



**Enmix** A.I.S.B.L.

## European Nanoporous Materials Institute of Excellence

Newsletter - N°1 - November 2010

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Welcome to the first edition of the ENMIX newsletter. Launched in February 2009, ENMIX wants to be an attractive platform for research organisations, governmental bodies and industries, acting as a unique competence and knowledge center in the field of nanoporous materials for applications in sorption/separations, catalysis and membranes.

The ENMIX mission statement can be formulated as: "As an independent and customer driven organisation, which can offer (a) Innovative technological solutions, (b) Unique scientific measuring expertise and facilities and (c) Science-based education, training and advice with the goal of stimulating sustainable development and strengthening the economic and societal fabric in Europe."

The objectives of the ENMIX newsletter are to inform researchers, to stimulate cooperations (including exchanges of scientists and training opportunities) in the field of nanoscience and nanotechnologies and to spread scientific knowledge on nanoporous materials.

The present members of the ENMIX consortium are: NCSR Demokritos (Gr), University of Antwerp (B), University of Stuttgart (D), University of Leipzig (D), SINTEF (N), CPERI-CERTH (Gr), University of Hannover (D), IFE (N), University of Alicante (E), TUDelft (NI), NCR (I), NIC (SI), CNRS (F).

In this issue three research groups present their research activities. In the following issues other ENMIX members will be presented.

E.F. Vansant



On October 4-5 2010, the 1st International Workshop of the 'European Nanoporous Materials Institute of Excellence' (ENMIX) – 'Nanostructured Materials for Sorption, Separation and Catalysis' was organised in Antwerp, Belgium. The conference was located at the city centre in the Elzenveld conference complex, hosting historical and listed buildings once belonging to the oldest hospital of Antwerp in the 13<sup>th</sup> century.

The workshop belongs to the activities of ENMIX A.I.S.B.L. The ENMIX organisation has arisen in 2009 from the EU-FP6 Network of Excellence 'IN-Situ Study and Development of NanoPORous Materials' (INSIDE-PORes), assembling European research groups in a coherent field of activities related to nanoporous materials. Following the successes of the workshops organised under the auspices of the Inside-Pores Network of Excellence, this is now the 1<sup>st</sup> ENMIX International Workshop devoted to the field of nanoporous materials.



Nanoporous materials play an increasingly important role in nowadays knowledge based society, as they can replace traditional materials and lead to a generation of new ones. They have great potential for improving sustainable development across a wide range of industrial sectors. Due to the unique structural and surface physicochemical properties, nanoporous materials are used as catalysts, sorbents and membranes.

Therefore, the scientific programme of the workshop was set up around four different topics: *Synthesis, Membranes, Sorption and Catalysis*, corresponding to the four scientific pillars of Inside-Pores and ENMIX.

The organisation of the workshop was made possible thanks to the support of ENMIX, FWO-Flanders, FWO-WOG (Scientific Research Community on Catalysis in Flanders), University of Antwerp, Thermo Fisher scientific and BASF.



The workshop was a great success, both in terms of number of participants and the high-quality and content of the presentations. In total there were 104 participants from 16 different countries, not only within Europe but also the United States, Iran and Ukrain.

Invited lectures were given by eminent specialists in the field. There were exciting invited contributions by *Prof. Bao-Lian Su* (University of Namur, Belgium) on synthesis; by *Prof. Gilbert Rios* (CNRS Montpellier, France and CEO of European Membrane House A.I.S.B.L.) on membranes; by *Prof. F. Rodriguez-Reinoso* (University of Alicante, Spain) on sorption and by *Prof. F. Schüth* (Max-Planck Institute, Germany) on catalysis.

Moreover, numerous other interesting scientific contributions were given in the form of oral and poster presentations during the 2-days workshop. Also, two special sessions were devoted to presentations by young researchers having the opportunity of introducing their poster in a short oral presentation of 8 minutes. The lectures covered many topics in the 4 main fields of synthesis, membranes, sorption and catalysis and a broad range of nanoporous materials, such as zeolites, mesoporous materials, hierarchically structured materials, carbons and organic-inorganic hybrid materials.

Three prizes for best poster presentations were awarded, to: D. Lutic (A.I. Cuza University, Romania) for her work on *Pt-doped mesoporous semiconductive oxides for gas sensing*; to C. Gücüyener (TUDelft, The Netherlands) for her work on *adsorption properties of the metal organic framework ZIF-7*; to A. Philippaerts (KULeuven, Belgium) for her work on the *shape-selective hydrogenation of FAMEs and vegetable oils*. Herewith a photograph of Prof. Etienne Vansant handing over one of the poster awards.



On Monday evening all participants were invited to an informal social dinner at the University club restaurant, located in the historical region of the Antwerp city centre. The location is called het 'Hof Van Liere' and is an important historical building in Antwerp. In fact, 'Hof van Liere' was once a city palace -dating from the 16<sup>th</sup> century- with an inner court with gardens and galleries, built in a Gothic style. Now it belongs to the University of Antwerp and it is used for administration and teaching, and it also hosts the University club restaurant. Hereby some photographs of the informal atmosphere at the dinner on Monday evening and the closing reception on Tuesday.





Thanks to all the interesting contributions presented, we were able to provide the participants with an interesting scientific programme. We are satisfied to have organised a workshop that stimulated the contacts, possible exchanges and collaborations between young researchers.

We are all looking forwards to the 2nd ENMIX workshop which will be organised in cooperation with the European Membrane House (EMH) A.I.S.B.L. at Myrdal, Norway in September 2011.

On behalf of the organising committee,

Pegie Cool

## Description of the research group SINTEF, N

SINTEF, established in 1950, is a private non-profit foundation, which performs contract research for industry, private organizations as well as public authorities. The field of research and development covers the entire range of applied science and technology, including medicine and social sciences. The customers are both Norwegian as well as foreign companies. With about 2000 employees, SINTEF is one of Europe's largest independent research organizations. SINTEF works in close operation with the Norwegian University of Science and Technology (NTNU) in Trondheim and with the University of Oslo. The intention of SINTEF is to assist the customers in developing and promoting new technological applications thereby contribute to a more competitive situation in the field of international technological applications. As a foundation, our objective is to ensure that knowledge is used to promote sustainable value creation in society. Our work is to contribute to the competitiveness of Norwegian industry and public sector productivity.

The SINTEF group consists of several institutes, where SINTEF Materials and Chemistry and is one of those. The Department of Hydrocarbon Process Chemistry is one of the eight departments within SINTEF Materials and Chemistry.

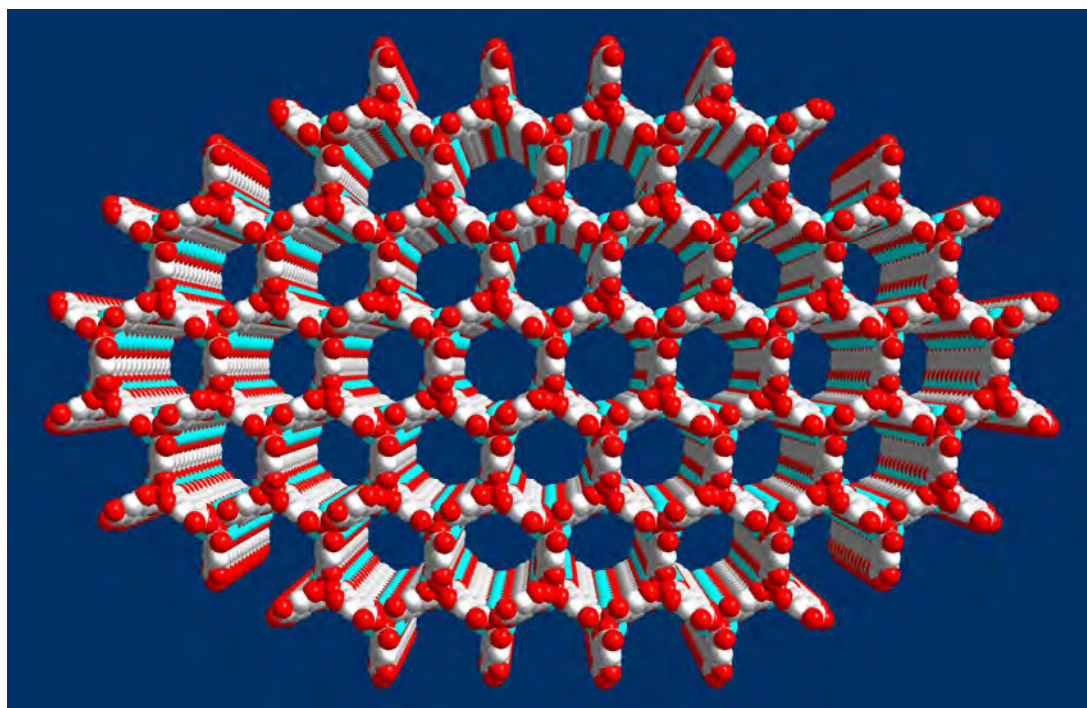


Figure 1: Structure of the metal organic framework CPO-27

### **Research Activities related to Synthesis at SINTEF's Department of Hydrocarbon Process Chemistry:**

Current research fields at the Department of Hydrocarbon Process Chemistry cover upgrading of oil and natural gas, petroleum refinery, petro-chemistry, catalysis related to polymeric materials, removal of sulfur and NO<sub>x</sub>, biorefinery processes, CO<sub>2</sub> capture as well as separation and purification of product streams.

Focus has been on the development of new or improved micro- and mesoporous materials, including metal organic frameworks (MOFs, see Figure 1), usable as catalysts and adsorbents for chemical conversion of suitable feed stocks. This covers new or improved



technologies with respect to preparation of micro- and mesoporous materials, including high throughput experimentation.

## Results

- Proprietary new and/or improved micro- and mesoporous materials, including metal organic frameworks (MOFs), have been synthesized, characterized and tested in co-operation with Norwegian and international companies, as well as with national and international universities.

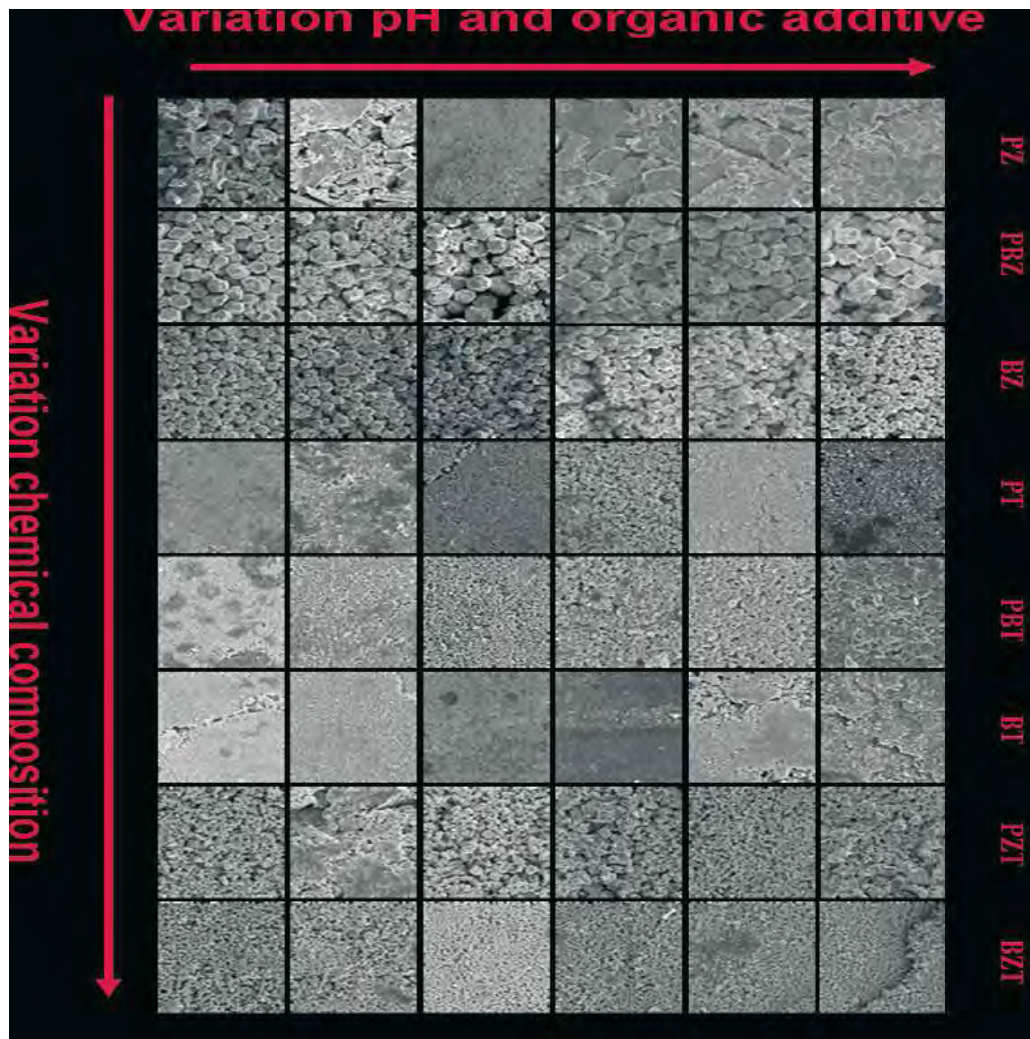


Figure 2: Scanning Electron Micrographs of 48 different materials prepared in parallel under hydrothermal conditions.

- New synthesizing methods have been developed (including combinatorial approaches), yielding compounds with enhanced catalytic activity, selectivity and improved stability as well as improved separation abilities. Entirely new structures have been identified through extensive characterization (see Figure 2).
- Synthesis equipment has been developed and built (see Figure 3), including high throughput facilities, for different types of processes, with high degree of automatic process control and advanced analytical instrumentation.

- A wide, international contact net has been established through co-operation projects, lectures in Norway and abroad, student supervision and exchange of students and post-docs. Several patents have been issued, and non-proprietary results have been published in international scientific journals and presented at international conferences.

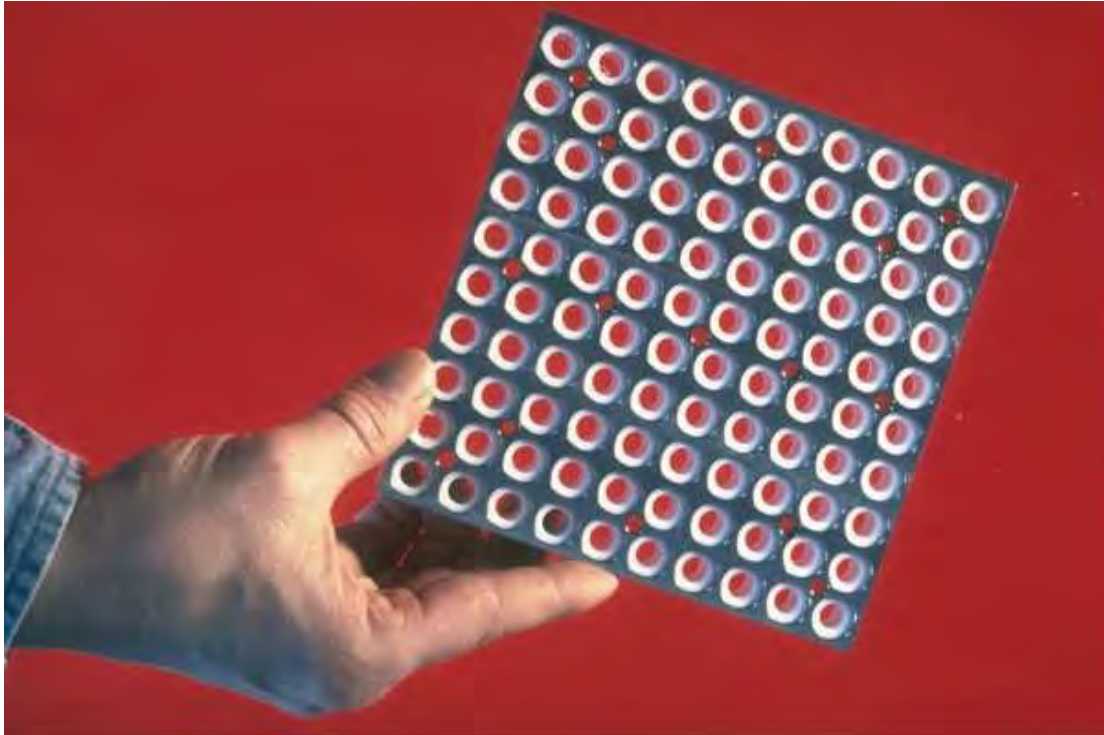


Figure 3: System for miniaturized parallel synthesis of catalyst materials under high temperatures and pressures.

### **Key personnel involved within ENMIX at the Department of Hydrocarbon Process Chemistry**

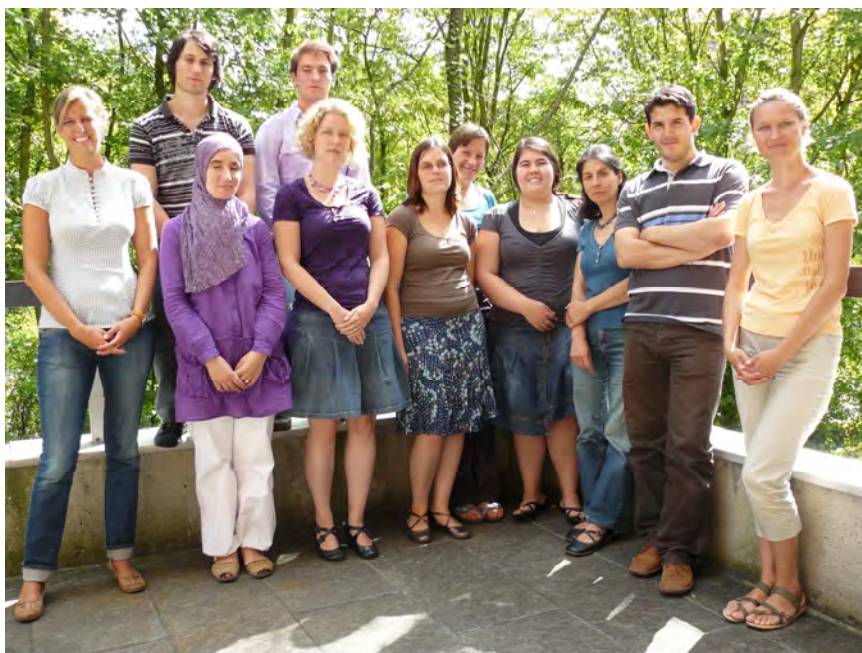
**Dr. rer. nat. Michael Stöcker**, Chief Scientist, graduated from the University of Münster (Germany) in Chemistry and joined the Center for industrial Research (now SINTEF) in 1982. He is the Editor-in-Chief of the international scientific journal "Microporous and Mesoporous Materials".

Michael Stöcker

## **Description of the research group Adsorption and Catalysis, University of Antwerp, BE**

The **University of Antwerp (UA)** is located in the heart of the Belgian chemical and pharmaceutical industry. This unique industrial region with leading chemical companies and multinationals is situated at the second largest harbour of the world. Academic and industrial research and development are in close contact and work together on technological, economical and environmental improvement.

The **Laboratory of Adsorption and Catalysis (LADCA)** within the Chemistry Department of University of Antwerp is an internationally recognized and specialized research laboratory focusing on the development of new nanoporous inorganic materials. The research group was founded by Prof. Etienne Vansant in 1979, and today the group is headed by Prof. Pegie Cool.



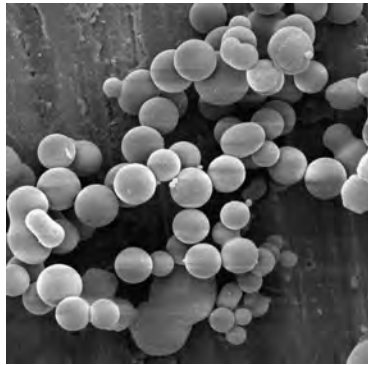
The current research topics related to synthesis include:

- **The synthesis of micro-, meso- and combined micro-/mesoporous siliceous materials**

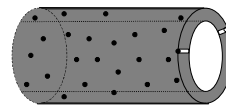
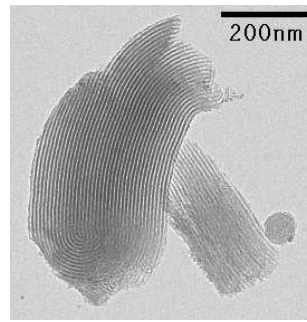
Different types of combined zeolitic/mesoporous materials are prepared. Zeolitic nanoparticles (beta or silicalite type) are introduced into mesoporous materials (e.g. SBA, MCF type) by post-synthesis impregnation methods or in-situ.

The obtained materials have both open and narrowed sections in the mesopores which gives them unique properties (diffusion, stability,...) that can be adjusted in function of the application (encapsulation, separation, controlled release, sorption, catalysis, ...). Also research is performed towards the control of morphology of the different materials (spheres, rods, tubes,...).





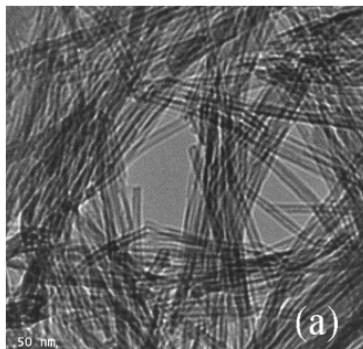
SBA silica spheres



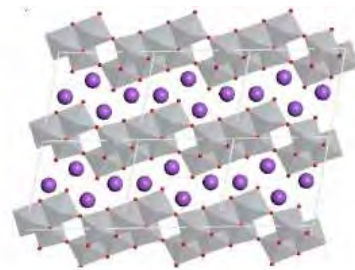
pore structure of SBA-15

- **The synthesis of mesoporous photocatalytically active transition metal oxides**

Mesoporous transition metal oxides ( $\text{TiO}_2$ ,  $\text{SnO}_2$ ,  $\text{ZnO}$ , ...) are prepared via different synthesis approaches such as sol-gel route, EISA method, carbon replicas or other templated routes. Also inexpensive template free hydrothermal methods are applied to obtain  $\text{TiO}_2$  nanotubes or nanorods. Fast synthesis routes are developed through the application of microwave conditions. The developed materials find interesting environmental applications (e.g. removal of organic pollutants from waste water) and applications in the photocatalytic reduction of  $\text{CO}_2$ .



$\text{TiO}_2$  nanotubes

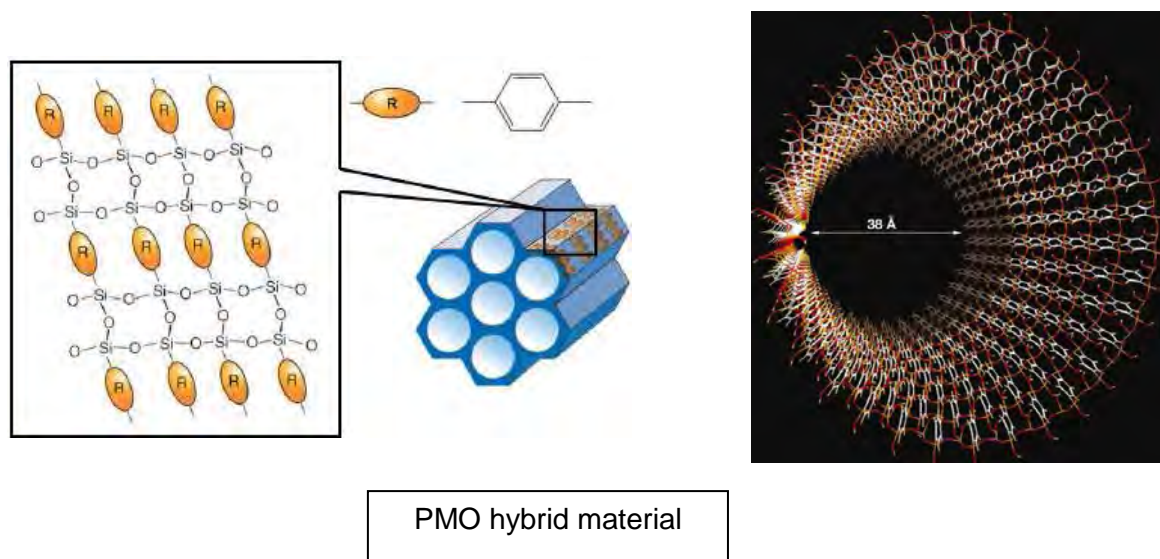


$\text{Na}_2\text{Ti}_3\text{O}_7$

- **The synthesis of mesoporous organic-inorganic hybrid materials**

Periodic mesoporous organosilicas (PMOs) are one of the latest innovations in the field of ordered mesoporous materials due to their feasibility in many different applications (catalysis, metal scavenging, sorption, low-k applications, ...). The synthesis of PMOs is based on the use of organic templates as structure directing agents and the use of an organosilane as precursor. A typical precursor molecule contains an organic functional group (R) bridged between two silsesquioxanes, generally written as  $(\text{R}'\text{O})_3\text{SiRSi}(\text{OR}')_3$ . The organic functional group in PMOs is fully and homogeneously imbedded within the inorganic silica walls of the

mesoporous material. PMO materials have many advantages such as a good accessibility of functional groups in the pore walls, a high crystallinity and stability and many possibilities for fine tuning towards the desired functionality as a function of the application.



- **Pore size engineering and surface modification of porous materials by impregnation techniques, ion-exchange processes and chemical modification reactions (both in-situ and post modification)**

An example of one of the latest modification processes developed by LADCA, is the enhancement of chemical stability of the top layer of inorganic ceramic membranes, to obtain a higher resistance towards leaching of the functional surface groups and towards the decrease of fluxes through the membranes. A new method has been developed based on organometallic chemistry allowing a direct covalent bond of the organic functional group on the transition metal oxide membrane surface (in collaboration with VITO, Belgium). Other methods that are applied for the catalytic activation of silica support materials with metals or metaloxides are dry and wet impregnation and incipient wetness method. A successful technique developed by LADCA for obtaining disperse metaloxide monolayers on the silica surface is the 'Molecular Designed Dispersion' method, in which a metal acetylacetonate complex of the type  $M^{n+}(acac)_n$  is reacted with the surface hydroxyls of the porous support material.

The **skills of this research group** are focused on the development of new synthesis routes, new chemical modification processes to tune the material properties towards applications in the area of sorption, separation and catalysis. A very important reference work on the verification of synthesis procedures towards mesoporous materials has recently been published by the LADCA group as a **special issue in Microporous and Mesoporous Materials** (Vol. 125/3 (2009) 169-224) as a result of a collaboration among many research groups participating in the Inside-Pores project (EU NoE-FP6).

An extensive experience is present for **detailed characterization** and evaluation of the performance of porous solids. Equipment is available for the analysis of different types of porous and non-porous compounds ranging from spectroscopic techniques (FT-IR, FT-RAMAN, FT-IR-PAS, UV-VIS-DR and in-situ-DRIFT) and sorption techniques (automated nitrogen sorption and manual sorption systems) over elemental analysis (f-AAS, UV-VIS, TOC) and thermal analysis (TGA) to catalysis (catalytic set-up, GC and UV-lamps for photocatalysis). Based on a close collaboration with the physics department within the

University of Antwerp, LADCA has access to the use of advanced EPR-techniques (CW and HYSCORE) and high-resolution 2D and 3D-TEM (tomography) techniques for characterization of the nanoporous materials.

### **Key personnel involved within ENMIX**

**Prof. Dr. Pegie Cool** received her Diploma in Chemistry in 1993 and her PhD degree in Science in 1998 - both from the University of Antwerp (Belgium). She kept positions as a postdoctoral researcher in the Laboratory of Adsorption and Catalysis, performing research in the area of clay-based micro- and mesoporous materials. In 2003 she was appointed Associate Professor in Inorganic Chemistry at the University of Antwerp. Beside teaching and research activities, she is involved in several national and international research projects in collaboration with industry and academia. She performed invited research visits at the Texas A&M University, USA and at the University of Queensland, Australia. She is the author/co-author of 110 A1-papers in internationally recognised leading journals and of 14 review papers and handbook chapters.

She is member of the Board of Directors of ENMIX.

**Em. Prof. Dr. Ir. Etienne Vansant** from the Laboratory of Adsorption and Catalysis is member of the Board of Directors of ENMIX and has been elected as CEO of the Institute.



### **Contact**

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## **Description of the research group Laboratory for Inorganic Chemistry and Technology, National Institute of Chemistry, SI**

Laboratory for Inorganic Chemistry and Technology, headed by *Professor Venčeslav Kaučič*, is one of 17 laboratories of the National Institute of Chemistry (NIC) in Ljubljana. NIC is the leading institution in the field of chemistry and related disciplines (materials and life sciences) in Slovenia. It has approximately 300 employees of which about 60 are senior researchers and about 80 are PhD students.



Figure 1. The group of the Laboratory of Inorganic Chemistry and Technology

### **Catalysts**

The group headed by professor Kaučič has a 20-years long tradition in synthesis and characterization of zeolitic materials. For most of the time the group has worked on the development of new materials for catalysis. Catalysts are important to the chemical industry and environmental remediation because they can effectively convert one chemical to another. Our recent work is focused on the development of *supported catalysts*, based on porous silicate and aluminophosphate supports and transition metals as catalytic components. To prepare stable mesoporous catalysts, we combine mesoporous matrices with microporous zeolitic nanoparticles. We developed the synthesis route for incorporation of isolated transition-metal ions (Ti, Mn, Fe) into zeolitic particles (Silicalite-1, ZSM-5, BETA) of typical dimensions of up to 100 nm. Preparation of such functionalised zeolitic seeds allowed us to develop hierarchical porous structures, that is *microporous/mesoporous composite materials* exhibiting bimodal pore distribution. For example, we organized microporous zeolitic seeds containing isolated Ti centres into mesoporous silicate structures with hexagonal and cubic mesopore arrangement (TiBeta/MCM-41, TiBeta/MCM-48, TiBeta/SBA-15). These composite solids enabled diffusion of larger molecules through their

mesopores than did pure microporous zeolitic materials. The composites were also thermally more stable than pure mesoporous materials and were not prone to transition-metal leaching. This means that the materials could be readily recovered after catalytic reactions. More recently we have started to use mesoporous supports for immobilization of metal oxides. We have homogeneously distributed  $MnO_x$  nanoparticles onto a new mesostructured silicate support with textural porosity, KIL-2. The material is a potential catalyst in environmental friendly applications due to its structural characteristics, hydrothermal stability, and its non-toxic and cost-effective nature. Ordered (SBA-15) and disordered (KIL-2) mesoporous silicate supports were used also for the immobilization of  $TiO_2$  nanoparticles.  $TiO_2$  supported on SBA-15 and KIL-2 shows high photocatalytic activity towards degradation of volatile organic compounds such as isopropanol and toluene.

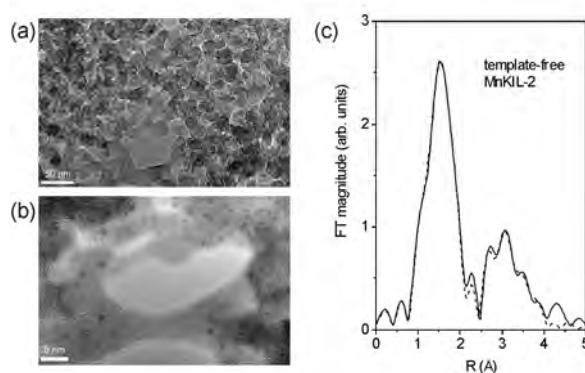


Figure 2. Homogeneously distributed Mn-oxide nanoparticles on mesostructured silicate support KIL-2. (a,b) HRTEM micrographs and (c) Fourier-transformed Mn EXAFS spectrum of template-free Mn/KIL-2 (solid line – experiment, dashed line – best-fit), confirming that manganese is incorporated in the form of oxides.

### Natural zeolites for waste-water treatment

We approached the topic of environmental protection even more closely through the investigations about possibilities of *using natural zeolitic tuffs for the removal of toxic metals from waste-waters and drinking waters*. The interest in natural zeolites was raised by their availability and non-toxicity, as well as by their robustness and high selectivity. In collaboration with the groups from Zagreb and Belgrade we performed detailed structural and kinetic studies of unmodified and metal-loaded zeolitic tuffs from two deposits (Vranjska Banja, Serbia and Donje Jesenje, Croatia), with the aim to determine the sorption behaviour of selected zeolites for zinc, copper, manganese, nickel, chromium and arsenic. Furthermore we wanted to help in the development of new cost-effective zeolite-based sorbent materials with optimized sorption performance for pollutant species in groundwater, surface water and waste-waters. The studies showed that the higher content of clinoptilolite in zeolitic tuff from Serbia (more than 70% vs. 50% for Croatian tuff) resulted in its better sorption performance

for selected metals. The pre-treatment of zeolites with  $\text{Na}^+$  significantly enhanced the uptake of all metal cations, while the pre-treatment with  $\text{Fe}^{3+}$ , which resulted in stable iron-oxo-species on the zeolite surface, enabled the sorption of anions. The spectroscopic and microscopic analyses (XAS, XPS, SEM, TEM) showed that metal concentrations in all samples decreased from the surfaces towards the subsurface region of crystallites suggesting that the metal atoms first accumulate at surfaces of crystallites. Metals arrange in the pores as cations coordinated with framework oxygen atoms or some water molecules (e.g.  $\text{Cu}^{2+}$  and  $\text{Zn}^{2+}$ ) or in a form of larger poly-oxo-complexes (e.g.  $\text{Cr}^{3+}$ ). Anionic species form smaller oxo-complexes, which are attached to the iron cations on the surface of the clinoptilolite. The post-treatment of the samples with HCl, NaCl or  $\text{NH}_4\text{Cl}$  solutions revealed irreversible sorption of chromium and arsenic and mostly reversible sorption of zinc and copper.

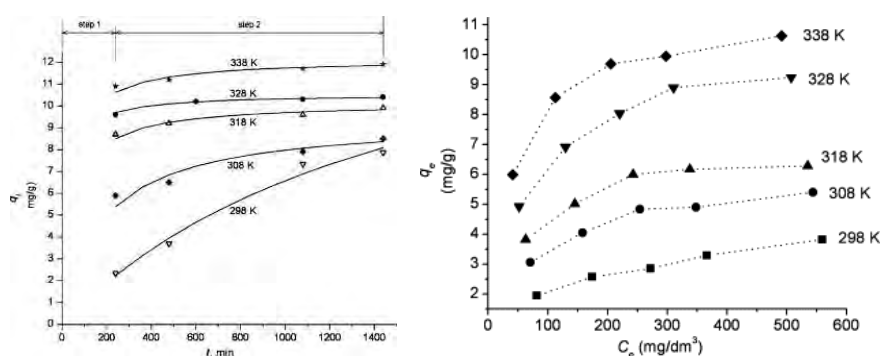


Figure 3. Sorption kinetics for Mn(II) on clinoptilolite (left);  $q_t$  is the amount of the sorbed metal (mg) per 1 g of the clinoptilolite. Symbols mark the experimental points, solid lines are the corresponding fits obtained by the pseudo-second-order kinetic model. The sorption isotherms for Ni(II) on clinoptilolite (right);  $q_e$  is the amount of the sorbed metal (mg per 1 g of the clinoptilolite) and  $C_e$  is the solution concentration at equilibrium.

### Hydrogen storage materials

Relatively recently we have directed our research into three additional topics, *materials for hydrogen storage*, *materials for heat storage*, and *drug-delivery systems*. Hydrogen is foreseen as an efficient and clean energy carrier and is already widely tested as an alternative to fossil fuels. The main obstacle toward the commercialization of hydrogen technology is safe storage of  $\text{H}_2$  at moderate conditions. A family of promising materials for hydrogen storage applications are *metal-organic framework (MOF) materials*. They are crystalline materials built of metal units linked through organic linkers with strong covalent bonds forming three-dimensional porous structure with exceptional adsorptive properties. At low temperatures (77 K) they already suffice the 2010 U.S. Department of Energy targets for  $\text{H}_2$  storage capacity, but at ambient conditions their storage capacities are still low.



We focused our research mostly on MOF materials that are based on light and/or non-toxic metal atoms, such as *magnesium, calcium, titanium, iron, and zinc*. We developed four new 3-dimensional metal-organic open-framework materials: two zinc-based 1,3,5-benzenetricarboxylates, a magnesium succinate and a calcium pimelate. The structures of new materials were solved by using either CCD single-crystal diffractometer or synchrotron facility (SOLEIL, Paris, France). Some of the above-mentioned materials could have a significant H<sub>2</sub> storage capacity. In a collaboration with Institute Lavoisier, University of Versailles, we attempted to enhance the interaction between H<sub>2</sub> molecules and MOF materials and thus to increase the H<sub>2</sub> storage capacity at ambient temperatures by modification of the selected MOF materials via *post-synthetic or in-situ introduction of functional groups*. Iron terephthalate (Fe<sup>III</sup>(bdc)OH or MIL53(Fe)) was modified by CF<sub>3</sub>, titanium terephthalate (Ti<sub>8</sub>O<sub>8</sub>(OH)<sub>4</sub>(bdc)<sub>6</sub> or MIL125(Ti)) was modified by NH<sub>2</sub> group, and zirconium terephthalate (Zr<sub>6</sub>O<sub>6</sub>(OH)<sub>4</sub>(bdc)<sub>6</sub> or UiO-66(Zr)) by NH<sub>2</sub> and CF<sub>3</sub> groups. We found out that modification of the organic linker caused an increase of H<sub>2</sub> storage capacity only in the case of MIL53(Fe). Another attempt to increase the storage capacity for H<sub>2</sub> is through the *introduction of charged metal species onto the pore surface*. Mg<sup>2+</sup>, Ni<sup>2+</sup> and Li<sup>+</sup> is introduced into highly porous iron terephthalate and trimesate (MIL-101(Fe) and MIL-100(Fe)) through anionic functional groups. H<sub>2</sub> storage capacity will be tested on a new high-pressure gas analyzer, which will be installed in our laboratory by the end of November 2010.

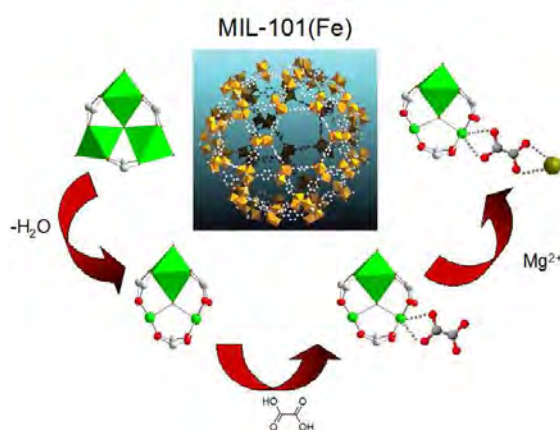


Figure 4. Schematic representation of Mg<sup>2+</sup> introduction into MIL-101(Fe) structure through oxalate functional group.

### Materials for heat storage and drug-delivery

Development of new *heat-storage systems* is extremely important for wider solar-energy and waste-heat utilization for domestic and industrial use. By improving the effectiveness of thermal storage, we can improve the effectiveness of most of energy conversion and utilization technologies. This is especially important in the case when there is a mismatch in

the time, when a renewable energy or waste heat is available, and the time, when the energy or heat is required (e.g. solar energy is available in summer and one would like to somehow store it until winter).

A class of promising materials for heat storage are also adsorbents, among which microporous and mesoporous materials with high surface area play an especially important role. The utilization of water adsorbents for heat storage is schematically explained in Figure 5. The aim of our research is to study the dependency between the structure of novel microporous and mesoporous materials and their energy storage abilities (energy density, thermal power, transport properties, and the temperature levels for charging and discharging). After establishing and understanding these dependencies, we would like to improve microporous and mesoporous storage materials. One of the attempts to enhance the heat storage abilities of porous materials is also by incorporating hygroscopic inorganic-salt-hydrates and metal active sites into their pores.

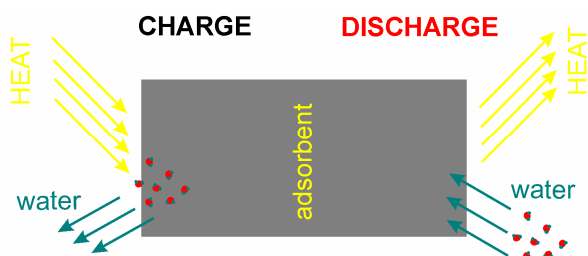


Figure 5. Schematic presentation of the performance of a heat storage system. The heat collected and stored in an adsorbent during summer could be released and used during winter. Potential adsorbents include aluminophosphates, zeolites, mesoporous metal silicates, porous metal-carboxylates, and composites with salt hydrates.

*Drug-delivery systems based on mesoporous silicates* have recently attracted a lot of attention, because they could enable controlled release of drugs. This means that by using such drug-delivery systems in combination with proper endogenic or exogenic physical or chemical stimuli, drugs could be released in the proper part of the human body within proper time period and in an adequate concentration. In our group we are studying incorporation and release of a model drug of indomethacin to and from two carriers, SBA-15 and MCM-41. We are particularly interested in *the physical state of the drug within the mesopores* of different diameters. We are investigating how this state depends on the concentration of the drug within the pores and how it influences the drug release. We are also studying the influence of the functionalization of the silicate walls on the drug incorporation and release. Detailed description requires inspection by XRD, NMR, thermal (TG and DSC) analysis, nitrogen adsorption and helium picnometry. The preliminary results show very interesting and unexpected effects due to confining a drug into mesopores of different diameters.

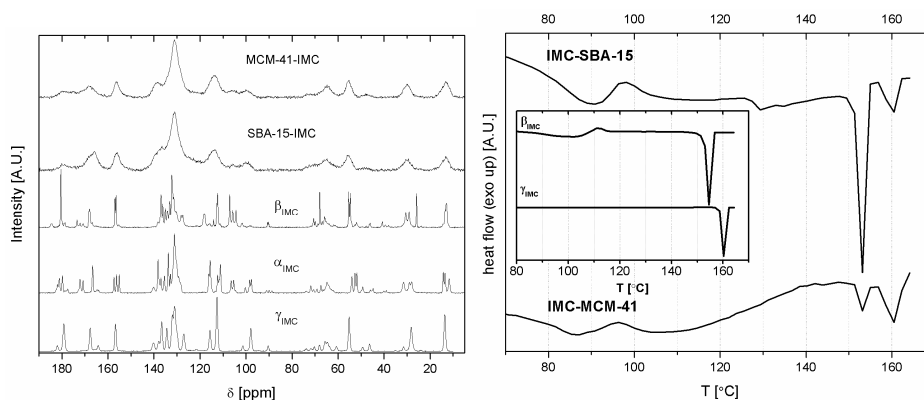


Figure 6.  $^{13}\text{C}$  MAS NMR spectra (left) and DSC curves (right) of two polymorphs ( $\alpha$ ,  $\gamma$ ) and a solvate ( $\beta$ ) of indomethacin, and of indomethacin incorporated into the mesopores of MCM-41 and SBA-15.

*Characterization* of the obtained materials is an extremely important part of our research. It allows us to understand the structure and function of the materials. We combine diffraction techniques with microscopy and with spectroscopic methods probing the local short-range arrangements of constituents. In the past years we have installed (or gained access to) valuable *equipment* such as three X-ray powder diffractometers (Siemens D5000, PANalytical X'Pert PRO, and PANalytical X'Pert PRO HTK), microscopes SEM (Zeiss SupraTM 3VP), TEM (200-kV field-emission gun microscope JEOL JEM 2100), and AFM (Agilent 5500), NMR spectrometer (600 MHz Varian NMR systems with several MAS probeheads), gas adsorption analyzer (Micromeritics, ASAP), system for thermal analysis (SDT 2960), and synchrotron facilities for X-ray absorption spectroscopy (DESY in Hamburg, Elettra in Trieste).

The majority of our research is carried out within the frame of two national projects, Nanoporous materials and Novel Thermal Energy Storage Materials, and through participation within two centres of excellence co-funded by European funds for regional development (one centre is dealing with hydrogen technology and another one with the development and application of NMR techniques). We have participated in European network of excellence Inside Pores, and have collaborated or are still collaborating in an EUREKA project (PUREWATER) and in several bilateral projects (with research groups from Argentina, Croatia, France, Hungary, Norway, and USA). We also established successful long-term collaboration with three Slovenian companies, Silkem, a producer of synthetic zeolite, Krka, Slovenian pharmaceutical company, and Salonit, a cement-producing company.

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