivity as high as 98 %, whereas 1-exanol produced hexanal and hexanoic acid. The oxidation products are consistent with a heterolytic mechanism of H_2O_2 activation. The stability of **1** under turnover conditions was confirmed by IR spectroscopy. The presence of titanium atoms in the belt of **1** is vital for the catalytic performance. The titanium-free analogue, $[As_2W_{21}O_{67}(H_2O)]^{6-}$ (**2**), revealed lower activity and selectivity in the oxidation of alcohols and was not active in the oxidation of alkenes. The catalytic performance of **1** contrasts that of polyoxometalates containing hexacoordinate Ti atoms, for example, $TBA_7[(PTiW_{11}O_{39})_2OH]$, $TBA_8H_7[\{Ti_3PW_9O_{37}(OH)\}_3(PO_4)]$, and $TBA_{17}[(\alpha\text{-}Ti_3SiW_9O_{37}OH)_3\{TiO_3(OH_2)_3\}]$, for which a homolytic oxidation mechanism was manifested by the formation of cyclohexene allylic oxidation products. The unique ability of **1** to catalyze heterolytic oxidations with the use of hydrogen peroxide most likely originates from the specific coordination number (5) and geometry (square-pyramidal) of the Ti atoms in this polyanion.

KINETICS AND MECHANISM OF THE OXIDATION OF ALKYL SUBSTITUTED PHENOLS AND NAPHTHOLS WITH ^{t}BuOOH IN THE PRESENCE OF SUPPORTED IRON PHTHALOCYANINE

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New J. Chem.,
33(5) (2009) pp. 1031-1037.

2,3,5-Trimethylbenzoquinone (precursor of vitamin E) and 2-methylnaphthoquinone (vitamin K_3) were obtained in good yields by oxidation of 2,3,6-trimethylphenol and 2-methyl-1-naphthol, respectively, with ^{t}BuOOH catalyzed by supported iron tetrasulfophthalocyanine. The mechanism of this heterogeneous oxidation was studied using $^{18}O_2$ labeling experiments, EPR spectroscopy with spin traps, kinetic studies, and complete analysis of reaction products including minor ones. $^{18}O_2$ labeling experiments did not indicate the involvement of O_2 in the oxidative process. EPR study of reaction mixtures of 2,3,6-trimethylphenol and 2-methyl-1-naphthol oxidations in the presence of 3,5-dibromo-4-nitrosobenzenesulfonic acid spin trap showed no formation of any radical intermediates. Besides the target quinones, epoxyquinones and formyldimethyl-1,4-benzoquinones, as over-oxidation minor products have been found. C—C and C—O coupling products relevant to one-electron oxidation pathways were detected in trace amounts. Based on the experimental results, a mechanism of oxidation of alkyl-substituted phenols and naphthols mediated by the supported iron phthalocyanine catalyst has been proposed which involves two successive electron transfers without escape of radical species in solution.

OXYGEN SPECIES AND THEIR REACTIVITY IN THE MECHANOCHEMICALLY PREPARED SUBSTITUTED PEROVSKITES $La_{1-x}Sr_xCoO_{3-y}$ ($x = 0–1$)

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50(2) (2009) pp. 275-283.

The oxygen species and their reactivity in the mechanochemically prepared substituted perovskites $La_{1-x}Sr_xCoO_{3-y}$ were studied using temperature-programmed reduction (TPR) of the samples with hydrogen. The experimental data were compared with data on the catalytic activity of the series of $La_{1-x}Sr_xCoO_{3-y}$ catalysts in the oxidation of CO, as well as with the real structures and surface compositions of the samples, which were studied in detail previously. As the strontium content was increased, the degree of reduction of the samples increased in the course of TPR and the TPR peaks shifted to the region of lower temperatures, except for the last sample containing no lanthanum ($x = 1$). An increase in the calcination temperature and time resulted in a decrease in TPR peak intensities and in a shift of the peaks to the region of higher temperatures. A reaction scheme was proposed for the reduction. In accordance with this reaction scheme, Co^{4+} in substituted cobaltites was reduced to Co^0 at temperatures lower than 400°C. In the temperature region of 400–500°C, the $Co^{3+} \rightarrow Co^{2+}$ bulk reduction, as well as the deep reduction processes $Co^{3+} \rightarrow Co^0$ and $Co^{4+} \rightarrow Co^0$, occurred; substitution facilitated the above processes. At temperatures higher than 500°C, $Co^{2+} \rightarrow Co^0$ bulk reduction occurred. The observed reduction of the mechanochemically prepared samples depended on their microstructure, which was described previously. It was found that the activity of the samples in the oxidation of CO depends on the amount of the most weakly bound reactive surface oxygen species, which were removed in TPR with hydrogen to 150°C. No correlation between the amount of strongly bound (lattice) oxygen removed upon TPR and the activity of $La_{1-x}Sr_xCoO_{3-y}$ samples in the oxidation of CO was found.

RADICAL CATIONS OF AROMATIC MOLECULES WITH HIGH IONIZATION POTENTIALS ON THE SURFACES OF OXIDE CATALYSTS: FORMATION, PROPERTIES, AND REACTIVITY

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50(2) (2009) pp. 314-324.

The results obtained by the authors on the formation and properties of the radical cations of benzene and other molecules with high ionization potentials on the surfaces of ZSM-5 zeolites and sulfated zirconium dioxide are analyzed in this paper. It was found that radical cations of aromatic compounds can be obtained both by a thermal process and under illumination with visible light. Radical cations were found to be key intermediates in the low-temperature polycondensation of aromatic compounds on these catalysts. A possible mechanism of the formation of radical cations and the nature of the sites responsible for these processes are discussed.

IN SITU HIGH TEMPERATURE MAS NMR STUDY OF THE MECHANISMS OF CATALYSIS. ETHANE AROMATIZATION ON Zn-MODIFIED ZEOLITE BEA

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Solid State Nucl. Magn. Reson.,
35(2) (2009) pp. 113-119.

Ethane conversion into aromatic hydrocarbons over Zn-modified zeolite BEA has been analyzed by high-temperature MAS NMR spectroscopy. Information about intermediates (Zn-ethyl species) and reaction products (mainly toluene and methane), which were formed under the conditions of a batch reactor, was obtained by ^{13}C MAS NMR. Kinetics of the reaction, which was monitored by ^{1}H MAS NMR *in situ* at the temperature of 573 K, provided information about the reaction mechanism. Simulation of the experimental kinetics within the frames of the possible kinetic schemes of the reaction demonstrates that a large amount of methane evolved under ethane aromatization arises from the stage of direct ethane hydrogenolysis.

METHANE AROMATIZATION ON Zn-MODIFIED ZEOLITE IN THE PRESENCE OF A CO-REACTANT HIGHER ALKANE: HOW DOES IT OCCUR?

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144(3-4) (2009) pp. 265-272.

By using ^{13}C solid-state NMR and GC–MS, the analysis of the ^{13}C-label transfer from methane-^{13}C into the products of methane and propane co-aromatization on Zn/H-BEA zeolite at 823–873 K has been performed. A high degree involvement of ^{13}C-carbon atoms of methane into aromatic products (benzene, toluene, xylenes) has been demonstrated. The main pathway of methane conversion into aromatics has been determined to consist in the methylation of aromatics, which is produced exclusively from propane, by methane. The methoxy species formed by the dissociative adsorption of methane on ZnO species of the zeolite is responsible for the methylation.

REMARKABLY FAST OXIDATION OF ALKANES BY HYDROGEN PEROXIDE CATALYZED BY A TETRACOPPER(II) TRIETHANOLAMINATE COMPLEX: PROMOTING EFFECTS OF ACID CO-CATALYSTS AND WATER, KINETIC AND MECHANISTIC FEATURES

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The kinetic and mechanistic features of alkane oxidations to the corresponding alkyl hydroperoxides (main primary products), alcohols and ketones (secondary products) in the systems composed of tetracopper(II) triethanolaminate catalyst $[O\subset Cu_4\{N(CH_2CH_2O)_3\}_4(BOH)_4][BF_4]_2$ (**1**), aqueous hydrogen peroxide, acetonitrile solvent and an acid promoter (co-catalyst), have been investigated based on the combination of experimental kinetic, selectivity, ESR and UV–vis methods. The nature of acid promoter (hydrochloric, sulfuric, nitric and trifluoroacetic acid) is shown to be a key factor affecting significantly the rate of alkane oxidation. Although all these acids exhibit noticeable promoting effect, it has been observed that in the presence of HCl the reaction proceeds extremely rapidly, being one order faster than those promoted by the other acids, and allowing to achieve the remarkably high turnover frequencies (TOFs) of ca. 600 h^{-1}. The unusual rate-accelerating role of water has also been disclosed in the oxidation of cyclohexane catalyzed by **1** + HCl or **1** + CF_3COOH systems. Furthermore, uncommon second-order reaction kinetics with respect to the catalyst have been found. A mechanism of the alkane oxygenation has been proposed, which includes the formation of hydroxyl radicals attacking the alkane molecule. Hydroxyl radicals are formed via the interaction between H_2O_2 and catalytically active Cu(I) species, the latter being reversibly generated from **1** under the action of an acid, H_2O_2 and water.

EPR SPECTROSCOPIC TRAPPING OF THE ACTIVE SPECIES OF NONHEME IRON-CATALYZED OXIDATION

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J. Am. Chem. Soc.,
131(31) (2009) pp. 10798–10799.

The key intermediate of a bioinspired iron catalyst for selective hydrocarbon oxidation based on hydrogen peroxide and an iron complex with a tetradentate aminopyridine ligand was trapped by EPR. On the basis of EPR and reactivity data this intermediate is tentatively proposed to be an oxoiron(V) complex.

REACTIVITY OF METHOXY SPECIES TOWARD CO ON KEGGIN 12-$H_3PW_{12}O_{40}$: A STUDY WITH SOLID STATE NMR

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J. Phys. Chem. C,
113(45) (2009) pp. 19639-19644.

By using ^{13}C solid-state NMR, monitoring the reactivity of methoxy species with carbon monoxide has been performed on 12-tungstophosphoric acid, $H_3PW_{12}O_{40}$, at 423-473 K. Surface methoxy species have been selectively prepared from methanol at 293-23 K. Almost quantitative conversion of surface methoxy species into acetyl groups bound to the Keggin anion and acetic acid has been observed at 473 K. These data provide unambiguous evidence for the role of methoxy species as the intermediate of methanol and dimethyl ether carbonylation on solid $H_3PW_{12}O_{40}$. The formation of a trimethyloxonium ion has been detected on the surface of heteropolyacid for the first time. The carbonylation of trimethyloxonium proceeds via the analogous mechanism through the methoxy species intermediate.

CONCEPT OF ACID–BASE CATALYSIS BY METAL SULFIDES

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The structure of the active component of the sulfide hydrodesulfurization (HDS) catalysts is considered. The active component consists of a single slab of MoS_2 (WS_2) with Ni or Co atoms being located in its edge plane. The essential element of the active component is hydrogen occluded into the MoS_2 matrix, which localizes in the center of “empty” trigonal prism of a MoS_2 matrix made from six sulphur atoms under Ni (Co) atom at the distance of 1.5 A. Occluded hydrogen appeared in the active component during the catalyst suffixation as a result of its oxidative addition to the Ni (Co) atoms. This occluded hydrogen creates an electronic d^6 configuration of Ni (IV) or Co (III) atoms, which are the Lewis acid sites with two vacant d_{z2} and d_{ay} orbitales. Therefore, these active centres can adsorb donor molecules of thiophen. The activation of hydrogen occurs on the couplet sulphur atoms at the edge planes of a single MoS_2 (WS_2) slab. The driving force of this process is the presence of the S─S chemical bond at the terminal sulphur atoms that restrict an electrically neutral macromolecule of the active component and act as the Lewis basic sites. An excess of electron density is transferred from these atoms to a hydrogen molecule, which results in its homolytic dissociation. The high rate of catalytic transformation is ensured by the fast transfer of electrons and protons along the system of chemical bonds within an electrically neutral macromolecule of the active component.

CATALYTIC OXIDATION OF CO ON CuO_x REVISITED: IMPACT OF THE SURFACE STATE ON THE APPARENT KINETIC PARAMETERS

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Analysis of kinetic features of the copper oxides reduction by CO pulses as related to mechanism of CO catalytic oxidation by oxygen combined with monitoring the state of the surface by an electrochemical technique using a solid electrolyte—Pyrex glass and high resolution TEM data on the defect structure of CuO allowed us to suggest a partially “flexible” model of CuO surface. This model, with a due regard for the data of FTIR spectroscopy of adsorbed CO test molecules, assigns the most active surface sites able to coordinate highly reactive CO and O forms to clusters of Cu^+ cations located at outlets of extended defects (dislocations, twins). Variation of the number, size and structure of these clusters under the reaction medium effect allows explaining the difference between quasi-steady and true steady states of copper oxides in catalytic CO oxidation reaction as well as the difference between kinetic parameters of reaction estimated at quasi-steady and constant states of the surface following Boreskov's approach. Kinetic features of the reaction agree with the Langmuir–Hinshelwood reaction mechanism operating for clustered defect centers of CuO.

NONOXIDATIVE CONVERSION OF METHANE INTO AROMATIC HYDROCARBONS ON Ni-Mo/ZSM-5 CATALYSTS

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Kinet. Catal.,
50(5) (2009) pp. 725-733.

The nonoxidative conversion of methane into aromatic hydrocarbons on high-silica zeolites ZSM-5 containing nanosized powders of molybdenum (4.0 wt %) and nickel (0.1–2.0 wt %) was studied. Data on the acid characteristics of the catalysts and the nature and amount of coke deposits formed on the surface of the catalysts were obtained using the thermal desorption of ammonia and thermal analysis. The microstructure and composition of Ni-Mo/ZSM-5 catalysts were studied by high-resolution transmission electron microscopy and energy-dispersive X-ray analysis. The formation of various chemical species in the samples was detected: oxide-like clusters of Mo within zeolite channels (<1 nm), molybdenum carbide particles (5–30 nm) on the outer surface of the zeolite, and Ni-Mo alloy particles with different compositions (under reaction conditions, carbon filaments grew on these particles). It was found that, as the Ni content was increased from 0.1 to 2.0 wt %, the rate of deactivation of the catalytic system increased because of blocking pores in the zeolite structure by filamentous carbon up to the formation of condensed coke deposits.

O_2 ISOTOPIC EXCHANGE IN THE PRESENCE OF O^- ANION RADICALS ON THE FeZSM-5 SURFACE

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Oxygen anion radicals O^- play an important role in various physico-chemical processes, including those taking place on solid surfaces, where their behavior is especially difficult to follow. This work presents a detailed study on the behavior of O^- radicals adsorbed on FeZSM-5 surface in the reaction of O_2 isotopic exchange carried out over a wide range of temperatures (198–513 K). High concentration of O^- in this unique system (called alpha-oxygen) made it possible to identify two exchange mechanisms, R_0 and R_1, operating in the presence of O_α. The R_1 mechanism (E=15 kJ/mol) dominates in the high-temperature region and can be represented by the following equation:

$$^{18}O_2 + {}^{16}O^-_\alpha \rightleftarrows [O^-_3]_\alpha \rightleftarrows {}^{16}O^{18}O + {}^{18}O^-_\alpha.$$

This mechanism is convincingly supported by several arguments: the first order reaction with respect to O_2; the linear dependence of reaction rate on O_α concentration; the ESR observation of O^-_3 species (in other systems); and, according to isotopic exchange theory, by a twofold excess of the rate of homoexchange over the rate of heteroexchange.

The R_0 exchange (E=0.8 kJ/mol) is a new reaction pathway that appears at cryogenic temperatures and leads to isotope redistribution only between O_2 molecules, not affecting the isotopic composition of O_α. This exchange mechanism is not quite clear. The necessity of the presence of O_α without its direct involvement in the exchange process seems to be quite surprising. The reaction may proceed via a hypothetical complex $[O^-_5]_\alpha$, which allows cleavage and redistribution of chemical bonds between two O_2 molecules adsorbed on the α-oxygen. The cleavage and redistribution of such strong bonds at cryogenic temperatures with nearly zero activation energy are an intriguing phenomenon worthy of further study.

PECULIARITIES OF DEHYDROAROMATIZATION OF CH_4–C_2H_6 AND CH_4 OVER Mo/ZSM-5 CATALYSTS

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React. Kinet. Catal. Lett.,
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The peculiarities of dehydroaromatization (DHA) of a CH_4–C_2H_6 mixture over 1–10% Mo/ZSM-5 catalysts (Si/Al = 17, 30, 45) were studied in comparison with reactions of individual hydrocarbons. For a (90 vol.% CH_4 + 10 vol.% Ar) mixture, the methane conversion passes through a maximum and decreases with the time-on-stream, being about 12% after 6 h. Experiments with a mixture (85 vol.% CH_4 + 5 vol.% C_2H_6 + 10 vol.% Ar) demonstrated that the presence of ethane leads to the suppression of the methane conversion practically to zero and to an increase in the benzene formation rate in comparison with the (CH_4 + Ar) mixture. Characterization of the spent Mo/ZSM-5 catalysts by DTA and TGA showed that both the condensation degree of the carbonaceous deposits (C/H ratio) and their content are higher in the presence of ethane. For both feed compositions, the maximal benzene formation rate was observed over the catalyst with 2 wt% Mo and Si/Al = 17. It is proposed that the formation of C2 intermediates from methane and their further transformations in DHA compete with the ethane aromatization, presumably on the same Mo active sites.

HOMOGENEOUS AND HETEROGENEOUS CATALYTIC OXIDATION OF SULFIDES BY H_2O_2 OVER ZINC(II) COMPOUNDS

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Dalton Trans.,
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It has been recently shown that zinc compounds are effective catalysts for the oxidation of alkyl aryl sulfides to the corresponding sulfoxides in the presence of hydrogen peroxide. In this paper, it has been investigated homogeneous and heterogeneous catalytic oxidation of sulfides by H_2O_2 over $Zn(NO_3)_2{\cdot}6H_2O$ and the metal–organic porous material $[Zn_2(bdc)(L\text{-}lac)(dmf)]{\cdot}DMF$ (where H_2bdc = *p*-benzenedicarboxylic acid, H_2lac = lactic acid), respectively. The experimental data can be explained by the proposed catalytic cycle which includes the activation of H_2O_2 *via* coordination to Zn(II) ions followed by oxygen transfer step. In homogeneous conditions, the presence of a large amounts of H_2O_2 results in the coordination of two molecules of hydrogen peroxide to Zn(II), so that sulfone is formed *via* transfer of two oxygen atoms from $Zn(H_2O)_4(H_2O_2)_2^{2+}$ active species. Contrary to the homogeneous system, the use of $[Zn_2(bdc)(L\text{-}lac)(dmf)]{\cdot}DMF$ as catalyst does not lead to the formation of sulfone in the initial period of reaction. This is consistent with the proposed catalytic cycle of sulfoxidation as each Zn(II) center in the crystalline framework is able to activate only one H_2O_2 molecule. The investigations indicate that the sorption and activation of H_2O_2 molecules by microporous framework $[Zn_2(bdc)(L\text{-}lac)(dmf)]$ occur faster than sulfide sorption and oxygen transfer.

SIZE EFFECTS IN CATALYSIS BY SUPPORTED METAL NANOPARTICLES

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Catal. Ind.,
1(1) (2009) pp. 17-28.

This paper is concerned with the study of size effects in reactions of low-temperature CO oxidation on the catalysts Au/γ-Al_2O_3 and Au/δ-Al_2O_3 and complete oxidation of methane on the catalysts Pt/γ-Al_2O_3. For the synthesis of gold catalysts, four techniques have been applied: ionic adsorption, deposition-precipitation, chemical liquid-phase grafting, and decomposition of volatile gold complexes. Platinum catalysts have been prepared by aluminum oxide impregnation with aqueous solutions of $H_2[Pt(OH)_6]$ that, depending on preparation conditions, contained mono- or oligonuclear hydroxocomplexes of platinum. Series of catalyst samples with a narrow size distribution of particles and a mean size variation from 0.5–1 to 20–25 nm have been prepared. The study of the catalytic properties of the prepared catalysts has shown that a decrease in mean size of supported metal particles leads to a sharp increase in specific catalytic activity in both systems. The activity maximum has been achieved for active component particles of 2–3 nm. A conclusion has been made that the application of nanosize catalysts is promising for the cleaning of air in closed rooms and vehicle exhaust gases from CO, for the utilization of methane, and for the obtaining of energy by the combustion of natural gas.

PLATINUM NANOPARTICLE SIZE EFFECT ON SPECIFIC CATALYTIC ACTIVITY IN *n*-ALKANE DEEP OXIDATION: DEPENDENCE ON THE CHAIN LENGTH OF THE PARAFFIN

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Kinet. Catal.,
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The specific activity of 0.8% Pt/Al_2O_3 catalysts in the deep oxidation of C_1–C_6 *n*-alkanes increases with an increase in the Pt particle size from 1 to 3–4 nm. Further coarsening of the particles insignificantly changes the specific activity. The size effect was studied for a series of catalysts containing platinum nanoparticles 1 to 11 nm in diameter. The specific catalytic activity variation range depends on the size of the reacting hydrocarbon molecules. As the platinum particle size increases, the specific catalytic activity increases 3–4 times for the oxidation of CH_4 and C_2H_6 and by a factor of 20–30 for the oxidation of *n*-C_4H_{10} and *n*-C_6H_{14}.

CATALYTIC CONDENSATION OF GLYCOLALDEHYDE AND GLYCERALDEHYDE WITH FORMALDEHYDE IN NEUTRAL AND WEAKLY ALKALINE AQUEOUS MEDIA: KINETICS AND MECHANISM

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Kinet. Catal.,
50(2) (2009) pp. 297-303.

The kinetics of glycolaldehyde and glyceraldehyde condensation with formaldehyde in a neutral aqueous medium in the presence of homogeneous phosphates and in a weakly alkaline medium in the presence of MgO was studied. The temperature dependences of the observed kinetic constants and the apparent activation energies of the reactions were determined. A reaction scheme for the interaction of lower monosaccharides with formaldehyde was derived from analyses of the reaction products.

HYDROGENATION OF LACTIC ACID TO PROPYLENE GLYCOL OVER COPPER-CONTAINING CATALYSTS

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React. Kinet. Catal. Lett.,
97(1) (2009) pp. 157-162.

Catalytic properties of different copper-containing catalysts synthesized from different precursors were studied in the hydrogenation of lactic acid at mild conditions. The most active catalyst was found to be chrysocolla-like copper hydroxysilicate with the copper loading of about 50 at%. At the optimal reaction conditions ($T = 473$ K, WHSV = 0.08 h^{-1}), 95% conversion of lactic acid over this catalyst and 65% selectivity to 1,2-propylene glycol were achieved. The effect of the weight hourly space velocity (WHSV) on the lactic acid conversion and selectivity to propylene glycol was studied. It is found that the formation of propylene glycol and propanoic acid as a byproduct proceeds via parallel pathways.

CATALYTIC BEHAVIOUR OF CARBON SUPPORTED PLATINUM GROUP METALS IN THE HYDROGENATION AND ISOMERIZATION OF METHYL OLEATE

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Appl. Catal., A,
357(2) (2009) pp. 170-177.

Kinetic peculiarities of *cis* methyl oleate hydrogenation over Pd/C, Ru/C, Rh/C, Pt/C and Ir/C were studied. Liquid phase hydrogenation was carried out at temperature $298 \le T \le 373$ K and under hydrogen pressure within 1–10 bar using *n*-octane as a solvent. GLC and FTIR techniques were employed to characterize the reaction products. The effects of the catalyst amount, *cis* methyl oleate concentration, reaction temperature, and hydrogen pressure on the rates of methyl oleate hydrogenation and *cis-trans* isomerization were investigated. A simple reaction scheme involving *cis* methyl oleate hydrogenation and *cis-trans* isomerization was proposed for kinetic modeling. A good agreement was obtained between the calculated kinetic curves and the experimental data for the isomerization and hydrogenation of methyl oleate. It was shown that the second-row metals (Ru, Rh, Pd) display high activity in isomerization whereas the third-row metals (Ir, Pt) exhibit minor activity.

MODELING OF KINETICS AND STEREOSELECTIVITY IN LIQUID-PHASE α-PINENE HYDROGENATION OVER Pd/C

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Appl. Catal., A,
356(2) (2009) pp. 216-224.

The hydrogenation of α-pinene was investigated in a batch reactor over 4 wt% palladium on carbon (Pd/C) catalyst in temperature range of 50–90°C and 2–11 bar of hydrogen pressure using *n*-octane as a solvent. The reaction network involves hydrogenation of α-pinene to two stereoisomers. The reaction kinetics was established in conditions free from diffusional limitations. The rate was temperature, pressure and reactant concentration dependent. Stereoselectivity was independent on conversion, but was sensitive to temperature and hydrogen pressure. A reaction network and mechanisms were proposed and corresponding kinetic equations were derived. The parameters of the mechanistic kinetic models were determined by using non-linear regression analysis. The kinetic models described the formation of the products with satisfying accuracy.

KINETICS, CATALYST DEACTIVATION AND MODELING IN THE HYDROGENATION OF β-SITOSTEROL TO β-SITOSTANOL OVER MICROPOROUS AND MESOPOROUS CARBON SUPPORTED Pd CATALYSTS

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Chem. Eng. J.,
154(1-3) (2009) pp. 45-51.

Kinetics of the hydrogenation of β-sitosterol to β-sitostanol is of industrial interest, since the desired product is used for suppressing cholesterol absorption in human body. The main drawback when using microporous Pd/C catalyst in this reaction is catalyst deactivation. In the current work the performance of microporous and mesoporous Pd catalysts in the hydrogenation of β-sitosterol was compared. The catalytic hydrogenations were performed in a shaking batch reactor in 1-propanol as a solvent. With larger amounts of catalyst less catalyst deactivation occurred due to the fact that the catalyst support acted also as an adsorbent. The mesoporous 4 wt.% Pd/C (Sibunit) catalyst showed higher sitosterol conversions and less catalyst deactivation compared to a microporous 5 wt.% Pd/C catalyst. The kinetics of the hydrogenation of β-sitosterol to β-sitostanol was studied over 4 wt.% Pd/C (Sibunit) catalyst at different temperatures between 60°C and 80°C and by reusing the catalyst. The origin for catalyst deactivation was poisoning by phosphorus and sulphur, as well as coking. *In situ* catalyst potential measurements showed that there is a correlation between catalyst deactivation and decreasing catalyst potential with increasing sitosterol conversion. A mechanistic kinetic model including a deactivation factor was successfully applied to this reaction and the kinetic parameters were determined.

DECALIN CYCLE OPENING REACTIONS ON RUTHENIUM-CONTAINING ZEOLITE MCM-41

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Pet. Chem.,
49(1) (2009) pp. 90-93.

Ruthenium catalysts supported on the mesoporous carrier MCM-41 were synthesized and characterized. It was found that the presence of the metal markedly accelerated decalin ring opening reactions as compared to the acid support alone, and it increases the selectivity owing to inhibition of protolytic cracking. It was shown that activity of ruthenium catalytic systems is lower than that of platinum catalysts supported on MCM-41 and much lower than the activity of platinum on zeolites. The low activity of catalytic systems based on the mesoporous support MCM-41 is due to its substantially lower acidity in comparison with zeolites beta and Y. Thus, it is concluded that the use of MCM-41 in decalin ring opening reactions is limited even in the presence of the metal, first of all, because of the low acidity of the support.

CONVERSION OF TETRALONES INTO NAPHTHOLS IN SUPERCRITICAL WATER

K.Yu. Koltunov, S.I. Abornev

Supercrit. Fluids: Theory Practice,
4(4) (2009) pp. 97-103.

The reactivity of several 1- and 2-tetralones in subcritical and supercritical water (T = 400°C, $p = 0.2\ g/cm^3$) is studied. Tetralones as well as tetralin are shown to undergo dehydrogenation that leads to corresponding naphthols and naphthalene, respectively.

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

COMPLEXES FORMING FROM AMMONIUM PARAMOLYBDATE, ORTHOPHOSPHORIC ACID, COBALT OR NICKEL NITRATE, AND CARBAMIDE IN SOLUTION AND THEIR USE IN THE PREPARATION OF DIESEL FUEL HYDRODESULFURIZATION CATALYSTS

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Kinet. Catal.,
50(6) (2009) pp. 867-873.

It is demonstrated by ^{14}N, ^{17}O, ^{31}P, and ^{95}Mo NMR spectroscopy that the heteropolyanion $[P_2Mo_5O_{23}]^{6-}$ forms in impregnating solutions containing orthophosphoric acid, ammonium paramolybdate, cobalt or nickel nitrate, and carbamide. The anion forms labile complexes with Co^{2+} or Ni^{2+} cations by coordinating to them through terminal oxygen atoms of the MoO_6 octahedra and outer oxygen atoms of the PO_4 groups. The catalysts prepared by supporting these complexes on Al_2O_3 are highly active in diesel fuel hydrodesulfurization. They compare well with the best foreign analogues and are superior to most of the Russian commercial hydrodesulfurization catalysts.

FORMATION OF MONONUCLEAR RHODIUM(III) SULFATES: ^{103}Rh AND ^{17}O NMR STUDY

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Russ. J. Coord. Chem.,
35(8) (2009) pp. 577-581.

It was shown that the monomeric rhodium sulfate complexes $[Rh(H_2O)_4(SO_4)]^+$, *trans*-$[Rh(H_2O)_2(SO_4)_2]^-$, *cis*-$[Rh(H_2O)_2(SO_4)_2]^-$, and $[Rh(SO_4)_3]^{3-}$ were not predominant forms in aqueous solutions. The ^{103}Rh NMR chemical shifts of the complexes were assigned, and the conditions for their formation in solutions, concentration parameters, and acidity at which the fraction of the monomers was maximal were determined. The constants of formation of the complexes and ion pair (IP) were estimated: $K_{IP} = 8 \pm 3.5$, $K_1 \approx 8$, $K_{2trans} \approx 1$, $K_{2cis} \approx 1$, and $K_3 \approx 2$.

^{103}Rh AND ^{17}O NMR STUDY OF OLIGOMER RHODIUM(III) SULFATES IN AQUEOUS SOLUTIONS

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Russ. J. Coord. Chem.,
35(11) (2009) pp. 824-829.

By virtue of ^{103}Rh-, ^{17}O-NMR, electrophoresis in agarose gel, and pH-metry, the authors report on the formation of rhodium(III) sulfate complexes in aqueous solutions. At higher concentrations of sulfuric acid (above 3 M), more than 90% of metal was found to stay in the state of symmetric polynuclear complexes containing magnetically equivalent rhodium atoms. The authors also labeled the ^{103}Rh-NMR chemical shifts for the complexes with 3, 4 and 6 metal atoms in the spectra.

NITRATION OF RHODIUM(III) SULFATES

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Russ. J. Inorg. Chem.,
54(8) (2009) pp. 1290-1295.

Nitration of sulfate complexes of rhodium has been investigated by NMR ^{103}Rh, ^{14}N, ^{15}N, and ^{17}O NMR. At high pH, $[Rh(NO_2)_6]^{3-}$, dimer $[Rh_2(\mu\text{-OH})_2(NO_2)_8]^{4-}$, and trimer $[Rh_3(\mu\text{-OH})_4(OH)(NO_2)_9]^{5-}$ are the dominant species in solutions.

POTASSIUM PEROXOSTANNATE NANOPARTICLES

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Russ. J. Inorg. Chem.,
54(6) (2009) pp. 824-828.

Stable amorphous potassium peroxostannate nanoparticles with controlled sizes (10–100 nm), morphology, and hydrogen peroxide percentage (19–30 wt %) were synthesized for the first time. The compounds were characterized by vibrational spectroscopy, ^{119}Sn MAS NMR spectroscopy, powder X-ray diffraction, and thermogravimetry. These characteristics were compared to those for $K_2Sn(OH)_6$ and $K_2Sn(OOH)_6$. Potassium peroxostannate particles are mainly built of peroxo-bridged polymer chains. The particles are stable when stored in a dry state or suspended in nonaqueous solvents; in contact with water, they release hydrogen peroxide.

HYDROGEN PEROXIDE INDUCED FORMATION OF PEROXYSTANNATE NANOPARTICLES

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J. Sol-Gel Sci. Technol,
50(2) (2009) pp. 229-240.

Stable, amorphous potassium peroxystannate nanoparticles of controlled average size in the range 10–100 nm and of controlled hydrogen peroxide content in the range of 19–30 wt% were synthesized by hydrogen peroxide induced polymerization in water–potassium hexahydroxostannate solutions. The sol phase and the precipitate were characterized by vibrational spectroscopies, ^{119}Sn NMR, XPS and XRD using crystalline $K_2Sn(OH)_6$ and $K_2Sn(OOH)_6$ reference materials. This is the first study to show that peroxocoordination induces polymerization of a main group element. ^{119}Sn NMR studies show that peroxotin coordination and polymerization took place already in the hydrogen peroxide– water phase. The high abundance of peroxotin bonds revealed by ^{119}Sn MAS NMR, vibrational spectroscopy, and XPS suggest that the particles are predominantly made of peroxo bridged tin networks. Although the particles are highly stable in the dry phase as well as in alcohol solutions and do not lose hydrogen peroxide upon storage, they release their stored hydrogen peroxide content by exposure to water.

EFFECT OF THE ACIDITY OF A ZEOLITE AND ITS MODIFICATION WITH CERIUM AND ZIRCONIUM ON THE ACTIVITY AND THERMAL STABILITY OF Pd/Beta IN THE REACTION OF DEEP TOLUENE OXIDATION

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Russ. J. Appl. Chem.,
82(1) (2009) pp. 34-39.

Acidities of beta zeolites with various moduli and the catalytic activities of Pd/beta in the reaction of deep toluene oxidation were compared.

SOLID-STATE ^{51}V NMR AND ITS POTENTIALITY IN INVESTIGATION OF VANADIA SYSTEMS WITH PARAMAGNETIC CENTERS

A.A. Shubin, D.F. Khabibulin, O.B. Lapina

Catal. Today, 142(3-4) (2009) pp. 220-226.

In this article the potentiality of solid-state ^{51}V NMR technique to characterize heterogeneous catalysts containing paramagnetic cations located on the surface or incorporated in the bulk of heterogeneous catalysts will be discussed. ^{51}V NMR data for a number of 3d and rare earth vanadates, where paramagnetic effects are caused by the presence of paramagnetic cations, are considered. The results were summarized available for vanadium bronzes, where some vanadium is in a paramagnetic V^{4+} state, and for some vanadium compounds, where closely spaced paramagnetic centers form diamagnetic pairs. The comprehensive ^{51}V chemical shift scale (database) for paramagnetic solids was compiled.

PRECURSOR EFFECT ON THE MOLECULAR STRUCTURE, REACTIVITY, AND STABILITY OF ALUMINA-SUPPORTED VANADIA

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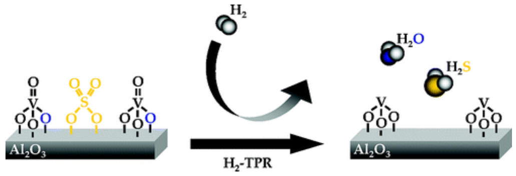
J. Phys. Chem. C,
113(48) (2009) pp 20648-20656.

This work studies the influence of specific vanadium precursors on alumina-supported vanadium oxide catalysts. Vanadyl sulfate, ammonium metavanadate, and vanadyl acetylacetonate were used as precursors. All catalysts have the same surface vanadium coverage (4 V atoms/nm^2). The structural features of surface vanadium oxide species were studied by ^{51}V-NMR, UV–vis, and Raman spectroscopy. The vanadium precursor determines the strength of interaction between supported VO_x species and alumina. The reducibility of vanadium ions was estimated by conventional TPR/TPO cycles. The structural and electronic changes during reduction/oxidation cycles were studied by *in situ* Raman and UV–vis spectroscopy (TPR/TPO-Raman, TPR/TPO-UV–vis). Surface sulfate species reduce concomitantly to surface vanadium oxide species.



POTENTIAL OF ^{129}Xe NMR SPECTROSCOPY OF ADSORBED XENON FOR TESTING THE CHEMICAL STATE OF THE SURFACE OF MESOPOROUS CARBON MATERIALS ILLUSTRATED BY THE EXAMPLE OF AGGREGATES OF DIAMOND AND ONION-LIKE CARBON NANOPARTICLES

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Kinet. Catal.,
50(1) (2009) pp. 26-30.

The chemical shift in the ^{129}Xe NMR spectrum of adsorbed xenon is very sensitive to the presence of oxygen-containing functional groups on the surface of mesoporous carbon materials. Well-characterized, structurally similar nanodiamond and onion-like carbon samples are considered here as model objects.

MAGNETIC RESONANCE IMAGING STUDIES ON CATALYST IMPREGNATION PROCESSES: DISCRIMINATING METAL ION COMPLEXES WITHIN MILLIMETER-SIZED γ-Al_2O_3 CATALYST BODIES

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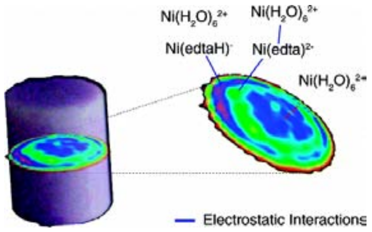
J. Am. Chem. Soc.,
131(18) (2009) pp. 6525-6534.

Magnetic resonance imaging (MRI) was used to study the impregnation step during the preparation of Ni/γ-Al_2O_3 hydrogenation catalysts with Ni^{2+} metal ion present in different coordinations. The precursor complexes were $[Ni(H_2O)_6]^{2+}$ and $[Ni(\mathit{edta}H_x)]^{(2-x)-}$ (where x = 0, 1, 2 and *edta* = ethylenediaminetetraacetic acid), representing a nonshielded and a shielded paramagnetic complex, respectively. Due to this shielding effect of the ligands, the dynamics of $[Ni(H_2O)_6]^{2+}$ or $[Ni(\mathit{edta}H_x)]^{(2-x)-}$ were visualized applying T_2 or T_1 image contrast, respectively. MRI was applied in a quantitative manner to calculate the $[Ni(H_2O)_6]^{2+}$ concentration distribution after impregnation when it was present alone in the impregnation solution, or together with the $[Ni(\mathit{edta}H_x)]^{(2-x)-}$ species. Moreover, the combination of MRI with UV–vis microspectroscopy allowed the visualization of both species with complementary information on the dynamics and adsorption/desorption phenomena within γ-Al_2O_3 catalyst bodies. These phenomena yielded nonuniform Ni distributions after impregnation, which are interesting for certain industrial applications.



AN NMR IMAGING STUDY OF STEADY-STATE AND PERIODIC OPERATION MODES OF A TRICKLE BED REACTOR

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Top. Catal.,
52(10) (2009) pp. 1371-1380.

NMR imaging has been applied to study the steady-state and the periodic operations of a functioning trickle bed reactor. It has been revealed that under conditions of the continuous supply of a liquid reagent to the catalyst bed, the bed was mostly filled with the liquid phase and was characterized by the uniform and stationary distribution of a liquid phase, whereas under conditions of the periodic supply of a liquid reagent to the catalyst bed with the same liquid flow velocity the bed was mostly dry and was characterized by a non-stationary distribution of the liquid phase. The oscillations of the liquid phase content within the bed, corresponding to the modulated liquid flow, have been observed. It has been shown that performing the hydrogenation reaction in a trickle bed reactor under conditions of the periodic supply of a liquid reagent to the catalyst bed leads to the intensification of the hydrogenation process. It becomes apparent in the significant increase of the temperature of the catalyst bed as well as in the increase of the conversion degree in the regimes under forced time-varying liquid flow rates in comparison to the steady-state regime of the reactor operation.

WATER DYNAMICS IN BULK AND DISPERSED IN SILICA $CaCl_2$ HYDRATES STUDIED BY NEUTRON SCATTERING METHODS

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Micropor. Mesopor. Mater.,
125(1-2) (2009) pp. 46-50.

Quasi-elastic and inelastic neutron scattering techniques have been used to study the dynamics of water in $CaCl_2$ x nH_2O (n = 1/3, 2, 4, 6, 9) hydrates dispersed in silica, in comparison with the bulk hydrates. Inelastic scattering shows that the water molecules in dispersed hydrates are in an amorphous state. Quasi-elastic scattering indicates a higher mobility of water in the hydrates dispersed in silica, compared to bulk hydrates. The self-diffusivity of water in the dispersed hydrates increases with water concentration, while remaining below the one of pure water. The neutron and pulsed-field gradient NMR diffusivities are similar, which indicates that the hydrates form a thin and continuous layer on the pore walls of silica.

ADSORPTION AND DIFFUSION OF H_2 IN THE MOF TYPE SYSTEMS MIL-47(V) AND MIL-53(Cr): A COMBINATION OF MICROCALORIMETRY AND QENS EXPERIMENTS WITH MOLECULAR SIMULATIONS

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J. Phys. Chem. C,
113(18) (2009) pp. 7802-7812.

Quasi-elastic neutron scattering measurements are combined with molecular dynamics simulations to determine the self-diffusivity (Ds) profile of hydrogen in the metal organic framework materials MIL-47(V) and MIL-53(Cr) (MIL, Materials Institut Lavoisier) as a function of loading. Experimentally, a sudden increase in Ds for H_2 at low loading (≤1 H_2/unit cell) was observed with values at least two orders of magnitude higher than in zeolites. This unusual behavior has been denoted as “super-mobility”. Here, two different force fields available in the literature to represent the H_2/H_2 and H_2/MOF framework interactions have been considered to capture such experimental findings via molecular dynamics simulations. It has been shown that (i) a similar magnitude of the energetic contribution for the H_2/H_2 and H_2/MOF framework interactions and (ii) a smoothness of the potential energy surfaces are required in order to match the supermobility of H_2 at low loading. The diffusion mechanism at the microscopic scale was successfully simulated in both materials and described in terms of the chemical features of the MIL framework, i.e., the presence or absence of the μ_2 hydroxyl group. It appears that a one-dimensional (1D) diffusion along the *z* axis and purely random three-dimensional (3D) diffusion processes are observed for MIL-53(Cr) and MIL-7(V), respectively. The adsorption properties were then simulated using the different force fields initially fitted to the diffusion results and compared to manometry measurements. Finally, the comparison of diffusion and adsorption results for the different force fields leads to choose the best compromise to describe both dynamic and thermodynamic properties.

In situ XRD STUDY OF NANOCRYSTALLINE COBALT OXIDE REDUCTION

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S.V. Tsybulya**

Kinet. Catal.,
50(2) (2009) pp. 192-198.

The reduction of nanocrystalline cobalt oxide samples (single-phase and supported on γ-Al_2O_3) was studied using *in situ* X-ray diffraction (XRD) analysis. The atomic structures of single-phase and supported Co_3O_4 samples were refined, and the occurrence of cationic vacancies was demonstrated. A set of methods (XRD, temperature-programmed reduction, and differential dissolution) was used to find that the reduction of supported and unsupported model cobalt oxide was considerably different. The single-phase sample was reduced in undiluted hydrogen to cobalt metal with a hexagonal closely packed structure. The reduction of the supported sample (unlike the single-phase sample) occurred through the formation of a crystalline CoO phase to the formation of cobalt metal with a face-centered cubic structure. Interaction of cobalt oxide with the γ-Al_2O_3 support, which hinders the reduction to cobalt metal, was detected.

In situ XRD INVESTIGATION OF Co_3O_4 REDUCTION

**O.A. Bulavchenko, S.V. Cherepanova,
S.V. Tsybulya**

Z. Kristallogr.,
30 (2009) pp. 329-334.

In situ X-ray diffraction (XRD) method was used to investigate the Co_3O_4 reduction in catalyst supported on γ-Al_2O_3 and oxide monophase sample. Initial structure of Co_3O_4 in both samples is similar and contains vacancies in the cation positions. In spite of this reduction process of Co_3O_4 is different for supported and monophase samples as has been shown by *in situ* XRD. In monophase sample the cobalt oxide is reduced to metallic cobalt (hcp) directly. On the contrary the two-stage reduction is observed for supported sample: Co_3O_4 is reduced to CoO and then to metallic Co (fcc). In both cases nanoparticles of metallic cobalt contains high concentration of stacking faults.

HIGH-TEMPERATURE X-RAY STUDY OF THE FORMATION AND DELAMINATION OF MANGANESE-ALUMINA SPINEL $Mn_{1.5}Al_{1.5}O_4$

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S.V. Cherepanova, T.N. Afonasenko*,
P.G. Tsyrulnikov*** (**Institute of Hydrocarbons Processing, Omsk, Russia*)

J. Struct. Chem.,
50(3) (2009) pp. 474-478.

The behavior of the manganese-alumina system with Mn:Al = 1:1 on heating in air and vacuum was studied. The starting samples were mixtures of β-Mn_3O_4, α-Mn_2O_3, and γ-Al_2O_3. On heating to 950°C in air, the samples were partially oxidized into α-Mn_2O_3, and corundum α-Al_2O_3 formed along with mixed manganese-alumina cubic spinel, whose composition was close to Mn_2AlO_4. In vacuum at 1200°C, the starting sample with a ratio of Mn:Al = 1:1 transformed into the manganese-alumina spinel $Mn_{1.5}Al_{1.5}O_4$, which retained its cubic structure after slow cooling in vacuum. When cooled in air, this solid solution delaminated, and a nanocrystalline $Mn_{2.8}Al_{0.2}O_4$ phase formed, whose structure was β-Mn_3O_4 type tetragonal spinel.

A STUDY OF THE MICROSTRUCTURE OF $La_{1-x}Ca_xMnO_3$ (x=0.5, 0.8) SOLID SOLUTIONS

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J. Surf. Invest. - X-Ray Synchrotron Neutron Techn.,
3(5) (2009) pp. 756-760.

HREM and XPD methods are used to study the microstructure of $La_{1-x}Ca_xMnO_3$ perovskite-like oxides synthesized by pyrolysis of polymer-salt compositions. The XPD data show that the samples studied are single-phase solid solutions. Morphological transitions are observed in series of samples depending on the substitution parameter x. At $0 \leq x \leq 0.4$ the samples can be characterized by rhombic symmetry (space group *Pnmb*); at $0.5 \leq x \leq 0.8$ the symmetry increases to the tetragonal space group I4/*mmm*; at $0.9 \leq x \leq 1$ the symmetry lowers to the monoclinic space group *P*1121. Heating of samples with $x = 0.5$ and 0.8 up to 1200°C in air does not lead to noticeable changes in the sample structure, which indicates their high thermal stability. When the $La_{0.2}Ca_{0.8}MnO_3$ sample is heated to 1100°C in vacuum nanocrystalline states with particle disintegration into microphases of different structures form because of a partial decomposition of the solid solution. As a result, a system of Mn_3O_4 nanoparticles appears, which is coherently bound with the perovskite phase of a defective structure.

STRUCTURAL FEATURES, NONSTOICHIOMETRY AND HIGH-TEMPERATURE TRANSPORT IN $SrFe_{1-x}Mo_xO_{3-\delta}$

A.A. Markov*, O.A. Savinskaya, M.V. Patrakeev*, A.P. Nemudry**, I.A. Leonidov*, Yu.T. Pavlyukhin**, A.V. Ishchenko, V.L. Kozhevnikov*** (**Institute of Solid State Chemistry, Yekaterinburg, Russia; **Institute of Solid State Chemistry and Mechanochemistry, Novosibirsk, Russia*)

J. Solid State Chem.,
182(4) (2009) pp. 799-806.

The oxide solid solutions $SrFe_{1-x}Mo_xO_{3-\delta}$, where x=0.05, 0.1 and 0.2, are studied in this work. It is shown that substitution of iron for molybdenum results in stabilization of a cubic quasi-perovskite locally inhomogeneous structure, which is evidenced by HREM and Mössbauer spectroscopy. The coulometric titration is used in order to determine changes of oxygen nonstoichiometry in the obtained solutions with temperature and ambient oxygen partial pressure. Partial molar thermodynamic functions of the labile oxygen are calculated from the measured data. The variations of partial molar entropy with oxygen content follow the ideal gas model reasonably well thus demonstrating approximately random distribution of oxygen vacancies in the doped ferrite at high temperatures. The partial molar enthalpy is found to increase with doping, which is indicative of a progressive decrease in average values of the bonding energy of labile oxygen ions. The measurements of total conductivity are used in order to determine partial contributions of charge carriers. The oxygen ion component is shown to increase at small level of doping, x=0.05 while further increase in molybdenum content is accompanied with the decline in the ion conductivity. The electron contribution in reducing conditions tends to increase with molybdenum content, which is interpreted as a manifestation of involvement of Mo^{5+} cations in electron transport. Concentration and mobility of electron carriers are calculated. Some increase in mobility of electron holes at small doping is explained as related to the filling of oxygen vacancies.

PROPERTIES OF Nb-DOPED $SrCo_{0.8}Fe_{0.2}O_{3-d}$ PEROVSKITES IN OXIDIZING AND REDUCING ENVIRONMENTS

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Catal. Today,
141(3-4) (2009) pp. 270-274.

The phase stability of $SrCo_{0.8}Fe_{0.2}O_{3-d}$ perovskite doped with niobium was studied by *in situ* high-temperature X-ray diffraction in the temperature range of 30–1000°C and oxygen partial pressure $0.2–10^{-5}$ atm. The stability of the cubic perovskite structure in a wide range of oxygen partial pressures is the main advantage of $SrCo_{0.8-x}Fe_{0.2}Nb_xO_{3-d}$ ($x = 0.1–0.3$) system in comparison with $SrCo_{0.8}Fe_{0.2}O_{3-d}$. It is suggested that equilibrium of the thermal expansion with changes of the oxygen non-stoichiometry leading to the same lattice parameters in the oxidizing and reducing environments at the catalytic temperatures is a necessary requirement for stable operation of perovskite as an oxygen-conducting membrane. In the case of $SrCo_{0.8-x}Fe_{0.2}Nb_xO_{3-d}$ perovskite this condition is met at $x = 0.2$. This makes the $SrCo_{0.6}Fe_{0.2}Nb_{0.2}O_{3-d}$ composition promising for application as oxygen-conducting membrane.

HIGH-TEMPERATURE PHASE TRANSITIONS IN THE $La_{0.25}Sr_{0.75}FeO_{3-\delta}$ SOLID SOLUTION WITH A PEROVSKITE STRUCTURE

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J. Struct. Chem.,
50(1) (2009) pp. 108-113.

Phase transformations in a sample from the series $La_{1-x}Sr_x FeO_{3-\delta}$ (x = 0.75) with cubic symmetry and perovskite type structure were studied by high-temperature X-ray diffraction. In air, the solid solution was stable to the synthesis temperature (1200°C), despite the loss of some part of oxygen. Heating in vacuum led to a two-phase state at 900°C (or even at 600°C when a small amount of palmitic acid was added to the sample); the unit cell parameter changed abruptly. The amount of the new phase increased with temperature, and at 1000°C, the sample, as well as the starting phase, was monophase perovskite with cubic symmetry; i.e., an isosymmetric phase transition occurred, which was accompanied by an abrupt change in the cell volume. Cooling in vacuum to ~800°C led to the ordering of the oxygen vacancies and the formation of a Grenier phase type structure. When the sample was heated again in vacuum, a reversible phase transition occurred from the Grenier phase type structure to a perovskite structure (again at ~800°C).

PECULIARITIES OF THE X-RAY DIFFRACTION OF OXYGEN-DEFICIENT PEROVSKITE-RELATED MATERIALS WITH PARTIAL VACANCY ORDERING

S.V. Cherepanova, S.V. Tsybulya

Z. Kristallogr.,
27 (2008) pp. 5-12.

X-ray diffraction patterns have been simulated for perovskite-Grenier phase and perovskite-brownmillerite coherent heterogeneous systems. Such systems occur as a result of partial ordering in oxygen-deficient perovskite-related materials. Calculations have been carried out on the basis of the model of 1D-disordered crystal. Simulation shows that main diffraction effect characteristic for both systems is appearance of broad peak of diffuse scattering between 110 diffraction peak of cubic perovskite and 113 diffraction peak of Grenier phase or 114 diffraction peak of brownmillerite.

STRUCTURAL TRANSFORMATIONS IN MECHANOCHEMICAL SYNTHESIS OF SOLID SOLUTIONS IN THE Cu-Ga SYSTEM

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Russ. J. Appl. Chem.,
82(5) (2009) pp. 779-782.

Formation of solid solutions in the copper-gallium system under mechanical activation is considered as an example of a mechanochemical interaction between a solid metal and a liquid metal.

An X-ray phase analysis was made using a URD-63 diffractometer equipped with a graphite monochromator (CuKα radiation).

The X-ray diffraction studies were carried out *in situ* using a detector system based on a Marresearch MAR-345 storage screen.

Precision X-ray diffraction studies were carried out using the method of recording in parallel beams.

Electron-microscopic studies were performed on a JSM-T20 electron microscope and JEM-2010 and JEM-400 high-resolution electron microscopes.

MECHANOCHEMICAL PREPARATION OF Ni_xAl_y/Al_2O_3 NANOCOMPOSITES

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Russ. Metall.,
4 (2009) pp. 87-91.

The mechanochemical preparation of nickel aluminide/corundum (Ni_xAl_y/Al_2O_3) powder nanocomposites is shown to be possible during the mechanochemical aluminum reduction of nickel oxide at various weight proportions of the components.

STRUCTURAL TRANSFORMATIONS UPON THE MECHANOCHEMICAL INTERACTION BETWEEN SOLID AND LIQUID METALS

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Phys. Met. Metallogr.,
107(5) (2009) pp. 457-465.

The process of mechanochemical interaction of solid and liquid metals is examined. It is shown that the mechanochemical formation of solid solutions in all the cases occurs through the stages of the formation of stable intermetallic compounds.

MECHANOCOMPOSITES BASED ON TUNGSTEN FOR OBTAINING PSEUDO-ALLOYS

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Chem. Sustain. Devel.,
17(6) (2009) pp. 553-558.

Structure and morphology of mechanocomposites based on tungsten with copper, nickel or iron added were studied by means of X-ray diffraction studies, electron microscopy and micro-X-ray spectral analysis. The possibilities of pressing dense samples from the obtained mechanocomposites were demonstrated.

MECHANOCHEMICAL PRODUCTION OF NANOCOMPOSITES OF METAL/OXIDE AND INTERMETALLIC/OXIDE SYSTEMS

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J. Phys.: Conf. Ser.,
144 (2009) 012076 (4 pp).

Addition of nanosized intermetallic or metallic phases into corundum considerably raises mechanical behavior of the material. In this work, the nanocomposites of α-Al_2O_3/intermetallic and α-Al_2O_3/metal systems were obtained by mechanochemical reduction of α-Fe_2O_3 by Al (and by solid solution of Al in Fe). The mechanochemical reduction process of hematite by various amount of metal-reducer was studied by IR and Mössbauer spectroscopies, and by X-ray synchrotron radiation diffraction technique.

STRUCTURE OF CATALYTICALLY ACTIVE GOLD NANOPARTICLES BY XAFS SPECTROSCOPY

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J. Phys.: Conf. Ser.,
190 (2009) 012121 (4 pp.).

A series of Au/Al_2O_3 catalysts prepared by the different procedures and series of model system Au/Si(100), Au/Si(111) differing in the mean Au particle sizes (from 2 nm up to 30 nm) were studied by EXAFS and XANES techniques. No visible changes in electron states of Au for all samples prepared by different methods in comparison with bulk material were detected. Oxygen atoms were not detected around Au atoms within the detection limits of study (a few per cents of surrounding atoms). A gradual reduction of the Au-Au bond length and first shell Au-Au coordination number and an increase in the Debye-Waller factor are observed as the size of supported Au particles decreases. The significant increase of structural disorder for the smallest Au particles comparatively to the bulk Au metal and sizeable particles was detected. These variations in micro-structural parameters of Au nanoparticles are in line with an increase in their catalytic activity in CO oxidation. For model systems some coordination of Au-atoms from islands and Si-atoms from crystal surface was detected for thin Au "films".

OPTICAL PROPERTIES OF THE $HfO_{2-x}N_x$ AND $TiO_{2-x}N_x$ FILMS PREPARED BY ION BEAM SPUTTERING

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Opt. Spectrosc.,
106(1) (2009) pp. 72-77.

Optical characteristics of the $HfO_{2-x}N_x$ and $TiO_{2-x}N_x$ films obtained by reactive ion beam sputtering have been investigated by spectral ellipsometry. The chemical composition of the films was determined using X-ray photoelectron spectroscopy. The nitrogen content in the oxynitride films (determined by the N_2/O_2 ratio in the gas mixture during synthesis) reached ~9 at % for $TiO_{2-x}N_x$ and ~ 6 at % for $HfO_{2-x}N_x$. It is found that the dispersion relations $n(\lambda)$ and $k(\lambda)$ for the $TiO_{2-x}N_x$ films change from those characteristic of titanium dioxide to those typical of titanium nitride with an increase in the nitrogen content from 0 to ~9 at %. The optical parameters of the $HfO_{2-x}N_x$ films depend weakly on the nitrogen content in the range 0–6 at %.

THE USE OF X-RAY ABSORPTION SPECTROSCOPY FOR DEVELOPING NEW GENERATION Co-Mo CATALYSTS OF HYDROTREATING OF DIESEL FUEL

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Doklady Phys. Chem.,
424(2) (2009) pp. 35-39.

In this paper, the results of the EXAFS and XANES study of the structure of bimetallic oxide precursors of the active phases of hydrotreating Co–Mo catalysts are reported.

Based on X-ray absorption spectroscopy data, it was determined, for the first time, the structure of compounds that form during different steps of the preparation of hydrotreating catalysts. In the impregnating solution, the ammonium salt $(NH_4)_4[Mo_4(C_6H_5O_7)_2O_{11}]$ formed first, which then reacted with $Co(CH_3COO)_2{\cdot}4H_2O$ to give the bimetallic compound $Co_2[Mo_4(C_6H_5O_7)_2O_{11}]$. The basic structural unit of this compound is the tetranuclear molybdenumcontaining anion coordinated to the Co^{2+} cations. The distance between Co and the nearest Mo atom is 3.41 Å. When $Co_2[Mo_4(C_6H_5O_7)_2O_{11}]$ is deposited on the Al_2O_3 surface, the structure of the bimetallic compound remains unaltered. These findings underlie the technology of preparation of the first new-generation domestic catalyst ICK-GO-1 for hydrotreating of diesel fuel. This catalyst has been in operation since the end of 2007 at Saratov oil refinery plant.

EXAFS STUDY OF OXIDE PRECURSORS OF THE HIGH ACTIVE Co–Mo HYDROTREATING CATALYSTS: EFFECT OF DRYING CONDITIONS

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Nucl. Instrum. Methods Phys. Res., Sect. A,
603(1-2) (2009) pp. 119-121.

The structure of the surface Mo compounds was studied by EXAFS for the hydrotreating catalysts prepared from bimetallic Co–Mo complexes and dried at different temperatures. The decomposition of the primary bimetallic compound and the formation of the inactive components were observed in the catalysts heated above 220°C.

SYNTHESIS AND STRUCTURE OF MONORIBBED-FUNCTIONALIZED DISULFIDE IRON(II) CLATHROCHELATES AND THEIR COORDINATION AS THE LIGANDS TOWARD PLATINUM(II) AND PLATINUM(IV) IONS

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Inorg. Chim. Acta,
362(1) (2009) pp. 149-158.

Disulfide monoribbed-functionalized clathrochelates (*i.e.*, fuctionalization of one of the three α-dioximate fragments) with ribbed thioalkyl, S_3-thioalkyl and hydroxythioalkyl substituents have been synthesized starting from the $FeBd_2(Cl_2Gm)(BF)_2$ precursor (where Bd^{2-} and Cl_2Gm^{2-} are α-benzyldioxime and dichloroglyoxime dianions) using the corresponding thiol/triethylamine system in dichloromethane solution. Clathrochelate S_6-dithiol in basic media underwent the intramolecular dealkylation to yield the S_3-thiocrown etheric clathrochelate. Clathrochelates obtained have been studied as the ligands toward Pt^{2+} and Pt^{4+} ions. The *S*-demethylation reaction of the methylsulfide complex with $[PtCl_4]^{2-}$ dianion produced the polynuclear complexes of the dianionic clathrochelate dithiolate ligand. The reaction of *n*-butylsulfide clathrochelate with the *trans*-$Pt^{IV}Cl_4(C_6H_5CH_2CN)_2$ afforded the binuclear compound with the disulfide iron(II) clathrochelate as a monodentate ligand. The obtained macrobicycles, their clathrochelate derivatives, and polynuclear complexes have been characterized using elemental analysis, MALDI-TOF and PD mass, IR, UV–Vis, and NMR spectra, and X-ray crystallography. The encapsulated iron(II) ion coordination polyhedra distortion angle φ values and the main distances in the molecules of polynuclear complexes have been deduced (obtained) using ^{57}Fe Mössbauer parameters and EXAFS data, respectively.

COMBINED *IN SITU* EXAFS AND ELECTROCHEMICAL INVESTIGATION OF THE OXYGEN REDUCTION REACTION ON UNMODIFIED AND Se-MODIFIED Ru/C

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Catal. Today,
147(3-4) (2009) pp. 260-269.

The kinetics of the oxygen reduction reaction on carbon-supported Ru nanoparticles modified with various amounts of Se has been studied. Rotating disk electrode is used to determine kinetic currents for the ORR in 0.1 M H_2SO_4 at 298 K and O_2 partial pressures from 1 to 0.01 atm. The dependence of the ORR activity on Se/Ru ratio shows volcano-type behavior with ca. 10 fold increase of the mass activity at $0.1 < Se/Ru < 0.3$. The reaction order in O_2 is close to 1 in the interval of overpotentials from 0.4 to 0.7 V, and is independent of the presence of Se. Regardless the amount of Se, the Tafel slope demonstrates continuous increase from ca. 70 mV/dec at 0.4 V to ca. 140 mV/dec at 0.6 V overpotential. *In situ* EXAFS spectra are measured at Ru K-edge (in the transmission mode) and Se K-edge (in the fluorescence mode) in argon and oxygen saturated 0.1 M H_2SO_4 solutions in the interval of electrode potentials from 0.050 to 0.750 V RHE. The data are used to explore the surface state changes of Ru and Ru_xSe_y particles and clarify the promoting role of Se during the ORR.

FORMATION OF ACTIVE CATALYSTS IN THE SYSTEM: CHLOROCUPRATES−CCl_4−*n*-$C_{10}H_{22}$

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J. Phys. Chem. A,
113(38) (2009) pp 10219–10223.

Transformations of anionic Cu^{II} chlorocomplexes have been studied under conditions of catalytic exchange reactions between carbon tetrachloride and *n*-alkanes. It was shown that chlorocuprates are just precursors and are easily reduced to the genuine catalysts, that is, to the respective Cu^{I} complexes. Both the composition and the geometric structure of the precursor ($CuCl_4^{2-}$) and, probably, the active site ($CuCl_3^{2-}$) have been investigated by several techniques (UV−vis spectroscopy, electron spin resonance (ESR), extended X-ray absorption fine structure (EXAFS), X-ray absorption near-edge structure (XANES), and static magnetic measurements). The dependence of the metathesis velocity on the $[Cl^-]/[Cu]$ ratio was found to exhibit a maximum most likely corresponding to the highest content of trichlorocuprite $CuCl_3^{2-}$.

EuroPt-1 CATALYST: X-RAY RADIAL DISTRIBUTION AND EXAFS STUDIES

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Russ. J. Struct. Chem.,
50(6) (2009) pp. 1082-1087.

The supported Pt/SiO_2 (EuroPt-1) catalyst has been studied by the radial distribution of electron density (RDED) and EXAFS techniques. The starting sample of the catalyst was stored in air, not subjected to any further treatment, and contained metal platinum Pt^0 and platinum oxide PtO in a ~1:2 ratio. An analysis of the EXAFS data was based on three possible structural models of platinum particles. Model 1 suggested that there was one Pt-Pt short contact, which was the same in the bulk of the particles and on the surface. Model 2 considered two different Pt-Pt distances for the particle volume and surface. For model 3, it was additionally assumed that the corresponding Debye-Waller factors differed. For the oxidized sample, model 2 was most reliable, and the Pt-Pt distance between the surface atoms was shortened by ~0.14 Å. For the reduced samples, the structural data obtained are consistent with model 3.

XRD STUDY OF THE LOCAL STRUCTURE AND PHASE COMPOSITION OF CATALYSTS WITH THE USE OF SYNCHROTRON RADIATION: METHOD OF THE RADIAL DISTRIBUTION OF ELECTRON DENSITY

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Nucl. Instrum. Methods Phys. Res., Sect. A,
603(1-2) (2009) pp. 99-101.

Some examples illustrating the use of the method based on the radial distribution function (RDF) of electronic density for investigation metal and oxide catalysts are reported. Using monoatomic unsupported metal catalysts with crystallite sizes D=40–450 Å, it was shown that the areas of coordination peaks in RDF directly define the absolute values of coordination numbers *Z* and depend on crystallite sizes. The investigation of the phase composition and local structure of supported metal catalysts revealed that the interaction between the active component and the support was responsible for the thermal stability of small supported metal particles. The local structure of ceria with different crystallite sizes was investigated.

STRUCTURE AND STATE OF COPPER OXIDE SPECIES SUPPORTED ON YTTRIA-STABILIZED ZIRCONIA

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J. Phys. Chem. C,
113(51) (2009) pp 21368-21375.

The structure and state of the active component - copper species in zirconia-supported copper catalysts with yttria-stabilized zirconia as a support were investigated. Catalysts with different copper contents (2−10 wt % of Cu) prepared by an impregnation technique were characterized by X-ray diffraction (XRD) analysis, radial distribution function (RDF) of electronic density method, X-ray absorption fine structure (XAFS) analysis, UV−visible diffuse reflectance spectroscopy (UV−vis DRS), and X-ray photoelectron spectroscopy (XPS). It was established that a small amount of copper could be incorporated in the zirconia fluorite lattice, while the main portion of the copper was situated on the support surface as finely dispersed copper oxide chain clusters.

COPPER–CERIUM OXIDE CATALYSTS SUPPORTED ON MONOCLINIC ZIRCONIA: STRUCTURAL FEATURES AND CATALYTIC BEHAVIOR IN PREFERENTIAL OXIDATION OF CARBON MONOXIDE IN HYDROGEN EXCESS

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Appl. Catal., A,
365(2) (2009) pp. 159-164.

Copper–cerium oxide catalysts supported on monoclinic zirconia $CuO/CeO_2/ZrO_2$ were tested in the preferential oxidation of CO in hydrogen-rich gases and the optimal catalyst composition was determined. The state and the structure of copper species in the catalysts were investigated by X-ray diffraction (XRD) and X-ray absorption fine structure (XAFS) analysis. While a small amount of copper incorporated into the surface layers of the CeO_2 lattice, the disordered oxide clusters were the main copper species in the catalysts with copper content ≤5 wt.%. The increase in the copper loading (>10 wt.%) led to predominant formation of bulk CuO phase coupled with catalyst deactivation.

PHOTOELECTRON STUDIES OF ADSORPTION AND CATALYSIS ON THE SURFACE OF METAL SINGLE CRYSTALS AND POLYCRYSTALS

A.I. Boronin

J. Struct. Chem.,
49(Suppl.) (2009) pp. S138-S158.

The potential of photoelectron spectroscopy (XPS) and its use in studies of the state of the surface and adsorption processes on bulk metal samples (disk single crystals, polycrystalline foil and plates) are discussed. Methods for determining the electronic state of the surface layer and geometrical localization of adsorbed atoms on the metal surface using angle-resolved photoelectron spectroscopy are described. The spectrokinetic use of the dynamic mode of XPS with high accuracy and time resolution for kinetic studies of fast processes on the surface (adsorption-desorption, reconstruction, reaction, diffusion-segregation) is considered in detail.

X-RAY PHOTOELECTRON SPECTROSCOPY STUDY OF Pd OXIDATION BY RF DISCHARGE IN OXYGEN

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Appl. Surf. Sci.,
255(22) (2009) pp. 9248-9254.

The low-temperature oxidation of polycrystalline palladium by RF oxygen plasma was studied via X-ray photoelectron spectroscopy (XPS) and scanning electron microscopy (SEM). Detailed information about the electronic states of palladium and oxygen was obtained based on the XPS curve fitting analysis of Pd3d and Pd3p + O1s lines. The results showed that Pd oxidation by oxygen plasma was different from Pd oxidation in pure O_2 at high temperature. SEM shows well-structured submicron PdO particles result from oxidation in pure O_2, whereas plasma oxidation results in the predominant formation of two-dimensional PdO structures covering the initial crystallites of the Pd foil. Further oxidation to a three-dimensional PdO phase occurs under prolonged treatment with oxygen plasma. The formation of a PdO_x ($x > 1$) species, characterized by a $E_b(Pd3d_{5/2})$ = 338.0–338.2 eV value that is close to the Pd^{4+} oxidation state, was also observed. This PdO_x species was found to have low thermal stability ($T < 400$ K). It is proposed that the PdO_x species can be localized within the boundaries of crystallites.

INVESTIGATION OF PALLADIUM INTERACTION WITH CERIUM OXIDE AND ITS STATE IN CATALYSTS FOR LOW-TEMPERATURE CO OXIDATION

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Catal. Today,
144(3-4) (2009) pp. 201-211.

Palladium catalysts supported on nanosized CeO_2 supports were synthesized by different methods. The catalysts showed high low-temperature activity (LTA) in CO oxidation. The synthesized palladium–ceria catalysts for low-temperature CO oxidation were investigated by a complex of physicochemical methods, and their catalytic performance was determined in the light-off regime. It was shown using high-resolution transmission electron microscopy (HRTEM) and EDX microanalysis that the catalysts with high LTA are characterized by exceptionally high dispersity of palladium on the surface of the supports. Two different states of palladium were observed by XPS. They correspond to the surface interaction phases (SIPs) as $Pd_xCeO_{2-\delta}$ and small metal clusters (<10 Å). According to diffraction images obtained by HRTEM, the latter have flattened shape due to epitaxial binding between (1 1 1) facets of palladium and CeO_2. Two types of CO adsorption sites (Pd^{2+} and Pd^0) were distinguished by FTIR. They can be attributed to SIP (Pd^{2+}) and palladium in flat metal clusters ($Pd^{\delta+}$ and Pd^0). The drop of LTA in CO oxidation is related to the loss of the palladium chemical interaction with the surface of the support and palladium sintering to form PdO nanoparticles. The formation of PdO particles is stimulated by crystallization of CeO_2 particle surface due to the calcination of support at temperatures above 600°C. The XPS, HRTEM and FTIR data give reliable evidence that PdO particles are not responsible for LTA in CO oxidation.

In this work, the structure of the active sites consisting of two phases: atomically dispersed palladium within the SIP and palladium metal nanoclusters is proposed. The catalyst pretreatment in hydrogen was found to improve significantly its catalytic (LTA) properties. The effect of the hydrogen pretreatment was supposed to be related to the formation of hydroxyl groups and their effect on the electronic and geometrical state of the surface active sites and their possible direct participation in CO oxidation.

STRUCTURE OF Ir-Al_2O_3 COATINGS OBTAINED BY CHEMICAL VAPOR DEPOSITION IN THE HYDROGEN ATMOSPHERE

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J. Struct. Chem.,
50(5) (2009) pp. 955-959.

Chemical vapor deposition is used to obtain Ir and Ir-Al_2O_3 coatings with a thickness of up to 5 μm and growth rate of 2.5 μm/h on steel substrates previously covered with an alumina layer. *Tris*-acetylacetonates of Ir(III) and Al(III) are used as precursors. The deposition process is carried out at the atmospheric pressure in the presence of hydrogen. The coatings obtained are studied by X-ray diffraction, X-ray photoelectron spectroscopy, and scanning electron microscopy. A dependence of the structure and composition of coatings on their preparation conditions is found.

IN SITU FTIR STUDY OF THE FORMATION AND CONSUMPTION ROUTES OF NITROORGANIC COMPLEXES - INTERMEDIATES IN SELECTIVE CATALYTIC REDUCTION OF NITROGEN OXIDES BY PROPENE OVER ZIRCONIA-BASED CATALYSTS

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Catal. Today,
145(1-2) (2009) pp. 152-162.

The results of this study show that the adsorption and catalytic properties of bulk ZrO_2 and nanoscale ZrO_2 pillars in pillared clay are significantly different. The NO + O_2 interaction with the surface of bulk zirconia results in the formation of three types of surface nitrate complexes (bridging, bidentate and monodentate), while over ZrO_2 pillars monodentate nitrates are not revealed.

The interaction of the propene-oxygen mixture with the surface of bulk zirconia leads to the formation of the acetate complexes only. Meanwhile, the main activation form of propene over ZrO_2 pillars is the isopropoxide complex; the acetate complexes are observed only in small amounts at elevated temperatures. Complexes of three types are formed on the surface of Pt,Cu/ZrPILC after interaction with the propene-oxygen mixture flow: isopropoxide complexes, coordination-bound acetone, and acetates. Modification of ZrO_2 pillars by Pt and Cu results in a significant change in the concentration and the temperature range of existence of C-H-O surface compounds.

The difference in the forms of reagents activation results in different structures of nitroorganic complexes. Over bulk zirconia the acetate and nitrate complexes form surface complexes with the structure similar to that of adsorbed nitromethane, monodentate nitrate being the most reactive species. Under reaction conditions at relatively low temperatures, over both modified and unmodified ZrO_2 pillars, interaction of isopropoxide and nitrate complexes produces surface complexes structurally similar to adsorbed dinitropropane.

IR spectral kinetic studies revealed that the rates of surface nitrate and nitroorganic complexes transformation are in general close to each other and to the rate of catalytic reaction, significantly increasing due to Pt + Cu supporting on pillars. This demonstrates that these complexes are real intermediates of NO_x HC-SCR over studied catalysts.

THE STATE OF ABSORBED HYDROGEN IN THE STRUCTURE OF REDUCED COPPER CHROMITE FROM THE VIBRATION SPECTRA

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Phys. Chem. Chem. Phys.,
11(29) (2009) pp. 6090-6097.

The reduction of copper chromite, $CuCr_2O_4$, is followed by means of thermogravimetric analysis. The reduced state is studied by means of FTIR spectroscopy, Raman spectroscopy and inelastic neutron scattering. The reduction of copper occurs in two stages: absorption of hydrogen at 250-400°C and dehydration of the reduced state at above 450°C. The measured vibrational spectra prove that a considerable amount of hydrogen is absorbed by the oxide structure with absorbed protons stabilized in OH and HOH-groups (geminal protons). Three groups of vibration bands are observed in the INS spectra, which can be assigned to stretching, bending and libration vibrations. An increase in the reduction temperature of copper chromite results in softening of the stretching and hardening of the bending vibrations, what can be related to the strengthening of hydrogen bonding.

CATHODIC ELECTROCRYSTALLIZATION AND ELECTROCHROMIC PROPERTIES OF DOPED RECHARGEABLE OXOTUNGSTATES

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Electrochim. Acta,
54(23) (2009) pp. 5439-5448.

The technique of preparing oxotungstate thin films by cathodic electrocrystallization from metastable acidic solutions to fabricate vanadium- and molybdenum-doped films on platinum and fluorine-doped tin oxide (FTO) is extended. By means of cyclic voltammetry, it is demonstrated a wider potential interval of highly reversible redox activity for these doped films, as compared to purely tungstate films. Electrochromic behavior in this region is dependent on the nature of the doping element. The microstructure of these films is characterized using scanning tunneling and atomic force microscopy. Raman and UV–vis spectroscopy is also applied to characterize the acidic deposition solutions. In tungstate–vanadate bath, it is observed evidence of mixed isopolyanions. The authors also found some features of less stable mixed anions in the tungstate–molybdate bath, characterized phase composition of these films by means of XRD, and explain the results in the terms of parallel deposition from coexisting molecular precursors.

THE EFFECTS OF TABLETING WITH POTASSIUM BROMIDE ON THE INFRARED ABSORPTION SPECTRA OF INDOMETHACIN

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Pharm. Chem. J.,
43(1) (2009) pp. 68-70.

The effects of tableting with KBr on the IR absorption spectra of molecular crystals were studied at room temperature. Study substances were mechanically activated mixtures of crystalline indomethacin and the excipient polyethylene glycol at ratios of 1:1 and 1:3. There was a marked widening of absorption bands in the range 2000-500 cm^{-1} in the IR spectra of mechanically activated mixtures in tablets with KBr. These experimental results are assessed within the framework of possible interactions between study samples and the KBr matrix during tableting and storage as powered mixtures.

PHASE TRANSITION AT 204-250 K IN THE CRYSTALS OF β-ALANINE: KINETICALLY IRREPRODUCEABLE, OR AN ARTEFACT?

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Phase Transitions,
82(7) (2009) pp. 497-506.

The crystals and the powder samples of β-alanine were studied in the wide temperature range by adiabatic calorimetry, differential scanning calorimetry, IR- and Raman spectroscopy and X-ray diffraction. No phase transitions could be observed. A small anomaly observed at about 256 K in the $Cp(T)$ dependences measured for the samples recrystallized from water was shown to be due to the presence of a small (about 0.14% of the total sample mass) amount of solvent inclusions. This anomaly was not observed in the solvent-free samples, either powders or single crystals.

A CONFORMATIONAL POLYMORPHIC TRANSITION IN THE HIGH-TEMPERATURE ε-FORM OF CHLORPROPAMIDE ON COOLING: A NEW ε'-FORM

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Acta Crystallogr., Sect. B: Struct. Sci.,
65(6) (2009) pp. 770-781.

Structural changes in the high-temperature ε-polymorph of chlorpropamide, 4-chloro-*N*-(propylaminocarbonyl)benzenesulfonamide, $C_{10}H_{13}ClN_2O_3S$, on cooling down to 100 K and on reverse heating were followed by single-crystal X-ray diffraction. At temperatures below 200 K the phase transition into a new polymorph (termed the ε'-form) has been observed for the first time. The polymorphic transition preserves the space group $Pna2_1$, is reversible and is accompanied by discontinuous changes in the cell volume and parameters, resulting from changes in molecular conformation. As shown by IR spectroscopy and X-ray powder diffraction, the phase transition in a powder sample is inhomogeneous throughout the bulk, and the two phases co-exist in a wide temperature range. The cell parameters and the molecular conformation in the new polymorph are close to those in the previously known α-polymorph, but the packing of the z-shaped molecular ribbons linked by hydrogen bonds inherits that of the ε-form and is different from the packing in the α-polymorph. A structural study of the α-polymorph in the same temperature range has revealed no phase transitions.

TEMPERATURE EFFECTS ON THE IR SPECTRA OF CRYSTALLINE AMINO ACIDS, DIPEPTIDES, AND POLYAMINO ACIDS.
V. L-SERYLGLYCIN

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J. Struct. Chem.,
50(6) (2009) pp. 1059-1065.

The IR spectra of L-serylglycine ($H_3^+N\text{-}CH(CH_2\text{-}OH)\text{-}CO\text{-}NH\text{-}CH_2\text{-}COO^-$), recorded at 413–93 K, have been studied, and the observed frequencies were assigned. Based on the results, we concluded that the stability of hydrogen bonds in the structure changed as a result of variation of temperature. The conclusions were compared with the results of IR spectroscopic studies of the crystals of α-glycylglycine, DL-serine, and glycine under the similar conditions.

IR SPECTROSCOPIC STUDY OF THE BASICITY OF AMINATED SILICA GELS

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Russ. J. Inorg. Chem.,
54(11) (2009) pp. 1798-1803.

The aminated silica gels $SiO_2/SOCl_2/NH_3$ (I), $SiO_2/SiCl_4/NH_3$ (II), $SiO_2/BCl_3/NH_3$ (III), and SiO_2/γ-aminopropyltriethoxysilane (SiO_2/APTES, IV) have been synthesized. According to DRIFT spectroscopy and chemical analysis data, the surface amino groups of I–III are “free,” while those of IV interact with the surface OH groups of the silica gel and with one another. The strength of basic sites has been measured on the proton affinity (PA) scale as the shift of the ν (CD) band of adsorbed deuterochloroform. The basicity of an aminated silica gel depends on its chemical composition. Silica gel IV (PA = 938 kJ/mol) is a stronger base than I–III (PA = 829 kJ/mol). As the basicity of the NH_2 group decreases, the N–H stretching band shifts to higher frequencies.

ADSORPTION SITES OF AN IRON-ALUMINUM CATALYST FOR AMMONIA OXIDATION AS STUDIED BY THE IR SPECTROSCOPY OF THE ADSORBED NO PROBE MOLECULE

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Kinet. Catal.,
50(2) (2009) pp. 264-269.

The state of surface adsorption sites in the IC-42-1 oxide catalyst for ammonia oxidation depending on catalyst preparation conditions (the nature of raw materials and the temperature of calcination) was studied in this work with the use of the diffuse reflectance IR spectroscopy of the adsorbed NO probe molecule. Hematite, which was prepared by a sulfate or chloride technology, was used as the starting raw material; Al_2O_3 binding agents were prepared by the reprecipitation or hydration of thermally activated gibbsite; and acetic or nitric acid was used as an electrolyte. The samples were calcined at 900–1000°C. It was found that mono- and dinitrosyl complexes with reduced coordinatively unsaturated Fe^{2+} cations and nitrite-nitrate complexes were formed upon the adsorption of NO on the catalyst surface (regardless of the catalyst preparation conditions). The samples differed in the amount and degree of coordinative unsaturation of adsorption sites depending on the preparation conditions. It was concluded that the most coordinatively unsaturated Fe^{2+} adsorption sites observed were formed on the surface of a solid solution of iron cations in aluminum oxide, which was formed in the course of catalyst preparation. It was found that an increase in the catalyst calcination temperature resulted in a decrease in the number of coordinatively unsaturated adsorption sites, which correlated with the observed decrease in the yield of NO. This correlation had the shape of a saturation curve, which can reflect the occurrence of a reaction in the diffusion mode at high degrees of conversion for the majority of catalysts.

STRUCTURE AND COMPOSITION OF ALUMINUM OXIDE FILMS IN CONTACT WITH THE LIQUID In–Ga EUTECTIC

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Inorg. Mater.,
45(12) (2009) pp. 1346-1350.

The oxide layers on the surface of aluminum metal and the aluminum alloys AD-1 and A-5 have been studied by attenuated total reflection infrared spectroscopy. The Al-O and Al-OH surface vibrational modes and A-O modes of the AlO_4 and AlO_6 groups have been identified. The structural inhomogeneity of the surface oxide layers is shown to contribute to their disruption when the sample is brought into contact with the liquid In-Ga eutectic. *In situ* scanning electron microscopy and X-ray microanalysis have been used to follow the dynamics of oxide layer disruption and the morphological and compositional changes in the eutectic alloy and the surface layer of aluminum.

RUTHENIUM-CARBON NANOCOMPOSITE

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J. Struct. Chem.,
50(2) (2009) pp. 268-272.

TEM, XRPA, and EXAFS studies showed that pyrolysis of $[Ru(dipy)_3](Cl)_2$ formed planar, two-dimensional, nanoparticles of ruthenium metal, stabilized in the carbon matrix.

STRUCTURAL DETERMINATION OF PALLADOUS OXIDE–CERIA NANOSYSTEM SUPPORTED ON γ-ALUMINA

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Nucl. Instrum. Methods Phys. Res., Sect. A,
603(1-2) (2009) pp. 178-181.

Recently great efforts are being devoted to develop new methods of preparation of high-disperse Pd–CeO_2 containing nanosystems stabilized on an oxide matrix. A new approach of synthesis consists in using the heterometallic $Pd^{II}_2Ce^{IV}_2(\mu\text{-OOCMe})_{12}(H_2O)_2$ complex as a precursor to anchor Pd nanoparticles on the surface of γ-alumina in direct contact with CeO_2. The present work is devoted to a structural study of this disperse Pd–CeO_2 containing nanosystem after oxidative or reductive pretreatments in comparison with monometallic alumina-supported samples by XAFS and TEM. A strong interaction between Pd and ceria in the catalyst produced in the studied system affects reducibility of both PdO and CeO_2, which in turn results in an increased low-temperature activity in CO oxidation along with a dramatic change of the ignition–extinction curve.

XAFS STUDY OF Pt/Al_2O_3 NANOSYSTEM WITH METAL-OXIDE ACTIVE COMPONENT

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Nucl. Instrum. Methods Phys. Res., Sect. A,
603(1-2) (2009) pp. 108-110.

A study of monodisperse $Pt/\gamma\text{-}Al_2O_3$ catalysts was carried by XAFS. Key factors of the particle size control were the composition of the precursor solutions and the pretreatment of the carrier. XAFS revealed that variation of preparation methods caused formation of three types of Pt particles (metal, oxide, metal-oxide) located on the $\gamma\text{-}Al_2O_3$ surface directly affecting the catalytic activity in CH_4 complete oxidation. A method of reliable estimation of the phase composition of the active component is developed taking into account the nano-size effects and Pt oxidation state.

UNIQUE STABILITY OF μ-HYDROXO LIGANDS IN Pt(IV) COMPLEXES TOWARDS ALKALINE HYDROLYSIS

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Nucl. Instrum. Methods Phys. Res., Sect. A,
603(1-2) (2009) pp. 182-184.

Pt(IV)-nitrate solutions are widely used as precursors for mobile and stationary emission control catalysts for being chlorine-free but are unfit for the preparation of highly dispersed catalysts due to hydrolysis taking place after dilution and/or interaction with a basic carrier surface. The alkalization of the commercial Pt(IV)-nitrate solutions was found not to lead to light-yellow mononuclear $[Pt(OH)_6]^{2-}$ solution because the bridged OH ligands stabilizing oligomeric Pt nitrate complexes are not cleaved even after refluxing with excess of alkali. The alkalization results in dilution-resistant polynuclear precursor solutions fit for highly dispersed Pt/Al_2O_3 catalyst preparation.

PLATINUM NANOPARTICLES ON Al_2O_3: CORRELATION BETWEEN THE PARTICLE SIZE AND ACTIVITY IN TOTAL METHANE OXIDATION

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J. Catal.,
268(1) (2009) pp. 60-67.

Catalytic activity of the size-controlled platinum nanoparticles supported on the acid-pretreated γ-alumina has been tested in complete methane oxidation under lean conditions. The mean sizes of platinum particles varied from 1.3 to 10 nm with the narrow size distribution (TEM data). It has been found that the reaction under study is strongly size sensitive. The size dependence of the specific catalytic activity is narrow and bell-shaped, with the maximum TOF value observed for the catalysts containing partially oxidized platinum with the mean particle sizes of about 2 nm. The observed strong size sensitivity is shown to originate from the size dependence of the apparent activation energy of the methane oxidation and/or the platinum oxidation state in the catalytically active nanoparticles.

XAFS STUDY OF A Au/Al_2O_3 CATALYTIC NANOSYSTEM DOPED BY Ce AND Ce–Zr OXIDES

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Nucl. Instrum. Methods Phys. Res., Sect. A,
603(1-2) (2009) pp. 185-187.

This work is devoted to a structural study of the finely dispersed nanosized gold species supported on nanosized mixed oxides prepared by the sol–gel method. An analysis of the XANES (Au-L_3) spectra has revealed mainly Au^{3+} cations located on the oxide matrix surface in distorted octahedral coordination for the catalysts reduced at 150°C. For the catalysts reduced at 300°C, a highly distorted metallic gold species has been mainly found, within the method limitation. The synthesized catalysts have also been characterized by transmission electron microscopy (TEM), BET, X-ray diffraction (XRD) and XPS.

STRUCTURAL STUDY OF $CuCr_{1-x}V_xS_2$ DISULFIDE

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Nucl. Instrum. Methods Phys. Res., Sect. A,
603(1-2) (2009) pp. 188-190.

XAFS was first used to study the changes in the local structure and oxidation state of metal ions in $CuCr_{1-x}V_xS_2$ ($0 \le x < 0.25$). XANES showed identical monovalent state of $Cu^{1+}$and Cr^{3+} ions in this family. EXAFS revealed local structure distortions of Cr and Cu as compared to mother $CuCrS_2$ provided the V content was minor for Cr and more pronounced for Cu. Analysis of the XAFS data showed that the V ions are partly inserted into unfilled sites of the Cr sublattice, but they are mainly located in non-regular sites, saving slightly the distorted octahedral sulfur nearest surrounding. The vacancies in CrS_2 slabs, the V distribution and the lattice reduction are considered as factors that can modify the magnetic state of the samples.

PHASE TRANSFORMATIONS OF $CuCrS_2$: STRUCTURAL AND CHEMICAL STUDY

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J. Struct. Chem.,
50(2) (2009) pp. 288-295.

The structure and composition of the $CuCrS_2$ powder synthesized by sulfidation of a mixture of oxides $Cu_2O:Cr_2O_3 = 1:1$ at 850°C and cooled to room temperature at a rate of 60°C/min were studied by X-ray powder diffraction and differentiating solution. A rhombohedral $CuCrS_2$ phase (space group $R3m$) was found, which was stoichiometric in composition and had disordering in the copper sublattice because copper was arranged at the tetrahedral and octahedral sites with occupancy 10% at the latter. The structure of $CuCrS_2$, in which the octahedra were occupied by copper atoms at room temperature, was found for the first time; in known structures, the copper atoms occupied only the tetrahedral sites, while the probability of octahedral occupation appeared around 400°C (order-disorder transition). The partially disordered $CuCrS_2$ phase is intermediate on the route to complete ordering. The quickly cooled $CuCrS_2$ powder is unstable; after the second heating to 500°C with prolonged annealing at 390°C→180°C→80°C→25°C, its transition to the stable state was accompanied by liberation of 2–4 wt.% Cu_9S_5. The real composition of ternary sulfide after isolation of the Cu_9S_5 phase is discussed using the data of the structural method, differential dissolution, and magnetic measurements.

REVISED PHASE DIAGRAM OF Li_2MoO_4–$ZnMoO_4$ SYSTEM, CRYSTAL STRUCTURE AND CRYSTAL GROWTH OF LITHIUM ZINC MOLYBDATE

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J. Solid State Chem.,
182(7) (2009) pp. 1935-1943.

Orthorhombic lithium zinc molybdate was first chosen and explored as a candidate for double beta decay experiments with ^{100}Mo. The phase equilibria in the system Li_2MoO_4–$ZnMoO_4$ were reinvestigated, the intermediate compound $Li_2Zn_2(MoO_4)_3$ of the

α-$Cu_3Fe_4(VO_4)_6$ (lyonsite) type was found to be nonstoichiometric: $Li_{2-2x}Zn_{2+x}(MoO_4)_3$ ($0 \le x \le 0.28$) at 600°C. The eutectic point corresponds to 650°C and 23 mol% $ZnMoO_4$, the peritectic point is at 885°C and 67 mol% $ZnMoO_4$. Single crystals of the compound were prepared by spontaneous crystallization from the melts and fluxes. In the structures of four $Li_{2-2x}Zn_{2+x}(MoO_4)_3$ crystals (x=0; 0.03; 0.21; 0.23), the cationic sites in the face-shared octahedral columns were found to be partially filled and responsible for the compound nonstoichiometry. It was first showed that with increasing the x value and the number of vacancies in $M3$ site, the average $M3$–O distance grows and the lithium content in this site decreases almost linearly. Using the low-thermal-gradient Czochralski technique, optically homogeneous large crystals of lithium zinc molybdate were grown and their optical, luminescent and scintillating properties were explored.

INTERACTION OF VANADIUM CONTAINING CATALYSTS WITH MICROWAVES AND THEIR ACTIVATION IN OXIDATIVE DEHYDROGENATION OF ETHANE

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Catal. Today,
141(3-4) (2009) pp. 300-305.

Microwave (MW)-activated oxidative dehydrogenation of ethane is studied using kinetic approach. It consists in the comparison of kinetic dependencies (shape of kinetic equations, “selectivity or yield vs. conversion” curves) and apparent parameters (activation energies) obtained in thermal and MW modes. In the case of VMo and VMoNb oxides a distinct difference between ethane yields was observed at given conversion of limiting reactant (oxygen). It was proven by X-ray diffraction that MW activation changes the catalyst microstructure forming phase distribution different from that formed under a conventional heating and thus changing catalytic behavior of VMo and VMoNb oxides.

THE STATE OF THE COMPONENTS IN COPPER–CERIUM CATALYSTS SUPPORTED ON DIFFERENT OXIDES

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Nucl. Instrum. Methods Phys. Res., Sect. A,
603(1-2) (2009) pp. 191-193.

The phase composition and the state of the active components in the catalysts used for preferential oxidation (PROX) of CO in hydrogen-containing mixtures are considered. Cu–Ce catalysts supported on different oxides (ZrO_2, TiO_2, Al_2O_3, MnO_2) before and after PROX reaction are characterized.

DNA, OLIGOSACCHARIDES, AND MONONUCLEOTIDES STIMULATE OLIGOMERIZATION OF HUMAN LACTOFERRIN

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J. Mol. Recognit.,
22(4) (2009) pp. 330-342.

Using small-angle X-ray scattering (SAXS), light scattering (LS), and soft laser ablation it has been shown that lactoferrin (LF) in solution at neutral pH is oligomerized in the absence of salt or at physiological salt concentrations.

DNA AND OLIGOSACCHARIDES STIMULATE OLIGOMERIZATION OF HUMAN MILK LACTOFERRIN

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Mol. Biol.,
43(1) (2009) pp. 142-149.

Lactoferrin (LF) is a Fe^{3+}-transferring glycoprotein and is contained in human barrier fluids, blood, and milk. LF is an acute phase protein, is involved in nonspecific defense, and displays a unique set of biological functions. Small-angle X-ray scattering and light scattering experiments demonstrated that DNA and oligosaccharides added to LF with various levels of initial oligomerization increased the oligomerization rate. Almost complete dissociation into monomers was observed when 1 M NaCl was added to LF oligomers obtained in the presence of DNA, oligosaccharides, and nucleotides, previously identified as oligomerization effectors. LF complexes obtained with different oligomerization effectors differed in stability.

FORMATION OF WO_3 FROM VARIOUS PRECURSORS

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J. Surf. Invest. X-Ray, Synchrotron Neutron Techn., 3(6) (2009) pp. 869-875.

A comparative study of tungsten oxide formation from various precursors has been carried out using a complex of physicochemical methods. Crystallization of WO_3 has been shown to pass through metastable states in all cases. For electrodeposited oxotungstate films, the structure of tungsten oxides depends on electrodeposition conditions and adsorption properties of substrate metal with respect to hydrogen; at temperatures below 600°C the stoichiometric composition of WO_3 is not attained.

$LiMn_2O_4$ AND $LiCoO_2$ COMPOSITE CATHODE MATERIALS OBTAINED BY MECHANICAL ACTIVATION

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Russ. J. Electrochem., 45(3) (2009) pp. 277-285.

New composite cathode materials xLiMn$_2$O$_4$/(1−x) LiCoO$_2$ (x = 0.7, 0.6, 0.5 и 0.4) were obtained by mechanical activation. According to scanning electron microscopy data, the process was accompanied by pronounced dispersion and fine mixing of the initial components. In the course of the preparation and electrochemical cycling of the composites, $LiMn_2O_4$ and $LiCoO_2$ partially reacted, leading to the replacement of manganese with cobalt in the structure of spinel, which was detected by powder X-ray diffraction (XRD), IR and X-ray photoelectron spectroscopy (XPS), and cyclic chronopotentiometry. The specific discharge capacity of composites was <100 mAh/g.

"CORE-SHELL" CATHODE MATERIALS FOR LITHIUM-ION BATTERIES PREPARED VIA MECHANOCHEMICAL ROUTE

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ECS Trans., 16(42) (2009) pp. 21-29.

Solid state mechanical activation method was applied for surface modification of $LiCoO_2$ by simple oxides (Al_2O_3, TiO_2, MgO and $Li_2O\bullet 2B_2O_3$) and of $LiMn_2O_4$ by $LiMO_2$ (M=Co, Ni). Pristine materials were ground with correspondent precursors and then heat treated at different temperatures. As prepared materials were studied by XRD, ^{6,7}Li and ^{27}Al MAS NMR spectroscopy, XPS, SEM and electrochemical tests. It has been shown that electrochemical properties of coated cathodes strongly depend on the composition and the thickness of coating layer.

BRØNSTED ACIDITY STUDY OF FIBERGLASS MATERIALS BY H/D-EXCHANGE

T.S. Glazneva, E.M. Sadovskaya, A.P. Suknev, V.B. Goncharov, L.G. Simonova, E.A. Paukshtis, B.S. Bal'zhinimaev

Appl. Catal. A, 366(2) (2009) pp. 262-268.

The kinetics of hydrogen isotopic exchange over fiberglass materials and reference samples (SiO_2, H_3PO_4/SiO_2 and HZSM-5) was studied at 500^{o}C under the dynamic adsorption-desorption equilibrium. The concentrations and H/D-exchange rates of different Brønsted acid sites (BAS) were determined using the numerical modeling of isotope exchange dynamics. The correlation between H/D-exchange rate of reference samples and their acidity determined by FTIR spectroscopy of adsorbed CO was established. Based on this correlation, the strength of BAS in fiberglass materials was shown to be comparable to that of HZSM-5 zeolite.

HfO_2-HIGH-k DIELECTRIC FOR NANOELECTRONICS

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ECS Trans.,
25(8) (2009) pp. 875-880.

Thermodynamic consideration of Hf-HfO_2-SiO_2-Si system, cross-sectional HR-TEM, XPS, IR-spectroscopy and Null ellipsometry technique were used to determine the chemical composition and structure of the HfO_2 thin films deposited on Si. The interface layer consists of the hafnium silicate with smoothly varying chemical composition along the film thickness. Formation of the IL occurs as during the HfO_2 film deposition so at annealing of the $HfO_2/SiO_2/Si$ structure. Kinetics of the hafnium silicate formation was studied by IR-spectroscopy.

Fe- AND Al-DOPED APATITE-TYPE LANTHANUM SILICATES: STRUCTURE AND PROPERTY CHARACTERIZATION

T.S. Kharlamova, S.N. Pavlova, V.A. Sadykov, T.A. Krieger, L.Ch. Batuev, V.S. Muzykantov, N.F. Uvarov*, Chr. Argirusis** (**Institute of Solid State Chemistry and Mechanochemistry, Novosibirsk, Russia; **Clausthal University of Technology, Germany*)

Solid State Ionics,
180(11-13)(2009) pp. 796-799.

For the first time the local structure of Al- and Fe-doped apatite-type lanthanum silicates (ATLS) of different stoichiometries was systematically studied by using IR spectroscopy. In addition to previous ^{29}Si MAS NMR study, data obtained confirm the possibility of [SiO_4] group interaction in ATLS, being favored by the presence of cation vacancies in the apatite. Transport properties of the systems were studied by impedance spectroscopy and oxygen isotope exchange technique. The latter indirectly shows that not only channel/interstitial oxygen atoms can be involved in the oxide-ion migration but also those of tetrahedrons.

THE EFFECT OF A MAGNETIC FIELD ON THE THERMAL DESTRUCTION OF COBALT FORMATE

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Russ. J. Phys. Chem.,
83(3) (2009) pp. 499-502.

The thermal decomposition of cobalt formate in a flow of an inert gas led to the formation of cobalt nanoparticles in pores of various substrates (silica gel, alumina, activated carbon, and montmorillonite). Electron microscopic studies showed that the particle-size distribution of cobalt depended on the external magnetic field strength; the average particle size and distribution variance decreased as the field strength increased. It was assumed that the external magnetic field affected the nucleation constant of cobalt nanoparticles.

NATURAL GAS CONVERSION ON ZSM-5 ZEOLITES MODIFIED WITH ZIRCONIUM AND MOLYBDENUM NANOPOWDERS

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Pet. Chem., 49(1) (2009) pp. 47-52.

The joint promoting action of zirconium and molybdenum nanosized powders on the catalytic properties of ZSM-5 zeolite with different silica ratios in the conversion of natural gas into liquid hydrocarbons was studied. By means of thermal analysis and high-resolution transmission electron microscopy, data on the amount and nature of the coke deposit produced during the reaction on the surface of the Zr-Mo/ZSM-5 catalyst were obtained. It was shown that the addition of zirconium to the molybdenum-containing catalyst leads to enhancement of the catalyst activity and selectivity in the formation of aromatic hydrocarbons from natural gas components. It was found that the maximal amount of aromatic compounds is formed in the presence of zeolite having a silica ratio of 40 and containing 0.5% Zr and 4.0% Mo.

EPR SPECTRA OF AEROSOL PARTICLES FORMED BY PYROLYSIS OF C_3H_8 plus Ar AND C_3H_8 plus $Fe(CO)_5$ plus Ar MIXTURES IN A FLOW REACTOR

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Appl. Magn. Reson,
35(4) (2009) pp. 625-637.

The paper is devoted to electron paramagnetic resonance investigation of nanoparticles and aggregates of nanoparticles formed by pyrolysis of propane as well as a mixture of propane and iron pentacarbonyl. The measurement showed that the pyrolysis of the C_3H_8 plus Ar mixture results in the formation of a carbonaceous phase (phase I), which is quite different from that formed by the C_3H_8 plus $Fe(CO)_5$ plus Ar mixture (phase II). In phase I there is a strong oxygen effect for as-prepared samples; 75% of spins are accessible to the environmental gas via the interconnected system of microvoids and microchannels. In phase II there was a weak oxygen effect for the as-prepared samples. However, after exposition of phase II to air for 160 h, the properties of phase II have become about the same as that of phase I. A strong oxygen effect was observed for the air-exposed phase II. The line width for phase II increases monotonically with the iron content in the sample. This increase is probably related to the dipole-dipole interactions between the radical centers and the iron atoms distributed throughout the carbon matrix.

INCREASE IN THE DIAMAGNETIC RESPONSE FROM LOW-DENSITY $Bi_{1.8}Pb_{0.3}Sr_{1.9}Ca_2Cu_3O_x$ HIGH-TEMPERATURE SUPERCONDUCTORS AND $Bi_{1.8}Pb_{0.3}Sr_{1.9}Ca_2Cu_3O_x$+Ag COMPOSITES

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Techn. Phys.,
54(8) (2009) pp. 1130-1134.

Low-density polycrystalline $i_{1.8}Pb_{0.3}Sr_{1.9}Ca_2Cu_3O_x$ high-temperature superconductors with a foamlike microstructure and composites consisting of this superconductor and silver in an amount of 20, 25, and 30 vol % are synthesized. The microstructure, as well as the temperature and field dependences of the magnetization, $M(T)$ and $M(H)$, are studied. It is found that, in $Bi_{1.8}Pb_{0.3}Sr_{1.9}Ca_2Cu_3O_x$ high-temperature superconductors and $Bi_{1.8}Pb_{0.3}Sr_{1.9}Ca_2Cu_3O_x$ + Ag composites, the diamagnetic response is enhanced and the screening properties are improved compared with high-temperature polycrystalline superconductors with the same composition that are prepared by the standard technology. The observed effect is explained by the features of magnetic flux penetration into a porous medium.

FORMATION OF GOLD AND GOLD SULFIDE NANOPARTICLES AND MESOSCALE INTERMEDIATE STRUCTURES IN THE REACTIONS OF AQUEOUS $HAuCl_4$ WITH SULFIDE AND CITRATE IONS

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Phys. Chem. Chem. Phys.,
11(26) (2009) pp. 5445-5454.

The effects of the molar ratio of sodium sulfide to chloroauric acid in the range of 0.5 to 5 and the time factor on the formation of the nanoparticles (NPs) of metallic Au, Au_2S or their mixtures have been studied applying *in situ* and *ex situ* techniques (UV-Vis absorption spectroscopy, potentiometry, TEM, SPM, SERS, XPS). The products and intermediates have been compared with those for the reduction of chloroaurate with citrate ions and combinations of citrate and sulfide ions.

PREPARATION OF COPPER AND SILICON/COPPER POWDERS BY A GAS EVAPORATION-CONDENSATION METHOD

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Bull. Mater. Sci. ©Indian Academy of Sci.,
32(5) (2009) pp. 543-547.

Pure and silicon-coated metal copper nano to submicron-sized powders were prepared by gas evaporation and condensation. This powder was synthesized by using an industrial electron accelerator, ELV-6, with Ar as the carrier gas. Vapour from the liquefied metal surface was transferred to the cold zone by the carrier gas and precipitated as spherical Cu metal and Si/Cu composite powders. The mean diameter of the resulting powder was 100–200 nm.

STRUCTURAL AND MAGNETIC ORDERING IN NANOPARTICLES OF COMPLEX 3*d*- AND 4*f*-ELEMENT OXIDES IN GERMANATE GLASS

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Russ. Chem. Bull.,
73(7) (2009) pp. 968-971.

Germanate glasses doped simultaneously with Fe and rare earth elements Gd, Tb, Dy, and Ho in concentrations of several percent have been investigated. It is shown that the phase state of paramagnetic elements changes from isolated ions and small clusters in the initial glass to crystalline magnetic nanoparticles in the samples subjected to heat treatments. The field dependences of the magnetization of such samples are characterized by magnetic saturation in fields of about 1 kOe and large Faraday rotation in the near IR region (up to 10 deg/cm in the range from 700 to 800 nm).

INTERACTION BETWEEN OXYGEN AND POLYCRYSTALLINE PALLADIUM AT O_2 PRESSURES OF 10^{-6}–10 Pa

A.N. Salanov, E.A. Suprun

Kinet. Catal.,
50(1) (2009) pp. 31-39.

The interaction between oxygen and polycrystalline palladium (Pd(poly)) at $P_{O2} = 2.6 \times 10^{-6}$–10 Pa and T = 300–1300 K was studied by the thermal desorption (TD) method. The interaction between O_2 and Pd(poly) is governed by the O_2 pressure and the sample temperature. At low pressures of P_{O2} ($\leq 1.3 \times 10^{-5}$ Pa), O_2 is chemisorbed dissociatively on the Pd(poly) surface. During chemisorption, the O_{ads}-surface bond energy and the O_2 sticking coefficient gradually decrease as the surface coverage θ increases. At $P_{O2} \geq 10^{-2}$ Pa and $T \leq 500$ K, after the saturation of the O_{ads} layer (θ ~ 0.5), O_{ads} atoms penetrate under the surface layer of the metal to form surface palladium oxide. At $P_{O2} \geq 1$ Pa and $T > 500$ K, after the saturation of the surface oxide film 2 ML in thickness (n ~ 2), O_{ads} atoms penetrate into the oxide film and then into the subsurface palladium layer and diffuse deep into the metal bulk. As a result, the oxygen uptake at 700 K is n ~ 50. Upon heating, the surface oxides decompose, desorbing O_2, which gives rise to a low-temperature TD peak with T_{max} = 715 K. The release of oxygen inserted in the subsurface layers of palladium shows itself as a distinct high-temperature TD peak with $T_{max} \geq 750$ K.

THE ROLE PLAYED BY AN IODINE-CONTAINING PROMOTER IN THE FORMATION OF ACTIVE POLYCRYSTALLINE SILVER CATALYST SURFACE

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Russ. J. Phys. Chem. A,
83(7) (2009) pp. 1088-1093.

The programmed temperature desorption method was used to study the interaction of oxygen with the surface of a polycrystalline silver catalyst promoted with iodine. Ethyl iodide almost did not interact with the unoxidized surface of silver. The adsorption of C_2H_5I on the oxidized catalyst surface resulted in the formation of two adsorbed iodine forms, silver iodide and iodine deeply dissolved in subsurface silver crystal lattice layers. The character of oxygen adsorption from the iodine-containing surface of the catalyst was determined by the amount and form of adsorbed iodine. In the presence of a iodine-containing promoter, the concentration of oxide-like oxygen sharply decreased, and the amount of strongly bound atomically adsorbed oxygen responsible for the selective transformation of ethylene glycol into glyoxal increased.

CHARACTERIZATION OF Rh/Al_2O_3 CATALYSTS AFTER CALCINATION AT HIGH TEMPERATURES UNDER OXIDIZING CONDITIONS BY LUMINESCENCE SPECTROSCOPY AND CATALYTIC HYDROGENOLYSIS

V.O. Stoyanovsky, A.A. Vedyagin, G.I. Aleshina, A.M. Volodin, A.S. Noskov

Appl. Catal., B, 90(1-2) (2009) pp. 141-146.

Rhodium/alumina catalysts with Rh loadings of 0.01–0.5 wt.% with substantially different thermal stability of supports with respect to phase transformation to corundum were studied. It was shown that the stability of alumina support with respect to its transformations to corundum is one of the important factors accounting for irreversible deactivation of Rh/Al_2O_3 catalysts. Laser-induced luminescence (LIL) of Cr^{3+} impurity and Rh^{3+} in α-Al_2O_3 and XRD was used for studying catalysts with different calcination temperatures. Catalytic ethane hydrogenolysis was used as a test for Rh loading on the catalyst surface. This method reliably detects Rh ions on the surface starting from concentrations as low as 0.01%. The results of LIL spectroscopy indicated that in all cases Rh^{3+} ions were incorporated in the bulk of the newly formed α-Al_2O_3 phase, and quite possible can initiates the formation of the corundum phase in catalysts with high-thermal stability.

LASER-INDUCED LUMINESCENCE ASSOCIATED WITH SURFACE HYDROXIDE GROUPS IN Al_2O_3

V.O. Stoyanovsky, V.N. Snytnikov

Kinet. Catal., 50(3) (2009) pp. 450-455.

Laser-induced luminescence of OH_s groups for undoped Al_2O_3 oxides of various phase compositions was excited by pulsed nitrogen laser radiation at 337.1 nm. The luminescence band at 500–650 nm assigned to hydroxide groups of Al_2O_3, actually, consists of several lines at 500–515, 553, 567, 577, 607, and 633 nm; these constituent bands can be assigned to various types of OH_s surface groups. In the low-temperature phases of the $\gamma\rightarrow\delta\rightarrow\theta$-$Al_2O_3$ series, excitation at a wavelength of 337.1 nm gave rise to a characteristic luminescence band associated with surface hydroxide groups of Al_2O_3 that appeared at 770 nm.

DETERMINATION OF THE TOLMAN LENGTH IN THE IMPROVED DERJAGUIN–BROEKHOFF–DE BOER THEORY FOR CAPILLARY CONDENSATION OF ETHANOL IN MESOPOROUS THIN FILMS BY ELLIPSOMETRIC POROSIMETRY

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Micropor. Mesopor. Mater.,
123(1-3) (2009) pp. 243-252.

Ethanol adsorption–desorption isotherms on well-organized mesoporous silica and titania films with hexagonal pores structure were studied by ellipsometric porosimetry. The mesopore volume was calculated from the change of the effective refractive index at the end points of the isotherm. An improved Derjaguin–Broekhoff–de Boer (IDBdB) model for cylindrical pores is proposed for the determination of the pore size. In this model, the disjoining pressure isotherms were obtained by measuring the thickness of the ethanol film on a non-porous film with the same chemical composition. This approach eliminates uncertainties related to the application of the statistical film thickness determined via *t*-plots in previous versions of the DBdB model. The deviation in the surface tension of ethanol in the mesopores from that of a flat interface was described by the Tolman parameter in the Gibbs–Tolman–Koening–Buff equation. A positive value of the Tolman parameter of 0.2 nm was found from the fitting of the desorption branch of the isotherms to the experimental data obtained by Low Angle X-ray Diffraction (LA-XRD) and Transmission Electron Microscopy (TEM) measurements in the range of pore diameters between 2.1 and 8.3 nm.

EXTRACTIVE COLORIMETRIC METHOD OF PALLADIUM DETERMINATION IN HOMOGENEOUS CATALYST (Pd + HETEROPOLY ACID)

V.F. Odyakov, E.G. Zhizhina

Factory Lab.,
75(8) (2009) pp. 22-24.

Presented is extractive-photometric rapid procedure of Pd (II) determination in Pd in homogeneous catalysts (Pd + Mo-V-phosphoric heteropolyacid. The interval of reliably determined concentrations of Pd (II) ranges from 0.011 to 1,06 mg/ml.

FARICH OPTIMIZATION FOR PRECISE VELOCITY MEASUREMENT

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Nucl. Instrum. Methods Phys. Res., Sect. A, 598(1) (2009) pp. 169-172.

Lately aerogel samples of refractive index as high as 1.13 were synthesized in Novosibirsk. Light scattering length for some samples approaches 12 cm.

A Geant4 simulation for a FARICH with aerogel of 1.10 index of refraction were performed. Two photodetector options were considered: Burle MCP PMT and a novel photodetector—Hamamatsu MPPC. It is shown that FARICH with MPPC photodetector is able to separate at 3σ level pions and kaons up to 8 GeV/*c*, muons and pions up to 1.5 GeV/*c* momentum.

The project of Forward RICH for SuperB factory based on focusing aerogel radiator is presented.

It is demonstrated that using a FARICH detector for the momentum measurement the momentum precision can be significantly improved.

HIGH DENSITY AEROGEL FOR ASHIPH SND - TEST RESULTS

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Nucl. Instrum. Methods Phys. Res., Sect. A, 598(1) (2009) pp. 163-165.

The paper is devoted to the cosmic tests of particle identification system for the SND detector at VEPP-2000. The goal of the tests is to measure main parameters of the system: average signal for the relativistic particles, nonuniformity of the light collection, and time resolution. The measurements of the average signal were done in five points of the counter. The obtained value of the signal is about 10 photoelectrons. The time resolution is about 2 ns. The time dependence of the average amplitude was measured also. It was shown that average amplitude of the signal decreases with characteristic time of about two years. The cause of the signal decrease is discussed.

DEVELOPMENT OF FARICH-DETECTOR FOR ALICE EXPERIMENT AT CERN

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Nucl. Instrum. Methods Phys. Res., Sect. A, 598(1) (2009) pp. 156-159.

In order to extend the momentum range of the charged particle identification up to 10 GeV/*c* for pion–kaon separation and to 14 GeV/*c* for kaon–proton separation of the ALICE experiment at LHC (CERN), the focusing aerogel ring imaging Cherenkov (FARICH) detector employing a multi-layer silica aerogel as radiator has been proposed. The design project of the FARICH Prototype is presented.

Results of the test of the avalanche photo-diodes with metal-resistance-semiconductor structure produced by the Centre of Perspective Technology and Apparatus (CPTA MRS APDs) are shown. Preliminary data of tests of the light-collecting capability for aluminum and stainless steel Winston cone holes of different types are presented.

The performance of the proposed detector was estimated using the GEANT4 simulation. Results of the optimization of characteristics of the aerogel radiators, CPTA MRS APDs with and without wavelength shifter paint are discussed.

THE EFFECT OF CHEMICAL MODIFICATION ON THE CHROMATOGRAPHIC PROPERTIES OF POLYMERIC ADSORBENTS POLYSORB-1 AND HISEP Q AND THE DIATOMITE CARRIER CHROMOSORB P NAW

E.Yu. Yakovleva, V.Yu. Belotserkovskaya, V.P. Fadeeva*, G.S. Litvak, E.A. Paukshtis, D.E. Babushkin (**Vorozhtsov Institute of Organic Chemistry, Novosibirsk, Russia*)

Russ. J. Phys. Chem. A, 83(5) (2009) pp. 849-853.

Polytrimethylsilylpropyne with the decomposition temperature 205°C was synthesized and characterized. The effect of chemical modification by polytrimethylsilylpropyne on the gas chromatographic properties of polymeric adsorbents and the diatomite carrier for the selective separation of C_1-C_{10} hydrocarbons, alcohols and aromatic and sulfur-containing compounds was studied.

SPECIFIC FEATURES OF THE DYNAMIC MODE OF DIFFERENTIAL DISSOLUTION AS OF A METHOD OF PHASE ANALYSIS

V.V. Malakhov

J. Anal. Chem.,
64(11) (2009) pp. 1097-1107.

The author considers the specific features of the separation properties of the stoichiographic method of differential dissolution (DD) and the role of thermodynamic and kinetic factors in the processes of the separation of solid mixtures. It is shown that the separation properties of DD should be characterized by selectivity and efficiency, as it is accepted in chromatography. The separation possibilities of DD are considered in characterizing uniformity, stoichiometry-nonstoichiometry of the elemental composition of phases, and also of their macro- and microstructures. The comparison of the principles and characteristics of DD, voltammetry, titrimetry, mass spectroscopy, and chromatography has led to the conclusion that the place of DD among the other methods is determined, primarily, by its unique property, namely, its standardless nature.

DETECTION, IDENTIFICATION, AND QUANTITATIVE DETERMINATION OF MINOR PHASES IN SOLID MULTIELEMENT MULTIPHASE COMPOUNDS AND MATERIALS

V.V. Malakhov, L.S. Dovlitova

Inorg. Mater.,
45(14) (2009) pp. 1571-1579.

The theoretical and experimental aspects that are connected with the use of the differential dissolution method (DD) for the identification and quantitative determination in complex matrices of the minor phases that occur in form of unintentional impurities, side products in the synthesis, or purposefully introduced additives whose concentration is from $n \times 10^{-3}$ to several percent were considered. The types of spatial distribution of the minor phases within the volume and surface of the matrices were classified. Mathematical simulation of DD processes for the systems with complex matrices was carried out. The conception of the selectivity and efficiency of the separation of minor and matrix phases was given. The factors that need to be considered during the analysis of the mixtures of crystalline and/or amorphous phases of constant and variable composition were classified. The concentration processes of the minor phases based on the selective and/or differential dissolution of the matrix phases of the compounds under investigation were studied. Examples of using DD method for the determination of the minor phases in catalysts, carriers, sorbents, various functional materials, archaeological finds, and other objects were given.

TRANSPORT PROPERTIES OF NAFION MEMBRANES MODIFIED WITH TETRAPROPYLAMMONIUM IONS FOR DIRECT METHANOL FUEL CELL APPLICATION

E.N. Gribov, I.M. Krivobokov,
E.V. Parkhomchuk, A.G. Okunev, G. Spoto*,
V.N. Parmon (**Department of Chemistry IFM, University of Turin, Torino, Italy*)

Russ. J. Electrochem.,
45(2) (2009) pp. 199-207.

This work presents a study of transport properties (proton conductivity, methanol permeability, and water uptake) and acid-base properties of commercial Nafion-112, -115, and -117 membranes modified with tetrapropylammonium (TPA) cations. In the interaction between TPA hydroxide and protons of sulfonate groups in the Nafion matrix, some of the protons are shown to be bound to sulfonate groups and do not participate in transport processes. These findings are confirmed by IR spectroscopy, acid-base titration, and data on proton conductivity of the modified membranes. Proton conductivity of the modified membranes is shown to be effectively described by a percolation model with parameters that agree with published data for commercial Nafion membranes. Based on these results, a model is proposed for the interaction of TPA cations with the sulfonate groups in Nafion membranes. According to this model, TPA cations form hydrophobic clusters in hydrophilic regions of the polymer matrix, thus preventing some of the protonated sulfonate groups from participating in transport processes.

Fundamental and Practical Application to Catalyst Preparation. The Effects of Preparation Methods on Catalyst Structure and Performance

CATALYSTS CONTAINING COMPOUNDS OF DEPLETED URANIUM

Z.R. Ismagilov, S.V. Kuntsevich

Chem. Sustain. Devel.,
17(5) (2009) pp. 445-469.

Features of the physicochemical properties of uranium oxide are considered in the review. Chemical processes that involve the catalysts containing the compounds of depleted uranium are considered. The data published within the years 1964 to 2009 are included in the review.

EFFECT OF THERMAL TREATMENT CONDITIONS ON THE PHASE COMPOSITION AND STRUCTURAL CHARACTERISTICS OF V-Mo-Nb-O CATALYSTS

T.Yu. Kardash, L.M. Plyasova, V.M. Bondareva, T.V. Andrushkevich, A.V. Ishchenko, Yu.A. Chesalov, L.S. Dovlitova

Kinet. Catal., 50(1) (2009) pp. 48-56.

The formation of an active phase in V-Mo-Nb oxide catalysts for the selective oxidation and ammoxidation of ethane during thermal treatment in air and in helium was studied using high-temperature *in situ* and *ex situ* X-ray diffraction analysis, transmission electron microscopy, IR spectroscopy, and the differential dissolution method. It was found that, in thermal treatment below 500°C, the formation occurred through the same irreversible steps with the formation of a unidimensionally ordered layered compound with structure elements like Mo_5O_{14} regardless of the calcination atmosphere. Above 500°C, the formation of crystalline phases took place; the composition and structure of these phases depended on the atmosphere of thermal treatment. The unidimensionally ordered V-Nb-Mo oxide with structure elements like Mo_5O_{14} exhibited the best catalytic properties.

THE INVESTIGATION OF CHEMICAL AND PHASE COMPOSITION OF SOLID PRECURSOR OF MoVTeNb OXIDE CATALYST AND ITS TRANSFORMATION DURING THE THERMAL TREATMENT

G.Ya. Popova, T.V. Andrushkevich, L.S. Dovlitova, G.I. Aleshina, Yu.A. Chesalov, A.V. Ishchenko, E.V. Ishchenko, L.M. Plyasova, V.V. Malakhov, M.I. Khramov* (**Solutia Inc., Gonzalez, FL, USA*)

Appl. Catal., A,
353(2) (2009) pp. 249-257.

This work presents the investigation of chemical and phase composition of solid precursor of $MoV_{0.3}Te_{0.23}Nb_{0.12}O_x$ catalyst and its changes during the thermal treatment. The properties of the precursors and of the catalysts obtained by calcinations of precursors at 220–600°C were characterized by BET, XRD, IR and Raman spectroscopy, HRTEM and differential dissolution methods. A MoVTeNb four-component heteropoly compound is formed upon mixing of the starting materials. Thermal decomposition of this precursor occurs in the temperature range from 220 to 350°C, and it results in formation of highly dispersed uniformly distributed particles with a layered structure. These particles progressively transform into two amorphous phases of variable composition. The first phase is a three-component $MoV_xTe_yO_n$ and the second one is a four-component $MoV_gTe_pNb_zO_m$ phase. The change of the chemical and phase compositions completes at 550°C; it yields well-crystallized phases with compositions corresponding to phases M2 ($MoV_{0.35}Te_{0.34}$) and M1 ($MoV_{0.28}Te_{0.1}Nb_{0.12}$). Crystalline materials show the best catalytic performances in propane ammoxidation.

FORMATION OF ACTIVE PHASES IN MoVTeNb OXIDE CATALYSTS FOR AMMOXIDATION OF PROPANE

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144(3-4) (2009) pp. 312-317.

The method of drying, heat evaporation or spray drying, of the aqueous suspension of starting chemicals has a pronounced effect on the phase composition of the final MoVTeNb catalyst, which ultimately influences the catalytic properties in propane ammoxidation reaction. The sample synthesized by spray drying is active and selective; it contains two main crystalline phases, orthorhombic M1 and hexagonal M2. The activity of the sample prepared by heat evaporation is low. This sample does not contain the active M1 phase and consists of hexagonal M2, $TeMo_5O_{16}$, and $Mo_{5-x}(V/Nb)_xO_{14}$ phases. The different mechanisms of phase composition formation in the samples synthesized by heat evaporation or spray drying arise from the different chemical nature of corresponding solid precursors.

EFFECT OF HYDROTHERMAL TREATMENT ON THE COMPOSITION AND STRUCTURE OF Pt(IV) HYDROXO COMPLEXES AS MODEL PRECURSORS OF THE ACTIVE COMPONENT IN Pt/Al_2O_3 CATALYSTS

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50(6) (2009) pp. 880-885.

The influence of hydrothermal treatment conditions (temperature, duration, acidity of the medium) on the structural and chemical transformations of the complex $H_2[Pt(OH)_6]$ was studied. The composition and structure of the resulting compounds were determined by several physicochemical methods. Thermal analysis coupled with mass spectrometry showed that, as the hydrothermal treatment temperature is raised from 25 to 120 and 150°C, the product composition in terms of empirical formulas changes as follows: $PtO_2 \cdot 4H_2O \rightarrow PtO_2 \cdot 3.5H_2O \rightarrow PtO_2 \cdot 1.5H_2O$. X-ray diffraction and UV and IR spectroscopy demonstrated that the changes in the chemical composition are accompanied by the amorphization of the structure and Pt-O bond strengthening. X-ray structure determination using the radial electron density distribution method showed that polynuclear species ~10–15 Å in size with a structure similar to that of orthorhombic PtO_2 form in the complexes subjected to “hard” hydrothermal treatment ($T \geq 150$°C).

INVESTIGATION OF THE THERMAL STABILITY OF THE CHROMIA-ALUMINA CATALYSTS FOR THE PROCESS OF THE ONE-STAGE DEHYDROGENATION OF n-BUTANE

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Catal. Ind.,
1(1) (2009) pp. 43-49.

The effect of the high-temperature (800–1000°C) treatment in air of the Cr_2O_3/Al_2O_3 catalyst, which contained $18Cr_2O_3 + 0.4Na^+$, wt % and was prepared under laboratory conditions with the use of Pural SB1 grade high-purity pseudoboehmite, on the variation in the phase composition of the catalyst, specific surface, and catalytic characteristics in the dehydrogenation reaction of *n*-butane (yield and selectivity by ΣC_4 olefins and 1,3-butadiene, conversion of *n*-butane) was investigated depending on the calcination temperature of the catalyst. It is shown that thermal stability depends on the following main factors: the method of catalyst preparation, the phase composition of the starting aluminum hydroxide, carrier texture, and the presence of modifying additions and impurities of other metals. In the case of the same chemical composition of the catalyst, the samples obtained by the wet mixing of pseudoboehmite with an aqueous solution of chromic anhydride are most thermally stable compared with the impregnation samples. It is established that the addition of cerium improves the thermal stability and activity of the impregnation Al-Cr catalyst, while the impurity of the Fe^{3+} ions (up to 0.1 wt %) does not worsen these characteristics. The investigated samples of the catalyst are more thermally stable than the imported industrial catalyst, which loses activity and specific surface after calcination at 900–1000°C. The determination of the thermal stability of fresh catalysts and the factors affecting it can be used as the preliminary evaluation of the future lifetime of catalysts.

THE INFLUENCE OF THE ACTIVE COMPONENT AND SUPPORT NATURE, GAS MIXTURE COMPOSITION ON PHYSICOCHEMICAL AND CATALYTIC PROPERTIES OF CATALYSTS FOR SOOT OXIDATION

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J. Mol. Catal. A: Chem.,
310(1-2) (2009) pp. 101-112.

Physicochemical and catalytic properties of compositions Fe(Ce)–Mn–O/support (γ-, θ-, α-Al_2O_3, SiO_2 as the support) and Pt/CeO_2/θ-Al_2O_3 for oxidation of soot were characterized. It was established that the phase composition of the initial catalysts depended mainly on the nature of the active component and preparation conditions. Non-isothermal treatment of the soot–catalyst compositions at the temperature up to 1000°C resulted in a change in the phase composition depending mainly on the final treatment temperature. The catalyst surface area was determined by the support nature. It was established that catalyst activities for oxidation of soot are determined by both catalyst nature and composition of gas mixture. The process of the soot oxidation is thought to involve oxygen from the catalyst surface. The higher proportion of weakly bound surface oxygen, the higher was the catalyst activity. An increase in the oxygen concentration from 5% O_2/N_2 to 15% O_2/N_2 is shown to lead to a decrease of the temperature of the soot oxidation. The influence of the oxygen concentration on the process of soot oxidation becomes weaker in the presence of water vapor. Results showed that the presence of NO in the gas mixture favors a decrease in the oxidation temperature of the soot, the higher being the nitrogen oxide concentration, the more pronounced effect. Introduction of SO_2 in amount of 50 ppm in the gas mixture has no noticeable effect on the process of the soot oxidation. Among the catalysts under study, Fe–Mn–K–O/γ-Al_2O_3 is most effective to oxidation of the soot at otherwise identical conditions.

PHYSICOCHEMICAL AND CATALYTIC PROPERTIES OF SYSTEMS BASED ON CeO_2

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50(6) (2009) pp. 798-815.

The effect of synthesis conditions, the nature of components, and the ratio between the components on the phase composition, the texture, and the redox and catalytic properties of the Ce-Zr-O, Ce-Zr-M_1-O (M_1 = Mn, Ni, Cu, Y, La, Pr, or Nd), *N*/Ce-Zr-O (*N* = Rh, Pd, or Pt), and Pd/Ce-Zr-M_2-O/Al_2O_3 (M_2 = Mg, Ca, Sr, Ba, Y, La, Pr, Nd, or Sm) was considered. A cubic solid solution with the fluorite structure was formed on the introduction of <50 mol % zirconium into CeO_2, and the stability of this solid solution depended on preparation procedure and treatment conditions. The presence of transition or rare earth elements in certain concentrations extended the range of compositions with the retained fluorite structure. The texture of the Ce-Zr-O system mainly depended on treatment temperature. An increase in this temperature resulted in a decrease in the specific surface area of the samples. The total pore volume varied over the range of 0.2–0.3 cm^3/g and depended on the Ce/Zr ratio. The presence of transition or rare earth elements either increased the specific surface area of the system or made it more stable to thermal treatment. The introduction of the isovalent cation Zr^{4+} into CeO_2 increased the number of lattice defects both on the surface and in the bulk to increase the mobility of oxygen and facilitate its diffusion in the $Ce_{1-x}Zr_xO_2$ lattice. The catalytic properties of the Ce-Zr-M_1-O or *N*/Ce-Zr-M_2-O systems were due to the presence of anion vacancies and the easy transitions Ce^{4+} ai Ce^{3+}, M_1^{2n+} ai M_1^{n+}, and $N^{\delta+} \rightarrow N^0$ in the case of noble metals.

SYNTHESIS AND PHYSICOCHEMICAL CHARACTERIZATION OF PALLADIUM-CERIUM OXIDE CATALYSTS FOR THE LOW-TEMPERATURE OXIDATION OF CARBON MONOXIDE

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Kinet. Catal.,
50(6) (2009) pp. 819-823.

The effect of CeO_2 preparation procedure on the electronic and structural states of the active component of Pd/CeO_2 catalysts and their activity in the low-temperature reaction of CO oxidation was studied. The following two nonequivalent states of palladium were detected in the catalysts having low-temperature activity using XPS and IR spectroscopy: $Pd^0(Pd^{\delta+})$ as the constituent of a palladium-reduced interaction phase and Pd^{2+} as the constituent of a palladium-oxidized interaction phase $Pd_xCeO_{2-\delta}$. It was found that the procedure used for preparing a

CeO_2 support considerably affected the formation of these phases and quantitative ratios between them. It was demonstrated that the palladium-oxidized interaction phase was responsible for low-temperature activity, whereas the palladium-reduced interaction phase was responsible for activity in the region of medium and high temperatures.

MECHANOCHEMICAL ACTIVATION AS A TOOL OF INCREASING CATALYTIC ACTIVITY

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144(3-4) (2009) pp. 212-218.

The role of structure–energy properties of mechanically induced defects in the crystal structure of heterogeneous catalysts is considered. The main concepts of the mechanochemical activation effect on the activity and selectivity of catalysts are discussed. The wasteless and energy-saving methods of preparation of some catalysts are presented. Rigorous experimental proofs of the influence of defects in the crystal structure of catalysts on their specific catalytic activity are obtained. High performance of mechanochemical catalysis application for hydrogenation, oxidation, amination and hydroalumination processes is shown. For the first time catalytic reactions under the conditions of mechanochemical activation at elevated temperature and increased pressure of gases are carried out.

THE INFLUENCE OF MECHANICAL ACTIVATION AND THE NATURE OF A CARRIER ON THE STRUCTURAL AND DIFFUSION PROPERTIES OF ZEOLITE-CONTAINING CATALYSTS

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Russ. J. Phys. Chem. A,
83(5) (2009) pp. 741-744.

The influence of mechanical activation and the nature of a carrier on the structural and diffusion properties of zeolite-containing catalysts, including the microporous and mesoporous structure of zeolites, was studied. Zeolite-matrix contacts were found to provide the accessibility of the inside volume of zeolite pores. In addition, new pores of a larger size appeared. The IR spectra of adsorbed molecules were used to determine the effective diffusion coefficients of methanol in the porous system of zeolites. It was shown that there was some optimum zeolite pore radius at which the largest diffusion coefficient was attained.

PROPERTIES OF Pt/C CATALYSTS PREPARED BY ADSORPTION OF ANIONIC PRECURSOR AND REDUCTION WITH HYDROGEN. INFLUENCE OF ACIDITY OF SOLUTION

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Appl. Catal., B,
355(1-2) (2009) pp. 115-122.

Carbon-supported Pt/C catalysts prepared with H_2PtCl_6 as metal precursor have been studied by temperature-programmed reduction (TPR), transmission electron microscopy (TEM) and CO chemisorption in order to investigate the effect of acidity of the solution on the catalyst properties. The catalytic activity of the samples has been tested in structure insensitive reaction of cyclohexene hydrogenation. The catalysts were prepared by adsorption of platinum precursor on activated carbons with different pore structure followed by drying and reduction in flowing hydrogen at 523 K. The use of acidic and highly basic solution during the impregnation of microporous carbons led to highly dispersed catalysts with low activity. It has been suggested that a part of the supported metal became inaccessible to the organic substrate due to localization of metal particles in narrow pores of the support. However, in the range of intermediate solution pH, the activity per surface metal atom remained at a high level. Such phenomenon is attributed to the formation of bulky species of platinum precursor, which hardly penetrates into the small pores of the support, thus preventing a blocking effect. Effect of acidity of the solution proved significant for Pt/C catalysts on the activated carbons with the smaller micropores and higher concentration of oxygen-containing groups.

LOW-TEMPERATURE SYNTHESIS OF SUPPORTED HYDRODESULFURIZATION CATALYSTS BASED ON CHEVREL PHASES

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Kinet. Catal.,
50(2) (2009) pp. 270-274.

A new method has been developed for the synthesis of finely dispersed, highly active, supported hydrodesulfurization catalysts based on Chevrel phases. It is hypothesized that the modification of MoS_2 with cobalt or nickel, which enhances the catalytic activity, and the same modification of Chevrel-type systems are of the same nature. The modifiers act through electron density donation into the conduction band of the active component. The increase in catalytic activity is due to the decrease of the effective charge of the molybdenum ion. The catalysts undergo partial restructuring during the reaction.

NANODISPERSED Au/Al_2O_3 CATALYSTS FOR LOW-TEMPERATURE CO OXIDATION: RESULTS OF RESEARCH ACTIVITY AT THE BORESKOV INSTITUTE OF CATALYSIS

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144(3-4) (2009) pp. 292-305.

This paper presents some important results of the studies on preparation and catalytic properties of nanodispersed Au/Al_2O_3 catalysts for low-temperature CO oxidation, which are carried out at the Boreskov Institute of Catalysis starting from 2001. The catalysts with a gold loading of 1–2 wt.% were prepared *via* deposition of Au complexes onto different aluminas by means of various techniques ("deposition-precipitation" (DP), incipient wetness, "chemical liquid-phase grafting" (CLPG), chemical vapor deposition (CVD)). These catalysts have been characterized comparatively by a number of physical methods (XRD, TEM, diffuse reflectance UV/vis and XPS) and catalytically tested for combustion of CO impurity (1%) in wet air stream at near-ambient temperature. Using the hydroxide or chloride gold complexes capable of chemical interaction with the surface groups of alumina as the catalyst precursors (DP and incipient wetness techniques, respectively) produces the catalysts that contain metallic Au particles mainly of 2–4 nm in diameter, uniformly distributed between the external and internal surfaces of the support granules together with the surface "ionic" Au oxide species. Application of organogold precursors gives the supported Au catalysts of egg shell type which are either close by mean Au particle size to what was obtained by DP and incipient wetness techniques (CVD of $(CH_3)_2Au(acac)$ vapor on highly dehydrated Al_2O_3 in a rotating reactor under static conditions) or contain Au crystallites of no less than 7 nm in size (CLPG method). Regardless of deposition technique, only the Cl-free Au/Al_2O_3 catalysts containing the small Au particles ($d_i \leq 5$ nm) reveal the high catalytic activity toward CO oxidation under near-ambient conditions, the catalyst stability being provided by adding the water vapor into the reaction feed. The results of testing of the nanodispersed Au/Al_2O_3 catalysts under conditions which simulate in part removal of CO from ambient air or diesel exhaust are discussed in comparison with the data obtained for the commercial Pd and Pt catalysts under the same conditions.

EFFECT OF ZrO_2 MORPHOLOGY IN COPPER–CERIUM–ZIRCONIUM OXIDE SYSTEMS ON THEIR CATALYTIC PROPERTIES IN THE REACTION OF CO OXIDATION IN HYDROGEN-RICH MIXTURES

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Theor. Exp. Chem.,
45(2) (2009) pp. 125-130.

The catalytic properties of copper–cerium oxide systems, deposited on supports obtained by calcination of yttrium-stabilized zirconium dioxide at 300-1000°C, have been studied in the reaction of selective oxidation of CO in a stream of hydrogen. It has been shown that the catalytic activity of the samples obtained correlates with the activity of the original supports in the reaction of CO oxidation: the highest CO conversion is observed on catalysts with the highest and the lowest specific surface area.

REGULATING THE PHYSICOCHEMICAL AND CATALYTIC PROPERTIES OF LAYERED ALUMINOSILICATES

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Kinet. Catal.,
50(1) (2009) pp. 57-64.

Basic methods of the introduction of metal ions into the interlayer space of natural layered aluminosilicate (LAS) are considered. The physicochemical and structural properties of pillared LAS's depend on the nature of the cation, as well as on the cation intercalation method and conditions. The catalytic properties and stability of an LAS in acid-catalyzed and oxidation reactions may depend on its textural and physicochemical properties.

THE EFFECT OF Fe/Cu RATIO IN THE SYNTHESIS OF MIXED Fe,Cu,Al-CLAYS USED AS CATALYSTS IN PHENOL PEROXIDE OXIDATION

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Appl. Catal., B,
90(3-4) (2009) pp. 618-627.

Mixed Fe,Cu,Al-clays (Fe,Cu,Al-MM) were prepared from Ca-montmorillonite (Mukhortala, Buryatia) at OH/(Fe + Cu + Al) 2.4 and Al/(Fe + Cu) 10/1. Effect of Fe/Cu ratio of Fe,Cu,Al-containing pillaring solution on structural properties of Fe,Cu,Al-MM was examined. Characterization studies were performed by use of XRD, FTIR, DR-UV–vis and N_2-adsorption/desorption analysis. The increase in copper loading leads to the decrease in total surface area, micropore volume and interlay distance of Fe,Cu,Al-MMs. It was found that the decrease in Fe/Cu ratio is favourable for the formation of oligomeric iron species. The relationships between preparation conditions, iron state, catalytic activity and stability to leaching were revealed in wet phenol oxidation with H_2O_2. The introduction of copper ions results in catalytic reaction acceleration that was interpreted in terms of the increase of radical generation rate.

SYNTHESIS OF Fe,Al-PILLARED CLAYS STARTING FROM THE Al,Fe-POLYMERIC PRECURSOR: EFFECT OF SYNTHESIS PARAMETERS ON TEXTURAL AND CATALYTIC PROPERTIES

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Appl. Catal., B,
88(1-2) (2009) pp. 127-134.

Effect of OH/(Fe + Al) ratio, aging time of Fe,Al-containing pillaring solution on physicochemical properties of Fe,Al-pillared clays (Fe,Al-PILCs) obtained from Mukhortala (Buryatia) montmorillonites and the change of these properties with the calcination temperature were examined. Characterization studies were performed by use of XRD, FTIR, DR-UV-vis, ^{27}Al NMR and N_2-adsorption/desorption analysis. It was found that the prolonged aging time of the Fe,Al-solution (Al/Fe = 10/1) favours the increase in total surface area, total pore-volume and micropore volume of Fe,Al-PILCs. The relationships between preparation conditions, iron state, catalytic activity and stability to leaching were revealed in hydrogen peroxide oxidation of phenol and monoazo dye acid chrome dark-blue (ACDB). The state of iron atoms can be controlled by Al/Fe and OH/(Fe + Al) ratios, aging period of Fe,Al-pillaring solution and calcination temperature. The increase in Al/Fe ratio and calcination temperature from 400°C to 500°C reduces to the decrease in the formation of oligomeric iron species. The increase in aging time of Fe,Al-pillaring solution reduced to decrease in iron leaching and increase in reaction rate due to the formation of isolated iron species.

DESIGN AND APPLICATION OF IRON-CONTAINING MESOPOROUS MOLECULAR SIEVES FOR PEROXIDE OXIDATION OF POLLUTANTS: EFFECT OF IRON ENVIRONMENT ON TEXTURAL, PHYSICOCHEMICAL AND CATALYTIC PROPERTIES

M.N. Timofeeva, M.S. Melgunov

In “*Mesoporous Materials*”,
Ed. L.T. Burness, Nova Sci. Publishers, Inc.,
2009, Chapter 10, pp. 1-28.

Homogeneous catalytic systems of the Fenton type ($Fe^{2+}/Fe^{3+}/H_2O_2$) are known active catalysts for their complete oxidation (or mineralization) with H_2O_2 to produce CO_2 and H_2O. Iron-containing mesoporous silicates have attracted considerable attention as heterogeneous oxidation catalysts due to their Fenton like behaviour. The authors review herein major aspects of their research in this field for iron-containing mesoporous molecular sieves (Fe-MMM-2 and Fe,Al-MMM-2) that were synthesized via sol-mesophase rout under weak acidic conditions. Catalyst design principles are summarized and discussed to illustrate the effective development of catalysts meeting the requirements of the catalytic application. The following parameters influencing textural, physicochemical, catalytic properties of the catalysts and stability to leaching of metal from the solid are considered: (1) pH of the synthesis solution; (2) agglomeration and isolation of iron ions; (3) iron and aluminum content in the samples. Results of model experiments are correlated with application properties in order to improve the understanding of the peroxide oxidation process. It has been established that the catalytic activity in oxidation of pollutants such as phenol and monoazo dye acid chrome dark-blue with hydrogen peroxide strongly depends on the presence of isolated iron species, which are stable to leaching. The increase in iron loading and pH of the synthetic solution leads to iron agglomeration, which, in turn, results in the reduction of the catalytic activity of Fe-MMM-2 and in the increase of iron leaching. It was shown that catalytic activity could be improved by insertion of Al species into the framework of the silicate matrix. It is demonstrated that activity of Fe,Al-MMM-2 is higher than that of Fe,Al-pillared clay due to the higher surface acidity and decrease of diffusion limitation.

EFFECT OF THE CONCENTRATION OF ALUMINUM ON THE ADSORPTION, TEXTURE, AND STRUCTURE CHARACTERISTICS OF A MESOPOROUS MINERAL MESOPHASE OF THE SBA-15 TYPE

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50(3) (2009) pp. 456-460.

The effect of Al ion implantation on the properties of mesoporous aluminosilicate mineral phases of the SBA-15 type was studied. The implantation of Al was performed immediately under conditions of the synthesis of SBA-15 in a weakly acidic medium (pH ~ 2.9). It was found that, under these conditions, the amount of Al that can be implanted into the SBA-15 framework is limited (a maximum of 7.2 mol %). According to XPS data, aluminum ions were implanted into the matrix of silica rather than occurring on the surface as an individual phase. The study of nitrogen adsorption at 77 K and the results obtained by X-ray diffractometry and high-resolution electron microscopy suggest that Al-SBA-15 materials exhibited a hexagonal structure of channel pores of the same diameter of 8.3 nm, and the unit cell parameter was 12.3 nm. The degree of crystallinity of the material increased with the concentration of Al.

MAGNETICALLY SEPARABLE TITANIUM-SILICATE MESOPOROUS MATERIALS WITH CORE-SHELL MORPHOLOGY: SYNTHESIS, CHARACTERIZATION AND CATALYTIC PROPERTIES

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J. Mater. Chem.,
19(39) (2009) pp. 7332-7339.

The preparation and characterization (XRD, N_2 adsorption, TEM, EDX, DRS-UV) of a novel catalytic material Ti-SMCMS (solid magnetic core-mesoporous shell) is reported. The material has quasi spherical particles with an ordered mesoporous silicate shell containing isolated Ti atoms and a silica core which, in turn, comprises superparamagnetic iron oxide nanoparticles. The magnetic kernels are embedded in the nonporous silica core and, thus, are completely protected from ambient medium. The material was found to combine the advantages of high activity and selectivity in H_2O_2-based selective oxidations with the merits of an easy magnetic separation from the reaction mixture.

TITANIUM-DOPED SOLID CORE-MESOPOROUS SHELL SILICA PARTICLES: SYNTHESIS AND CATALYTIC PROPERTIES IN SELECTIVE OXIDATION REACTIONS

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Catal. Lett.,
127(1-2) (2009) pp. 75-82.

Near monodisperse spherical particles composed of a nonporous silica core covered by a Ti-doped hexagonally arranged mesoporous silica shell, Ti-SCMS, as well as spherical submicron-size particles of Ti-MCM-41, have been synthesized for the first time and characterized by elemental analysis, N_2 adsorption, XRD, TEM and DR-UV spectroscopy. The mesoporous Ti,Si-shell has a thickness of about 45 nm and incorporates isolated Ti centers in tetrahedral coordination. Catalytic properties of Ti-SCMS and Ti-MCM-41 have been studied in selective oxidation of three representative bulky organic substrates, 2,3,6-trimethylphenol, methyl phenyl sulfide and caryophyllene, with aqueous H_2O_2 in MeCN medium. Ti-SCMS appeared to be more active and selective in the H_2O_2-based selective oxidation reactions compared to Ti-MCM-41, thus demonstrating an advantage of conducting a catalytic process in a thin mesoporous Ti,Si-shell.

NEW APPROACHES TO THE PREPARATION OF HIGHLY EFFICIENT CHROMIUM-CONTAINING OXIDE CATALYSTS FOR THE WATER GAS SHIFT REACTION

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Kinet. Catal.,
50(6) (2009) pp. 837-850.

It was found experimentally that the solutions of Cr^{3+} nitrate and the nitrates of other metals that are the constituents of Cr-containing catalysts can be prepared by dissolving a corresponding metal (for example, cast iron and electrolytic copper) in a solution of chromic anhydride and nitric acid to reach the quantitative reduction of Cr^{6+} without the formation of nitrogen oxides. Analogously, the oxidation of Fe^{2+} cations to Fe^{3+} coupled with the reduction of hexavalent chromium can be performed. The precipitation of Fe^{3+}, Cr^{3+}, and Cu^{2+} ions at a ratio of Fe: Cr = 9 and a concentration of Cu^{2+} to 20 at % can result in the formation of a partially hydrated oxide with the hydrohematite structure - a dispersed and highly defective oxide structure with a high specific surface area more than 300 m^2/g and a higher thermal stability, as compared with the goethite phase (α-FeOOH). The dehydration of hydrohematite occurred at a noticeable rate at temperatures higher than 400°C. Hydrohematite promoted with copper cations exhibited high activity below 400°C; this can decrease the starting temperature of the adiabatic high-temperature WGSR to 300°C or below.

PARTIALLY HYDRATED IRON–CHROMIUM OXIDE CATALYST FOR THE FISCHER-TROPSCH SYNTHESIS

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React. Kinet. Catal. Lett.,
97(2) (2009) pp. 371-379.

Due to their well-developed surface, partially hydrated iron–chromium oxides with the ferrihydrite structure exhibit much higher activity in the Fischer-Tropsch synthesis than those that have the hematite structure.

SYNTHESIS AND MESOPORE-MICROPORE STRUCTURE CHARACTERIZATION OF NANODISPERSE Fe^{3+} HYDROGELS (XEROGELS)

V.Yu. Gavrilov, O.P. Krivoruchko

Kinet. Catal., 50(1) (2009) pp. 122-130.

A preparation procedure was developed, and samples of nanodisperse Fe^{3+} hydroxide with a narrow particle-size distribution (2.5–3.5 nm) were synthesized. The occurrence of a substructure in the bulk of Fe^{3+} hydroxide nanoparticles was detected for the first time using light-field and dark-field transmission electron microscopy. It was found that structurally ordered regions with sizes of ~1.0 nm, which were disoriented with respect to each other at angles of a few degrees, occurred in the bulk of the nanoparticles. The empirical formula of nanodisperse iron hydroxide was $\sim Fe_2O_3 \cdot 1.8H_2O$; the structure of this hydroxide contained crystal water, OH^-, and O^{2-}. The coordination number of Fe^{3+} cations with respect to oxygen was 6. It was found that both structural and nonstructural water can be removed almost completely from the bulk of nanoparticles in the course of sample heating to 150–250°C in a vacuum with the retention of their amorphous character and observed sizes. In the course of dehydration, the mutual mobility of nanoparticles within aggregates was retained in Fe^{3+} xerogels; this resulted in a decrease in the total pore volume, whereas the volume of mesopores with diameters of 3.4–3.5 nm progressively increased. The micropore structure of the samples of nanodisperse iron hydroxides was studied by the molecular probe method using the low-temperature (77 K) sorption of nitrogen and molecular hydrogen. It was found that, along with micropores of volume ~0.02 cm^3/g, which are accessible to both of the sorbates, the sorption of H_2 exhibited an additional specific absorption of 1.0–1.7 cm^3(STP)/g, which can be interpreted as an additional ultramicropore volume accessible to only hydrogen molecules.

SYNTHESIS, ELECTRONIC STATE, AND PARTICLE SIZE STABILIZATION OF NANOPARTICULATE $[Co(OH)_2(H_3O)_\delta^+]^{\delta+}$

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Inorg. Mater.,
45(12) (2009) pp. 1355-1361.

Nanoparticulate cobalt(II) hydroxide have been synthesized containing Co^{2+} in tetrahedral oxygen coordination ($Co_{T\delta}^{2+}$) atypical of such systems: nano-

$[Co(OH)_2(H_3O)_\delta{}^+]^{\delta+}$. The $Co_{T\delta}{}^{2+}$ coordination in the hydroxide is inferred from its electronic diffuse reflectance spectrum, which shows a multiplet of strong absorption bands at 14 500, 15 000, and 16 000 cm^{-1} ($^4A_2(F)$- $^4T_1(P)$ transition). Nanoparticulate cobalt(II) hydroxide forms in a weakly acidic medium under essentially nonequilibrium conditions due to supersaturation (by three to four orders of magnitude) with the starting reagents ($CoCl_2$ and LiOH) at the instant of the formation of the poorly soluble phase $Co(OH)_2$. Presumably, colloidal particles of nanoparticulate cobalt(II) hydroxide in a weakly acidic aqueous medium have a positive surface charge, compensated by a counter-ion (Cl^-) layer: nano-$[Co(OH)_2(H_3O)_\delta{}^+]^{\delta+} \cdot \delta Cl^-$. The XRD patterns of pastes (gels) containing this hydroxide show three broadened lines with d = 5.31 (2θ = 16.7°), 2.77 (2θ = 32.3°), and 2.32 Å (2θ = 38.8°). According to small-angle X-ray scattering data, nano-$[Co(OH)_2(H_3O)_\delta{}^+]^{\delta+}$ has a narrow particle size distribution (1.0–2.0 nm). Synthesis and storage conditions are identified which ensure stabilization of the electronic state and particle size of nano-$[Co(OH)_2(H_3O)_\delta{}^+]^{\delta+}$ for a long time.

SELECTIVE CATALYTIC REDUCTION OF NO_x OVER NANO-SIZED GOLD CATALYST SUPPORTED ON ALUMINA AND TITANIA AND OVER BIMETALLIC GOLD-SILVER CATALYST SUPPORTED ON ALUMINA

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Top. Catal.,
52(13-20) (2009) pp. 1762-1765.

The reduction of NO with octane under lean conditions was examined over gold supported on alumina and titania and over alumina supported bimetallic gold–silver catalysts. The silver loading was either 1.2 or 1.9 wt% whereas 0.3, 1 or 5 wt% gold was used. The catalysts were characterized by means of EDXS, N_2-adsortion, UV–Vis and TEM to correlate recorded results with different preparation methods. UV–Vis measurements indicated that gold was present in the form of fine Au particles, single Au ions and small $(Au)_n{}^{\delta+}$ clusters on the catalysts and silver was mainly present in the form of single Ag ions. The highest NO to N_2 reduction activity was recorded over the 0.3Au–Al_2O_3 catalyst. The Au–TiO_2 catalysts did not result in significant NO to N_2 reduction.

EFFECT OF THE CERIUM CONTENT ON THE ACTIVITY OF Cu-Ce-Al-O CATALYSTS IN THE METHANOL STEAM REFORMING REACTION IN A FLOW REACTOR

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Kinet. Catal., 50(2) (2009) pp. 241-246.

The $Cu_{18.5}Ce_x Al_{81.5-x}$ (where x = 2, 7.4, and 14) oxide catalysts were synthesized by coprecipitation and tested in the methanol steam reforming reaction in an integral flow reactor at 270°C. It was found that the activity of the catalysts increased with the calcination temperature and catalysts with intermediate cerium contents exhibited the highest activity; these catalysts exhibited the greatest values of S_{BET} and S_{Cu}. The phase analysis demonstrated that copper in these samples occurred almost entirely as a CuO-CeO_2 solid solution. The concentration of carbon monoxide at the reactor outlet decreased with the calcination temperature. For the most active sample with a cerium content of 7.4% calcinated at 700°C, the concentration of CO reached a minimum of no higher than 0.3%.

SELECTIVE CATALYTIC REDUCTION OF NITROGEN OXIDE BY AMMONIA ON Mn(Fe)-SUBSTITUTED Sr(La) ALUMINATES

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Appl. Catal., A,
357(2) (2009) pp. 193-205.

Aluminates $SrMn_2Al_{10}O_{19}$, $Sr_{0.8}La_{0.2}Mn_2Al_{10}O_{19}$, $LaMn_2Al_9O_{18}$, $LaMnAl_{10}O_{18}$ and $SrFeMnAl_{10}O_{19}$ prepared by co-precipitation and calcined at 700–1400°C have been characterized by absorption methods, thermal analysis (TG–DTA), X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS) and temperature-programmed reduction (TPR). It has been shown that calcination at 1100°C results in the formation of multiphase systems containing mainly hexaaluminate. The specific surface area of the samples calcined at 1100°C has been ranged from 20 to 25 m^2/g, except for the $Sr_{0.67}Mn_{2.28}Al_{10}O_{19.1}$ hexaaluminate, whose specific surface area was twice as large. TPR-H_2 of the samples calcined at 700°C proceeded in several steps.

The first step was related to the reduction of $Mn(Fe)_2O_3$ to $Mn(Fe)_3O_4$ at 345–382°C. The second step was related to the reduction of Mn_3O_4 to MnO. The replacement of the part of Sr ($Sr_{0.67}Mn_{2.28}Al_{10}O_{19.1}$) by La and of the part of Mn by Fe leaded to the decrease in T_{TPR}. The amount of H_2 consumed during the reduction of the samples calcined at 700 and 1100°C increases in the series:

$Sr_{0.67}Mn_{2.28}Al_{10}O_{19.1}$ (1100°C) <
$Sr_{0.89}Fe_{0.93}Mn_{1.03}Al_{10}O_{18.8}$ (1100°C) <
$Sr_{0.89}Fe_{0.93}Mn_{1.03}Al_{10}O_{18.8}$ (700°C) <
$Sr_{0.67}Mn_{2.28}Al_{10}O_{19.1}$ (700°C) <
$Sr_{0.69}La_{0.33}Mn_{2.23}Al_{10}O_{19.5}$ (700°C).

Surface concentrations of elements in the samples $Sr_{0.69}La_{0.33}Mn_{2.23}Al_{10}O_{19.5}$ and $Sr_{0.89}Fe_{0.93}Mn_{1.03}Al_{10}O_{18.8}$ differ from the volume chemical contents. The main components on the surface are in oxidized state: Al^{3+}, La^{3+} and Fe^{3+}, Mn^{3+} and Mn^{2+}. Amount of Mn^{3+} ions is 65 and 49%, respectively. Sr is predominantly localized on the surface as $SrCO_3$. Among hexaaluminates, the most active ($X_{NO} \approx 78\%$) and selective sample in the selective catalytic reduction (SCR) of NO to N_2 was $Sr_{0.69}La_{0.33}Mn_{2.23}Al_{10}O_{19.5}$, This sample was characterized by the highest amount of accessible oxygen and ratio Mn^{3+}/Mn^{2+}, in which the amount of Mn^{3+} is 65%, determining the red-ox transformation.

PHASE COMPOSITION AND TEXTURE OF Sr(La)Mn HEXAALUMINATES

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Kinet. Catal.,
50(6) (2009) pp. 824-829.

Hexaaluminates synthesized by precipitation and calcined at 700–1400°C have been characterized by atomic absorption spectroscopy, thermal analysis, X-ray powder diffraction, and adsorption methods. The heat treatment of the Mn-substituted and unsubstituted materials at 1100 and 1200°C yields a hexaaluminate phase. The specific surface area of the samples calcined at 1100°C is 20–49 m^2/g. The texture of the samples calcined at 1000°C is characterized by a unimodal mesoporous pore size distribution with a mean pore diameter of 290 Å.

DEEP DESULPHURIZATION OF DIESEL FUELS ON BIFUNCTIONAL MONOLITHIC NANOSTRUCTURED Pt-ZEOLITE CATALYSTS

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Catal. Today,
144(3-4) (2009) pp. 235-250.

The preparation of Pt-zeolite catalysts, including choice of the noble metal precursor and loading (1.0–1.8 wt.%), was optimized for maximizing the catalytic activity in thiophene hydrodesulphurization (HDS) and benzene hydrogenation (HYD). According to data obtained by HRTEM, XPS, EXAFS and FTIR spectroscopy of adsorbed CO, the catalysts contained finely dispersed Pt nanoparticles (2–5 nm) located on montmorillonite and zeolite surfaces as: Pt^0 (main, ν_{CO} = 2070–2095 cm^{-1}), $Pt^{\delta+}$ (ν_{CO} = 2128 cm^{-1}) and Pt^{2+} (ν_{CO} = 2149–2155 cm^{-1}). It was shown that the state of Pt depended on the Si/Al zeolite ratio, montmorillonite presence and Pt precursor. The use of H_2PtCl_6 as the precursor (impregnation) promoted stabilization of an oxidized Pt state, most likely $Pt(OH)_xCl_y$. When $Pt(NH_3)_4Cl_2$ (ion-exchange) was used, the Pt^0 and hydroxo- or oxy-complexes $Pt(OH)_6^{2-}$ or PtO_2 were formed. The addition of the Ca-montmorillonite favoured stabilization of $Pt^{+\delta}$. The Cl^- ions inhibit reduction of oxidized Pt state to Pt particles. The Pt-zeolite catalyst demonstrated high efficiency in ultra-deep desulphurization of DLCO. The good catalyst performance in hydrogenation activity and sulphur resistance can be explained by the favourable pore space architecture and the location and the state of the Pt clusters. The bimodal texture of the developed zeolite substrates allows realizing a concept for design of sulphur-resistant noble metal hydrotreating catalyst proposed by Song [C. Song, Shape-Selective Catalysis, Chemicals Synthesis and Hydrocarbon Processing (ACS Symposium Series 738), Washington, 1999, p. 381; Chemtech 29(3) (1999) 26].

PREPARATION OF SUPPORTED IRON-CONTAINING CATALYSTS FROM A $FeSO_4$ SOLUTION: THE EFFECT OF THE SUPPORT

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Kinet. Catal.,
50(6) (2009) pp. 874-877.

The iron compounds resulting from the impregnation of the most common supports—alumina and silica gel-with an aqueous $FeSO_4{\cdot}7H_2O$ solution and subsequent heat treatment in air are identified by thermal analysis and Mössbauer spectroscopy. The state of the active component of the iron-containing catalysts depends strongly on the nature of the support.

NANOCRYSTALLINE AEROGEL VO_x/MgO AS A CATALYST FOR OXIDATIVE DEHYDROGENATION OF PROPANE

I.V. Mishakov, E.V. Ilyina, A.F. Bedilo, A.A. Vedyagin

React. Kinet. Catal. Lett.,
97(2) (2009) pp. 355-361.

Nanocrystalline aerogel VO_x/MgO catalysts for the oxidative dehydrogenation of propane with high surface area and uniform vanadium distribution were synthesized by co-gelation followed by supercritical drying. The catalysts were shown to have superior performance compared to nanocrystalline VO_x/MgO catalysts prepared by impregnation.

PREPARATION OF NANOCRYSTALLINE $VMg(OH)_x$ AND VO_x/MgO FROM ORGANOMETALLIC PRECURSORS

E.V. Ilyina, I.V. Mishakov, A.A. Vedyagin

Inorg. Mater.,
45(11) (2009) pp. 1267-1270.

A process is proposed for the synthesis of nanocrystalline $VMg(OH)_x$ and $VO_x{\cdot}MgO$ with specific surface areas of up to 1200 and 470 m^2/g, respectively. The synthesized VO_x MgO oxides consist of nanocrystals (2′–5 nm), which form platelike agglomerates. As distinct from conventional impregnation of magnesium oxide, the aerogel process for VO_x MgO synthesis ensures a uniform vanadium distribution in MgO.

AEROGEL VO_x/MgO CATALYSTS FOR OXIDATIVE DEHYDROGENATION OF PROPANE

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Catal. Today,
144(3-4) (2009) pp. 278-284.

VO_x/MgO aerogel catalysts were synthesized using three different preparation methods: by mixing the aerogel MgO support with dry ammonium vanadate, by vanadium deposition from a precursor solution in toluene, and by hydrolysis of a mixture of vanadium and magnesium alkoxides followed by co-gelation and supercritical drying. The latter aerogel technique allowed to synthesize mixed vanadium–magnesium hydroxides with the surface areas exceeding 1300 m^2/g. The synthesized catalysts were studied by a number of physicochemical methods (XRD, Raman spectroscopy, XANES and TEM). A common feature of all synthesized samples is the lack of V_2O_5 phase. In all cases vanadium was found to be a part of a surface mixed V–Mg oxide (magnesium vanadate), its structure depending on the synthesis method. The VO_x/MgO mixed aerogel sample had the highest surface area 340 m^2/g, showed higher catalytic activity and selectivity in oxidative dehydrogenation of propane compared to the catalysts prepared by impregnation and dry mixing. The addition of iodine vapor to the feed in 0.1–0.25 vol.% concentrations was found to increase to propylene yield by 40–70%.

SYNTHESIS AND STABILIZATION OF NANO-SIZED TITANIUM DIOXIDE

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Russ. Chem. Rev.,
78(9) (2009) pp. 873-885.

The published data on the preparation and the dispersion-structural properties of nano-sized TiO_2 are considered. Attention is focused on its sol–gel synthesis from different precursors. The possibilities for the purposeful control and stabilization of properties of TiO_2 nanopowders and sols are analyzed. Information on physicochemical methods used in studies of the particle size and the phase composition of nanodisperse TiO_2 is presented. The prospects of using nano-sized TiO_2 in medicine and nanobiotechnology are considered.

PREPARATION, CHARACTERIZATION, AND CATALYTIC PERFORMANCE OF MESOPOROUS ZSM-12 ZEOLITE

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Chinese J. Catal.,
30(6) (2009) pp. 531-536.

Mesoporous ZSM-12 zeolite was prepared by desilication treatment in alkaline medium. The ZSM-12 samples before and after alkaline treatment were characterized by X-ray diffraction, N_2 adsorption, solid state NMR, and NH_3 temperature-programmed desorption. The effects of desilication on the structure, acidity, and catalytic performance of ZSM-12 zeolite in the alkylation of naphthalene were investigated. The results showed that the mesopore distribution can be controlled by changing the NaOH solution concentration without destroying the micropore framework of ZSM-12 zeolite. The mesopore volume was increased and pore size distribution was widened with increasing NaOH concentration. Furthermore, the additional mesopores generated by desilication acted as transferring channels, which improved the diffusion of the reactants and products. Therefore, the catalytic performance was effectively improved, and the conversion of naphthalene and selectivity for 2,6 DMN were increased.

SYNTHESIS OF SAPO-31 AND ITS CATALYTIC PERFORMANCE IN HYDROISOMERIZATION OF n-DECANE

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Acta Petrolei Sinica (Petroleum Processing Section),
25(Spec. Issue 2) (2009) pp. 87-90.

A series of SAPO-31 molecular sieves with different silicon contents was synthesized under hydrothermal conditions using AIP, fumed silica, H_3PO_4 and DBA as sources of Al, Si, P and template, respectively. SAPO-31 samples synthesized at different silicon contents were characterized using XRD, BET, SEM, and NH_3-TPD. The bifunctional catalysts 0.5%Pd/SAPO-31 were prepared through the incipient wetness impregnation technique, and its catalytic performance of the hydroisomerization of n-decane was investigated on the fixed-bed continuous-flow reactor. The influence of silicon contents on the crystal morphologies, pore structure, acidic properties and catalytic performance of SAPO-31 samples was studied. The results indicated that gave rise to SAPO-31 molecular sieves with different acidities, conversion of n-decane increased but selectivity to *mono*-methylnonane isomers decreased. The maximal selectivity to *iso*-C_{10} was reached over 0.5%Pd/SAPO-31 ($n(SiO_2)/n(Al_2O_3)=0.5$).

SYNTHESIS AND CHARACTERIZATION OF COMPOSITE MOLECULAR SIEVES ZSM-12/MCM-41

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Rare Met.,
28(Spec. Issue) (2009) pp. 619-622.

A ZSM-12/MCM-41 composite molecular sieve has been hydrothermally synthesized through a novel process of the self-assembly of CTAB surfactant micelles with silica-alumina source which has been originated from the alkaline-treated zeolite ZSM-12 and contains secondary building units characteristic of ZSM-12 besides the zeolite crystals. The structure, morphology and acidity of the composite molecular sieves ZSM-12/MCM-41 samples were thoroughly characterized with XRD, N_2 adsorption, TEM, and NH_3-TPD. The influence of alkaline concentration on the distribution of micropores and mesopores in ZSM-12/MCM-41 composite molecular sieve was investigated.

THE INFLUENCE OF NATURE OF THE ANION IN TETRAALKYLAMMONIUM SALTS ON THE CRYSTALLIZATION OF ZEOLITE BETA

A.V. Toktarev, G.V. Echevsky

Pet. Chem., 49(1) (2009) pp. 30-35.

The influence of the nature of anion X in the tetraethylammonium salt $[N(C_2H_5)_4]$ X on the selectivity of the formation zeolite beta in the $SiO_2–Al_2O_3–Na_2O–[N(C_2H_5)_4]X–H_2O$ system under hydrothermal conditions without seed crystals was studied. It was shown that the test anions can be arranged in the following series in terms of the preferential formation of zeolite beta: F^-, SO_4^{2-}, PO_4^{3-}, $(CH_2COO)_2^{2-}$ > $HCOO^-$ > $(COO)_2^{2-}$, CH_3COO^- >> Cl^- ~ NO_3^->> Br^- >>I^- >> ClO_4^-. In particular, the fluoride and the anions of polybasic acids facilitate the formation of zeolite, whereas perchlorate, bromide, and iodide ions inhibit its formation. The experiments showed that anions facilitating the formation of zeolite beta make it possible to synthesize zeolite within the same broad range of chemical composition of the reaction mixture as in the case of $[N(C_2H_5)_4]OH$. The reveled classification of anions into groups coincides with their categorization by Samoilov into anions with positive and negative hydration.

FORMATION OF Pt/C CATALYSTS ON VARIOUS CARBON SUPPORTS

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Russ. J. Inorg. Chem.,
54(10) (2009) pp. 1531-1536.

Platinum catalysts with the active component content of 5–40 wt % on various carbon supports have been synthesized by the reductive hydrolysis of platinum chloro complexes. The degree of dispersion of the supported platinum decreases with an increasing weight percentage of the metal in the catalyst. The following mechanism of Pt/C catalyst formation is deduced from experimental data: H_2PtCl_6 adsorption on the support surface generates platinum nuclei, which then grow owing to the deposition of platinum ions under the action of an alkali and a reductant.

DEFECTIVE MAGNESIUM OXIDES WITH OXYGEN-CONTAINING ANION FRAGMENTS INCORPORATED IN THE OXIDE STRUCTURE

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Kinet. Catal.,
50(6) (2009) pp. 816-818.

The synthesis of defective magnesium oxides from different precursors is reported. The formation of the defective oxides (which are catalytically active in free-radical reactions) as substitutional solid solutions is possible only via MgO hydration in a salt solution.

MECHANISTIC FEATURES OF REDUCTION OF COPPER CHROMITE AND STATE OF ABSORBED HYDROGEN IN THE STRUCTURE OF REDUCED COPPER CHROMITE

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Russ. J. General Chem.,
78(11) (2009) pp. 2203-2213.

The review discusses the experimental data on the unusual mechanism of the reduction of copper cations from the copper chromite, $CuCr_2O_4$, structure. Treatment of copper chromite in hydrogen at 180–370°C is not accompanied by water formation but leads to absorption of hydrogen by the oxide structure with simultaneous formation of metallic copper as small flat particles which are epitaxially bound to the oxide. This process is due to the redox reaction $Cu^{2+} + H_2 \rightarrow Cu^0 + 2H^+$; the protons are stabilized in the oxide phase, which is confirmed by neutron diffraction studies. The reduced copper chromite which contains absorbed hydrogen in its oxidized state and the metallic copper particles epitaxially bound to the oxide phase structure exhibit catalytic activity in hydrogenation reactions.

EFFECT OF SILICON DIOXIDE ON THE FORMATION OF THE PHASE COMPOSITION AND PORE STRUCTURE OF TITANIUM DIOXIDE WITH THE ANATASE STRUCTURE

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Kinet. Catal.,
50(5) (2009) pp. 760-767.

The formation of the structure of titanium dioxide modified with silicon dioxide, which was introduced as tetraethyl orthosilicate, was studied. It was found that the formation of the nanocrystalline structure of TiO_2 occurred upon the modification of titanium dioxide with silicon dioxide. This nanocrystalline structure of TiO_2 was formed by highly dispersed anatase particles of size 6–10 nm stabilized by silicon oxide layers, which were formed upon the decomposition of tetraethyl orthosilicate. An increase in the modifier concentration resulted in a deceleration of the growth of anatase particles and an increase in the temperature of the phase transition of anatase to rutile. It was found that the anatase phase in the samples containing 5–15 wt % SiO_2 was stable up to 1000°C. The stabilization of highly dispersed anatase particles facilitated the retention of the developed fine-pore structure of xerogels with a pore diameter of 4 nm up to 900°C.

REINFORCED NICKEL CATALYSTS FOR STEAM REFORMING OF METHANE TO SYNTHESIS GAS

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React. Kinet. Catal. Lett.,
97(3) (2009) pp. 353-370.

XRD, mercury porosimetry, low-temperature nitrogen adsorption and electron microscopy were used to study peculiarities of the formation of reinforced composite nickel catalysts. The catalysts were prepared by sintering powdered metallic nickel with a supported nickel catalyst (GIAP-3 or NIAP-18) applied to a reinforcing stainless steel gauze. It was found that a metal matrix, in the pores of which supported catalyst particles were distributed, was formed in the composite catalysts. The NIAP-18-based catalyst exceeded the GIAP-3-based catalyst in activity toward the methane steam reforming. The NIAP-18-based catalyst was as active as the Cr_2O_3-doped NIAP-18-based catalyst, but showed a worse coke-resistance. A chromium oxide additive increased the activity of the GIAP-3-based catalyst.

DESIGN OF Pt–Sn CATALYSTS ON MESOPOROUS TITANIA FILMS FOR MICROREACTOR APPLICATION

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Catal. Today,
147(suppl. 1) (2009) pp. S81-S86.

A new generation of nanostructured Pt–Sn/TiO_2 catalytic thin films has been developed by deposition of Pt–Sn mixed-metal precursors from organic solvents on mesoporous TiO_2/Ti films with a thickness of 200–300 nm. The titania sol was obtained by templating a TiO_2 precursor with Pluronic F127 surfactant. The films were prepared on Ti substrates by spin-coating. The influence of the F127/Ti ratio in the range between 0.006 and 0.050, the pH of the titania sol between 1.5 and 2.0, and the aging time between 8 and 240 h on the morphology and porous structure of titania films was investigated. A TiO_2 film with the highest degree of the long-order structure was obtained at a surfactant/Ti molar ratio of 0.009, a pH of 1.5, and an aging time of 24 h. This film has a hexagonal pore structure with a mean pore size of 3.5 nm and a porosity of 25%. A powder titania support with a similar chemical composition and morphology was also produced and used for optimization of an active component deposition. The Pt–Sn carbonyl $[Pt_3(CO)_3(SnCl_3)_2(SnCl_2{\cdot}H_2O)]_n^{-2n}$ clusters were synthesized separately from monometallic precursors. They were loaded onto the TiO_2 supports by impregnation or adsorption. The adsorption of the Pt–Sn precursor for 24 h from an ethanol solution with concentrations of Pt and Sn of 2.0 and 1.2 mg/ml, respectively, followed by a vacuum treatment at 463 K, resulted in Pt–Sn nanoparticles embedded in the mesoporous titania network. An average size of bimetallic nanoparticles was 1.5–2 nm with a narrow particle size distribution. A reaction rate in terms of TOF between 0.2 and 3.3 min^{-1} was observed in the hydrogenation of citral over the Pt–Sn/TiO_2 catalysts. The selectivity to the unsaturated alcohols was as high as 90% at a citral conversion above 95%.

STUDY OF CHROMIUM ION SORPTION ON MODIFIED SILICA ORDERED MATERIALS

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Bull. Lebedev Phys. Inst.,
36(12) (2009) pp. 365-366.

The sensitivity of chemical sensors depends on the number of receptors on the surface; hence, an important role is played by the accessible support surface. Films based on silica mesoporous mesophase materials feature the highest specific surface area for silica materials, and their walls can be modified by various functional groups. In this paper, the authors propose to use mesoporous mesophase films as supports of sensitive elements for Cr^{3+} ions.

Carbon and Carbon Related Materials

SYNTHESIS OF MESOPOROUS CARBONS BY LEACHING OUT NATURAL SILICA TEMPLATES OF RICE HUSK

P.M. Yeletsky, V.A. Yakovlev, M.S. Melgunov, V.N. Parmon

Microporous Mesoporous Mater.,
121(1-3) (2009) pp. 34-40.

Carbons with developed mesoporosity were obtained from rice husk (RH) according to a three step procedure, that includes carbonization in fluidized catalyst bed reactor at 450–600°C or by pyrolysis in nitrogen atmosphere at 400–700°C, and removal of mineral (silica) part due to interaction with equimolar mixture of K_2CO_3 and Na_2CO_3 at 750–1000°C followed by washing out of soluble K and Na silicates. The obtained carbons have specific surface area up to 1676 $m^2\ g^{-1}$, and total pore volume up to 1.54 ml g^{-1}, more than 80% of which are mesopores. The structure and morphology of the carbons were characterized by N_2 adsorption (77 K), XRD and TEM. The influence of the synthetic parameters such as temperature of the thermal treatment, composition of blends with carbonates was investigated. The main approach was that the SiO_2 of RH appears to be able to play a template role for the preparing carbonaceous materials. Thus it seems possible to control the textural properties of preparing carbons via affecting on the SiO_2 template properties by preparation conditions.

The carbons are intended to be used as catalyst supports and adsorbents.

THE ROLE OF MOLYBDENUM IN Fe–Mo–Al_2O_3 CATALYST FOR SYNTHESIS OF MULTIWALLED CARBON NANOTUBES FROM BUTADIENE-1,3

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Appl. Catal., A,
363(1-2) (2009) pp. 86-92.

MoO_3–Fe_2O_3–Al_2O_3 catalysts were prepared by co-precipitation of mixed hydroxides from soluble salts. It was found that the 55%Fe_2O_3–Al_2O_3 aluminum–iron catalyst calcined in air at 500–700°C consisted of a solid solution based on hematite where a part of iron atoms was substituted for aluminum. The modification of the aluminum–iron catalyst with molybdenum results in the formation of a solid solution based on hematite where a part of iron atoms is substituted for aluminum and molybdenum ions. At 700°C the MoO_3–Fe_2O_3–Al_2O_3 catalysts are reduced under the action of the feed. The dependence of the multiwalled carbon nanotube (MWNT) yield over MoO_3–Fe_2O_3–Al_2O_3 catalysts on the molybdenum concentration has a maximum. Small concentrations of MoO_3 (up to 6.5 wt.%) added to the aluminum–iron catalyst increase the dispersity of the active metal particles and alter their properties due to the formation of a Fe–Mo alloy. Its formation leads to the decrease of the nanotube growth rate and makes the catalyst more stable. The overall yield of the carbon nanotubes increases. Further increase of the molybdenum concentration leads to the decrease of the nanotube yield because the catalytically active Fe–Mo particles are enriched too much with molybdenum. In addition, excess molybdenum forms molybdenum carbide α-Mo_2C, in which part of the molybdenum atoms are substituted for iron atoms, and which is inactive in the MWNT growth.

STRUCTURE AND ELECTRICAL CONDUCTIVITY OF NITROGEN-DOPED CARBON NANOFIBERS

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Carbon,
47(8) (2009) pp. 1922-1929.

The effect of nitrogen concentration in carbon nanofibers (CNFs) on the structural and electrical properties of the carbon material was studied. CNFs with nitrogen concentration varied from 0 to 8.2 wt.% (N-CNFs) with “herringbone” structure were prepared by decomposition of ethylene and ethylene mixture with ammonia over 65Ni–25Cu–10Al_2O_3 (wt.%) catalyst at 823 K. Detailed investigation of the CNFs and N-CNFs by XPS, FTIR and Raman spectroscopy showed that the nitrogen introduction in carbon material distorts the graphite-like lattice and increases the structure defectiveness. Both effects become more significant as the nitrogen concentration in N-CNFs grows.

The electrical conductivity of N-CNFs with different nitrogen concentrations is caused by the competition of the nanofiber graphite-like structure disordering after introduction of nitrogen atoms and doping of an additional electron into the delocalized π-system of the graphite-like material. As a result, the maximum electrical conductivity among the samples studied was observed at nitrogen concentration in N-CNFs equal to 3.1 wt.%.

CHIRAL CARBON NANOSCROLLS WITH A POLYGONAL CROSS-SECTION

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Carbon,
47(13) (2009) pp. 3099-3105.

Carbon nanoscrolls with a unique polygonal cross-section have been discovered in films synthesized by chemical vapor deposition in a hydrogen-methane gas mixture activated by a direct current discharge. The structure type has been revealed by high resolution transmission electron microscopy (TEM) combined with selected area electron diffraction patterning. The peculiarities observed in the TEM diffraction contrast have allowed for the identification of the nanoscroll shape as a twisted polygon. A three-dimensional atomic structure model of these complex nanocarbon species is based on a tilting series and the electron diffraction patterns. The proposed model adequately describes the structure of the nanoscrolls and the formation mechanism of the characteristic diffraction contrast in their corresponding electron microscopy images. The results point to the specific features of the electronic properties of nanoscrolls.

OPTICAL LIMITING AND BLEACHING EFFECTS IN A SUSPENSION OF ONION-LIKE CARBON

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Quantum Electron.,
39(4) (2009) pp. 342-346.

The effect of nanosecond laser pulses (λ = 1064 nm) on the optical properties of onion-like carbon (OLC) prepared by high-temperature vacuum annealing of detonation nanodiamond and dispersed in N,N-dimethylformamide (DMF) has been studied. The results demonstrate that, under low-intensity irradiation, the OLC suspension displays optical limiting behaviour. Increasing the incident intensity leads to bleaching of the suspension in the visible and near-IR spectral regions.

PHOTOINDUCED TRANSPARENCY OF A SUSPENSION OF ONION-LIKE CARBON NANOPARTICLES

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Tech. Phys. Lett.,
35(2) (2009) pp. 162-165.

The phenomenon of photoinduced transparency in a suspension of onion-like carbon (OLC) nanoparticles in N,N-dimethylformamide (DMF) under the action of high-power laser radiation (λ = 1064 nm) have been observed. The OLC particles were prepared from detonation nanodiamonds (NDs) by means of high-temperature annealing in vacuum and then ultrasonically dispersed in DMF. Upon exposure to laser radiation, the optical density of the suspension significantly decreases in the visible and near-infrared range, but increases in the range of wavelengths below 400 nm.

LOW-FREQUENCY (10-50 kHz) IMPEDANCE OF POLYSTYRENE-ONION-LIKE-CARBON COMPOSITES

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Tech. Phys. Lett.,
35(1) (2009) pp. 85-88.

The impedance of polystyrene-onion-like-carbon (PS-OLC) composites in the low-frequency (10-50 kHz) range has been studied as a function of the OLC weight fraction in the material. The composites were fabricated by rolling of PS filled with OLC powder obtained through the annealing of detonation nanodiamonds at 2140 K. The homogeneity of OLC distribution in the PS matrix has been studied as dependent on the number of rolling stages. It is established that the percolation threshold in PS-OLC composites is achieved at an OLC content of 35-40 wt %.

CATALYST STRUCTURE - PERFORMANCE TRENDS FOR SIBUNIT CARBON BASED CATHODES FOR PROTON EXCHANGE MEMBRANE FUEL CELLS

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ECS Trans.,
25(1) (2009) pp. 1908-1913.

Synthetic mesoporous carbon - Sibunit - offers a unique possibility to tune porous structure of the catalyst support without changing other textural parameters, enabling fundamental studies of the support structure - catalyst performance relationship. Three different Sibunit carbons with similar surface area of ca. 450-500 m^2/g but widely varying pore size distribution were used for the preparation of the 40 wt% Pt/C catalysts for the cathode in H_2/O_2 PEMFC. Porous structure of the catalyst support was shown to affect both Pt utilization and the transport properties of the catalyst. Presence of the small - 3-4 nm in diameter - pores was shown to increase the utilization of Pt, while impairing the transport properties of the catalyst. For Sibunit supports, featuring large meso- and macropores, a lower degree of Pt utilization was found. Electrodes prepared using the latter catalysts exhibit excellent transport properties, comparable to those of the commercial benchmark Vulcan XC72 material.

SIBUNIT CARBON-BASED CATHODES FOR PROTON-EXCHANGE-MEMBRANE FUEL CELLS

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Fuel Cells,
9(4) (2009) pp. 439-452.

Preparation, characterisation and evaluation of a novel cathode catalyst for H_2/O_2 PEMFC - 40 wt.% Pt/Sibunit 1562P carbon - is presented. The performance of the cathodes based on the latter material is compared to that of a widely used commercially available reference material - 40 wt.% Pt/Vulcan XC 72R - Hispec 4000 (Johnson Matthey, UK). It has been found that the mass activity of the 40 wt.% Pt/Sibunit 1562P catalyst exceeds that of a commercial benchmark by a factor of 2 due to a better dispersion of the Pt metal realised in the Sibunit-supported catalyst. Mass transport properties of the two catalysts studied are virtually equal.

NEW EFFECTIVE CATALYSTS BASED ON MESOPOROUS NANOFIBROUS CARBON FOR SELECTIVE OXIDATION OF HYDROGEN SULFIDE

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Appl. Catal., B,
85(3-4) (2009) pp. 180-191.

The systems based on granular mesoporous nanofibrous carbonaceous materials (NFC) synthesized by decomposition of hydrocarbons over nickel-containing catalysts are promising catalysts for the selective oxidation of hydrogen sulfide. Sample series of nanofibrous carbon with three main types of their fiber structures and different content of the metal catalyst inherited from the catalyst for their synthesis were studied in the reaction of direct oxidation of hydrogen sulfide into sulfur. The correlation between NFC structure and its activity and selectivity in hydrogen sulfide oxidation was determined. The metal inherited from the initial catalyst for synthesis of NFC influences the activity and selectivity of the resulting carbon catalyst. The particular influence is observed with the catalyst withdrawn from the synthesis reactor at the stage of stationary operation of the metal catalyst (low specific carbon yields per unit weight of the catalyst). The presence of the metal phase results in an increase in the carbon catalyst activity and in a decrease in the selectivity to sulfur. NFC samples with the highest activity and selectivity are those with graphite planes perpendicular to the axis of the fibers and nanotubes. Cabon nanotubes have high selectivity, though samples obtained on copper-nickel catalysts possess also high activity. The perspective NFC catalysts provide the high conversion and selectivity with the great excess of oxygen in the reaction mixture, which are almost independent of the molar oxygen/hydrogen sulfide ratio.

ONION-LIKE CARBON IN MICROWAVES: ELECTROMAGNETIC ABSORPTION BANDS AND PERCOLATION EFFECT

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J. Nanoelectron. Optoelectron.,
4(2) (2009) pp. 257-260.

Microwave absorption properties of onion-like carbons (OLC) have been studied in microwave frequency range. A consistent analysis of the electromagnetic (EM) attenuation provided by OLC-based suspensions with variable concentration of OLC has been carried out in $K\alpha$-band (26-37 GHz). The first study of spectral features of the given suspensions (7.2-28.6 volume percents of OLC stuff in vaseline) demonstrates that the percolation threshold of OLC is strongly dependent on the average nanocarbon cluster size. It has been found also that raising the annealing temperature of a precursor nanodiamond leads to the enhanced absorption ability of onion-like carbon. The way to manipulate the OLC absorption ability through the OLC cluster size and nanodiamond annealing temperature is proposed for producing the effective EM coatings.

NANO-SCALED ONION-LIKE CARBON: PROSPECTIVE MATERIAL FOR MICROWAVE COATINGS

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Metamater.,
3(3-4) (2009) pp. 148-156.

Microwave absorption properties of a novel electromagnetic material – onion-like carbon (OLC) produced by annealing of detonation nanodiamonds in vacuum – have been studied in microwave frequency range. The OLC electromagnetic (EM) absorption ability can be optimized by varying the nanocarbon cluster size and nanodiamond annealing temperature so that effective EM coatings can be produced. Attenuation of EM waves in the 26–38 GHz frequency range had been studies for both OLC powder and polymer composites. For powder samples it was concluded that OLC aggregates with higher conductivity (higher annealing temperature of the precursor nanodiamond particles) and larger aggregate sizes provide higher efficiency for attenuation of the EM radiation. At the same time, when dispersed in a polymer matrix, more efficient shielding properties had been observed for OLC aggregates of the smallest sizes as compared to larger-sized aggregates at the same OLC loading. This is attributed due to the better dispersion and formation of a continuous conductive network by smaller aggregates (reaching the percolation threshold).

ONION-LIKE CARBON BASED POLYMER COMPOSITE FILMS IN MICROWAVES

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Solid State Sci.,
11(10) (2009) pp. 1762-1767.

The electromagnetic (EM) absorbing properties of a novel technological material – onion-like carbon (OLC) and OLC-based polymer composites are studied experimentally in microwave frequency range. It has been found strong dependence of the electromagnetic response of the nanocarbon fillers and corresponding composites on the annealing temperature of the precursor detonation nanodiamonds and the cluster size of the OLC particles. The role of different hosts in the design of effective EM absorbing materials has also been discussed. Reported results show a high potential of onion-like carbon for the design of EM lossy materials in Ka band (26-37 GHz).

DIELECTRIC PROPERTIES OF ONION-LIKE CARBON BASED POLYMER FILMS: EXPERIMENT AND MODELING

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Solid State Sci.,
11(10) (2009) pp. 1828-1832.

Dielectric analysis of a novel polymer composite based on onion-like carbon (OLC) has been carried out by varying the temperature between 240 and 460 K within a wide frequency range (20 Hz–1 MHz). A small effect of the OLC inclusions on the beta transition of OLC/polymethylmethacrylate (PMMA) composite has been observed, which indicates a weak affinity of the OLC to the host matrix. At the same time, an increase in concentration of the OLC has been found to strongly influence the glass and melting temperatures of the nanocomposites. These structural properties of the OLC/PMMA composite materials should be taken into account when a new family of effective wide-band electromagnetic materials are designed and fabricated.

DIELECTRIC PROPERTIES OF MWCNT BASED POLYMER COMPOSITES CLOSE AND BELOW PERCOLATION THRESHOLD

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Phys. Status Solidi C,
6(12) (2009) pp. 2814-2816.

Results of broadband (20 Hz-1 MHz) dielectric spectroscopy of multi-wall carbon nanotubes/polymethylmethacrylate composites with low loading of the carbon fillers (up to 0.5 wt%) are reported. The temperature behaviour of complex dielectric permittivity of MWCNT/PMMA composites with 0.25 wt% is similar to the pure PMMA polymer. However, the dielectric permittivity and electric conductivity of composites with 0.5 wt% are very high and both parameters are almost temperature independent. The high complex dielectric permittivity of these composites indicates the onset of percolation threshold. The relaxation time of β relaxation is shorter in MWCNT/PMMA composites than in pure PMMA.

DIELECTRIC RESPONSE OF ONION-LIKE CARBON-BASED POLYMETHYL METHACRYLATE COMPOSITES

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J. Nanoelectron. Optoelectron.,
4(2) (2009) pp. 261-266.

The dielectric properties of polymethyl methacrylate films with embedded onion-like carbon particles have been studied from 20 Hz to 1 MHz to investigate the structural relaxations of the composite material by varying the temperature between 240 and 520 K. The temperature behaviour of the complex permittivity at a given frequency is found to be strongly dependent on the nanocarbon concentration. A small effect of the onion-like carbon concentration on the beta transition of the polymer composite has been observed, which indicates that the included onion-like carbon particles do not block the rotational degrees of freedom of the ester groups along the polymer chains. By contrast, it has been found that embedding the nanocarbon fillers to the polymethyl methacrylate matrix changes significantly the alpha transition and the melting temperatures in a non-monotonous way.

INFLUENCE OF HUMIDITY ON DIELECTRIC PROPERTIES OF PMMA NANOCOMPOSITES CONTAINING ONION-LIKE CARBON

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Ferroelectr.,
391(1) (2009) pp. 131-138.

Results of broadband dielectric spectroscopy (20 Hz-1 MHz) of a novel polymer composite based on onion-like carbon (OLC) are presented. The measurements were performed by cooling and heating in a wide temperature range (240 K-420 K). After heating the dielectric permittivity strongly increases, β relaxation slows down while γ relaxation becomes negligible. The observed behaviour of relaxations suggests that dielectric properties of presented composite without additional heating are strongly influenced by extrinsic humidity. The increase of the OLC fillers concentration (up to 1 wt%) has been found to strongly increases the glass transition temperature of OLC-based polymethyl methacrylate (PMMA) composite, however, an effect of the OLC inclusions on the beta transition of the given nanocomposites is less expressed.

COLLOIDAL STABILITY OF MODIFIED NANODIAMOND PARTICLES

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Diamond Relat. Mater.,
18(4) (2009) pp. 620-626.

Colloidal stability is one of the critical factors for the use of nanodiamonds as potential enterosorbents. Although nanodiamonds are believed to be a promising enterosorbent, colloidal stability in hydrosols of raw polydispersed nanodiamonds produced by detonation is typically low. Surface modification and fractionation significantly improves colloidal stability of nanodiamond suspensions within the physiological pH range. The modification of nanodiamonds can be completed either by physical means, i.e., plasma treatments, or by chemical methods. In the current paper an analysis of the colloidal stability of detonation nanodiamonds hydrosols, which have undergone different surface modifications, is presented based on zeta potential measurements and titration experiments.

ELECTROMAGNETIC SHIELDING PROPERTIES OF MWCNT/PMMA COMPOSITES IN Ka-BAND

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Phys. Status Solidi B,
246(11-12) (2009) pp. 2662-2666.

A novel lightweight multi-wall carbon nanotubes (MWCNT) based polymethylmetacrylate (PMMA) composite is shown to present high electromagnetic (EM) shielding. EM shielding effectiveness (SE) was investigated in the frequency range 26-37 GHz (Kα-band). It was found that SE of the given composites strongly depends on MWCNT content. The highest EM attenuation reaching -37 dB at 28 GHz was observed for polymer film comprising 10 wt.% of MWCNT. Data on SE correlates well with the electrical conductivity measured by four-probe technique and by broadband dielectric spectroscopy (20 Hz-1 MHz). Both microwave and low-frequency analysis demonstrate low percolation threshold (lower than 1 wt.%) for the produced MWCNT/PMMA composites. At the same time significant electromagnetic interference absorbance demonstrates the contribution of the absorbance mechanisms specific for carbon nanostructures different of the main conductance mechanism.

MULTI-WALL CARBON NANOTUBES IN MICROWAVES

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Recent Advances in Nanotechnology: Electrical and Computer Engineering Series,
A Series of Reference Books and Textbooks,
2009, pp. 92-95.

The electromagnetic (EM) response of multi-wall carbon nanotubes (MWCNT) prepared by chemical vapor decomposition (CVD) method has been analyzed in the microwave frequency range. EM absorption properties of MWCNT depend on their medium diameter related to their conductivity, concentration of highly dispersed Fe-Co metal particles localized inside and in the tips of nanotube. MWCNT display high EM absorption in microwaves and can be used as fillers for the design of the effective EM coatings.

INFLUENCE OF CURVATURE OF GRAPHENE LAYERS OF MULTI-WALLED CARBON NANOTUBES ON ELECTRICAL PROPERTIES

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Int. J. Nanosci.,
8(1-2) (2009) pp. 1-4.

Temperature and magnetic field dependences of conductivity of multi-walled carbon nanotubes (MWNTs) with different average outer diameter were investigated. The authors separated out the quantum corrections to magnetoconductivity for interaction electrons and determined the dependence of constant of electron–electron interaction λ versus curvature of graphene layers in MWNTs.

ELECTROPHYSICAL PROPERTIES OF MULTIWALLED CARBON NANOTUBES WITH VARIOUS DIAMETERS

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Phys. Status Solidi B,
246(11-12) (2009) pp. 2641-2644.

Temperature σ (T) and magnetic field σ (B) dependences of the conductivity σ of multiwalled carbon nanotubes (MWNTs) with different average outer diameter d have been investigated. From the experimental data the authors have separated the quantum corrections to σ (T) and σ (B). It has been discovered that only quantum corrections for interaction electrons (QCIE) to σ (B) take place. It is associated to the absence of the bulk formations of amorphous carbon in produced MWNTs. From the data of QCIE we have estimated the constant of electron-electron interaction λ which is negative (attraction between electrons) and gradually falling with decrease in MWNTs d. Influence of defectiveness and structure of MWNTs on electrophysical properties is discussed.

TEMPERATURE DEPENDENCIES OF CONDUCTIVITY OF MULTI-WALLED CARBON NANOTUBES AND ONION-LIKE CARBON IN DIFFERENT GASEOUS MEDIUM

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Int. J. Nanosci.,
8(1-2) (2009) pp. 19-22.

The conductivity of multi-walled carbon nanotubes (MWNTs) and onion-like carbon (OLC) in different gaseous media: helium, air, oxygen, hydrogen and methane is measured. The MWNTs had different mean outer diameters varied from 5 to 15 nm. The OLC was synthesized at 1400-1850 K. The adsorption of nitrogen, oxygen, hydrogen and methane leads to decrease of conductivity because these gases are the donors of electron for semi-metallic band structure of investigated objects.

PREPARATION AND CHARACTERIZATION OF NANOPOROUS CARBON SUPPORTS ON A NICKEL/SIBUNIT CATALYST

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Kinet. Catal.,
50(4) (2009) pp. 597-605.

A nanoporous composite carbon material was developed; this material was prepared by the synthesis of catalytic filamentous carbon (CFC) on a Ni catalyst supported onto the Sibunit carbon support. The texture characteristics (specific surface area and pore structure) of this material were studied. The effects of the conditions of supporting bivalent nickel compounds from aqueous or water-ethanol solutions in the presence of urea and the pretreatment of the parent Sibunit (oxidation and reduction) on the yield of synthesized carbon were considered. The distribution of Ni inside a Sibunit granule was studied using energy dispersive X-ray microanalysis. The surface morphology of the Ni/Sibunit catalyst, as well as the synthesized carbon layer, was studied by scanning electron microscopy. It was found that a maximum yield of carbon (50–60 g/(g Ni)) was obtained on the precipitation of nickel compounds from water-ethanol solutions with an ethanol concentration of 5 to 50 vol %. The preliminary surface oxidation or reduction of the parent Sibunit resulted in a considerable decrease in the yield of carbon (by a factor of 2 or more). The parent Sibunit phase occurred within the prepared nanoporous carbon material, whereas a shell formed by CFC occurred on the outside.

PREPARATION AND CHARACTERIZATION OF SUPPORTS WITH A SYNTHESIZED LAYER OF CATALYTIC FILAMENTOUS CARBON: IV. SYNTHESIS OF CARBON NANOFIBERS ON A Co/Al_2O_3 CATALYST

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Kinet. Catal.,
50(6) (2009) pp. 899-906.

A comparative study of the synthesis of carbon layers, including catalytic filamentous carbon, on the surface of various alumina modifications was made. The synthesis was performed by the pyrolysis of alkanes (a propane-butane mixture) on Co/Al_2O_3 supported catalysts. The texture characteristics (specific surface area and pore structure) of the starting supports and adsorbents with a synthesized carbon layer were studied. The surface morphology of Co/Al_2O_3 catalysts and the synthesized carbon deposits was studied by scanning electron microscopy. It was found that carbon nanofibers were formed only on the catalysts prepared by the homogeneous precipitation of Co compounds onto the surface of macroporous α-Al_2O_3, whereas carbon deposits on mesoporous aluminum oxides did not exhibit a pronounced fibrous structure. The applicability of $C/Co/Al_2O_3$ carbon-containing adsorbents to the immobilization of the nitrile hydratase enzyme and the preparation of a biocatalyst for acrylonitrile hydration to acrylamide was considered.

ADSORPTIVE IMMOBILIZATION OF ENZYMATIC ACTIVE SUBSTANCES ON ALUMINA–SILICA FOAM COATED BY CARBON NANOFIBERS

G.A. Kovalenko, L.V. Perminova, T.V. Chuenko, N.A. Rudina

Carbon,
47(2) (2009) pp. 420-427.

The adsorption abilities of an alumina–silica foam coated by a layer of catalytic filamentous carbon (CFC/ceramics) were studied with respect to enzymatic active substances (EAS) - enzymes glucoamylase and invertase, intracellular compartments of baker’s yeast and whole non-growing cells of various microorganisms (*Saccharomyces cerevisiae, Rhodococcus* sp., *Arthrobacter* sp.). The activity and stability of heterogeneous biocatalysts prepared by EAS adsorption on the CFC/ceramics were studied in hydrolysis, oxidation and isomerization reactions. Glucoamylase was found to retain up to 20% of the activity of a soluble enzyme after adsorption on the CFC/ceramics. Invertase and invertase-active yeast autolysate retained practically all their activity. The enzymatic activities of non-growing cells of baker’s yeast and *Rhodococcus* increased after adsorption on CFC/ceramics by a factor of 1.5–1.8 and 3.7, respectively. A “glucoamylase on CFC/ceramics” biocatalyst for the hydrolysis of dextrin was shown to have high long-term stability, retaining its activity after storage for 10–12 months at ambient temperature. A “yeast autolysate on CFC/ceramics” biocatalyst for sucrose inversion retained >50% of the activity after 2–3 month storage at ambient temperature. A “non-growing bacteria *Arthrobacter* sp. on CFC/ceramics” biocatalyst for glucose isomerization retained half its activity after 12 h use at 70°C.

TUNING SURFACE MORPHOLOGY OF INORGANIC SUPPORTS FOR ADSORPTIVE IMMOBILIZATION OF ENZYMATIC ACTIVE SUBSTANCES

G.A. Kovalenko, L.V. Perminova, T.V. Chuenko, N.A. Rudina

Compos. Interfaces,
16(4) (2009) pp. 293-305.

A comprehensive study of composite carbon-mineral supports was performed to determine specific features of the adsorptive immobilization for such enzymes as glucoamylase and invertase. The catalytic properties (activity and stability) of immobilized enzymes were found to depend significantly on the morphology of the carbon layer synthesized on the support surface. After tuning the surface morphology towards immobilizing enzymes, a layer of catalytic filamentous carbon (CFC) was found to ensure the highest stability and activity of the prepared biocatalysts due to its mesoporous structure and optimal hydrophilic-hydrophobic balance. The supports used for immobilization of enzymes can be arranged in the following order of observed enzymatic activity and stability: supports coated by graphite-like carbon layer ≈ non-carbonized supports << CFC-coated supports.

SYNTHESIS OF CATALYTIC FILAMENTOUS CARBON BY THE PYROLYSIS OF ALKANES ON ALUMINA–SILICA FOAM SUPPORTING NICKEL NANOPARTICLES

G.A. Kovalenko, N.A. Rudina, T.V. Chuenko, D.Yu. Ermakov, L.V. Perminova

Carbon,
47(2) (2009) pp. 428-435.

Nickel compounds were deposited on alumina–silica ceramic foam by different methods, in particular a homogeneous precipitation and a heterogeneous sol-gel processing to prepare catalyst for pyrolysis of alkanes (methane, propane–butane). Carbon deposits, in particular catalytic filamentous carbon (CFC) were synthesized on the Al/Si surface during the catalytic pyrolysis. Comparative analysis of the textural parameters of the initial support and composite carbon-mineral adsorbents was carried out. The effect of the method of catalyst preparation upon the morphology of supported nickel compounds and synthesized carbon deposits was examined in scanning electron microscope. The carbon yield for catalysts prepared by homogeneous precipitation was shown to be an order of magnitude higher than for the catalysts prepared by other methods. A uniform distribution of nickel nanoparticles deposited on the Al/Si foam by homogeneous precipitation led to the formation of a mesoporous CFC-layer with thickness of 0.5–2.0 μm and specific surface area ca. 500 m^2/g due to chaotic interlacing of carbon nanofibers with a diameter of 50–100 nm and length exceeding 1 μm.

Composite Sorbents

OPTIMAL ADSORBENT FOR ADSORPTIVE HEAT TRANSFORMERS: DYNAMIC CONSIDERATIONS

Yu.I. Aristov

Int. J. Refrigeration,
32(4) (2009) pp. 675-686.

Kinetic properties of the adsorbent can strongly affect dynamics of an adsorptive heat transformer. In this paper the authors describe a current methodology and start discussing a new approach for dynamic characterization and optimization of the system “adsorbent - heat exchanger” under conditions typical for heat transformation. Effect of the adsorbent nature, its grain size, residual non-adsorbable gas, heating rate, local shape of adsorption isobar on the adsorption dynamics and specific cooling (heating) power was studied for promising adsorbents of water (Fuji silica RD, FAM-Z02, SWS-1L). Based on these results some demands to an optimal adsorbent from the dynamic point of view have been discussed.

“SALT IN A POROUS MATRIX” ADSORBENTS: DESIGN OF THE PHASE COMPOSITION AND SORPTION PROPERTIES

Yu.I. Aristov, L.G. Gordeeva

Kinet. Catal.,
50(1) (2009) pp. 65-72.

This review formulates the concept of target-oriented synthesis of two-component “salt in a porous matrix” (SPM) adsorbents designed for processes such as gas dewatering, moisture control, heat conversion in adsorption heat pumps, and equilibrium shifting in catalytic reactions. In terms of this approach, the requirements imposed on an ideal adsorbent, which is optimal for a particular application, are initially formulated; then, a material with nearly optimal properties is synthesized. Methods for the target-oriented synthesis of SPM adsorbents with the required properties are considered. The effects of the nature of the salt and the matrix, the salt content, the pore size of the matrix, and the synthesis conditions on the phase composition and adsorption properties of the SPM adsorbents are studied.

ADSORPTION PROPERTIES OF COMPOSITE MATERIALS (LiCl + LiBr)/SILICA

L.G. Gordeeva, A.D. Grekova, T.A. Krieger, Yu.I. Aristov

Microporous Mesoporous Mater.,
126(3) (2009) pp. 262-267.

This paper presents experimental data on synthesis and the phase composition of novel composites “(LiCl + LiBr) confined to the silica gel pores” as well as their sorption equilibrium with water and methanol vapour. Phase transformation of the composites during methanol sorption was characterized *in situ* by an X-ray diffraction analysis. The isobars of sorption on the composites were measured in the temperature range $T = 303–383$ K at the methanol and water pressure $P = 107$ and 13 mbar, respectively, using a thermo-gravimetric technique. It was shown that the formation of solid solutions of LiCl and LiBr took place in limited ranges of LiBr ($C_{\mathrm{Br}} = 0–11$ mol.%) and LiCl ($C_{\mathrm{Cl}} = 0–36$ mol.%) content. These solutions absorbed water (methanol) at temperature that was intermediate between the individual solvation temperatures for confined LiCl and LiBr. In the composites with LiCl/LiBr molar ratio between the ranges of solubility a mixture of two solid solutions was formed. Each solution absorbed water (methanol) independently at a certain temperature. The use of the binary LiCl–LiBr system confined to the silica pores can be an effective tool for designing innovative materials with predetermined sorption properties.

SIMULATION OF A SOLID SORPTION ICE-MAKER BASED ON THE NOVEL COMPOSITE SORBENT “LITHIUM CHLORIDE IN THE SILICA GEL PORES”

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Appl. Them. Engn.,
29(8-9) (2009) pp. 1714-1720.

In this paper, a novel composite sorbent ‘‘lithium chloride in silica gel pores” is proposed for application in solar-powered adsorptive ice makers. A mathematical model was used in order to calculate the performance of an ice-maker using this material as adsorbent and methanol as adsorbate. The results of the model showed that a maximum solar coefficient of performance (COPs) of 0.33 and a maximum daily ice production (DIP) of 20 kg m^{-2} can be obtained for an ice-maker equipped with a solar collector area of 1.5 m^2 and 36 kg of adsorbent material. Such performance is noticeably higher than those obtained using commercial activated carbon, that is the adsorbent mostly proposed till now.

COMPOSITE SORBENT OF METHANOL “LITHIUM CHLORIDE IN MESOPOROUS SILICA GEL” FOR ADSORPTION COOLING MACHINES: PERFORMANCE AND STABILITY EVALUATION

L.G. Gordeeva, Yu.I. Aristov, A. Freni*, G. Restuccia* (**ITAE-CNR, Messina, Italy*)

Ind. Chem. Engn. Res.,
48(13) (2009) pp. 6197-6202.

In this paper, a novel composite sorbent of methanol “lithium chloride in mesoporous silica gel” is proposed for adsorption cooling machines. Methanol sorption isotherms were measured by a thermogravimetric technique. This composite demonstrated outstanding methanol sorption ability (up to 0.6 g of methanol per 1 g of dry sorbent). The thermodynamic coefficient of performance (COP), calculated by mathematical modeling for a basic cooling cycle, can reach 0.72 at desorption temperature of 343 K. The real performance was measured by testing this novel material in a lab-scale adsorption chiller. The specific cooling power of 210-290 W/kg and real cooling COP of 0.32-0.4 were obtained. Cycling stability of the sorbent was successfully verified. Such results indicate that the new composite sorbent “lithium chloride in mesoporous silica gel” can be recommended for application in adsorption cooling machines driven by low temperature heat.

KINETICS OF WATER ADSORPTION/DESORPTION UNDER ISOBARIC STAGES OF ADSORPTION HEAT TRANSFORMERS: THE EFFECT OF ISOBAR SHAPE

I.S. Glaznev, D.S. Ovoshchnikov, Yu.I. Aristov

Int. J. Heat Mass Transfer,
52(7-8) (2009) pp. 1774-1777.

Effect of the shape of equilibrium adsorption isobar on dynamics of water adsorption/desorption under isobaric stages of a basic cycle of an adsorption heat transformer has been studied. Selective water sorbent SWS-1L ($CaCl_2$ confined to mesoporous silica gel) was used for these tests as its water adsorption isobars have segments with both convex and concave shapes. From these experiments the conclusion has been drawn that the dynamics of adsorption on a single adsorbent grain is closely linked with the shape (convex or concave) of the segment of water adsorption isobar between initial and final temperatures of the isobaric process. In particular, under the same boundary conditions desorption is faster than adsorption for a concave isobar segment and vice versa for a convex one.

A NEW GENERATION COOLING DEVICE EMPLOYING $CaCl_2$-IN-SILICA GEL-WATER SYSTEM

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Yu.I. Aristov (**Kiushi University, Kiushi, Japan*)

Int. J. Heat&Mass Transfer,
52(1-2) (2009) pp. 516-524.

This article presents the dynamic modeling of a single effect two bed adsorption chiller utilizing the composite adsorbent “$CaCl_2$ confined to KSK silica gel” as adsorbent and water as adsorbate, which is based on the experimentally confirmed adsorption isotherms and kinetics data. Compared with the experimental data of conventional adsorption chiller based on RD silica gel + water pair, the authors found that the new working pair provides better cooling capacity and performances. From numerical simulation, it is also found that the cooling capacity can be increased up to 20 percent of the parent silica gel + water adsorption chiller and the coefficient of performance (COP) can be improved up to 25% at optimum conditions. It was also demonstrated that the best peak chilled water temperature suppression, and the maximum cooling capacity can be achieved by the optimum analysis for both cycles.

WATER SORPTION ON COMPOSITE “SILICA MODIFIED BY CALCIUM NITRATE”

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Microporous Mesoporous Mater.,
122(1-3) (2009) pp. 223-228.

Composite SWS-8L was synthesized by impregnating a mesoporous silica KSK with a saturated aqueous solution of $Ca(NO_3)_2$. Its equilibrium with water vapor was studied by measuring sorption isobars and isosters at $T = 30$–150°C. The water sorption increased up to 0.2–0.3 g/g due to the presence of the salt inside the silica pores. The water uptake was not a simple addition of those by the bulk salt and the silica, because the confined salt changed its properties. The isosteric sorption heat decreased from 52 to 47 kJ/mol when the water uptake increased from 0.07 to 0.23 g/g. The X-ray diffraction study revealed no formation of crystalline dihydrate in the confined state during the hydration process that can be caused by the dihydrate dissolution inside the pores. For approximation of the sorption equilibrium, a simple analytical equation based on the Polanyi principle of temperature invariance was suggested.

Sorption kinetics was studied by the Isothermal Differential Step method at $T = 50$ and 70°C and the pressure range 10–35 mbar. Confinement of calcium nitrate to the silica pores significantly hastened the sorption with respect to the bulk salt. The hydration of the confined salt was limited by the intraparticle water diffusion instead of intrinsic chemical transformation of the bulk salt.

Comparison of the new composite adsorbent with those based on $CaCl_2$, $MgSO_4$ and LiBr confined to the same host matrix showed that SWS-8L can be envisaging further development for adsorptive air conditioning driven by low temperature heat.

A NEW HUMIDITY BUFFER FOR SHOWCASES ARTIC-1

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Stud. Conservation,
54(3) (2009) pp. 133-148.

This paper considers an efficient approach to passive humidity control for art objects in exhibition cases, picture frames and transporting containers. This approach is based on a gas–solid chemical reaction between an inorganic salt and water vapor. Requirements for hydration/dehydration reactions and salt hydrates, which are optimal for hydrostats for safe display, conservation and transportation of various works of art, ancient manuscripts, books, archival documents, etc., have been formulated. New materials for maintaining the relative humidity between 40 and 70% have been synthesized and studied. Tests on a novel material (the so-called ARTIC-1) for maintaining the relative humidity between 50 and 60% at the Museum of the History and Culture of Siberian Nations (Novosibirsk, Russia) and the State Scientific Library of the Siberian Branch of the Russian Academy of Sciences showed its feasibility for the efficient smoothing of daily and seasonal variations of the relative humidity. These trials confirmed that ARTIC-1 is an effective adsorbent for use in sufficiently airtight showcases and containers.

NEW ADSORPTIVE MOISTURE BUFFER FOR LIBRARY, MUSEUM AND ARCHIVE VALUES CONSERVATION

Yu.I. Aristov, I.S. Glaznev, V.N. Alekseev*, L.G. Gordeeva, I.V. Salnikova, I.A. Shilova***, L.P. Kundo**, B.S. Elepov*** (**State Public Scientific-Technological Library SB RAS, Novosibirsk, Russia*; ***Institute of Archaeology and Ethnography, Novosibirsk, Russia; ***Novosibirsk State University, Novosibirsk, Russia*)

Bibliosfera,
1 (2009) pp. 85-88.

A new adsorbent for maintaining air relative moisture in show-cases, boxes for deposits and containers for trans-porting library, museum and archive values is described. The results of testing this material in SPSTL SB RAS and the Institute of archaeology and ethnography of SB RAS are discussed.

NON-ADSORBABLE COMPONENT INFLUENCE ON THE DYNAMICS OF WATER SORPTION ON THE COMPOSITE ADSORBENT

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Vestnik MGU, Chemistry,
4 (2009) pp. 232-237.

The analysis of the experimental data on water adsorption in the presence of a non-adsorbable component on the composite adsorbent “silica gel impregnated with calcium chloride” for different partial pressures of water and air was performed. It was established that the mass-transfer mechanism inside a grain is Knudsen diffusion; it was shown that in the system there is formed a dynamic structure with the increased concentration of non-adsorbable component close to the external surface of grain, causing externally diffusive resistance; the coefficient of heat exchange between grain and gas phase was defined.

Catalysis by Heteropoly Compounds

HYDROCARBON OXIDATION WITH AN OXYGEN-HYDROGEN MIXTURE: CATALYTIC SYSTEMS BASED ON THE INTERACTION OF PLATINUM OR PALLADIUM WITH A HETEROPOLY COMPOUND

N.I. Kuznetsova, L.I. Kuznetsova

Kinet. Catal.,
50(1) (2009) pp. 1-10.

A series of studies of hydrocarbon oxidation by the O_2 + H_2 mixture in the presence of catalytic systems based on Pt or Pd and a heteropoly compound (HPC) is reviewed. The catalytic systems were prepared from Pd(II) complexes with the heteropoly tungstate anions $PW_{11}O_{29}^{7-}$ and $PW_9O_{34}^{9-}$, the complex salt $[Pt(NH_3)_4][H_2Mo_{12}O_{40}]_2 \cdot 7H_2O$, mixtures of H_2PtCl_4 or H_2PtCl_6 with $H_{3+n}PMo_{12-n}V_nO_{40}$ (n = 0–3) heteropoly acids, or supported platinum dispersed in HPC solutions. The interaction of metal ions and particles with HPCs in the initial state and after thermal and redox treatments was investigated by NMR, IR spectroscopy, XPS, EXAFS, HREM, and TPR. The catalytic systems were tested in the liquid-phase oxidation of alkanes, cyclohexane, cycloalkenes, benzene, toluene, and phenol with the O_2 + H_2 mixture at low temperatures. Effective supported catalysts based on platinum nanoparticles associated with the redox-active HPCs $H_3PMo_{12}O_{40}$ and $H_4PMo_{11}VO_{40}$ were prepared for gas-phase benzene oxidation into phenol. The oxidation mechanism includes the interaction between dioxygen and platinum (or palladium) and the participation of the HPC in the formation of active oxygen species of radical nature.

CATALYTIC PROPERTIES OF PLATINUM-PROMOTED ACID CESIUM SALTS OF MOLYBDOPHOSPHORIC AND MOLYBDOVANADOPHOSPHORIC HETEROPOLY ACIDS IN THE GAS-PHASE OXIDATION OF BENZENE TO PHENOL WITH AN O_2 + H_2 MIXTURE

L.I. Kuznetsova, L.G. Detusheva, N.I. Kuznetsova, S.V. Koshcheev, V.I. Zaikovsky, Yu.A. Chesalov, V.A. Rogov, V.B. Fenelonov, V.A. Likholobov* (**Institute of Hydrocarbon Processing, Omsk, Russia*)

Kinet. Catal.,
50(2) (2009) pp. 205-219.

Acid salts $Cs_xH_{3+n-x}PMo_{12-n}V_nO_{40}$ (n = 0, 1, 2, or 3; x = 2.5 or 3.5) with coprecipitated or supported platinum were studied using thermogravimetry, IR spectroscopy, and temperature-programmed reduction. The thermal region of the full stability of these salts is limited by the decomposition temperature of the corresponding acid $H_3PMo_{12}O_{40}$ (~400°C) or $H_{3+n}PMo_{12-n}V_nO_{40}$ (~300–350°C). The degree of reduction of heteropoly anions with hydrogen is regulated by temperature. Deeply reduced heteropoly anions (at 300°C) are slowly oxidized with oxygen with structure and composition regeneration. The states of molybdenum and vanadium on the surface of samples with coprecipitated platinum $Pt_{0.1}$-$Cs_{2.5}H_{0.5}PMo_{12}O_{40}$ (**1**) and $Pt_{0.1}$-$Cs_{2.5}H_{2.5}PMo_{10}V_2O_{40}$ (**2**), which were studied using XPS, correspond to reduced or reoxidized heteropoly anions in the bulk. Platinum metal particles of ~5 nm in size were observed in high-resolution TEM images obtained after the reduction and storage of sample **1** in air. A heteropoly compound forms two texture levels: spherical nanoparticles of 10–20 nm in size are collected in closely packed globules of 100–300 nm in size. Detailed texture studies, which were performed using nitrogen adsorption isotherms, demonstrated texture mobility under the ambient conditions. The cesium salts of the heteropoly acids were tested in the gas-phase oxidation of benzene to phenol with an O_2 + H_2 mixture at 180°. The effect of platinum concentration on the specific catalytic activity in the presence of deeply reduced heteropoly anions was monitored. The samples containing the salt $Cs_{2.5}H_{0.5}PMo_{12}O_{40}$ exhibited the highest activity in the formation of phenol. The introduction of vanadium into the heteropoly anion impaired the catalytic performance of both deeply and slightly reduced samples.

ARSENIC SPECIATION IN NATURAL AND CONTAMINATED WATERS USING CZE WITH *in situ* DERIVATIZATION BY MOLYBDATE AND DIRECT UV-DETECTION

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Electrophoresis,
30(6) (2009) pp. 1088-1093.

A simple and sensitive procedure for simultaneous determination of arsenate, arsenite, monomethylarsonate (MMA) and dimethylarsinate (DMA) ions in waters using CZE with chemical derivatization *in situ* and UV-detection at 250 nm was developed. The separation was performed in a fused-silica capillary using solution containing sodium molybdate and sodium perchlorate as electrolyte. Molybdate forms heteropolycomplexes with arsenic species in low acidic media, while sodium perchlorate masks silicate ion. The analysis conditions were optimized; the best results were achieved with the electrolyte consisting of 10 mM Na_2MoO_4 and 10 mM $NaClO_4$ at pH 3.0 using negative voltage and pneumatic injection of the sample. Nevertheless, the signal of arsenite ion was not detected probably, because of its instability. Arsenite ion was quantified as a difference between arsenate ion contents after and before oxidation by bromine water. The detection limits for the fresh water at the level of 5.0 μg/L for As^{III} and As^{V}, 16 μg/L for DMA and 20 μg/L for MMA were achieved. The reprodusibility varied in the range of 0.06-0.25 relative units. To reduce the interferences of the sample salinity an addition of organic substances and isotachophoretic effect were used.

EFFECT OF IONIC LIQUID BMImBr ON PALLADIUM-CATALYZED OXIDATION OF ETHYLENE WITH $LiNO_3$

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React. Kinet. Catal. Lett.,
97(2) (2009) pp. 191-197.

Oxidative bromination of ethylene was found to proceed effectively in the presence of a catalytic system $Pd(OAc)_2$–$LiNO_3$ in two-component BMImBr–HOAc and three-component BMImBr–HOAc–H_2O solvents. At a large content of BMImBr, conversion of ethylene was found to yield 1,2-dibromoethane with a selectivity over 95%. The composition of the $Pd(OAc)_2$–$LiNO_3$ solutions was followed during the reaction of ethylene by means of UV–Vis spectroscopy.

STRUCTURE AND PROPERTIES OF $H_8(PW_{11}TiO_{39})_2O$ HETEROPOLY ACID

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J. Struct. Chem.,
50(4) (2009) pp. 618-627.

Heteropoly acid (HPA) $H_8(PW_{11}TiO_{39})_2O_xH_2O$ (I) is synthesized by three different ways and studied by chemical analysis, potentiometric titration, mass-spectrometry, IR, ^{31}P, ^{183}W, and ^{17}O NMR spectroscopy, thermogravimetry, and transmission electron microscopy. Anion *I* consists of two subparticles of the Keggin structure bridged by Ti-O-Ti. The dimeric anion exists in HPA aqueous solutions at [*I*] > 0.02 M. At pH > 0.6 it splits to a $[PW_{11}TiO_{40}]^{5-}$ monomer stable up to pH ~ 6. When heated (150–400)°C, *I* splits into $H_3PW_{12}O_{40}$ and, apparently, $H_3PW_{10}Ti_2O_{38}$ without phase separation. Thermolysis products are soluble and when dissolved in water turn again into *I*. Complete decomposition of *I* to oxides occurs at ~450°C.

CATALYTIC PROPERTIES AND STABILITY OF THE HETEROPOLYTUNGSTATE $[P_2W_{21}O_{71}(H_2O)_3]^{6-}$ IN H_2O_2-BASED OXIDATIONS

B.G. Donoeva, T.A. Trubitsina, G.M. Maksimov, R.I. Maksimovskaya, O.A. Kholdeeva

Eur. J. Inorg. Chem.,
2009(34) (2009) pp. 5142-5147.

Catalytic properties of the sandwich-type heteropolytungstate $[P_2W_{21}O_{71}(H_2O)_3]^{6-}$ (**1**) in the selective oxidation of three representative organic substrates, methyl phenyl sulfide, cyclohexene, and cyclohexanol, with aqueous H_2O_2 have been studied in acetonitrile. With one equivalent of hydrogen peroxide, methyl phenyl sulfide readily produced the corresponding sulfoxide with 92-94 % selectivity at 95-97 % conversion. The substituent effect on the oxidation rates of the set of aryl methyl sulfides followed the Hammett free-energy relationship ($\rho = -0.85$). The catalytic activity of heteropolyacid H-**1** in thioether oxidation was several times higher than the activity of its tetrabutylammonium salt, TBA-**1**. In contrast, H-**1** was completely inactive in the oxidation of cyclohexanol, while TBA-**1** catalyzed this reaction effectively to give cyclohexanone with 94 % yield. Neither H-**1** nor TBA-**1** was active in the oxidation of cyclohexene. The catalytic performance of **1** in H_2O_2-based oxidations in MeCN is similar to that of the Keggin heteropolyanion $[PW_{12}O_{40}]^{3-}$ (PW_{12}) and differs significantly from the performance of the Venturello complex $\{PO_4[W(O)(O_2)_2]_4\}^{3-}$. The stability of **1** and PW_{12} towards solvolytic destruction under turnover conditions (50-100 equiv. H_2O_2, 25-70°C, 2-72 h) was confirmed by using ^{31}P NMR, IR, and Raman spectroscopic techniques.

PHYSICOCHEMICAL PROPERTIES OF CATALYSTS BASED ON AQUEOUS SOLUTIONS OF Mo–V–PHOSPHORIC HETEROPOLY ACIDS

E.G. Zhizhina, V.F. Odyakov

Appl. Catal., A,
358(2) (2009) pp. 254-258.

Various organic substrates can be selectively oxidized with O_2 in two stages using aqueous solutions of Mo–V–phosphoric heteropoly acids (HPA) as catalysts. In stage (1), a substrate is oxidized with a HPA solution to the desired product that is separated from the reduced form of HPA. The latter is oxidized with O_2 in stage (2) closing a catalytic cycle. All physicochemical properties of the homogeneous catalysts based on HPA solutions continuously alter during these redox processes. Using as an example a solution of the modified high-vanadium HPA having gross composition $H_{16}P_3Mo_{17}V_7O_{84}$ (HPA-7′), it was shown that viscosity and pH of this solution reach their maxima after reaction (1) and attain their minima after reaction (2). The reverse situation is observed for redox potential of the HPA-7′ solution. Many-cycled testing of 1-butene oxidation in stages (1) + (2) in the presence of the Pd + HPA-7′ catalyst confirms that the alterations of physicochemical properties of the catalyst are completely reversible.

CORROSIVITY OF CATALYSTS BASED ON AQUEOUS SOLUTIONS OF Mo–V–P HETEROPOLY ACIDS

E.G. Zhizhina, V.F. Odyakov

React. Kinet. Catal. Lett.,
98(1) (2009) pp. 51-58.

Corrosion resistance of metallic titanium, alloys KhN65MV and 06KhN28MDT, and stainless steels 10Kh17N13M2T and 12Kh18N10T towards the action of homogenous oxidation catalysts based on aqueous solutions of Mo–V–P heteropoly acids was studied by an example of the 0.25 M aqueous solution of $H_{12}P_3Mo_{18}V_7O_{85}$. It was established that the corrosivity of this solution towards these materials grows in the series: Ti < 06KhN28MDT < 10Kh17N13M2T < 12Kh18N10T << KhN65MV. The highest corrosion resistance of the steels 06KhN28MDT and 10Kh17N13M2T was attributed to the fact that their Ni:Cr atomic ratio is in the range from 0.6 to 1.1.

NEW PROCESS FOR PREPARING AQUEOUS SOLUTIONS OF Mo-V-PHOSPHORIC HETEROPOLY ACIDS

V.F. Odyakov, E.G. Zhizhina

Russ. J. Inorg. Chem.,
545(3) (2009) pp. 361-367.

A new ecofriendly process is proposed for the synthesis of aqueous solutions of Mo-V-phosphoric Keggin heteropoly acids $H_{3+x}PV_xMo_{12-x}O_{40}$(HPA−*x*). First, V_2O_5 is dissolved in cooled H_2O_2 to form peroxyvanadium compounds, which then spontaneously decompose to yield the $H_6V_{10}O_{28}$ solution. The latter is stabilized by the addition of H_3PO_4 to yield an $H_9PV_{14}O_{42}$ solution. This solution is gradually added to a boiling H_3PO_4 + MoO_3 aqueous suspension. This suspension is gradually evaporated that is followed by dissolution of MoO_3 to produce an HPA−*x* solution. This process is reliable and almost non-waste and is promising for preparing HPA−*x* solutions with x = 2–6 on pilot and large scales.

Oxidation Chemistry of Nitrous Oxide

NITROUS OXIDE AS AN OXYGEN DONOR IN OXIDATION CHEMISTRY AND CATALYSIS

G.I. Panov, K.A. Dubkov, A.S. Kharitonov

In “*Modern Heterogeneous Oxidation Catalysis: Design, Reactions and Characterization*”,
Ed. N. Mizuno, WILEY-VCH Verlag GmbH & Co. KGaA, 2009, Weinheim. Part 7, pp. 217-252.

Theoretically dioxygen is an ideal oxidant: ecologically benign, cheap, with unlimited and renewable supply. However, at the current state of the art one is quite far from being able to use O_2 as a universal and easily controllable source for generating the active oxygen species necessary for selective oxidation of various organic compounds. Therefore, presently, of great importance is a search for alternative oxygen donors that can selectively perform the oxidations that are unfeasible with O_2.

The present chapter is a review of intensive studies made during two last decades on the oxidation chemistry of nitrous oxide. These studies discovered unique properties of the N_2O oxidant, which promises great prospects in the field. Many exciting reactions that become possible due to the N_2O are worth a particular attention of the catalytic researchers, especially the reactions which are currently conducted in a thermal way.

The economic consideration shows that N_2O application in the chemical processes is strongly advantageous compared to other oxygen donors.

THE ROLE OF α-SITES IN N_2O DECOMPOSITION OVER FeZSM-5. COMPARISON WITH THE OXIDATION OF BENZENE TO PHENOL

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Appl. Catal., B,
91(1-2) (2009) pp. 174-179.

The kinetics and the mechanism of nitrous oxide decomposition (NOD) were studied using a set of FeZSM-5 samples with a wide variation in concentration of α-sites, which are special complexes of bivalent iron $(Fe^{II})_\alpha$ stabilized in the micropore space of the zeolite matrix. The results are compared with the N_2O oxidation of benzene to phenol (OBP) studied earlier with the same set of samples. In spite of strong differences in turnover frequencies (ca. 100 times) and activation energies (92 kJ/mol), both reactions are catalyzed by the same active centers represented by α-sites. The rates of the reactions increase linearly with increasing α-sites concentration within two orders of magnitude.

The NOD and OBP reactions are shown to have kindred mechanisms, which can be described by two main steps. The first step is a common one and includes oxidation of the α-site by the deposition of α-oxygen from N_2O:

$$(Fe^{II})_\alpha + N_2O \rightarrow (Fe^{III}–O^-)_\alpha + N_2$$

The α-oxygen can be safely quantified, its properties being thoroughly studied in many previous works.

The second step is the reduction of the site. It proceeds due to the removal of α-oxygen by the interaction of the latter with either N_2O (NOD reaction) or benzene (OBP reaction). In both cases this step is the rate determining one.

Mechanistically, the OBP can be considered as an N_2O reduction by benzene. One may think that fundamental role of α-sites may hold also for N_2O reduction by other substrates like CO, methane, propane, etc., which are widely used for this purpose.

KETONIZATION OF 1,5-CYCLOOCTADIENE BY NITROUS OXIDE

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Adv. Synth. Catal.,
351(11-12) (2009) pp. 1905-1911.

The kinetics and mechanism of the liquid phase ketonization of 1,5-cyclooctadiene (COD) by nitrous oxide have been studied. The reaction proceeds without catalyst in the temperature range 473-553 K with the activation energy 113 kJ mol^{-1} and is first order with respect to the initial reactants. The mechanism includes consecutive ketonization of two C═C bonds in the COD molecule, with the intermediate formation of an unsaturated monoketone (MK). Further ketonization of MK leads to two isomeric diketones (DK): 1,4- and 1,5-cyclooctanedione. The 1,5-DK is a stable final product while the 1,4-DK undergoes further intramolecular aldol transformation leading to two bicyclic compounds, that retain the same number of carbon atoms. The distribution of mono- and diketones in the course of reaction is described by theoretical dependences pointing to identical reactivities of the C═C double bonds residing in COD and MK molecules. The ketonization of COD by nitrous oxide exemplifies a prospective way for the preparation of valuable organic products in perfect harmony with the strategy of green chemistry.

KETONIZATION OF A NITRILE-BUTADIENE RUBBER BY NITROUS OXIDE. COMPARISON WITH THE KETONIZATION OF OTHER TYPE DIENE RUBBERS

S.V. Semikolenov, K.A. Dubkov; D.P. Ivanov, D.E. Babushkin, M.A. Matsko, G.I. Panov

Eur. Polym. J.,
45 (2009) 3355–3362.

Noncatalytic ketonization of a nitrile-butadiene rubber (21 mol % acrylonitrile units) by nitrous oxide was shown to yield polymeric products functionalized with carbonyl (mainly ketone) groups. The reaction was conducted in a benzene solvent at 180-230° and pressure of 3-6 MPa. An assumed ketonization mechanism includes a 1,3-dipolar cycloaddition of N_2O to C=C bonds in butadiene units. According to the NMR and GPS data, the main route of the reaction (ca. 85%) proceeds without cleavage of the C=C bonds and yields ketone groups in the polymer backbone. The minor route (ca. 15%) includes the cleavage of C=C bonds resulting in fragmentation of the macromolecules that leads to a decrease in their molecular weight. The nitrile (–CN) groups remain untouched. The resulting product is a bifunctional low-molecular rubber containing, in addition to originally present nitrile groups, a regulated amount of new ketone groups randomly distributed along the polymer backbone. The results for nitrile-butadiene rubber are compared with the earlier studied ketonization of butadiene and isoprene rubbers. The molecular structure of monomeric units was shown to be an important characteristic of a parent rubber affecting the reaction rate, degree of fragmentation, and consistency (rubber-like, plastic, or liquid) of the resulting material.

Synthesis, Properties and Application of Nanostructured/Nanocomposite Materials

DESIGN AND PILOT-SCALE TESTS OF MONOLITHIC CATALYSTS FOR AUTOTHERMAL/STEAM REFORMING OF NATURAL GAS AND BIOFUELS

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AIChE Spring National Meeting Proceedings, (2009) pp. 1-6.

This work presents results of research aimed at design of monolithic catalysts for hydrogen/syngas production by autothermal/steam reforming/partial oxidation of natural gas and liquid fuels and characterization of their performance at the pilot-scale level. Active components comprised of $Ln_x(Ce_{0.5}Zr_{0.5})_{1-x}O_{2-y}$ mixed oxides (Ln=La, Pr, Sm), $La_{0.8}Pr_{0.2}Mn_{0.2}Cr_{0.8}O_3$ + yttria-doped zirconia (YSZ) promoted by precious metals and/or Ni were supported on several types of heat-conducting substrates (compressed Ni-Al foam, fechraloy foil or gauze protected by corundum layer, Cr-Al-O microchannel cermets, titanium platelets protected by oxidic layer) as well as honeycomb corundum monolithic substrate. For best catalysts, high and stable performance without degradation due to coking was demonstrated in different pilot-scale reactors. Main process parameters allowing obtaining high syngas yield were determined.

REAL STRUCTURE - OXYGEN MOBILITY RELATIONSHIP IN NANOCRYSTALLINE DOPED CERIA-ZIRCONIA FLUORITE-LIKE SOLID SOLUTIONS PROMOTED BY Pt

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Mat. Res. Soc. Symp. Proc., 1122 (2009) 1122-O05-03.

This work considers effect of type and content of dopant on the real structure, state of surface Pt species and oxygen mobility of nanocrystalline $Ln_x(Ce_{0.5}Zr_{0.5})_{1-x}O_{2-y}$ (Ln=La^{3+}, Gd^{3+}, $Pr^{3+}/^{4+}$) solid solutions prepared by Pechini route. For the reactions of methane selective oxidation and dry reforming into syngas, catalytic activity correlates rather well with either surface (in diluted feeds) or bulk (in realistic feeds) oxygen mobility as well as Pt dispersion controlled by the type and content of a dopant.

COMPOSITE CATALYTIC MATERIALS FOR STEAM REFORMING OF METHANE AND OXYGENATES: COMBINATORIAL SYNTHESIS, CHARACTERIZATION AND PERFORMANCE

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Catal. Today, 145(1-2) (2009) pp. 127-137.

Using robotic workstation, a number of composite materials based on NiO/yttria-doped zirconia (YSZ) cermet promoted with doped ceria–zirconia fluorite-like mixed oxides and Ru were synthesized. The materials were characterized by XRD, BET, TEM, H_2

TPR, and their catalytic performance was estimated in the reactions of methane, ethanol and acetone steam reforming at short (10–36 ms) contact times. The amount of deposited carbonaceous species was estimated by temperature-programmed oxidation. Complex fluorite-like oxides as promoters minimize coking even in stoichiometric fuel/steam feeds. Promotion by small (<1 wt.%) amount of Ru further decreases coking and facilitates activation of such molecules as CH_4 and acetone, thus ensuring a high level of performance in the intermediate temperature (500–600°C) range. Factors controlling performance of these composites in steam reforming reactions such as lattice oxygen mobility in complex oxide promoters controlled by their chemical composition, strong interaction between components in composites, state and reactivity of supported Ru were assessed.

DESIGN OF SOME METAL/OXIDE COMPOSITE SUPPORTS AND CATALYSTS

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Compos. Interfaces,
16(4) (2009) pp. 457–476.

Textural, mechanical and catalytic properties of porous composite materials Al_2O_3/Al, MeO_x(Me)/Al_2O_3/Al with metal particles homogeneously distributed in the alumina matrix were studied. These materials were prepared by mixing the powdered components with aluminum followed by hydrothermal treatment and calcination. The macroporous structure was shown to be controlled by the size of big (>microns) particles in starting blends. The mesoporous structure is primarily determined by the properties of alumina formed by dehydration of hydroxide produced in turn via aluminum oxidation by water. The mechanical strength of porous cermets is determined by the number and properties of contacts between micron-size components of composites. Improved catalytic performance of composites is ensured by the developed macroporous structure providing enhanced mass transfer inside the cermet granules.

SYNTHESIS OF HOMOGENEOUS $La_{1-x}Ca_xMnO_3$ SOLID SOLUTIONS BY THE PECHINI METHOD AND THEIR ACTIVITY IN METHANE OXIDATION

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Kinet. Catal.,
50(6) (2009) pp. 886-891.

Homogeneous $La_{1-x}Ca_xMnO_3$ solid solutions have been synthesized by the Pechini method (using polymer-solid compositions). Their microstructure, stability at high temperatures, and catalytic activity in methane oxidation are reported. A continuous series of solid solutions stable in air up to 1100°C forms in the system, and the particle surface is enriched with calcium. A distinctive microstructural feature of the particles is their microporosity. The catalytic activity of all calcium-containing samples (except for $x = 0.7$) below 700°C is lower than that of lanthanum manganite and decreases under the action of the reaction medium, which can be due to the decrease in the amount of weakly bound oxygen on the surface because of the enrichment of the surface with calcium and the formation of strongly bound surface carbonates. The higher activity and stability of the $La_{0.3}Ca_{0.7}MnO_3$ sample (calcined at 1100°C) above 500°C can be due to the formation of nanosized areas with an Mn_3O_4 structures on the perovskite particle surface in the reaction medium.

NANOCOMPOSITE CATALYSTS FOR INTERNAL STEAM REFORMING OF METHANE AND BIOFUELS IN SOLID OXIDE FUEL CELLS: DESIGN AND PERFORMANCE

V.A. Sadykov, N.V. Mezentseva, G.M. Alikina, R.V. Bunina, V.V. Pelipenko, A.I. Lukashevich, S.F. Tikhov, V.V. Usoltsev, Z.Yu. Vostrikov, O.F. Bobrenok*, A. Smirnova, J. Ross***, O. Smorygo****, B. Rietveld******* (**Institute of Thermal Physics, Novosibirsk, Russia; **Connecticut Global Fuel Cell Center, Storrs, CT, USA; ***University of Limerick, Limerick, Ireland; ****Powder Metallurgy Institute, Minsk, Belarus; *****Energy Research Center of the Netherlands, Petten, The Netherlands*)

Catal. Today,
146(1-2) (2009) pp. 132-140.

Nanocomposite catalysts comprised Ni particles embedded into the complex oxide matrix comprised Y- or Sc-stabilized zirconia (YSZ, ScCeSZ) combined with doped ceria–zirconia oxides or La–Pr–Mn–Cr–O perovskite and promoted by Pt, Pd or Ru were synthesized via different routes (impregnation of YSZ or NiO/YSZ composites with different precursors, one-pot Pechini procedure). Both composition and preparation procedure determining degree of interaction between components of composites were found to strongly affect performance of nanocomposites in steam reforming of methane at short contact times as well as their stability to coking in stoichiometric feeds. Temperature-programmed reduction of composites by CH_4 followed by temperature-programmed oxidation by H_2O revealed more efficient dissociation of CH_4 on promoted composites yielding loose surface CH_x species more easily removed by water as compared with unpromoted composites. Best active components highly active and stable to coking were supported as thin layers on different substrates (Ni/YSZ anode platelets, refractory dense/porous metal alloys, cermet or corundum monolithic carriers). These structured catalysts demonstrated high efficiency and stability in the reactions of steam reforming of methane and oxygenates (ethanol, acetone) in pilot-scale reactors.

STRUCTURED CATALYST SUPPORTS AND CATALYSTS FOR THE METHANE INDIRECT INTERNAL STEAM REFORMING IN THE INTERMEDIATE TEMPERATURE SOFC

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Int. J. Hydrogen Energy,
34(23) (2009) pp. 9505-9514.

Open cell metal foams made from Ni, Fe–Cr steel and Ni–Al intermetallic were studied as candidate catalyst supports for the internal indirect methane steam reforming. All the samples exhibited good corrosive resistance during 500–1000 h testing in H_2-H_2O–Ar environment at 600°C. NiO/8YSZ composite based catalysts doped with fluorite-like ($Pr_{0.3}Ce_{0.35}Zr_{0.35}O_2$) or perovskite-like ($La_{0.8}Pr_{0.2}Mn_{0.2}Cr_{0.8}O_3$) complex oxides with high lattice oxygen mobility and promoted with Pt or Ru were prepared and deposited on the foam-structure supports. Both good catalyst adhesion and stable catalyst performance were achieved in the case of the Ni–Al foam supported catalysts. The Fe–Cr support reacted with the catalytic active components resulting in fast catalyst deactivation. The foam supported catalyst performance was compared with the same catalyst prepared in a form of 0.25 mm fraction. Porous supports with different porosities were prepared by the metal foam deformation and the catalyst performance depending on the support porosity (75–95%) was studied.

$La_{0.8}Sr_{0.2}Ni_{0.4}Fe_{0.6}O_3$–$Ce_{0.8}Gd_{0.2}O_{2-\delta}$ NANOCOMPOSITE AS MIXED IONIC–ELECTRONIC CONDUCTING MATERIAL FOR SOFC CATHODE AND OXYGEN PERMEABLE MEMBRANES: SYNTHESIS AND PROPERTIES

V.A. Sadykov, T.S. Kharlamova, L.Ch. Batuev, V.S. Muzykantov, N.V. Mezentseva, T.A. Krieger, G.M. Alikina, A.I. Lukashevich, V.A. Rogov, V.I. Zaikovsky, A.V. Ishchenko, A.N. Salanov, A.I. Boronin, S.V. Koshcheev, S.N. Pavlova, N.F. Uvarov*, A. Smirnova, O. Vasylyev*****
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Compos. Interfaces,
16(4) (2009) pp. 407–431.

Mixed ionic-electronic conducting nanocomposite $La_{0.8}Sr_{0.2}Ni_{0.4}Fe_{0.6}O_3$ (LSNF)-$Ce_{0.8}Gd_{0.2}O_{2-\delta}$ (GDC) was prepared *via* ultrasonic dispersion of nanocrystalline powders of perovskite and fluorite oxides in water with addition of surfactant, followed by drying and sintering up to 1300°C. Analysis of the real structure of nanocomposite (studied by XRD and TEM with EDX) and its surface composition (studied by XPS) revealed moderate redistribution of elements between phases favoring their epitaxy. Results of impedance spectroscopy, oxygen isotope exchange, O_2 TPD and H_2 TPR experiments revealed a positive effect of composite interfaces on the oxygen mobility and reactivity agreeing with the ambipolar transport behavior of MIEC composite. Preliminary testing of button-size cell with functionally graded LSNF-GDC cathode layer supported on thin YSZ layer covering Ni/YSZ cermet demonstrated high and stable performance, which is promising for its practical application.

DESIGN AND CHARACTERIZATION OF FUNCTIONALLY GRADED CATHODE MATERIALS FOR SOLID OXIDE FUEL CELLS

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ECS Trans.,
25(2) (2009) pp. 2403-2412.

This work presents results on synthesis and characterization of mixed ionic-electronic conducting nanocomposite cathode materials for Intermediate Temperature Solid Oxide Fuel Cells. The corresponding materials are comprised of $La_{0.8}Sr_{0.2}MnO_3$ (LSM) or $La_{0.8}Sr_{0.2}Fe_{1-x}Ni_xO_{3-\delta}$ (x=0.1-0.4) ($LSFN_x$) perovskites and $Sc_{0.1}Ce_{0.01}Zr_{0.89}O_{2-x}$ (ScCeSZ) or $Ce_{0.9}Gd_{0.1}O_{2-\delta}$ (GDC) fluorites mixed via powerful ultrasonic dispersion of powders in isopropanol. Genesis of their structural properties was studied in details by transmission electron microscopy with elemental analysis and X-ray diffraction, surface features were characterized by X-ray Photoelectron Spectroscopy. Perovskite-fluorite nanodomain interfaces appear to provide a path for fast oxygen diffusion as revealed by the oxygen isotope exchange and temperature-programmed desorption. Testing in wet H_2/air feeds for button-size fuel cells with thin Y-doped zirconia layer and nanocomposite functionally graded cathodes demonstrated promising and stable performance of some combinations in the intermediate temperature range.

DESIGN AND CHARACTERIZATION OF LSM-ScCeSZ NANOCOMPOSITE AS MIEC MATERIAL FOR SOFC CATHODES AND OXYGEN-SEPARATION MEMBRANES

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Mater. Res. Soc. Symp. Proc.,
1126 (2009) S13-03.

LSM–ScCeSZ nanocomposite was prepared via ultrasonic dispersion of the mixture of perovskite and fluorite powders in acetone in 1:1 ratio with addition of polymethylmethylacrylate. Detailed studies of the real structure and surface properties evolution at sintering under air up to 1100°C revealed pronounced redistribution of elements between the phases without new phases formation. Despite a lower specific conductivity of nanocomposite as compared with LSM, the oxygen mobility estimated by the oxygen isotope exchange and O_2 TPD exceeds that of LSM demonstrating a positive role of interfaces as fast oxygen migration paths. Button-cell tests with the LSM–ScCeSZ interlayer between LSNF and a thin YSZ (ScYSZ) layer covering Ni/YSZ cermet demonstrated a high and stable performance promising for the practical application.

CATALYTIC PROPERTIES AND COKING STABILITY OF NEW ANODE MATERIALS FOR INTERNAL METHANE REFORMING IN THE INTERMEDIATE TEMPERATURE SOLID OXIDE FUEL CELLS

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Catal. Today,
146(1-2) (2009) pp. 141-147.

Catalytic properties and coking stability of composite materials based on NiO and doped apatite-type lanthanum silicates were studied for their using as anode materials for internal methane reforming in intermediate temperature solid oxide fuel cells. Samples based on Sr- or Al-doped lanthanum silicate were prepared by modified Pechini methods. The composite materials were characterized by X-ray diffraction, methane steam reforming and temperature programmed oxidation. The effects of $La_{0.8}Sr_{0.2}Mn_{0.8}Cr_{0.2}O_3$ addition, partial Ni substitution with $La_{0.1}Sr_{0.9}TiO_3$ ionic and electronic mixed conductor and composition of apatite-type lanthanum silicate on catalytic properties and coking resistance were considered.

DOPED APATITE TYPE LANTHANUM SILICATES: STRUCTURE AND PROPERTY CHARACTERIZATION

T.S. Kharlamova, S.N. Pavlova, V.A. Sadykov, T.A. Krieger, L.Ch. Batuev, V.S. Muzykantov, O.B. Lapina, D.F. Khabibulin, M.V. Chaikina*, N.F. Uvarov*, Yu.T. Pavlukhin*, S.A. Petrov*, Chr. Argirusis** (**Institute of Solid State Chemistry and Mechanochemistry, Novosibirsk, Russia; **Clausthal University of Technology, Clausthal-Zellerfeld, Germany*)

Mater. Res. Soc. Symp. Proc.,
1126 (2009) pp. 1126-S11-04.

Structural features and transport properties of Fe- and Al-doped apatite-type lanthanum silicates were studied with relation to a role of the $[SiO_4]$ substructure. XRD, TEM, Mössbauer, UV-Vis electron, IR, ^{29}Si and ^{27}Al NMR spectroscopy were used for structural studies; impedance spectroscopy and oxygen isotope heteroexchange were used for transport properties characterization.

STRUCTURE AND TRANSPORT PROPERTIES OF DOPED APATITE-TYPE LANTHANUM SILICATES PREPARED VIA MECHANOCHEMICAL ROUTE

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ECS Trans.,
25(2) (2009) pp. 1791-1800.

Structural features of Fe- and Al-doped apatite-type lanthanum silicates with different stoichiometry have been studied by using XRD, XPS, IR, UV–Vis, EXAFS, Mossbauer, ^{29}Si and ^{27}Al MAS NMR spectroscopy. The transport properties of the samples were also characterized by using impedance spectroscopy and oxygen isotope heteroexchange.

ELECTROCHEMICAL CHARACTERIZATION OF A $La_{0.8}Sr_{0.2}Ni_{0.4}Fe_{0.6}O_{3-\delta}$ ELECTRODE INTERFACED WITH $La_{9.83}Si_5Al_{0.75}Fe_{0.25}O_{26\pm\delta}$ APATITE-TYPE ELECTROLYTE

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ECS Transactions,
25(2) (2009) pp. 2681-2688.

The electrochemical characteristics of a $La_{0.8}Sr_{0.2}Ni_{0.4}Fe_{0.6}O_3$ cathode electrode interfaced to $La_{9.83}Si_5Al_{0.75}Fe_{0.25}O_{26\pm\delta}$ apatite- type electrolyte were studied using AC impedance spectroscopy under open circuit conditions, at temperatures 600 to 800°C and oxygen partial pressures in the range 0.1 to 20 kPa. The cathode and electrolyte powders were prepared via the Pechini method and the mechanochemical activation method, respectively. The electrocatalytic activity of the tested interface was assessed on the basis of its open circuit area specific polarization conductance R^{-1}_p determined from the impedance data. The polarization conductance was found to increase with increasing PO_2, following power law dependence. The impedance characteristics of the system were determined by at least two different processes, corresponding to two partially overlapping depressed arcs in the Nyquist plots. The relative contribution and degree of overlap of these arcs depended on temperature and oxygen partial pressure.

CONDUCTIVITY OF THE NANOSTRUCTURED CERAMIC MATERIAL $Zr_{0.88}Sc_{0.1}Ce_{0.01}Y_{0.01}O_{1.955}$ PREPARED FROM MECHANICALLY ACTIVATED POWDERS

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Inorg. Mater.,
45(1) (2009) pp. 90-98.

The structure of the $Zr_{0.88}Sc_{0.1}Ce_{0.01}Y_{0.01}O_{1.955}$ solid solution, a candidate for the use as a solid electrolyte in fuel cells with a low temperature, has been investigated using X-ray powder diffraction and

Raman spectroscopy. Single-phase ceramic materials have been produced from powders prepared by the mechanochemical synthesis from ZrO_2 nanoprecursors purified of the impurities introduced during grinding of commercial zirconia. The solid solution has a rhombohedral structure at room temperature owing to the partial ordering of oxygen vacancies. The electrical conductivity of the ceramic materials sintered at temperatures below 1570 K exhibits a hysteresis due to the delay of the martensitic transition from the cubic phase to the rhombohedral phase upon cooling of the sample. The nanostructured ceramic materials are characterized by a high mechanical strength and unusually close values of the activation energies for bulk and grain-boundary electrical conduction.

MECHANOSYNTHESIS, RADIATION-THERMAL MODIFICATION AND CHARACTERIZATION OF NANOSTRUCTURED SCANDIA STABILIZED ZIRCONIA CERAMICS

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Mater. Res. Soc. Symp. Proc.,
1122 (2009) O03-10.

SSZ-based ceramics were obtained by sintering of nanopowders derived at room temperature by mechanochemical synthesis from refined technical grade ZrO_2 nano-precursors. RT-treatment by 2.5 MeV electrons up to 1563 K was used for the modification of ceramics. Powders and ceramics were characterized by XRD, Raman, SEM and EDS, TEM, SIMS techniques. The phase composition of $Zr_{0.89}Sc_{0.1}Ce_{0.01}O_{1.95}$ ceramics was very close to cubic structure but better fitting of XRD patterns was obtained for rhombohedral lattice. Conductivity of solid electrolytes for IT SOFC was studied by complex impedance method. To stabilize cubic structure and increase conductivity at operation temperature of T_o ~ 1000 K, the composition of SSZ solid electrolyte was optimized by addition of yttria and sintering aids. The interaction of admixtures with minor dopants leading to intergrain phase was revealed. During fast sintering, ceramics keep a memory about inhomogeneous disordered solid solutions in a form of nanostructuring. Conductivity data indicate nanostructuring of ceramics too: activation energies of bulk and grain boundary conductivities are close (E_b ~ 0.9 eV, E_{gb} ~ 1.05 eV). Annealing of ceramics at high temperatures increases conductivity at T_o and promotes grain growth.

MECHANOCHEMICAL SYNTHESIS AND CONDUCTING PROPERTIES OF NANOSTRUCTURED RHOMBOHEDRAL SCANDIA STABILIZED ZIRCONIA CERAMICS

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J. Alloys Compd.,
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Studies of mechanochemical powders and sintered ceramics in scandia stabilized zirconia system have been carried out by means of XRD technique, Raman spectroscopy and SIMS. Nanostructured single phase ceramics of $Zr_{0.88}Sc_{0.1}Ce_{0.01}Y_{0.01}O_{1.955}$ composition with the room-temperature rhombohedral structure caused by ordering of oxygen vacancies possesses excellent mechanical strength and unusual conductivity properties with $E_b \sim E_{gb}$. Decreased level of conductivity at $T \sim 1000$ K was explained by incomplete stabilization of cubic phase and admixtures of silica and alumina present in starting ZrO_2 reagent of technical grade.

Photocatalytic and Related Processes

FAST ADSORPTIVE AND PHOTOCATALYTIC PURIFICATION OF AIR FROM ACETONE AND DIMETHYL METHYLPHOSPHONATE BY TiO_2 AEROSOL

A.S. Besov, A.V. Vorontsov, V.N. Parmon

Appl. Catal., B,
89(3-4) (2009) pp. 602-612.

A high concentration (1.5×10^6 cm^{-3}) TiO_2 aerosol of the average particle size 0.5 μm was generated by a sonic method inside 0.1 m^3 Plexiglas chamber and applied for the adsorptive and adsorptive-photocatalytic purification of air from vapors of acetone and chemical agents' model dimethyl methylphosphonate (DMMP). The adsorptive capture of acetone over the TiO_2 aerosol results in establishing equilibrium adsorption state and is limited by the rate of the aerosol admission into the chamber. A model derived from the Langmuir isotherm describes well the acetone concentration vs. aerosol mass curve and allows obtaining the adsorption constant and monolayer coverage of acetone in a 10 min experiment. The UV irradiation of TiO_2 aerosol accelerates dramatically the purification from acetone at the high relative humidity (RH) of the air. Increased RH of air decreases the rate of the acetone adsorption but has a little positive effect on the rate of photocatalytic oxidation of acetone over aerosol particles. The DMMP adsorption over TiO_2 aerosol is accompanied by the immediate ($\tau < 10$ s) and irreversible hydrolysis of DMMP with the formation of gas phase methanol and adsorbed methyl methylphosphonic acid. The irreversible reactive adsorption results in the very fast air purification ($\tau = 20$–40 s) due to very small diffusion distances of substrate to the TiO_2 surface in aerosol. The increase of the air RH from 4 to 37% (296 K) decreases the rate of adsorption but accelerates significantly the rate of photocatalytic oxidation. The complete air purification from organic compounds within 10 min is possible only with the photocatalytic oxidation because the adsorption alone does not remove methanol. The time needed for the air purification over the nanosized TiO_2 aerosol is directly determined by the rate of the aerosol generation which allows a further optimization of the TiO_2 aerosol air purification. The obtained results approve experimentally a suggestion that the photocatalytic oxidation over solid atmospheric aerosols actually takes part in the Earth atmosphere and serves as an important sink for airborne organics.

PRODUCTS DISTRIBUTION DURING THE GAS PHASE PHOTOCATALYTIC OXIDATION OF AMMONIA OVER THE VARIOUS TITANIA BASED PHOTOCATALYSTS

P.A. Kolinko, D.V. Kozlov

Appl. Catal., B,
90(1-2) (2009) pp. 126-131.

The adsorption and photooxidation of gaseous ammonia have been investigated by the FTIR spectroscopy method in a static reactor using TiO_2 and modified TiO_2 as photocatalysts. Two types of modified TiO_2 were tested: TiO_2 impregnated with transition metal oxides (NiO, V_2O_5, MoO_3, and Fe_2O_3) and TiO_2 doped with noble metals like Ag, Au, Pt, Pd by the chemical and photochemical deposition methods. Molecular nitrogen, NO_3^-, N_2O and water were found as the major final products of ammonia photocatalytic oxidation (PCO). One of the purposes of the current study was to minimize the N_2O formation during PCO. The pure unmodified TiO_2 (anatase, $S_{BET} \sim 350$ m^2/g) turned out to be the best photocatalyst for this purpose giving only 7% of initial NH_3 quantity converted into N_2O. Long-term experiments were carried out to determine the photocatalysts stability in PCO of NH_3. All the catalysts exhibited stable performance; the rate of deactivation was low due to the transformation of NH_3 nitrogen mainly into N_2. Only ≈30% of initial NH_3 quantity was oxidized into HNO_3 which was adsorbed on the catalysts surface in all cases.

OVERALL WATER SPLITTING OVER Pt/TiO_2 CATALYST WITH Ce^{3+}/Ce^{4+} SHUTTLE CHARGE TRANSFER SYSTEM

E.A. Kozlova, T.P. Korobkina, A.V. Vorontsov

Int. J. Hydrogen Energy,
34(1) (2009) pp. 138-146.

The experiments on photocatalytic hydrogen production from aqueous solution of $Ce_2(SO_4)_3$ and photocatalytic oxygen production from aqueous solution of $Ce(SO_4)_2$ were carried out on the same catalyst platinized Degussa P25. Pure oxygen was shown to evolve from $Ce(SO_4)_2$ solution and pure hydrogen from $Ce_2(SO_4)_3$ solution with stoichiometric formation of $Ce_2(SO_4)_3$ and $Ce(SO_4)_2$, respectively.

The rate of photocatalytic hydrogen production was lower than the rate of photocatalytic oxygen production on the same catalyst. The influence of initial cerium salt concentration, acidity and catalyst loading was checked for photocatalytic oxygen and hydrogen production. The dependences of the hydrogen and oxygen production rate on Ce^{3+} and Ce^{4+} concentration have maxima. Kinetic equation for the rate of photocatalytic oxygen production was proposed that takes into account the inner filter effect; constants of this equation were calculated. Optimal conditions for photocatalytic reactions were found and explained.

The shuttle charge transfer regime was tested for consecutive O_2 and H_2 production over the same and different portions of catalyst and same Ce^{3+}/Ce^{4+} system. It was found that separate catalyst batches are needed to avoid mixed evolution of hydrogen and oxygen.

ENHANCEMENT OF THE O_2 OR H_2 PHOTOPRODUCTION RATE IN A Ce^{3+}/Ce^{4+}-TiO_2 SYSTEM BY THE TiO_2 SURFACE AND STRUCTURE MODIFICATION

E.A. Kozlova, T.P. Korobkina,
A.V. Vorontsov, V.N. Parmon

Appl. Catal., A,
367(1-2) (2009) pp. 130-137.

The effect of structural and surface changes in the suspended TiO_2 on the dioxygen and dihydrogen evolution rates was studied with the purpose to improve photogenerated charges utilization efficiency for future solar light water splitting systems which use reversible inorganic electron relays. Prepared rutile and anatase catalysts are characterized by N_2 adsorption–desorption measurements, X-ray diffraction (XRD) and transmission electron microscopy (TEM). It was discovered that the dioxygen photoproduction rate from the Ce^{4+}-containing TiO_2 suspension increased linearly with the rutile specific surface area. A further increase in the O_2 generation rate provides a modification of TiO_2 surface with Pt. Photodeposition (PD) of Pt resulted in a higher O_2 evolution rate than Pt deposition by soft chemical reduction (SCR). The highest photocatalytic activity at pH equal to 0.4 was obtained for 1 wt.% Pt/TiO_2 Degussa P25 platinized by PD.

In contrast to O_2, the H_2 photocatalytic production from the Ce^{3+}-containing TiO_2 suspension solution was faster over Pt/TiO_2 prepared by SCR. Uniform coverage of the TiO_2 surface with Pt particles turned out to be more important for the H_2 generation than rather for the O_2 one. A lower quantum efficiency of the H_2 generation associated with the low Ce^{3+} surface coverage on Pt/TiO_2 was attempted to be improved by treating TiO_2 surface with inorganic acids. Sulfuric acid increased the rate by 20% while phosphoric and hydrofluoric acids did not. The best photocatalytic dihydrogen production catalyst at pH equal to 1.0 was found to be 1 wt.% Pt/Degussa P25 prepared by SCR and treated with H_2SO_4. The results demonstrate that the bulk structure (phase composition), morphology (surface area), as well as the surface morphology (Pt distribution) and composition (acid additives) are all important for the O_2 and H_2 photogeneration in the separate TiO_2—suspended based.

INCREASE IN ENERGETIC EFFICIENCY OF PHOTOCATALYTIC OXIDATION OF ORGANIC VAPORS UNDER THE ACTION OF NEGATIVE ATMOSPHERIC CORONA DISCHARGE

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In "*Nonequilibrium Phenomena: Plasma, Combustion, Atmosphere*", Eds. G.D. Roy,
S.M. Frolov, A.M. Starik,
Moscow: TORUS PRESS Ltd., 2009, pp. 190-197.

The study is devoted to influence of atmospheric corona discharge on the course of photocatalytic oxidation of vapors of acetone (83 ppm), acetaldehyde (108 ppm) and toluene (12-46 ppm) over TiO_2 under the action of soft UV light in a closed chamber. Corona discharge was created around the wires of the charging section of an electric precipitator situated upstream of a photocatalytic filter. Substantial acceleration of removal and deep oxidation of the compounds into CO_2 is revealed, the highest effect being observed for acetaldehyde. In the presence of corona discharge, an increase in the rate of deep oxidation per unit of consumed electric power attains 1.2-3.6 times. The results point out to bright prospects of combining photocatalytic and plasma-chemical oxidations for purification of gas exhausts.

GENERATOR OF NANOSECOND 200-kilovolt PULSES FOR PLASMOCHEMICAL REACTORS

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In "*Nonequilibrium Phenomena: Plasma, Combustion, Atmosphere*", Eds. G.D. Roy,
S.M. Frolov, A.M. Starik,
Moscow: TORUS PRESS Ltd., 2009, pp. 198-203.

The construction and a principle of work of short pulse high voltage generator for creation of the plasma discharge in gases are presented. The generator is constructed on the basis of the transformer of Tesla with a sharpening of pulses on output. Voltage of output pulses about 200 kV, raising time – 2 ns, energy in one pulse – 0,2 J, following frequency 0÷1024 Hz. By means of the generator in the barrier discharge cell with efficient diameter of electrodes 19 mm, a gap between surfaces of a dielectric 5 mm, thickness of a dielectric on electrodes 3 mm, current about 20 A and power ~ 1 megawatt are obtained.

PHOTOCHEMICAL PROPERTIES OF POWDER TITANIUM DIOXIDE PRODUCED FORM RUTILE CRYSTAL UNDER AMBIENT AIR

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Atmos. Oceanic Opt.,
22(6) (2009) pp. 611-614.

It was shown that the photochemical activity under tropospheric solar irradiation ($\lambda > 300$ nm) of titanium dioxide, produced by fine grinding of rutile crystal under ambient air, is essentially higher than the photochemical activity of titanium dioxide prepared by traditional ways. The quantum yield and spectral dependencies of oxygen photoadsorption and carbon dioxide photodesorption of titanium dioxide were determined. The mechanism of photoprocess activity increasing under illumination in the surface absorption region was suggested.

ACTION SPECTRA AND THE MECHANISM OF PHOTOINDUCED FORMATION OF HYDROGEN AND OXYGEN ON POTASSIUM NIOBATES

V.S. Zakharenko, S.V. Bogdanov

High Energy Chem.,
43(1) (2009) pp. 34-37.

Powdered samples of complex oxides – potassium niobates with a layered structure were prepared via high-temperature synthesis followed by grinding. The action spectra of photoinduced processes on potassium niobates with different phase compositions in aqueous and aquo-organic systems were determined. It was shown that the evolution of oxygen was due to its photodesorption and occurred upon illumination with light at an energy near the edge of intrinsic niobate absorption. The formation of hydrogen in aqueous systems is due to the photocatalytic dehydrogenation of trace organic compounds adsorbed on potassium niobate, rather than to the photodecomposition of water. The quantum efficiency of the photocatalytic dehydrogenation increased by an order of magnitude when hydrogen-containing organic compounds were added to the reaction mixture.

PUTATIVE PREBIOTIC PHOTOCATALYTIC SYNTHESIS OF MONOSACCHARIDES IN AQUEOUS SOLUTION OF FORMALDEHYDE

A.N. Simonov, I.V. Delidovich, O.P. Pestunova, V.N. Snytnikov, V.N. Parmon

Orig. Life Evol. Biosph.,
39(3-4) (2009) pp. 268-269.

It was demonstrated that glycolaldehyde (hereinafter, GA) could be formed in aqueous formaldehyde (hereinafter, FA), solution under the UV-irradiation (Pestunova et al., 2005). From the other hand higher monosaccharides were shown to be synthesized via condensation of formaldehyde and lower carbohydrates catalyzed by phosphates in neutral aqueous solution at mild temperatures. (Simonov et al., 2007). In order to combine these processes an experimental photo-catalytic flow installation was designed.

The starting solution for all experiments contained FA with different concentrations and a catalyst-homogeneous phosphates (Na_2HPO_4 + KH_2PO_4), at pH=8. That is, the sole substrate for the synthesis of monosaccharides was FA known to be an abundant compound of the prebiotic environment. The consecutive photosynthesis of GA and catalytic condensation of FA with lower monosaccharides resulted in the formation of significant amounts of higher monosaccharides. The HPLC analysis of the reaction mixture revealed that erythrulose (tetra-ketose) and 3-pentulose (penta-3-ketose) with maximum yields of 10% and 5%, respectively, were the major products of the process. At the same time the isomerization of 3-pentulose results in the formation of reasonable amounts of ribulose (4% yield). Finally, under the catalytic action of phosphates ribulose is isomerized into ribose and arabinose. The detected concentration of ribose in the reaction mixture was not very high. Nevertheless, it is the first evidence of the possibility of the synthesis of these vitally important monosaccharides from FA in putative prebiotic conditions.

PROBABLE PATHWAYS TO PREBIOTIC CARBOHYDRATES AND THEIR DERIVATES

O.P. Pestunova, A.D. Simonov, V.N. Parmon

Orig. Life Evol. Biosph.,
39(3-4) (2009) pp. 262-263.

In this article the authors summarize and discuss the most significant experimental results on the plausible prebiotic synthesis of carbohydrates and other vitally important organic substances from carbohydrates as initial substrates for such synthesis. It is demonstrated that lower carbohydrates necessary for the synthesis of monosaccharides can be formed in formaldehyde aqueous solutions under the action of UV-irradiation (Pestunova, 2005). It has been shown (Simonov, 2007) that higher monosaccharides can be formed directly from formaldehyde in the course of the combined photochemical and catalytic reactions in plausible prebiotic conditions. Aminoacids and heterocycles can be obtained from carbohydrates and NH_3 in the presence of thiols (Weber, 1995).

Polymerization Catalysts and Polymer Materials

HOMOGENEOUS CATALYSTS FOR ETHYLENE POLYMERIZATION BASED ON BIS(IMINO)PYRIDINE COMPLEXES OF IRON, COBALT, VANADIUM AND CHROMIUM

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E.P. Talsi

Catal. Today,
144(3-4) (2009) pp. 334-340.

Results of a comparative study of ethylene polymerization activity and the structure of polyethylene (PE) produced over homogeneous catalysts based on bis(imino)pyridine complexes with close ligand frameworks and different transition metal centers (Fe(II), Co(II), Cr(III) and V(III)) are reported. The effects of the activator nature and polymerization conditions on the activity of these complexes and the resulting PE structure (molecular weight, molecular weight distribution, content of methyl and vinyl groups) have been studied. The experimental data obtained under comparable conditions demonstrate a pronounced effect of transition metal center on the catalytic properties of bis(imino)pyridine complexes (polymerization activity, copolymerization reactivity, thermal stability, PE structure, composition of optimal activator, formation of single-site or multiple-site catalytic system).

FORMATION AND NATURE OF THE ACTIVE SITES IN BIS(IMINO)PYRIDINE IRON-BASED POLYMERIZATION CATALYSTS

K.P. Bryliakov, E.P. Talsi, N.V. Semikolenova,
V.A. Zakharov

Organomet.,
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The activation of 2,6-bis(2,6-diisopropylphenylimino)ethylpyridine iron-based catalysts of ethylene polymerization with methylalumoxane (MAO) and aluminum trialkyls has been studied in detail by ^{1}H NMR and EPR spectroscopy. Neutral catalytically active species are formed in $\mathrm{L^{iPr}FeCl_2/Al(Alk)_3}$ systems. They are complexes $\mathrm{[L^{iPr(-)}Fe^{(+)}(\mu\text{-}Me)_2AlMe_2]}$ (6) or $\mathrm{[L^{iPr(-)}Fe^{(+)}(\mu\text{-}\mathit{i}Bu)(\mu\text{-}X)Al(\mathit{i}Bu)_2]}$ (7 or 8, X = *i*Bu or Cl), depending on the activator used (either $\mathrm{AlMe_3}$ or $\mathrm{Al(\mathit{i}Bu)_3}$). On the contrary, when “$\mathrm{AlMe_3}$-free” methylalumoxane (PMAO) is used as the activator, catalytically active ion pairs of the type $\mathrm{[LFe^{II}(\mu\text{-}Me)_2AlMe_2]^+[MeMAO]^-}$ (3) are formed. Intermediates 6 and 8 are relatively unstable and decay within minutes at room temperature, giving rise to an EPR-active iron species with a signal at g = 2.08, presumably of the type $\mathrm{L'Fe^{I}{-}Alk}$ ($\mathrm{L'Fe^{I}(\mu\text{-}Alk)_2Al(Alk)_2}$) in the low-spin state S = 1/2 (where L′ is a modified bis(imino)pyridine ligand). Polymer properties for the different catalyst/activator systems are presented.

INVESTIGATING THE NATURE OF THE ACTIVE SPECIES IN BIS(IMINO)PYRIDINE COBALT ETHYLENE POLYMERIZATION CATALYSTS

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Organometallics,
28(20) (2009) pp. 6003-6013.

In the past decade, there has been much interest in iron and cobalt ethylene polymerization catalysts $\mathrm{LM^{II}Cl_2/MAO}$, where M = Fe or Co, L = bis(imino)pyridine ligand. The nature of the active species of these catalyst systems was debatable. The reason of the different molecular mass distribution (MMD) of the produced polyethylene (monomodal for cobalt and bimodal for iron) was unclear. It has been shown that ion pairs $\mathrm{[LFe^{II}(\mu\text{-}Me)_2AlMe_2]^+[Me\text{-}MAO]^-}$ predominate in the reaction solution at the initial stage of interaction of $\mathrm{LFe^{II}Cl_2}$ with MAO. Then this ion pair slowly converts into the low-spin iron(I) species with proposed structure $\mathrm{L'Fe^{I}((\mu\text{-}Me)_2AlMe_2}$. The presence of two various types of iron species in the reaction solution can explain the bimodal MMD of the produced polyethylene. In the case of cobalt, the only one type of cobalt species is present in the catalyst system $\mathrm{LCo^{II}Cl_2/MAO/C_2H_4}$ – ion pair $\mathrm{[LCo^{I}(\eta_2\text{-}C_2H_4)]^+[Me\text{-}MAO]^-}$. This is in good agreement with monomodal MMD of the produced polyethylene.

AN EPR STUDY OF THE V(IV) SPECIES FORMED UPON ACTIVATION OF A VANADYL PHENOXYIMINE POLYMERIZATION CATALYST WITH AlR_3 AND AlR_2Cl (R = Me, Et)

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Macromol. Chem. Phys.,
210(7) (2009) pp. 542-548.

Vanadium(IV) complexes formed in the systems $L_2V^{IV}O/AlR_3$, $L_2V^{IV}O/AlR_3$/ETA, $L_2V^{IV}O/AlR_2Cl$, and $L_2V^{IV}O/AlR_2Cl$/ETA, where L is a phenoxyimine ligand (Scheme 1), R = Me or Et, and ETA is ethyltrichloroacetate, were monitored by EPR. It was shown, that at 20°C, V(IV) complexes with proposed structures $(L')V^{IV}(O)(R)(AlR_3)$ are formed in the systems $L_2V^{IV}O/AlR_3$ and $L_2V^{IV}O/$ AlR_3/ETA, whereas complexes with proposed structures $(L')V^{IV}(O)(R)(AlR_2Cl)$ are formed in the systems $L_2V^{IV}O/AlR_2Cl$ and $L_2V^{IV}O/AlR_2Cl$/ETA (L' is the modified initial ligand). The systems $L_2V^{IV}O/AlR_3$/ETA are inert towards polymerization of ethylene, whereas the systems $L_2V^{IV}O/AlR_2Cl$/ETA are moderately active in this reaction. The polymerization (activity of the systems $L_2V^{IV}O/AlR_2Cl$/ETA correlates with the concentration of V(IV) species present in the reaction solution. V(IV) complexes of the type $(L')V^{IV}(O)(R)(AlR_2Cl)$ can be precursors of the active species of polymerization.

AN EPR STUDY OF THE VANADIUM SPECIES FORMED UPON INTERACTION OF VANADYL N AND C-CAPPED TRIS(PHENOLATE) COMPLEXES WITH $AlEt_3$ AND $AlEt_2Cl$

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J. Mol. Catal. A: Chem.,
303(1-2) (2009) pp. 23-29.

Reactions of vanadyl N and C-capped tris(phenolate) complexes **1** and **2** (Scheme 1) with $AlEt_3$ and $AlEt_2Cl$, in the absence and in the presence of ethyltrichloroacetate (ETA), were monitored by EPR. It was shown, that vanadium(IV) complexes with proposed structures $L^NV^{IV}(Et)(AlEt_3)$ and $L^NV^{IV}(Et)(AlEt_2Cl)$ are formed at the initial stage of interaction of **1** with $AlEt_3$ and $AlEt_2Cl$, respectively (L^N are unidentified products of interaction of the initial N-capped ligand and V═O moiety with aluminum alkyls). In the absence of re-activator (ETA), $L^NV^{IV}(Et)(AlEt_3)$ and $L^NV^{IV}(Et)(AlEt_2Cl)$ rapidly convert at room temperature to V(III) species, which relatively slowly transform to complexes $L^NV^{II}Et_2$ and $L^NV^{II}(Et)Cl$. Concentration of $L^NV^{IV}(Et)(AlEt_2Cl)$ dramatically increases in the presence of ETA, that correlates with the increase of the polymerization activity of the catalyst system **1**/$AlEt_2Cl$. The complex $L^NV^{IV}(Et)(AlEt_2Cl)$ can be precursor of the active species of polymerization. Similarly, ETA increases concentration of vanadium(IV) complex $L^CV^{IV}(Et)(AlEt_2Cl)$ in the catalyst system **2**/$AlEt_2Cl$ (L^C are unidentified products of interaction of the initial C-capped ligand and V═O moiety with aluminum alkyls). The complex $L^CV^{IV}(Et)(AlEt_2Cl)$ can be precursor of the active species of polymerization.

EPR MONITORING OF VANADIUM(IV) SPECIES FORMED UPON ACTIVATION OF VANADIUM(V) POLYPHENOLATE PRECATALYSTS WITH AlR_2Cl AND AlR_2Cl/ETHYLTRICHLOROACETATE (R = Me, Et)

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Organometallics,
28(23) (2009) pp. 6714-6720.

Reactions of the vanadyl polyphenolate complexes {VO[*p*-*tert*-butylcalix[4]arene(O)$_3$(OMe)]} (**1**) and $[VO(OArCH_2Ar'CH_2OAr)]_2$ (Ar = 4,6-*tert*-butylphenol; Ar′ = 4-*tert*-butylphenol) (**2**) with AlR_3 and AlR_2Cl, in the absence and in the presence of reactivator (ethyltrichloroacetate, ETA), were monitored by EPR (R = Me, Et). It was shown, that vanadium(IV) complexes with proposed structures $L_1V^{IV}R(AlR_3)$ and $L_1V^{IV}R(AlR_2Cl)$ are formed upon interaction of **1** with AlR_3 and AlR_2Cl, respectively (20 °C, L_1 is the initial oxo-calix[4]arene ligand). Similarly, vanadium(IV) complexes with proposed structures $L_2V^{IV}R(AlR_3)$ and $L_2V^{IV}R(AlR_2Cl)$ are formed upon interaction of **2** with AlR_3 and AlR_2Cl (L_2 is the initial ligand of **2**). The growth of the concentration of $L_1V^{IV}R(AlR_2Cl)$ and $L_2V^{IV}R(AlR_2Cl)$ correlates with the increase of the ethylene polymerization activity of the catalyst systems **1**,**2**/AlR_2Cl/ETA. It is proposed therefore that vanadium(IV) species of the type $L_1V^{IV}R(AlR_2Cl)$ and

$L_2V^{IV}R(AlR_2Cl)$ can be the active species (or their immediate precursors) of the catalyst system **1**,**2**/AlR_2Cl/ETA.

COMPLEXES OF COPPER(II) AND COBALT(II) HALIDES WITH 4-(3,5-DIMETHYL-1*H*-PYRAZOL-1-YL)-6-METHYL-2-PHENYLPYRIMIDINE: SYNTHESIS, STRUCTURES, AND PROPERTIES

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Russ. J. Coord. Chem.,
35(8) (2009) pp. 597-608.

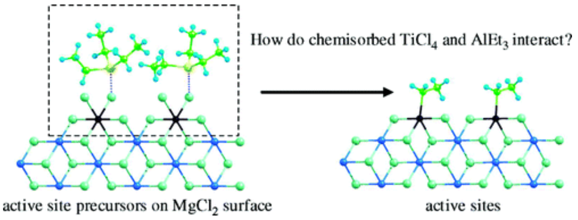
Copper(II) and cobalt(II) complexes with 4-(3,5-dimethyl-1*H*-pyrazol-1-yl)-6-methyl-2-phenylpyrimidine (L) of the general formula MLX_2 (M = Cu(II), X = Cl and Br; M = Co(II), X = Cl, Br, and I) were obtained. According to X-ray diffraction data, $CuLBr_2$ and $CoLX_2$ (X = Cl, Br, and I) are mononuclear molecular complexes. The ligand L is coordinated to the metal atom in a chelating bidentate fashion through theNatoms of the pyrimidine and pyrazole rings. The coordination polyhedron of the metal atom is extended to a distorted tetrahedron by two halide ions. In solution, $CuLBr_2$ undergoes slow transformation into $CuL_{(1-x)}L'_xBr_2$ and the binuclear (X-ray diffraction data) Cu(I) complex $[CuL_{(1-x)}L'_xBr]_2$ (L' is 4-(4-bromo-3,5-dimethyl-1*H*-pyrazol-1-yl)-6-methyl-2-phenylpyrimidine). The complexes MLX_2 show weak antiferromagnetic interactions between the M^{2+}ions.

ACTIVE SITE FORMATION IN $MgCl_2$–SUPPORTED ZIEGLER–NATTA CATALYSTS. A DENSITY FUNCTIONAL THEORY STUDY

D.V. Stukalov, V.A. Zakharov

J. Phys. Chem. C,
113(51) (2009) pp 21376-21382.

All types of chemical reactions (reduction, alkylation, and complexation of Ti species by organoaluminum compounds) leading to formation of active sites in heterogeneous Ziegler–Natta catalysts have been studied within density functional theory. A generally accepted mechanism of active site formation was found to be less preferable as compared to the alternative ways considered. Based on the calculation implemented, a whole active site formation scenario was postulated for the different active center precursors on the $MgCl_2$ surface. The mechanism proposed allows to rationalize the domination of Ti^{3+} over Ti^{2+} under reduction of Ti^{4+} surface species by AlR_3, the absence of an electron spin resonance signal for the $TiCl_4/MgCl_2$ + AlR_3 system with low Ti content, the stronger reduction ability of $Al(i\text{-}Bu)_3$ than $AlEt_3$, the deactivation effect of AlR_2Cl, and the reactivation ability of AlR_3.



SUPPORTED ZIEGLER–NATTA CATALYSTS FOR PROPYLENE POLYMERIZATION. STUDY OF SURFACE SPECIES FORMED AT INTERACTION OF ELECTRON DONORS AND $TiCl_4$ WITH ACTIVATED $MgCl_2$

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J. Catal.,
266(1) (2009) pp. 39-49.

Adsorption of various internal electron donors (IDs) and coadsorption of these IDs with $TiCl_4$ on activated $MgCl_2$ have been studied by DRIFT and analytical techniques. As a result, the structures of the ID species on the (1 0 4) and (1 1 0) $MgCl_2$ surfaces were proposed. Based on the ν(C=O) band resolution data for the adsorbed ID, it was estimated that the activated $MgCl_2$ surface contains ca. 90% of the five-coordinated Mg cations residing on the (1 0 4) surface and ca. 10% of the four-coordinated Mg cations residing on the (1 1 0) surface. The size of ID molecule was found to influence its adsorption more significantly on the (1 0 4) $MgCl_2$ surface than on the (1 1 0) $MgCl_2$ surface. Coadsorption of the various ID and $TiCl_4$ demonstrated that $TiCl_4$ formed greatly weaker surface complexes than the ID; obviously, upon coadsorption $TiCl_4$ occupies mainly the adsorption sites that are inaccessible for the ID because of steric reasons.

DRIFTS STUDY OF THE INTERACTION OF THE $AlEt_3$ COCATALYST WITH THE INTERNAL DONOR ETHYL BENZOATE IN SUPPORTED ZIEGLER-NATTA CATALYSTS

A.G. Potapov, G.D. Bukatov, V.A. Zakharov

J. Mol. Catal. A: Chem.,
301(1-2) (2009) pp. 18-23.

The interaction of $AlEt_3$ cocatalyst with the internal donor (ethyl benzoate-EB) in the supported Ziegler-Natta ($TiCl_4$/EB/$MgCl_2$) catalysts and the EB/$MgCl_2$ support was studied by DRIFT spectroscopy. For EB/$MgCl_2$ samples, it was shown that $AlEt_3$ firstly removed the more weak surface complexes of EB from the $MgCl_2$ surface and competed with EB at adsorbtion for releasing sites of the $MgCl_2$ surface. EB, as the external donor, keeps constant the initial content and distribution of surface complexes of EB at the interaction of the $AlEt_3$/EB mixture with the EB/$MgCl_2$ support. At the interaction of $AlEt_3$ with the $TiCl_4$/EB/$MgCl_2$ catalysts one type of surface complexes of EB is mainly removed. A part of removed EB adsorbs again on the catalyst surface in the form of complexes with aluminum-containing compounds. At the interaction of the catalyst with the $AlEt_3$/EB mixture the EB content in the catalyst changes slightly, but the distribution of different surface complexes of EB in the catalyst changes noticeably compared to that in the initial catalyst. EB, as the external donor, adsorbs both on surface $MgCl_2$ sites released at the removal of the internal donor and on aluminum-containing compounds adsorbed on the catalyst surface.

STUDY OF MULTI-SITE NATURE OF SUPPORTED ZIEGLER-NATTA CATALYSTS IN ETHYLENE-HEXENE-1 COPOLYMERIZATION

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Macromol. Symp.,
282(1) (2009) pp. 157-166.

Heterogeneity of active centers (AC) of titanium-magnesium catalysts (TMC) and vanadium-magnesium catalyst (VMC) in ethylene-hexene-1 copolymerization has been studied on the base of data of polymer molecular weight distribution (MWD) deconvolution technique and copolymer fractionation onto narrow fractions. It was found that 3 and 4 Flory components (groups of active centers) are required to describe experimental MWD curves of copolymers produced over TMC with different Ti content. In the case of VMC MWD of homopolymer and copolymer are characterized by set of 5 Flory components (5 groups of AC).

Different character of inter-relationship between MW and short chain branching (SCB) was found for ethylene-hexene-1 copolymers produced over different catalysts: heterogeneous type in the case of TMC and more uniform for copolymer prepared over VMC. The content of Ti affects on the slope of that profile in copolymers produced over TMC.

The results indicated that TMC and VMC are different greatly on the heterogeneity of active centers to the formation of polymers with different molecular weights and to formation of copolymers with different inter-relationship between MW and short chain branching. TMC produces polymers with more narrow MWD but it contains highly heterogeneous centers regarding comonomer reactivity ratios. VMC produces polymers with broad and bimodal MWD but it contains more homogeneous centers regarding comonomer reactivity ratios.

SYNTHESIS OF FUNCTIONALIZED LIQUID RUBBERS FROM POLYISOPRENE

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J. Appl. Polym. Sci.,
114(2) (2009) pp. 1241-1249.

Noncatalytic transformation of *cis*-1,4-polyisoprene rubber (M_n = 320,000) into functionalized liquid rubbers containing various amounts of carbonyl groups was studied. The process is performed via selective carboxidation of the polymer C═C bonds by nitrous oxide (N_2O) in the temperature range of 180-230°C and under 3-6 MPa pressure. The carboxidation proceeds by the nonradical type mechanism involving the 1,3-dipolar cycloaddition of N_2O to the C═C bond. The main route of the reaction (ca. 65%) proceeds without cleavage of the internal C═C bonds and leads to the formation of ketone groups in the polymer backbone. The second route (ca. 35%) includes the cleavage of C═C bonds, yielding the molecules of a smaller size. This route results in a manifold decrease of the molecular weight, which, depending on the carboxidation degree, may be more than two orders of magnitude less than that of the parent rubber. A series of functionalized liquid rubbers having M_n value from 1000 to 19,000, and the oxygen content from 0.3 to 3.9 wt % was obtained in the form of the liquid unsaturated polyketones. Similar polyketones can also be prepared by carboxidation of the natural rubber.

USE OF ULTRAHIGH-MOLECULAR-WEIGHT POLYETHYLENE FOR BEARING MATERIALS ON A STEEL SUBSTRATE

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J. Frict. Wear,
30(1) (3009) pp. 58-61.

The paper presents results on the usage of ultrahigh-molecular-weight polyethylene for manufacture of antifrictional materials on a steel substrate. Hot molding regimes are determined for the production of polymeric components. The optimum degree of ultrahigh-molecular-weight polyethylene filling with graphite is determined (about 50 mass %) for the purpose of achieving perfect triboengineering parameters of sheet antifrictional materials during operation against a steel counterbody under boundary lubrication by water.

COMPOSITE POLYMER HYDROGELS

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Polymer. Sci. Ser. A,
51(7) (2009) pp. 743-760.

Methods of preparing and properties of composite hydrogels based on various hydrophilic polymers and their mixtures with inorganic nanosized additives are considered. The effect of the type of physical or covalent bonding between components on the formation of composite hydrogels and their characteristics is discussed. The biphasic character of composite hydrogels determines as a rule their advantages as supersorbents, membrane materials, living-tissue substitutes, drug carriers, and soft-contact-lens materials.

NEW SILICONE HYDROGELS BASED ON INTERPENETRATING POLYMER NETWORKS COMPRISING POLYSILOXANE AND POLY(VINYLALCOHOL) NETWORKS

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Polym. Adv. Technol.,
20(4) (2009) pp. 367-377.

A method for the synthesis of a new silicone hydrogel as a biphase material for soft contact lenses is considered. The method is based on the synthesis of sequential interpenetrating polymer networks (IPN) and includes the following stages: (1) cross-linked silicone synthesis by the reaction of vinyl- and hydride-containing oligosiloxanes; (2) silicone network saturation with vinyl acetate and cross-linking monomer followed by UV-initiated polymerization to form an IPN comprising the silicone and cross-linked poly(vinyl acetate) (PVAc) network; (3) PVAc network alcoholysis with methanol to obtain silicone hydrogels comprising the silicone and cross-linked poly(vinyl alcohol) (PVAl). A study of hydrophilic, optical, mechanical, and structural features of the silicone hydrogels showed that optical transparency is achieved for materials with the highest density of silicone network cross-linking where the size of IPN structural units does not exceed 100 nm. The water content in hydrophilic networks of silicone hydrogel is found to be below the values typical of cross-linked PVAl, leading to non-additivity of IPN mechanical properties. Indeed, the elasticity moduli (*E*) of the hydrophilic and silicone networks are 0.4-0.7 and 0.7-1.8 MPa, respectively, whereas for some IPN this value reaches 3.0 MPa. The optimal parameters of synthesis providing the reduction of *E* to 0.8-1.6 MPa without deterioration of the required performance characteristics (optical transparency 90-92%, water content 20-39 wt%) are determined.

SUPPORTED TITANIUM-MAGNESIUM CATALYSTS FOR ETHYLENE POLYMERIZATION: A COMPARATIVE STUDY OF CATALYSTS CONTAINING ISOLATED AND CLUSTERED TITANIUM IONS IN DIFFERENT OXIDATION STATES

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J. Polym. Sci., Part A: Polym. Chem.,
47(23) (2009) pp. 6362-6372.

Supported titanium-magnesium catalysts (TMC) comprising isolated and clustered titanium ions in different oxidation states, which are obtained using titanium compounds of different composition ($TiCl_4$, $TiCl_3$•*n*DBE (DBE - dibutyl ether), [η^6-BenzeneTiAl_2Cl_8]), were synthesized and tested in ethylene polymerization. The state of titanium ions was studied by the ESR method both for the procatalysts and after their interaction with triisobutilaluminum. For identification of ESR-silent Ti^{3+} ions and Ti^{2+} ions, special procedures of additional catalyst treatment with pyridine, water, and chloropentafluorobenzene were used to obtain Ti^{3+} ions that are observable in ESR spectra. In distinction to numerous earlier works performed with the $TiCl_4/MgCl_2$ catalyst comprising after the interaction with AlR_3 the Ti^{3+} surface compounds both as isolated ions and clusters (ESR-silent), this work considers the [η^6-BenzeneTiAl_2Cl_8]/$MgCl_2$ catalyst (TMC-3) comprising mainly the isolated Ti^{2+} ions and a new catalyst TMC-4 obtained by treating the TMC-3 with chloropentafluorobenzene. This catalyst comprises only the isolated Ti^{3+} ions both before and after the interaction with triisobutylaluminum. It was shown that in spite of sharp distinctions between the catalysts under consideration concerning titanium oxidation state and the ratio of isolated Ti^{3+} ions to clustered ones, all these catalysts produce polyethylenes with similar molecular weights and molecular-weight distributions.

MONODISPERSE PARTICLES BASED ON COPOLYMERS OF METHYL METHACRYLATE OR STYRENE WITH N-VINYLFORMAMIDE

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Macromol. Symp.,
281 (2009) pp. 61-68.

Various techniques of one-step batch emulsion copolymerization of methyl methacrylate (MMA) or styrene (St) with N-vinylformamide (NVF) initiated by 2,2' -azobis(2-methylpropanamidine) (AIBA) or 2,2'-azobis[4,5-dihydroimidazol-2-yl] (AIP) dihydrochlorides in the presence of dextran, cetyltrimethylammonium bromide (CTAB) or without any stabilizers were examined to obtain monodisperse submicron and nano- particles, having both positive surface charge and hydrophilic surface. After hydrolysis in acidic media, the particle surface contained amino groups originating from NVF units along with carboxylic groups from hydrolyzed initiator residues. The obtained particles were tested as building blocks of hierarchic structures. Owing to their amphiphilic surface, the particles were capable of self-assembling from their dispersions into three-dimentional (3D) ordered arrays.

HYBRID POLYMER PROTON CONDUCTING MEMBRANE MATERIALS: INTERRELATION BETWEEN STRUCTURE AND CONDUCTING PROPERTIES

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Preprint *PIYaF* № 2813, *Gatchina, 2*009, pp. 1-24.

Hybrid polymer proton conducting membranes have been synthesized via copolymerization of monomers to form interpenetrating networks and studied by chemical and physical methods including small-angle neutron scattering (SANS). The relationship between multilevel nanostructure, water content and conductivity of materials was established. The dependence of conductivity of membranes on their mechanical modules, strength limit at a stretching and their structure details, based on mechanical tests, IR-spectroscopy, measurements of amounts of free and associated water and small-angle neutron scattering was found.

POLYMERIZATION OF ETHYLENE WITH SELF-IMMOBILIZING BIS(PHENOXYIMINE) CATALYTIC SYSTEMS

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J. Polym. Sci., Part A: Polym. Chem.,
51(7-8) (2009) pp. 276-282.

The kinetic features of polymerization of ethylene with five methylaluminoxane_activated selfimmobilizing bis(phenoxyimine) complexes of titanium chloride, namely, bis{2-[(4-allyloxyphenylimino) methyl]-6-*tert*-butylphenoxy}$TiCl_2$, bis{2-[(4-allyloxyphenylimino)methyl]-4,6-di-*tert*-butylphenoxy}$TiCl_2$, bis{2-[(4-allyloxyphenylimino)methyl]-6-cumylphenoxy}$TiCl_2$, bis{2-[(4-allyloxyphenylimino)methyl]-4,6-dicumylphenoxy}$TiCl_2$, and bis{2-[(4-allyloxyphenylimino)methyl]-6-1-(4-*tert*-butylphenyl)ethylphenoxy}$TiCl_2$ have been studied.

The activity of these complexes in the polymerization of ethylene in the temperature range 20–80°C and an ethylene pressure of 0.3 MPa has been investigated both in the homogeneous and polymer matrix-bound states. The self-immobilizing catalytic systems possess high activity (up to 40000 kgPE/molcat MPa h) and give rise to ultrahigh-molecular-weight PE (M = (2–7) • 10^6) with an improved morphology of polymer particles.

CATALYTIC ACTIVITY OF NEW BINUCLEAR TITANIUM CHLORIDE BIS(PHENOXYIMINE) COMPLEXES IN ETHYLENE POLYMERIZATION

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Doklady Phys. Chem.,
424(1) (2009) pp. 17-20.

Therefore, in this work, it was synthesized and studied a new class of binuclear titanium chloride complexes based on tetradentate salicylaldimine ligands with various aryl-containing bridges between imino groups of different structure and identical substituents in phenoxy groups (R1= R2= cumyl).

Thus, the studies of the catalytic activity of the titanium tetradentate binuclear complexes synthesized for the first time confirmed the validity of the assumption that high-activity and more thermostable bis(phenoxyimine) catalytic systems can be obtained on the basis of modified tetradentate ligands.

DESIGN OF POSTMETALLOCENE SCHIFF BASE-LIKE CATALYTIC SYSTEMS FOR POLYMERIZATION OF OLEFINS: XII. SYNTHESIS OF TETRADENTATE BIS-SALICYLALDEHYDE IMINE LIGANDS

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Russ. J. Org. Chem.,
45(4) (2009) pp. 528-535.

Reactions of salicylaldehyde, 3-*tert*-butylsalicylaldehyde, and 3,5-di-*tert*-butylsalicylaldehyde with 1,4-diaminobutane, 1,6-diaminohexane, 4,4′-diaminodiphenylmethane, 4,4′-diamino-3,3′,5,5′-tetramethyldiphenylmethane, 4,4′-diamino-5,5′-dicyclopentyl-3,3′-dimethyldiphenylmethane, 4,4′-diamino-5,5′-dicyclohexyl-3,3′-dimethyldiphenylmethane, bis(4-aminophenyl) sulfone, *o,o*′- and *p,p*′-diaminodiphenyl ethers, 1,4-bis(4-aminophenoxy)benzene, 2,2-bis[4-(4-aminophenoxy)phenyl]propane, and 4,4″-diamino-*p*-terphenyl gave a series of the corresponding Schiff bases which can be used as tetradentate ligands for the synthesis of titanium and zirconium complexes

DESIGN OF SCHIFF BASE-LIKE POSTMETALLOCENE CATALYTIC SYSTEMS FOR POLYMERIZATION OF OLEFINS: X. SYNTHESIS OF PHENOXY IMINO LIGANDS WITH BULKY SUBSTITUENTS

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Russ. J. Org. Chem.,
45(1) (2009) pp. 30-36.

Reactions of primary amines with salicylaldehydes containing bulky substituents (*tert*-butyl, 2-phenylpropan-2-yl, triphenylmethyl) in positions *3* and *5* gave a number of new Schiff bases as ligands for complex formation with transition metals.

DESIGN OF POSTMETALLOCENE SCHIFF BASE-LIKE CATALYTIC SYSTEMS FOR POLYMERIZATION OF OLEFINS: XI.* SYNTHESIS OF SCHIFF BASES CONTAINING CYCLOALKYL SUBSTITUENTS FROM 2-ACETYL-6-BROMOPYRIDINE

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Russ. J. Org. Chem., 45(1) (2009) pp. 44-47.

Reactions of substituted cycloalkylanilines with 2-acetyl-6-bromopyridine in methanol gave a number of the corresponding Schiff bases that are promising as ligands for the synthesis of cobalt(II) and iron(II) complexes.

ELECTRON BEAM INITIATED GRAFTING OF METHACRYLOXYPROPYL-TRIMETHOXYSILANE TO FUSED SILICA GLASS

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Appl. Surf. Sci., 255(12) (2009) pp. 6391-6396.

The effect of electron beam pretreatment of fused silica glass upon its surface functional composition and possibility for subsequent immobilization of methacryloxypropyl-trimethoxysilane (MOPTMS) layer is studied using FTIR spectroscopy and adsorption of acid–base indicators. The content of Brensted acidic centers (silanol groups) on the irradiated fused silica surface is found to follow an “oscillatory” trend as function of the absorbed dose below 100 kGy at electron beam processing due to the alternating reactions of hydroxylation (probably as a result of Si–O–Si bond disruption and interaction with radiolyzed physically adsorbed water) and thermal dehydration/dehydroxylation at radiation heating. The best conditions for MOPTMS layer formation are based on the increased acidity of both silica surface (formation of acidic hydroxyls) and the reaction medium (MOPTMS deposition from acetic acid solution). The optimal value of absorbed dose at electron beam processing providing the highest efficiency of MOPTMS grafting is 50 kGy at accelerated electron energy 700 keV. Electron beam pretreatment of fused silica surface is shown to provide more efficient MOPTMS immobilization in comparison with conventional chemical and thermal grafting procedures. The obtained results are promising for the enhancement of the processes for the production of fused silica glass capillaries for electrochromatography and electrophoresis at the stage of an intermediate bifunctional layer formation required for the subsequent deposition of specific polymer coatings.

THE STRUCTURE OF ELECTRON BEAM-MODIFIED POLY(ETHYLENE TEREPHTHALATE) FILMS

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High Energy Chem., 43(2) (2009) pp. 92-99.

The effect of electron-beam irradiation on the surface properties and the parameters of the semicrystalline structure of biaxially oriented poly(ethylene terephthalate) (PET) films was studied. It was shown that the crystallinity and the surface tension of the irradiated films at the interfaces with isooctane and water vary in a nonmonotonic manner over the dose range D= 25–300 kGy. As the absorbed dose increases, the dispersion and polar terms of surface energy increase, exhibiting an extremum as a result of the competing chain crystallization and amorphous-phase formation processes, as well as oxidative degradation and crosslinking of PET samples.

Biocatalysts, Biochemical Phenomena, Biochemical Ecology

CATALYTICAL PROPERTIES OF ARTHROBACTER NICOTIANAE CELLS, A PRODUCER OF GLUCOSE ISOMERASE, IMMOBILIZED INSIDE XEROGEL OF SILICIUM DIOXIDE

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Appl. Biochem. Microbiol.,
45(4) (2009) pp. 389-394.

Arthrobacter nicotianae cells, producers of glucose isomerase, were immobilized inside xerogel of silicium dioxide, and properties of the resulted heterogeneous biocatalysts were investigated in the process of isomerization of monosaccharide (glucose and fructose). The glucose isomerase activity of the resulted biocatalysts was shown to be 10 U/g, on average, taking into account the loss of the activity upon the immobilization, which amounted to 50% of the cell activity in suspension. The rate of the fructose isomerization increased linearly in the range of 55–80°C with the temperature coefficient 1.3. The biocatalysts were stable in this range; they were rapidly inactivated, however, at increasing temperature. The half time for inactivation was 6-7 h and $\leq$5 min at 80 and 85°C, respectively. The half time for inactivation of heterogeneous biocatalysts was 50–90 h in the periodic process of isomerization of 2 M monosaccharides at 60°C in the presence of the immobilized *Arthrobacter nicotianae* cells.

DEVELOPMENT OF NEW CATALYTIC SYSTEMS FOR UPGRADED BIO-FUELS PRODUCTION FROM BIO-CRUDE-OIL AND BIODIESEL

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V.O. Dundich, D.Yu. Ermakov,
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Catal. Today,
144(3-4) (2009) pp. 362-366.

The investigation of upgraded bio-fuels production processes was carried out via the development of efficient catalysts for oxy-organic hydrodeoxygenation (HDO) processes. It was found that Ni–Cu catalysts are more attractive than single Ni catalysts in HDO under mild conditions. Copper facilitates the nickel oxide reduction at temperatures lower than 300°C. Moreover, copper prevents methanization of oxy-organics at 280–350°C. The catalyst supports play also a key role in hydrotreatment of oxygen-containing compounds. Screening of catalyst supports showed that CeO_2 and ZrO_2 are most effective in the target processes because of possible additional activation of oxy-compounds on the support surface. The prepared catalysts have non-sulfided nature and can be used for upgrading of bioliquids with a low sulfur content.

HYDRODEOXYGENATION OF BIODIESEL IN THE PRESENCE OF NOBLE METALS

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Chem. Sustain. Devel.,
17(5) (2009) pp. 527-532.

Reaction of the catalytic hydrodeoxygenation of biodiesel - a mixture of methyl esters of fatty acids - is investigated for the purpose of obtaining linear alkanes. Precious metals (Ru, Rh, Pd, Ir, Pt) deposited on the binary oxide system CeO_2-ZrO_2 were used as catalysts. It is shown that these catalysts allow one to carry out efficient deoxygenation of methyl esters of fatty acids under hydrogen pressure 0.5 MPa within a broad temperature range (300-380^{o}C) resulting mainly in heptadecane ($C_{17}H_{36}$). However, only in the presence of Rh and Ru catalysts the yield of alkanes is quantitative. The process of obtaining linear alkanes of the C_{14}-C_{18} series using this method is positioned as a stage of production of high-quality kerosene fuel of plant origin.

MICROBIAL COMMUNITIES OF ALKALINE HOT SPRINGS AS A MODEL FOR STUDYING EARLY STAGES OF BIOSPHERE EVOLUTION

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Orig. Life Evol. Biosph.,
39(3-4) (2009) pp. 305-306.

According to the hypothesis of the first Precambrian prokaryotic communities origin and development and their attendant environment (Zavarzin, 2004; Gerasimenko, 2004), chemical and gas composition and primary phototrophes structure of Barguzin valley hot springs in Baikal rift zone might represent analogs to relict Precambrian biocenoses. The research concerned microbial communities structure and composition, hot springs macro- and microelements composition, minerals formed in microbial mat and a wide range of elements distribution between organic and mineral parts of mats.

Basing upon studying of structure and some specific features of Baikal rift zone hydrotermes microbial communities functioning, it is possible to get a notion about the processes which occurred in Precambrian primary prokaryotic community and its significance for the modern biosphere formation.

ELEMENTS REDISTRIBUTION BETWEEN ORGANIC AND MINERAL PARTS OF MICROBIAL MATS: SR-XRF RESEARCH (BAIKAL RIFT ZONE)

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Nucl. Instrum. Methods Phys. Res., Sect. A,
603(1-2) (2009) pp. 137-140.

In article minerals formation and elements accumulation in microbial mats of some hot springs of the Barguzin basin (Baikal Rift Zone) is discussed. The content of a wide spectrum of elements in microbial mats is studied by means of the method SR-XRF. Regularity of elements accumulation by community depending on geochemical features of hot spring's waters are discussed. These elements are distributed in different ways between organic and mineral substance of the microbial mats. The distribution of K, Mn, Ni, Cu, Zn, Fe is regular, Ca, Rb, Sr are almost totally related with the mats mineral part, while Ga, Ge and Br are accumulated in mats organic substance. Germanium element is concentrated in considerable amounts in the cyanobacterial communities, that develop in sulphideless springs with a higher radon concentration.

MODERN THERMOFILIC CYANOBACTERIAL MATS: GEOCHEMICAL FEATURES

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*Goldschmidt Abstr. 2009 – B,
Geochim. Cosmochim. Acta*,
73(13) Suppl. 1 (2009) p. A731.

This study considers cyanobacterial communities of the Barguzin Valley (Baikal Rift Zone) hot springs and a complex research of minerals forming and redistribution of elements, C isotopes and ^{226}Ra, ^{228}Ra between the organic and mineral parts of microbial mars. Hot springs are alkaline hydrotherms. The Alla type waters (Alla, Kuchiger, Umkhei, Seya) are SO_4-HCO_3-Na solutions with high HS- and low Rn (4 eman) content. The Garga, Uro and Gusikha spring waters are HCO_3-SO_4-Na and characterized by the absence of HS- in the solution and Rn content is of 30-10 eman. In all hot springs solutions alkaline, alkaline-earth (Li, Rb, Sr, Cs, Ba) and anionogenic elements (Si (30-45 ppm), Ge, Mo, W) prevail. SiO_2 precipitation proceeds as the result of the thermal solution evaporation from the microbial mat surface and of the silicic acid gel coagulation. The adhesion of SiO_2 flakes upon cyanobacteria filaments results in forming of covers. Rhombohedral and prismatic calcite crystals are precipitated in a microbial communities which develop in the waters with Ca concentration more than 10 mg/l (Alla, Garga, Gusikha). Calcite which is being formed in a microbial community is enriched with δ^{13}C relatively to the organic matter. The distribution of K, Mn, Ni, Cu, Zn, Fe between the organic and mineral parts of cyanobacterial mats is regular; Ca, Rb, Sr are almost totally related with the mineral part, while Ga, Ge and Br are accumulated in organic substance. Ge (up to 600 ppm) and Ra isotopes (up to 7000 Bk/kg) are concentrated in the cyanobacterial communities, which develop in sulphideless, Rn-bearing springs. There is an opportunity examined of a complex use of the cyanobacterial mat geochemical characteristics as biosignatures in terms of the Garga spring carbonate deposits.

ANTIOXIDATIVE AND IMMUNE-RESPONSE MODULATING PROPERTIES OF NATURAL ZEOLITES

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Pacific Med. J.,
3 (2009) pp. 62-64.

The local immunity system in lungs is permanently exposed to injurious action of a number of chemical, physical and biological factors. Among other things, these are mineral microparticles. This paper gives consideration to assessment of effects produced by the Vanginskiy and Kulikovskiy zeolite deposits on the local immunity in lungs. The authors used low energy laser radiation as a factor very likely to induce lipid peroxidation and reduce functional activity of alveolar macrophages and lymphocytes. The studies allow to conclude that unlike the Kulikovskiy deposit zeolites appeared to intensify free radical reactions and suppress the local immunity system, the Vanginskiy deposit zeolites exhibited immunoprotective and antioxidative properties.

AN EXAMINATION OF THE ABILITY OF TITANIUM DIOXIDE NANOPARTICLES AND ITS CONJUGATES WITH OLIGONUCLEOTIDES TO PENETRATE INTO EUCARIOTIS CELLS

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Nanotechn. Russ.,
4(9-10) (2009) pp. 732-735.

In this study the possibility that TiO_2 nanoparticles and their conjugates can penetrate into cultivated cells without any special transfection procedures was investigated. Oligonucleotides and their derivates were conjugated with the TiO_2 nanoparticles, which were obtained as colloidal solutions at a concentration of TiO_2 0.3M by $TiCl_4$ hydrolysis. The electronic microscopy of various cell cultures (KCT, Vero, and MDCK) treated with nanoparticle solutions (20 µg/µl) showed that nanoparticles could enter the cells and accumulate in the vacuoles and phagosomes and form inclusions in cytoplasm. Thus, it was demonstrated the penetration of TiO_2 nanoparticles and their oligonucleotide conjugates into intracellular space without any auxiliary operations. Most other researches used electroporation techniques for similar purposes.

Catalysis in Organic Synthesis

METHODS OF OBTAINING AND OUTLOOKS FOR THE DEVELOPMENT OF MODERN LOW-WASTE PRODUCTION OF GUANAMINES IN RUSSIA

L.L. Gogin, E.G. Zhizhina, Z.P. Pai, V.N. Parmon

Chem. Sustain. Devel.,
17(4) (2009) pp. 339-348.

Known laboratory and industrial methods of obtaining guanamines (6-alkyl- or aryl-substituted 2,4-diamino-1,3,5-triazines) based on modification of substituents in the triazine ring and synthesis from acyclic precursors are described. It is shown that at present the most convenient method of obtaining guanidines is the reaction of dicyandiamide with nitriles in the presence of a strong base, with high-boiling alcohols as solvents. The reaction conditions are considered in detail, the data on its kinetics and mechanism are presented. The conditions of the industrial implementation of this reaction allowing one to develop an up-to-date low-waste technology of obtaining high-quality guanamines are described.

CATALYTIC OXIDATION OF FATTY ACIDS. I. EPOXIDATION OF UNSATURATED FATTY ACIDS

Z.P. Pai, T.B. Khlebnikova, Yu.V. Mattsat, V.N. Parmon

React. Kinet. Catal. Lett.,
98(1) (2009) pp. 1-8.

Unsaturated fatty acids and their derivatives were oxidized with aqueous hydrogen peroxide in two-phase systems in the presence of homogeneous catalysts $Q_3\{PO_4[WO(O_2)_2]_4\}$, where Q is $[Bu_4^n]_3^+$, $[C_5H_5N(n\text{-}C_{16}H_{33})]_3^+$ or $[Me(n\text{-}C_8H_{17})_3N]_3^+$. The reactions occurred under mild conditions ($T < 100°C$, atmospheric pressure), without organic solvents, and exhibited high conversion and selectivity.

CATALYTIC OXIDATION OF FATTY ACIDS.

2. EPOXIDATION AND OXIDATIVE CLEAVAGE OF UNSATURATED FATTY ACID ESTERS CONTAINING ADDITIONAL FUNCTIONAL GROUPS

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React. Kinet. Catal. Lett., 98(1) (2009) pp. 9-17.

Methyl esters of monounsaturated fatty acids were oxidized with aqueous hydrogen peroxide in biphasic organic-aqueous systems in the presence of catalyst $[Me(n\text{-}C_8H_{17})_3N]_3\{PO_4[WO(O_2)_2]_4\}$. Epoxidation and oxidative cleavage of the C=C bond were found to proceed with high conversion and selectivity in the absence of organic solvents and under rather mild conditions ($T < 100°C$, 1 atm).

ABOUT SELECTIVE METHODS OF SYNTHESIS OF 6-TERT-BUTYL-2-METHYLPHENOL AND 6-TERT-BUTYL-2,4-DIMETHYLPHENOL

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Russ. J. Gen. Chem., 79(6) (2009) pp. 1156-1162.

Vapor phase catalytic methylation with methanol of 2-*tert*-butylphenol at the temperature 280–300°C proceeds selectively with formation of 6-*tert*-butyl-2-methylphenol. Elevating reaction temperature above 300°C leads to formation of 2,6-dimethylphenol. Reaction of 2-*tert*-butylphenol with methanol in alkaline medium in the presence of zinc oxide is shown to lead initially to formation of a mixture of calixarenes and methylenebisphenols that at elevated temperature exert splitting leading in future to 6-*tert*-butyl-2,4-dimethylphenol. Obtaining it in this reaction from 2,2′-methylenebis-(6-*tert*-butyl-4-methylphenol) proceeds selectively. Pathways of the reductive methylation of methylenebisphenols with methanol in alkaline medium is considered.

CATALYTIC DEHYDROCHLORINATION OF 1,2-DICHLOROETHANE TO VINYL CHLORIDE ON INDIVIDUAL OXIDES AND TWO-COMPONENT SILICATE CATALYSTS

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Chem. Sustain. Devel., 17(4) (2009) pp. 423-428.

Catalytic dehydrochlorination of 1,2-dichloroethane on a series of two-component silicate catalysts with regularly changing acid-base properties (Al_2O_3/SiO_2, Ga_2O_3/SiO_2, ZiO_2/SiO_2, BeO/SiO_2, Y_2O_3/SiO_2) and on oxides (γ-Al_2O_3, MgO) is investigated in a flow set-up at a temperature within the range 250-450^{o}C. It is established that the degree of 1,2-dichloroethane transformation into vinyl chloride is determined by the strength of Lewis acid centres. It is shown that catalyst deactivation is due to the formation of 1,3-dichlorobutene-2.

SYNTHESIS OF POLYFLUORINATED 4-PHENYL-3,4-DIHYDROQUINOLIN-2-ONES AND QUINOLIN-2-ONES VIA SUPERACIDIC ACTIVATION OF *N*-(POLYFLUOROPHENYL)CINNAMAMIDES

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Tetrahedron Lett., 50(37) (2009) pp. 5245-5247.

The cyclization reactions of a series of polyfluorocinnamanilides in triflic acid (CF_3SO_3H) yield 4-phenyl-3,4-dihydroquinolin-2-ones, which include a polyfluorinated benzene moiety as a part of the quinoline scaffold. These compounds undergo dehydrophenylation in the presence of $AlCl_3$ to give the corresponding polyfluoroquinolin-2-ones which are converted into polyfluorinated 2-chloroquinolines on treatment with $POCl_3$. A mechanism for the cyclization reaction presuming the intermediacy of a superelectrophilic O,C-diprotonated form of the starting material is suggested.

ACTIVITY OF CATALYSTS IN THE SYNTHESIS OF DIMETHYL SULFIDE FROM DIMETHYL DISULFIDE

A.V. Mashkina, L.N. Khairulina

Kinet. Catal., 50(3) (2009) pp. 414-419.

Dimethyl disulfide conversion at T = 190–350°C over catalysts containing acid and basic sites is reported. The products of this reaction are dimethyl sulfide, methanethiol, hydrogen sulfide, carbon disulfide, methane, and ethylene. At 190°C, these products form via parallel reactions. At higher temperature of up to 350°C, dimethyl sulfide can form by the condensation of the resulting methanethiol. The strong basic sites of the catalysts are uninvolved in dimethyl sulfide formation. Over catalysts whose surface has only strong protonic or strong Lewis acid sites, dimethyl sulfide formation does take place, but slowly and nonselectively. The highest dimethyl sulfide formation activity and selectivity are shown by catalysts having medium-strength basic sites along with strong protonic and strong Lewis acid sites.

THE FEATURES OF THE CATALYTIC SYNTHESIS OF METHANETHIOL FROM DIMETHYL SULFIDE

A.V. Mashkina

Pet. Chem.,
49(5) (2009) pp. 420-426.

The characteristic features of methanethiol synthesis from dimethyl sulfide and H_2S in the presence of Al_2O_3 at atmospheric pressure and T = 320–500°C have been studied. It has been shown that the yield of methanethiol increases with an increase in the temperature, the H_2S-to-dimethyl sulfide ratio, and the contact time, attaining equilibrium values. The methanethiol formation rate is proportional to the dimethyl sulfide partial pressure raised to a power of 0.4 and the H_2S partial pressure raised to a power of 0.8. An increase in the specific surface area and the volume of transport pores and a decrease in the particle sizes of Al_2O_3 facilitate the augmentation of the catalyst activity in methanethiol formation. At T ~ 400°C, a low H_2S concentration, and a long contact time, the side reaction of dimethyl sulfide cracking occurs to result in the release of methane and the deposition of sulfur-containing and carbonaceous compounds on the surface, which lower the activity of alumina. The deactivated catalyst can be regenerated by oxidation.

MODIFICATION OF 1,4-cis-POLYBUTADIENE RUBBER WITH OLIGODIENES

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News University Chem. Chem. Techn.,
52(1) (2009) pp. 94-97.

Modifying effect of oligobutadienes and low-molecular unsaturated polyketone (functional oligomer), obtained by treatment of SKD rubber with N_2O, on technological and physico-mechanic properties of 1,4-cis-polybutadiene rubber and elastomer composition on its base is studied.

EFFECT OF ADDITION OF OLIGOMERS (LIQUID RUBBERS) ON THE PROPERTIES OF RUBBER COMPOUNDS AND VULCANIZED RUBBERS

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Russ. Rubbers,
5 (2009) pp. 25-28.

Modifying effect of two oligobutadienes and functional oligomer of new type (unsaturated polyketone) on the properties of row and vulcanize butadiene rubbers was studied. It was shown that addition of the polyketone into raw compound improves some of processing and mechanical properties of obtained rubbers.

THE FIRST EXAMPLE OF THE *ORTHO*-DIRECTING EFFECT OF THE WEAKLY COORDINATING SUBSTITUENT $[–BF_3]^-$ IN THE CATALYTIC HYDRODEFLUORINATION OF THE PENTAFLUOROPHENYL-TRIFLUOROBORATE ANION

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Mendeleev Commun.,
19(5) (2009) pp. 260-262.

The pentafluorophenyltrifluoroborate anion undergoes hydrodefluorination under the action of zinc in the presence of nickel complexes turning into the 2,3,4,5-tetrafluoroborate anion.

POLYFLUORINATED ORGANOBORON COMPOUNDS. PECULIAR REACTIVITY IN THE PD-CATALYZED CROSS-COUPLING REACTIONS

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Chimica Oggi–Chemistry Today, 27(3) (2009)
Suppl. “Focus on Fluorine Chemistry”, pp. 31-33.

Application of polyfluorinated organoboron compounds as partners in the Pd-catalyzed cross-coupling reactions (the Suzuki-Miyaura reaction) and key distinguishes from reactivity of their non-fluorinated and less fluorinated analogues are reviewed

FLUORINE-CONTAINING HETEROCYCLES: XIX. SYNTHESIS OF FLUORINE-CONTAINING QUINAZOLIN-4-ONES FROM 3,1-BENZOXAZIN-4-ONES

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Russ. J. Org. Chem., 45(6) (2009) pp. 913-920.

Reactions of fluorine-containing 3,1-benzoxazin-4-ones with ammonium acetate, hydrazine, and heteroaromatic amines gave new 3*H*-, 3-amino-, and 3-hetarylquinazolin-4-ones, respectively. Differences in the conditions of formation of benzoxazinones from anthranilic acids with different fluorination patterns and in the reactions of fluorinated 3,1-benzoxazinones with nitrogen-centered nucleophiles were revealed.

TRANSFORMATIONS OF ALLYLAMINE AND N-ALLYLACETAMIDE ON TRIOSMIUM CLUSTER COMPLEXES WITH HEMILABILE LIGANDS

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Polyhedron, 28(13) (2009) pp. 2754-2758.

The cluster complexes $(\mu\text{-H})Os_3(\mu\text{-O}{=}CR)(CO)_{10}$ (R = Me (**1**); R = Ph (**2**)) catalyze the allylic rearrangement of N-allylacetamide at room temperature. Microwave irradiation greatly (by more than several thousand-fold) accelerates the reaction between the solution of **2** and allylacetamide. The interaction of cluster **2** with allylamine both at room and elevated temperatures results in cleavage of the C–N bond and coordination of the allylic fragment to one of the osmium atoms with the formation of $Os_3(CO)_9(\mu,\eta^2\text{-O}{=}C\text{-Ph})(\eta^3\text{-}C_3H_5)$ (**4**) cluster.

Mathematical Simulation, Calculating Models

MATHEMATICAL SIMULATION OF THE OXIDATION OF ORTHO-XYLENE TO PHTHALIC ANHYDRIDE IN A FLUIDIZED CATALYST BED

S.I. Reshetnikov, A.A. Ivanov, V.P. Gaevoi

Kinet. Catal., 50(2) (2009) pp. 325-331.

The oxidation of *ortho*-xylene to phthalic anhydride in a catalytic fluidized-bed reactor was simulated. The mathematical model implied that the reactor was separated into two temperature zones throughout the height of the bed and the catalyst circulated between these zones. The parameters responsible for the regulation of the catalyst state and, correspondingly, the process selectivity, were analyzed. It was found that the temperatures in the zones and the rate of catalyst circulation between them affected the selectivity of the reaction.

TRANSFORMATIONS OF ORGANIC COMPOUNDS IN SUPERCRITICAL FLUID SOLVENTS: FROM EXPERIMENTS TO KINETICS, THERMODYNAMICS, SIMULATION, AND PRACTICAL APPLICATIONS

V.I. Anikeev

Kinet. Catal., 50(2) (2009) pp. 284-296.

The results of fundamental studies performed by the author on the reactivity of supercritical fluid solvents are systematized and generalized; practical applications of these solvents are considered. Methods for performing kinetic and physicochemical experiments, processing the data, constructing kinetic models, and calculating the thermodynamics of nonideal supercritical fluids (the parameters of critical points, binodal and spinodal lines, and critical point drifts; the thermophysical properties of reaction mixtures under supercritical conditions; etc.) are described. Attention is focused on the effect of supercritical fluid pressure on the rate constants of chemical reactions. The kinetics and mechanisms of the reactions of 2-propanol dehydration and decomposition of aliphatic nitro compounds in supercritical water and the isomerization of terpene compounds (α-pinene, β-pinene, and turpentine) in supercritical lower alcohols are studied. The formation of nanoparticles in supercritical fluid solvents and the treatment of ultradisperse diamonds in supercritical water are considered. The results formed the basis for the simulation and calculation of acetic acid and phenol oxidation processes in supercritical water and the Fischer-Tropsch synthesis in a supercritical solvent and for the development of a pilot plant for the complete oxidation of trinitroglycerol and diethylene glycol dinitrate in supercritical water.

HIGH-PRESSURE THERMOLYSIS OF SULFATE TURPENTINE

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I.V. Kozhevnikov, V.I. Anikeev (**Vorozhtsov Novosibirsk Institute of Organic Chemistry, Novosibirsk, Russia*)

J. Supercrit. Fluids,
48(2) (2009) pp. 139-145.

Thermolysis of sulfate turpentine was studied in the pressure range of 40–280 atm. The conversion kinetics of main turpentine components (α-pinene, 3-carene) and their isomerization products (limonene, alloocimenes, pyronenes) was investigated and compared with the conversion kinetics of these compounds in supercritical ethanol. Thermodynamic parameters $\Delta H^{\#}$, $\Delta S^{\#}$ and $\Delta G^{\#}$ of the reversible reaction “α-pinene-[TS]” were estimated, the observed discrepancies being interpreted in the context of transition state theory. The thermolysis rate of α-pinene as a turpentine component was compared to that in a supercritical diluted solution of ethanol versus pressure. The rate of α-pinene conversion was shown to depend on the density of working medium.

THERMODYNAMICS OF MULTICOMPONENT REACTION MIXTURES AT SUB- AND SUPERCRITICAL CONDITIONS

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Supercrit. Fluids, Theor. Practice,
4(1) (2009) pp. 18-48.

The mathematical and calculating models were developed to solute the specific research problems on thermodynamics of multicomponent and multiphase mixtures. The features of chemical and phase equilibria were examined both for the ideal-gas approximation and the non-ideality. The conditions of equilibrium phase stability were studied for multi phase systems. The calculation results of specific phase diagrams, binodal and spinodal are demonstrated for the appropriate reaction mixtures. The new interpretation of mathematical model was suggested for the localization of critical point, especially for fixed multicomponent chemical mixtures. The new variation of classical homotopy method was developed for the soluting of complex nonlinear equations systems, which are the principles of concerned mathematical models. The some anomalous phase profiles and the critical curves were analyzed to demonstrate the necessity of taking into account the pointed features at the planning of experiments, the reactor parameters calculations. The examples of specific multicomponent non-ideal mixtures indicate the possibility of thermodynamic and thermophysical behaviors calculations (entropy, enthalpy and heat capacity; thermal reaction effect, adiabatic warming up). It was noted that the cubic state equations are quite suitable for the prediction of property deviations from the ideal-gas state and can be used for the prognosis of behavior anomaly at the critical point surrounding.

SIMULATION OF GAS DIFFUSION IN POROUS LAYERS OF VARYING STRUCTURE

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J. Struct. Chem.,
50(3) (2009) pp. 456-460.

Gas diffusion in porous layers of varying structure was simulated numerically. Mesoporous mesophase material (MMM) and silica gel layers were studied. The former were a set of ordered cylinders; the latter were disordered packings of spheres. The average residence time of a molecule in a layer (return time) and dispersion of this time in relation to the layer depth were calculated. For the same porosity and specific surface of layers, the average return time is independent of the pore structure and increases with the layer depth as a linear function. This is the consequence of the general theoretical result, according to which the duration of molecule wandering in a pore depends only on the ratio of the pore volume to the section area of its windows. Dispersion of the wandering time is sensitive to the pore structure; it is slightly smaller for regular pores than for a complex pore system. The functional dependence of return time dispersion on the layer depth is the same for different layers (the cubic root of dispersion changes with the layer depth as a linear function). This work helps to understand recent experimental data, which showed that using MMM for gas chromatographic columns increased the efficiency of the latter compared with other columns based on silicon oxide.

MODELING OF CHAOTIC DYNAMICS IN A HETEROGENEOUS CATALYTIC REACTION

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AIP Conf. Proceed.,
Int. Conf. on Numer. Anal.and Appl. Mathem.,
1168 (2009) pp. 264-266.

This paper addresses the phenomenon of homoclinic chaos in a kinetic model with fast, intermediate, and slow variables. The model describes the behavior of the heterogeneous catalytic reaction of interaction of hydrogen and oxygen on metallic catalyst. The subharmonic period-doubling cascade that is observed under a parameter variation in the system of three nonlinear ordinary differential equations leads to the generation of a global attractor. The skeleton of the attractor consists of a family of unstable Möbius orbits of large periods. Numerical experiments show that a typical trajectory on the attractor under consideration is chaotic.

CHAOTIC DYNAMICS IN THE THREE-VARIABLE KINETIC MODEL OF CO OXIDATION ON PLATINUM GROUP METALS

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Chem. Eng. J.,
154(1-3) (2009) pp. 82-87.

The paper addresses the development of a simple three-variable kinetic model of CO oxidation on metallic catalysts under isothermal conditions and the numerical study of chaotic dynamics of the reaction rate. It is suggested that, at some concentrations of the adsorbed oxygen, a surface modification occurs and the reaction capability of adsorbed oxygen changes, so that the activation energy of the interaction between the adsorbed species increases sharply. Moreover, oxygen atoms can penetrate into the subsurface catalyst layer. At some critical values of the subsurface oxygen concentration, the catalyst surface reconstructs and the probability of oxygen adsorption decreases sharply. Based on these suggestions, a three-variable kinetic model of CO oxidation on the metals of the platinum group is developed, which has the hierarchy of characteristic times and one slow variable. For the purpose of studying the dynamics of the model, the one-parameter family of two-variable subsystems with fast variables is considered, and the steady states and periodic solutions of these subsystems were investigated. The conditions are described under which the dynamics of the model becomes chaotic by means of a cascade of period-doubling bifurcations or owing to complex multipeak oscillations.

PROCESSES OF SELECTIVE OXIDATION IN FLUIDIZED BED REACTOR AT A NON-STATIONARY CONDITION OF THE CATALYST

S.A. Pokrovskaya

Chem. Ind. Today,
9 (2009) pp. 37-41.

On the basis of methods of mathematical modeling dependence of selectivity of the process on a catalyst condition in reactors with fluidized bed is studied. For a number of processes of selective oxidation of hydrocarbons operating conditions at which the non-stationary condition of the catalyst is formed are defined, allowing to raise an yield of a target product.

CHEMPAK SOFTWARE PACKAGE AS AN ENVIRONMENT FOR KINETICS SCHEME EVALUATION

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Chem. Prod. Process Model.,
4(4) (2009) Article 3.

A new technology for the simulation of physico-chemical processes in reactive medium is suggested, which allows optimizing and adjusting kinetic schemes of chemical reactions. To implement the technology, a ChemPAK software tool, which is used to solve the primal problems of chemical kinetics, was developed. The proposed technological solution proved to be efficient in a series of studies. Schemes presented in the literature for the gas-phase pyrolysis of ethane and the Butlerov reaction of organic synthesis of sugars were considered along with their modifications.

A 3D-CODE FOR MODELLING OF THE ULTRA RELATIVISTIC BEAMS

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Comput. Techn.,
14(5) (2009) pp. 18-29.

An algorithm for modeling of a collision of beams of charged particles is presented for the three-dimensional ultra relativistic case. A new algorithm for imposing of boundary and initial conditions, based on a new artificial potential, is proposed. A comparison with calculations on the Guines-Pig code for the case of focusing colliding beams is presented.

Chemical Engineering

CATALYSIS AND NANOTECHNOLOGIES: FROM FUNDAMENTAL RESEARCH TO MODERN RUSSIAN LARGE-SCALE INDUSTRY

V.N. Parmon

Catal. Ind.,
1(1) (2009) pp. 1-10.

The role and innovation potential of the newest functional nanomaterials—catalysts—and catalytic technologies in the now day economics of Russia and the influence of up-to-date nanotechnological approaches to designing new materials on the progress of catalysis science and practice is discussed. The priority areas are identified where the most intense development is expected in the nearest decade. Among them are modernization of oil-refining and petrochemical industries aimed at an increase in the commercial yield of the value-added products, implementation of innovative catalytic technologies for processing of oil associated gases, structural modernization of the Russian nitrogen industry, accelerated development of the production of polymers and tires of the new generation, and a particular attention to modern environmental protection technologies. With the state support, the large-scale catalytic technologies may provide the real contribution to the Russian GNP growth in amount more than a trillion roubles by 2015.

EFFICIENCY OF Zn/TiO$_2$ CATALYST OPERATION IN A MICROCHANNEL REACTOR IN METHANOL STEAM REFORMING

A.G. Gribovskii, L.L. Makarshin, D.V. Andreev, S.V. Korotaev, P.M. Dutov*, R.M. Khantakov*, S.I. Reshetnikov, V.N. Parmon (**Novosibirsk State University, Novosibirsk, Russia*)

Kinet. Catal.,
50(1) (2009) pp. 11-17.

The activity of a Zn/TiO$_2$ catalyst deposited on metal microchannel plates in methanol steam reforming was studied. The catalyst exhibited maximum activity upon deposition on microchannel plates made of copper foam. In this case, the specific hydrogen production of a microreactor at 450°C was 78.6 l (g cat)$^{-1}$ h^{-1}. The catalysts deposited on a microchannel plate of nickel foam and on corrugated brass foil exhibited lower activity because of the lower efficiency of heat transfer to the reaction zone. A correlation between the thermal conductivity of the microchannel plate material and the activity of the catalyst was observed in the following order: copper, brass, and nickel. The kinetic parameters of the process of methanol steam reforming in a microreactor were calculated with the use of a plug-flow reactor model. In this case, the calculated formal activation energy of 132 kJ/mol was independent of the microchannel plate material. A comparison of the equilibrium concentrations of reaction products at the reactor outlet, which were calculated from thermodynamic data, with the experimental data demonstrated that methanol steam reforming at a temperature higher than 400°C occurred in the nonequilibrium region. The concentration of carbon monoxide at the microreactor outlet was lower than 1 mol %, which is lower than the equilibrium concentration by one order of magnitude. This effect was attributed to the suppression of the reversed water gas shift reaction on the catalyst.

DEACTIVATION OF A Zn/TiO_2 CATALYST IN THE COURSE OF METHANOL STEAM REFORMING IN A MICROCHANNEL REACTOR

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Kinet. Catal.,
50(3) (2009) pp. 444-449.

The service life tests of a Zn/TiO_2 catalyst deposited on the microchannel plates of copper foam, nickel foam, and corrugated brass foil in the process of methanol steam reforming demonstrated that the catalyst stability and operation time depend on microchannel plate material. The rate of catalyst deactivation correlated with the thermal conductivity of the microchannel plate material. It was found that catalyst deactivation resulted from the decomposition of zinc titanates, which are active components, and it was accompanied by the appearance of a zinc oxide phase. The best results in the service life tests were obtained with the microchannel plates of copper foam. A microchannel reactor containing 16 copper plates continuously operated at 400°C for 150 h; in this case, the conversion of methanol decreased by 8%. The subsequent microreactor operation for 500 h caused a decrease in the methanol conversion by 26%. It was found that the loss of the catalyst activity was a reversible process, and the activity can be restored by annealing in air.

A REACTOR FOR THE STUDY OF HOMOGENEOUS PROCESSES USING LASER RADIATION ENERGY

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Chem. Eng. J.,
150(1) (2009) pp. 231-236.

A flow reactor with the reactants heated by continuous CO_2 laser radiation is proposed for studying the gas phase homogeneous reactions. Ethane pyrolysis yielding ethylene is considered as an example of such chemical process. For this process, a mode of ‘energetic catalysis’ is feasible, where increasing energy absorption in the volume is related with the increased content of target product. The mode of ‘energetic catalysis’ was implemented at transformation of laser energy into thermal power using the sensitization properties of ethylene. In the pyrolysis reaction zone, temperature measurements were made for a gas mixture in the laser radiation field with a power density up to 10^2 W/cm^2. The walls and windows were isolated from the high-temperature zone by argon feeding and configuration of the reactor. Three-dimensional calculation of the gas-dynamic reactants flows and their mixing made with the FLUENT software package showed the presence of the modes where reaction zone with a high content of C_2 hydrocarbons is localized in the center of reactor, which was confirmed by experiments. High values of ethane conversion up to 80 vol.% were obtained at nearby 53% selectivity for ethylene.

PRODUCTION OF HYDROGEN BY METHANE CATALYTIC DECOMPOSITION OVER Ni–Cu–Fe/Al_2O_3 CATALYST

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Int. J. Hydrogen Energy,
34(7) (2009) pp. 2979-2985.

Catalysts with high nickel concentrations 75%Ni–12%Cu/Al_2O_3, 70%Ni–10%Cu–10%Fe/Al_2O_3 were prepared by mechanochemical activation and their catalytic properties were studied in methane decomposition. It was shown that modification of the 75%Ni–12%Cu/Al_2O_3 catalyst with iron made it possible to increase optimal operating temperatures to 700–750°C while maintaining excellent catalyst stability. The formation of finely dispersed Ni–Cu–Fe alloy particles makes the catalysts stable and capable of operating at 700–750°C in methane decomposition to hydrogen and carbon nanofibers. The yield of carbon nanofibers on the modified 70%Ni–10%Cu–10%Fe/Al_2O_3 catalyst at 700–750°C was 150–160 g/g. The developed hydrogen production method is also efficient when natural gas is used as the feedstock. An installation with a rotating reactor was developed for production of hydrogen and carbon nanofibers from natural gas. It was shown that the 70%Ni–10%Cu–10%Fe/Al_2O_3 catalyst could operate in this installation for a prolonged period of time. The hydrogen concentration at the reactor outlet exceeded 70 mol%.

MODELING OF A HEAT-INTEGRATED CATALYTIC REFORMER/COMBUSTOR OF METHANE: FINE BALANCING BETWEEN HOT SPOTS AND EXTINCTION

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Chem. Eng. Technol.,
32(9) (2009) pp. 1367-1375.

Modeling is used to study the parametric sensitivity of a heat-integrated methane steam reformer/combustor for hydrogen production. The simulated reformer design is based on structured metal-porous catalysts and the co-current flow mode operation. The modeling results indicate that the reformer has high intrinsic sensitivity to the kinetic activity of reforming and combustion catalysts and to the axial or transversal coefficients of heat conductivity. Moreover, a hysteresis effect with critical points of ignition and extinction was found under synchronous load variation in the reforming and combustion units. These results show that fine tuning of the main design parameters is necessary to avoid both hot spot formation and extinction.

COMPARISON OF EXTERNAL MASS TRANSFER APPROACHES FOR HETEROGENEOUSLY CATALYZED HYDROGENATION OF VAPORIZED HYDROCARBONS

A.B. Shigarov, V.A. Kirillov

Chem. Eng. J.,
154(1-3) (2009) pp. 120-130.

The catalytic hydrogenation of vaporized α-methylstyrene (AMS) and 1-octene under external transfer control is tested by different interphase mass transfer approaches. The previously reported experimental data on temperature measurements in the single-pellet reactor and the new experimental data in a fixed-bed reactor are analyzed theoretically. The independent diffusion (Fick) approach absolutely failed to fit the experiments. Three methods that take into account multicomponent diffusion (and are based on the Maxwell–Stefan equations) give close results and agree well with the experimental data. Nevertheless, for hydrogenation (or dehydrogenation) of vaporized hydrocarbons the pseudo-binary approach considered in this study is recommended. It is very simple to use even in a case of complex mixture of hydrocarbons and/or several hydrogenation reactions, when general approaches demanded cumbersome matrix operations. Another more fundamental advantage of the pseudo-binary treatment is that the usage of traditional empirical correlations $Sh(Re, Sc)$, $Nu(Re, Pr)$ for gas–solid transfer (which, as a rule, are based on the Chilton–Colburn analogy) gets the solid theoretical background.

CATALYTIC MEMBRANE REFORMERS

B.N. Lukyanov, D.V. Andreev

Chem. Ind. Today,
9 (2009) pp. 27-32.

The basic are described catalytic processes of hydrogen reception in which application of reactors with hydrogen-permeable membranes is expedient. Various types of membranes for hydrogen extraction and design of catalytic reformers are considered. Advantages of such devices in comparison with traditional reactors with a fixed beds are shown. Prospects of their use in onboard fuel processors are estimated.

CATALYTIC REACTORS WITH HYDROGEN MEMBRANE SEPARATION

B.N. Lukyanov, D.V. Andreev, V.N. Parmon

Chem. Eng. J.,
154(1-3) (2009) pp. 258-266.

The main catalytic hydrogen-generation processes efficiently utilizing hydrogen-permeable membranes are described. Different membranes for hydrogen separation from gas mixtures and preparation methods are considered. The catalytic reactors with an integrated membrane module and their advantages over traditional fixed-bed reactors are demonstrated. The prospects of membrane catalytic reactors in hydrogen energy are analyzed.

MODELING MEMBRANE CO-REFORMING REACTOR FOR RECEPTION OF PURE HYDROGEN FROM SYNTHESIS GAS

V.A. Kirillov, V.D. Meshcheryakov

Chem. Ind. Today,
9 (2009) pp. 33-36.

Modeling of membrane reactor of CO steam conversion is carried out. It is shown, that membrane reactor on the basis of disk diffusion separators of hydrogen and medium-temperature catalyst of steam conversion possesses a number of advantages, such as efficiency of use of the catalyst and membranes, simplicity of a design and reliability in operation.

AUTOTHERMAL REFORMING OF DIESEL FUEL IN A STRUCTURED POROUS METAL CATALYST: BOTH KINETICALLY AND TRANSPORT CONTROLLED REACTION

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Catal. Today,
144(3-4) (2009) pp. 341-349.

Autothermal reforming (ATR) of diesel fuel into syngas was studied experimentally and theoretically. The experiments were performed in a reactor consisting of two cylindrically shaped monoliths 50 × 55 mm. Different catalytically active components and supports (Co, Mn, Rh, BaO, La_2O_3/Al_2O_3 and SiO_2) were tested. The reactor parameters were as follows: $O_2/C = 0.5$, $S/C = 1.5–1.7$, $T_{in} = 350–400$ C. The regularly structured catalytic monoliths were prepared using various metal porous supports. The most active and coke resistant catalyst was determined. The original modeling approach was based on the assumption that ATR involves two parallel reaction routes: (1) complete hydrocarbon oxidation, (2) steam reforming of hydrocarbon. The experimental data and the results of reactor modeling agreed well and allowed a conclusion that the ATR rate is controlled by inter-phase mass transfer. However, the contribution of the reaction routes (1) and (2), i.e., the distribution of hydrocarbon flux between these reactions is determined by the ratio of the reaction rate constants and oxygen concentration near the surface.

REDUCTION OF NITROGEN OXIDES IN DIESEL EXHAUST: PROSPECTS FOR USE OF SYNTHESIS GAS

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Kinet. Catal.,
50(1) (2009) pp. 18-25.

Already commercialized and some of the most promising technologies of nitrogen oxide reduction in automotive diesel exhaust are compared. The Boreskov Institute of Catalysis is developing an advanced method for the selective catalytic reduction of NO_x with synthesis gas produced on board by the catalytic conversion of diesel fuel. The activity of the Ag/Al_2O_3 catalytic system in NO_x reduction by H_2 + CO admixtures is studied for both a model composition of the exhaust gas and under real diesel operation conditions.

CONVERSION OF HYDROCARBON FUELS TO SYNGAS IN A SHORT CONTACT TIME CATALYTIC REACTOR

L.N. Bobrova, N.V. Vernikovskaya, V.A. Sadykov

Catal. Today,
144(3-4) (2009) pp. 185-200.

Some results of the theoretical and experimental research on the oxidative production of syngas from hydrocarbon fuels in catalytic reactors which operate at high temperatures and short contact times are presented. Pilot scale tests of the partial oxidation of methane, isooctane and gasoline have been carried out in nearly adiabatic conditions on structured catalysts developed at the Boreskov Institute of Catalysis and characterized by a low (≤0.5 wt./wt.%) content of noble metals. High yield of syngas and stable performance of the catalysts were revealed in the experiments. The details of interaction between chemical and physical processes inside adiabatic monolith reactor have been elucidated by mathematical modeling of the partial oxidation reaction on the base of catalyst detailed chemistry. The problems that emerged from the short contact time reactor operating on a pilot scale are also discussed.

CHARACTERISTICS OF A CHEMICAL REACTOR THAT IS A LOADED MICROWAVE RESONATOR

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J. Commun. Technol. Electron.,
54(2) (2009) pp. 231-233.

Relationships for calculation of the following parameters of reactors with microwave heating of chemical reagents are presented: the coefficient of absorbed microwave energy distribution in the reactor (in the cavity between the resonator walls and reagents) and the efficiency of transformation of microwave energy into heat of the reagents. The range of variation of microwave properties of the reactor during variation of reagent properties is estimated, and the coupling parameters of the resonator with the external line that provide for a high efficiency are determined.

MAIN FEATURES OF PERFORMING THE CHEMICAL REACTIONS UNDER MICROWAVE IRRADIATION

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Vestnik NGU, ser. Fizicheskaya,
4(2) (2009) pp. 78-83.

The main features of performing the chemical reactions, which take place with heating of reagents under microwave irradiation, are examined. Relationships for calculation of the efficiency of transformation of microwave energy to heat for different chemical objects in the high-Q cavity are presented. Lab-scale set-up (prototype of chemical reactor with the high-frequency heating) for studying the endothermic chemical reactions has been designed and manufactured. The catalytic thermal decomposition of *n*-hexadecane has been carried out with the high selectivity of *α*-olefins formation.

COKING CAUSES OF BOILING-BED ISOBUTANE DEHYDROGENATION REACTOR

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Catal. Ind.,
1(3) (2009) pp. 196-200.

The formation of disperse metal particles and existing dead spaces in reactors were found to cause intense coking in boiling-bed dehydrogenation reactors. These disperse metal particles result from abrasive wear of the reactor construction parts with corundum particles existing in the IM-2201 catalyst due to jet flows accounted for by the geometry of the reactor interior devices. On these disperse metal particles, hydrocarbons are catalytically decomposed into hydrogen and carbon nanofibers of two types: fibers with a pile arrangement of graphene layers and bamboo-like tubes. The amount of formed carbon exceeds by two orders of magnitude the amount of metal resulting from abrasive wear. It was recommended to redesign the reactor in order to minimize abrasive wear of its parts, to line the reactor parts with ceramic materials, and to use a corundum-free catalyst. The results of this work can be used to prevent coking in boiling-bed reactors for the synthesis of isobutylene, isoamylenes, and n-butylene at Sibur Open JSC, Nizhnekamskneftekhim Open Joint-Stock Company, etc.

DEHYDROGENATION OF C_3–C_4 PARAFFINS ON Cr_2O_3/Al_2O_3 CATALYSTS IN FLUIDIZED AND FIXED BED REACTORS

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Chem. Eng. J.,
154(1-3) (2009) pp. 185-188.

Thermodynamic calculations for dehydrogenation of a model C_3–C_4 paraffin mixture indicate that there is a complex mutual effect of the components on the equilibrium yield of olefins. Each component can act simultaneously: (i) as a diluent shifting the dehydrogenation equilibrium of the mixture components to the desired olefin and (ii) as an additional source of hydrogen to the reaction zone, which shifts the dehydrogenation equilibrium to the left. Experiments on dehydrogenation of model C_1–C_4 paraffin mixtures on Cr_2O_3/Al_2O_3 catalysts in fluidized and fixed beds show an increase of the total conversion of C_3–C_4 paraffins to olefins and an increase of the total process selectivity to olefins.

STRUCTURED REACTOR WITH A MONOLITH CATALYST FRAGMENT FOR KINETIC STUDIES: THE CASE OF CH_4 PARTIAL OXIDATION ON LaNiPt-CATALYST

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Chem. Eng. J.,
154(1-3) (2009) pp. 17-24.

Analysis of the behavior of a structured reactor of small scale has been fulfilled at millisecond contact times and high temperatures. A single channel fragment of corundum honeycomb substrate with supported LaNiPt active component has been tested in partial oxidation of methane as a model reaction at high gas velocities. Rather high rates of mass transfer are achieved for the conditions under study and the regions are defined with the operation mode where chemical kinetics is rate-controlling. The numerical analysis allows contribution of the direct route of methane oxidation into synthesis gas to be evaluated and some kinetic parameters of main reactions to be estimated.

Suggested design of the lab-scale reactor with separate one-channel structural element of a real monolithic catalyst is shown to be promising for kinetic studies under severe conditions.

VARIANTS OF THE ORGANIZATION OF A CONTROLLED-TEMPERATURE-PROFILE CATALYST BED IN A TUBULAR REACTOR FOR THE SINGLE-STEP

N.A. Baronskaya, A.A. Khassin, E.I. Smirnov, T.M. Yurieva

Theor. Found. Chem. Eng.,
43(4) (2009) pp. 366-373.

The possibility of bringing the actual temperature profile along the catalyst bed to the profile maximizing the CO conversion in the water gas shift (WGS) reaction in a tubular reactor is considered for new, highly efficient catalysts as heat-conducting composite plates (HCCPs). It is demonstrated by the example of a controlled-temperature-profile (CTP) tubular reactor integrated into an experimental model of a fuel cell of a 5-kW power plant that the efficiency of the WGS process can be raised by using the catalyst as HCCPs. Use of these catalysts in CTP apparatuses can markedly increase the efficiency of the WGS stage of natural gas reforming for ammonia synthesis, hydrogen production, and the production of fuel gas for fuel cells.

UP-TO-DATE APPROACH TO THE TESTING OF THE DEEP HYDROTREATING CATALYSTS FOR DIESEL-RANGE DISTILLATES

G.A. Bukhtiyarova, A.V. Pashigreva, O.V. Klimov, A.S. Noskov

World Oil Prod. The Oil Comp. Bull.,
3 (2009) pp. 28-33.

The problem arising during the investigation of granulating catalysts for deep hydrotreating of diesel oil is discussed. The generation of reliable and repeatable data in the small-scale hydrotreating reactors is complicated by different factors such as pour wetting of catalysts, reactor wall effect and back mixing of liquid, which cause the axial heterogeneity of liquid velocity and feed bypassing. It is reported that the recent development of the technique, based on dilution of catalyst granule with the appropriate size of inert particles (silicon carbide) provide for the hydrodynamic characteristic identical to that in the industrial scale reactors. An attempt has been made to provide guidelines for selecting the appropriate reactor and methodology to evaluate deep hydrotreating catalysts.

INVESTIGATION OF DEEP METHANE OXIDATION WITH THE USE OF THE IMPROVED FLOWING CIRCULATION METHOD

I.Yu. Pakharukov, N.N. Bobrov, V.N. Parmon

Catal. Ind.,
1(1) (2009) pp. 38-42.

The most correct method for studying the peculiarities of stationary kinetics of heterogeneous reactions is the flowing circulation method which has been substantially improved in recent years. The core of the introduced improvement lies in the direct determination of reaction velocity (catalytic activeness) at the given compound of the contact reaction mixture. The improved flowing circulation method was applied for the first time to study the deep oxidation of methane by molecular oxygen in order to obtain one-parameter dependences of catalytic reactivity on the temperature and concentration of reaction medium components and the solution of the inverse kinetic problem. Catalyst IC-12–72 compound, wt %: 16 ± 2 Cr_2O_3, 4 ± 0.6 MgO and the remaining Al_2O_3 has been studied. In the kinetic area, the apparent energy of reaction activation (110 kJ/mol) has been determined. On the basis of experiments and ideas on the mechanism of the Langmuir-Hinshelwood reaction of competitive absorption type, the equation well describing experimental data in the overall studied range of concentrations (average relative deviation 8%) was selected. The simplicity of the improved flowing circulation method allows for it to be recommended for the selection of the most active catalysts and analysis of dependences of stationary velocities of heterogeneous catalytic reactions on various process parameters.

REACTION KINETICS OF METHANOL OXIDATION AND DECOMPOSITION ON AN SNM-1 CATALYST AS STUDIED USING AN IMPROVED FLOW-CIRCULATION SYSTEM

I.Yu. Pakharukov, N.N. Bobrov, V.N. Parmon

Catal. Ind.,
1(2) (2009) pp. 95-101.

Methanol is a product of the large-scale chemical industry; it is promising for use as an alternative motor fuel and a hydrogen source for fuel cells. In actual practice, an approximate integral method, which does not allow one to measure the rates of reactions, is used to study the kinetics of catalytic reactions of methanol conversion. The kinetics can be most correctly studied experimentally using differential methods, primarily, a flow-circulation method, which has been considerably improved in the past few years. The improvements consist in the possibility of directly determining the rate of reaction (catalytic activity) at a specified composition of the contact reaction mixture. For the single-path process of the deep oxidation of methanol at 260–320°C, the absolute values of rates and apparent activation energy were obtained. For the multipath process of methanol decomposition, it was found that the activity and selectivity of reaction changed under variations in the fictitious contact time, and the catalytic conversion products of methanol exerted a considerable effect on these parameters. The advantages of the improved experimental system proposed, based on a flow-circulation method for the rapid determination of the activity and selectivity of methanol conversion, were demonstrated.

STRUCTURED CATALYST AND COMBINED REACTOR LOADING FOR METHANE COMBUSTION IN A GAS TURBINE POWER PLANT

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Catal. Today,
147(suppl. 1) (2009) pp. S237-S243.

Modeling of methane combustion in several types of catalyst packages composed of 2 or 3 beds of granulated catalysts with different chemical compositions, shapes and sizes of pellets has been performed. It has been shown that the high efficiency of methane combustion (>99.97%) and low emission of HC < 10 ppm can be achieved for all types of catalyst packages at variation of the inlet temperature. Experimental runs in a model reactor (1.3 kg catalyst) have demonstrated very good correlation with the results of the modeling.

HYDROISOMERIZATION OF DIESEL FRACTIONS ON PLATINUM-CONTAINING SILICOALUMINOPHOSPHATE SAPO-31: FROM THE LABORATORY TO THE PILOT SCALE

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Pet. Chem.,
49(1) (2009) pp. 74-78.

The properties of the Pt-SAPO-31 catalyst in hydrocarbon feed conversion depending on the catalyst chemical composition and crystallinity has been studied on the laboratory scale. The results of pilot testing of a Pt-SAPO-31 catalyst specimen in hydroisomerization reactions of real diesel fractions are presented. It is shown that the catalyst has high activity and selectivity in feedstock transformation as confirmed by a decrease in the cloud- or freezing point of diesel fuel with a yield of 96–98%. Catalyst life tests have shown high operation stability over more than 800 h without variations in the catalytic activity and the temperature characteristics of the product. Hydrogenation of unsaturated compounds present in the feedstock also results in diesel fuel upgrade.

EFFECT OF THE REACTION MEDIUM ON THE PHYSICOCHEMICAL PROPERTIES OF THE OXIDE MONOLITH CATALYST IC-42-1 FOR AMMONIA OXIDATION

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Kinet. Catal.,
50(6) (2009) pp. 892-898.

The changes in the properties of the oxide monolith catalyst IC-42-1 for ammonia oxidation upon 3540-h-long operation as the second stage of a UKL-7 industrial reactor were studied by X-ray diffraction, chemical analysis, secondary ion mass spectrometry, temperature-programmed reduction, and IR spectroscopy. The spent catalyst shows a lower activity and a lower nitrogen oxide selectivity than the initial catalyst: the decrease in the NO yield is about 25%. As the catalyst operates, the state of its surface changes under the action of the reaction medium. The specific surface area of the catalyst decreases, and the total pore volume increases, which can cause a slight decrease in the ammonia conversion and diminish the mechanical strength of the monoliths. Under the ammonia oxidation conditions, there can be partial reduction of the catalyst surface and the local formation of a spinel structure (spinel-like defects). Oxygen adsorbed on these areas/defects is characterized by a high activation energy of desorption, which favors the reaction route yielding molecular nitrogen. According to IR spectroscopic data, the spent catalyst has a lower concentration of active, coordinatively unsaturated Fe^{2+} sites of adsorption, which are responsible for ammonia oxidation into NO. The decrease in the number of active sites can be due to both the breakdown of the solid solution of iron oxide in aluminum oxide during the reaction and the blocking of these sites by silicon, alkali and alkaline-earth metal, chromium, and rhodium atoms present on the surface of the spent catalyst.

CORDIERITE-LIKE MIXED OXIDE MONOLITH FOR AMMONIA OXIDATION PROCESS

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Catal. Today,
147(Suppl. 1) (2009) pp. S319-S323.

The paper deals with modeling of a two-stage catalytic system for ammonia oxidation in an UKL-7 plant, preparation and characterization of cordierite-like honeycomb monoliths (~230 cpsi) modified with (Mn, Co, Fe, V, Bi)O_x oxides as the secondary catalysts. The NO yield for as prepared monoliths was shown to depend not only on the testing temperature, nature and content of the active oxides but also on the cordierite crystallinity degree and on the inlet NH_3 concentration. The best data on NO yield (>80%) at 880–900°C were obtained with the iron and cobalt based modified monoliths used as the secondary catalysts in the two-stage catalytic system. The data obtained by modeling indicated the possibility to use 8 (instead of 12) gauzes and one layer of as prepared cordierite-like catalysts in the UKL-7 plant to provide the NO yield typical of 12 gauzes.

DETOXICATION OF NITROSE GASES FORMED IN THE PRODUCTION OF ADIPIC ACID: THE TWO-STAGE CATALYTIC CLEANING PROCESS

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Catal. Ind., 1(1) (2009) pp. 76-84.

A two-stage process was developed for the catalytic cleaning of the nitrose gases formed in the production of adipic acid. The process involves the decomposition of N_2O over a Co-ZSM-5-type catalyst and the reduction of NO_x with ammonia over a V_2O_5/Al_2O_3 catalyst. The proposed process provides for the cleaning of the effluent gases from N_2O and NO_x at an efficiency of over 99% and has the advantage of natural gas economy over the thermal process. The Co-ZSM-5-type zeolite was used to make an N_2O decomposition catalyst characterized by high moisture resistance. The kinetic characteristics of the process were determined, and hydrothermal endurance tests conducted with the catalysts (Co-ZSM-5 and V_2O_5/Al_2O_3) were recommended for the process. Technical and design parameters were established for the catalytic reactors forming part of the system for the cleaning of the effluent gases in the production of adipic acid.

CATALYTIC FLUE GAS CONDITIONING IN ELECTROSTATIC PRECIPITATORS OF COAL-FIRED POWER PLANTS

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Chem. Eng. J.,
154(1-3) (2009) pp. 325-332.

The paper is dedicated to development of the catalytic process for controlled SO_3 production for conditioning of flue gases from coal-fired power plants. On the basis of comparative technological analysis performed for different variants of the process arrangement, it was shown that optimal technological decision is catalytic sulfur dioxide oxidation in synthetic SO_2-containing stream in a separate reactor with two adiabatic beds of catalyst with following introduction of SO_3-containing flow into the main coal boiler gas duct. Undoubted benefits of this process are: complete independence of the process from the flue gases properties; possibility to control SO_3 concentration in the flue gases in the wide range; possibility to process gases with high SO_2 concentration, providing low gas flow rate and low catalyst loading; absence of limitations for the pressure drop of the catalyst bed. Among catalysts on the basis of fiber–glass support the best catalytic performance was observed for platinum-containing catalyst on support modified with zirconium dioxide. It was demonstrated that optimal catalyst solution is combination of conventional granular vanadia and platinum woven fiber–glass catalysts, providing high activity both at high and low temperatures.

LIQUID-PHASE HYDRODECHLORINATION OF CHLOROBENZENE BY MOLECULAR HYDROGEN: THE INFLUENCE OF REACTION MEDIUM ON PROCESS EFFICIENCY

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Pure Appl. Chem.,
81(11) (2009) pp. 2107-2114.

Catalytic hydrodechlorination (HDCl) of chlorobenzene was carried out in a two-phase aqueous–organic solvent system and a single-phase solvent composed of saturated KOH solution in a secondary alcohol over Pd-based catalysts at 50°C and atmospheric pressure of H_2. It was shown that an aqueous–organic solvent system containing propan-2-ol and aqueous KOH increases catalyst activity by promoting mass transfer of the formed chloride ions to water phase that prevents catalyst deactivation. It is inferred that propan-2-ol favors hydrogen activation during the HDCl process. Use of the Pd catalysts based on hydrophobic carbon support enables chlorobenzene HDCl to proceed in a two-phase solvent at a satisfactory rate, even in the absence of phase-transfer catalysts.

METALS IN COMMERCIAL CATALYSTS: I. MOLYBDENUM AND TUNGSTEN

V.I. Simagina, L.P. Milova, V.N. Parmon

Catal. Ind.,
1(4) (2009) pp. 261-266.

Information on the production and consumption of molybdenum and tungsten in the world and in Russia is provided. The lowering of domestic consumption of these metals in the Russian industry is demonstrated. The use of these metals in catalysts manufacturing in Russia is considered. The assortment of domestic molybdenum- and tungsten-containing catalysts is discussed, and their consumption is estimated. The current demand for molybdenum and tungsten in catalyst manufacturing is small because of the high impact of imported catalysts and low level in the development of chemical catalytic processes. Certain applications of molybdenum- and tungsten-containing catalysts are considered. It is shown that the consumption of molybdenum and tungsten in the manufacturing of high-added value products (catalysts) may grow at the expense of the replacement of imported catalysts by competitive domestic molybdenum- and tungsten-containing catalysts and their use in new processes.

OVERVIEW OF CATALYTIC METHODS FOR PRODUCTION OF NEXT GENERATION BIODIESEL FROM NATURAL OILS AND FATS

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Russ. J. Phys. Chem. B,
3(7) (2009) pp. 1035-1043.

Production of biodiesel from natural oils and fats can be achieved using various technologies briefly discussed in this review. A particular appealing concept for production of green diesel is selective catalytic deoxygenation of renewables leading to diesel fuel products. This reaction can be performed over Pd on active carbon supports with saturated and unsaturated fatty acids and their derivatives.

CATALYTIC DEOXYGENATION OF STEARIC ACID IN A CONTINUOUS REACTOR OVER A MESOPOROUS CARBON-SUPPORTED Pd CATALYST

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Energy Fuels,
23(8) (2009) pp. 3842-3845.

Catalytic deoxygenation of neat stearic acid was studied at 360°C under 10 bar argon or 5 vol % hydrogen in argon in a fixed-bed reactor (down flow) using mesoporous-supported Pd/C (Sibunit) beads as a catalyst. The results showed stable catalyst performance, giving about 15% conversion level of stearic acid. The main liquid-phase product was heptadecane, while the main gaseous products were CO and CO_2.

CATALYTIC DEOXYGENATION OF STEARIC ACID AND PALMITIC ACID IN SEMIBATCH MODE

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Catal. Lett.,
130(1-2) (2009) pp. 48-51.

The deoxygenation experiments of different reactants, i.e., pure palmitic acid, stearic acid, and a technical grade stearic acid containing a mixture of 59% of palmitic and 40% of stearic acid were successfully performed over 4 wt% Pd/C mesoporous catalyst at 300°C under 17 bar of 5% H_2 in argon. The main product in catalytic deoxygenation of saturated fatty acids, C16 and C18, were aliphatic chain length hydrocarbons containing one less carbon than the corresponding acid. Additionally it was found that the deoxygenation rates of different reactant were independent on carbon chain length of its fatty acids.

DEOXYGENATION OF PALMITIC AND STEARIC ACID OVER SUPPORTED Pd CATALYSTS: EFFECT OF METAL DISPERSION

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Appl. Catal., A, 355(1-2) (2009) pp. 100-108.

Catalytic deoxygenation of palmitic and stearic acids mixture was studied over four synthesized Pd catalysts supported on synthetic carbon (Sibunit) in a semibatch reactor and dodecane as a solvent at 260-300°C. The catalysts were prepared by precipitation deposition method using Pd chlorides as metal precursors. All catalysts contained 1 wt.% Pd, however, the metal dispersion was systematically varied. An optimum metal dispersion giving the highest reaction rate was observed. The main liquid phase products were n-heptadecane and n-pentadecane, which were formed in parallel. In addition to the particle size effect the impact of mass transfer was elucidated and a detail discussion on temperature programmed desorption of CO from the fresh and spent samples was provided.

GAS CHROMATOGRAPHY ON ZEOLITE-BASED ADSORBENTS CHROMATOGRAPHIA

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Chromatogr.,
69(7-8) (2009) pp. 701-708.

Adsorption and gas chromatographic properties of zeolites, both modified with carbon and supported as a surface-porous layer, have been studied. Carbonization of zeolites changes the chemical nature of their surface and their molecular sieve properties. Adsorbents obtained in this way combine the surface properties of both the initial zeolite and of carbon-containing materials. Coating of zeolites on the solid support surface as a porous layer of small depth allows to obtain highly selective adsorbents required for the separation and determination of the components in the various mixtures. In their separation ability of a number of substances formed in catalytic reactions, zeolite-based adsorbents exceed the performance of well-known chromatographic stationary phases, including the highly effective porous-layer open tubular columns based on Porapak. Compared to the initial volume zeolites, zeolite-based adsorbents permit to reduce the temperature of analysis and the chromatographic analysis time. Zeolite-based adsorbents have been applied in the gas chromatographic analysis of mixtures whose analysis presented a problem or was impossible at all with the traditional chromatographic columns. In comparison with known adsorbents (carbons, polymers, etc.), modified and supported zeolites are characterized by better selectivity with respect to separation of light-boiling compounds formed in many catalytic reactions.

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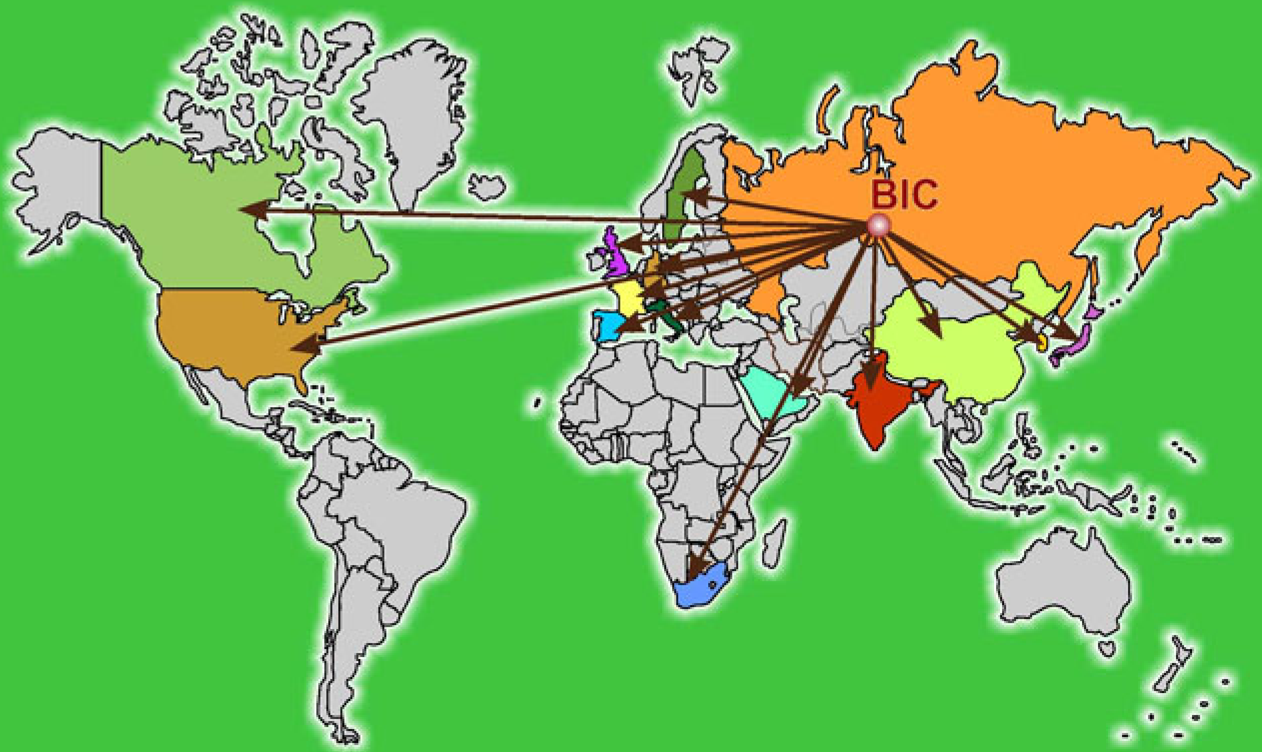

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