

## Eduard Hála Laboratory of Separation Processes

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### Main fields of research

- Thermodynamic properties and phase behavior of ionic liquids and their mixtures with molecular solvents
- Experimental determination of vapor–liquid equilibria in mixtures containing components of low and high molecular mass
- Mass transport in polymeric membranes, mutual influence of permeating substances
- Membrane separation of CH<sub>4</sub> and CO<sub>2</sub> mixtures
- Separation of gases by ionic liquids membranes
- Condensation in porous membranes during vapor permeation
- Separation of volatile organic compound from air
- Pertraction – separation of enantiomers
- Supercritical fluid extraction and pressurized liquid extraction of bioactive compounds from plants and their mathematical modelling; integration of extraction and fractionation
- Study on preparation of nanostructured metal oxides using supercritical and pressurized fluids
- Study on preparation of polymeric foams by pressurization with supercritical CO<sub>2</sub> followed by rapid depressurization
- Kinetic studies of hydrogenation reactions in a packed-bed microreactor
- Microreactors application for photosensitive reactions

- Methodology development for integration of 3D printing technologies in the advanced design, modelling and manufacture in chemical

### **Applied research**

- Separation of unwanted components from raw biogas
- Separation of volatile organic compound from air
- Microtechnology application for kinetic studies and process intensification
- Utilization of microreactors as efficient tools for kinetic studies and process intensification
- Advanced separation methods developments for liquid-liquid systems

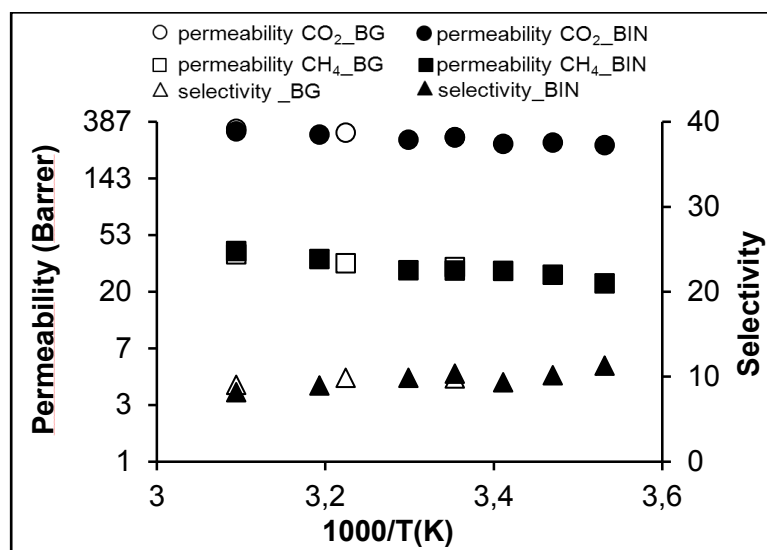
## Research projects

### Enrichment of raw biogas by methane

(P. Izák, [izak@icpf.cas.cz](mailto:izak@icpf.cas.cz); joint project with UCT, Prague; supported by MEYS, project No. LH14006)

Membrane gas permeation found its place among the biogas upgrading methods some years ago. Here, we try to summarize the progress in the implementation of gas permeation in biogas upgrading. Gas permeation has been already accepted as a commercially feasible method for CO<sub>2</sub> removal. Many different membranes and membrane modules have been tested and also some commercial devices are available. On the other hand, utilization of gas permeation in other steps of biogas upgrading like desulfurization, drying, or VOC removal is still rather rare. This work shows that membrane gas permeation is able to compete with classical biogas upgrading methods and tries to point out the main challenges of the research.

The influence of the temperature and stage cut on permeation of CO<sub>2</sub> and CH<sub>4</sub> through two different supported ionic liquid membranes were studied (see Fig.). The measurements were performed with binary mixture of CH<sub>4</sub> and CO<sub>2</sub> and with real pre-dried biogas collected in sewage plant. The influence of temperature on permeability followed the Arrhenius behavior in agreement with solution diffusion model of transport. The influence of stage cut was also very small, what helped to confirm that the membrane was operated under optimal conditions. [Refs. 12, 39]



**Temperature dependence of permeability and selectivity of CH<sub>4</sub> and CO<sub>2</sub> in binary mixture (denoted as BIN) and raw biogas (denoted as BG) in [emim][Tf<sub>2</sub>N] membrane (permeability is displayed as a semilogarithmic plot) [Ref. 12]**

### Membrane separation – the more effective separation of a pure enantiomer from a racemic mixture

(P. Izák, [izak@icpf.cas.cz](mailto:izak@icpf.cas.cz); joint project with UCT, Prague and IMC; supported by GACR, project No. P106/12/0569)

A different behavior of particular enantiomers was typically observed in the prepared membranes (although this behavior does not imply resolution of racemic mixtures in all cases) and hypotheses about the reason of these facts were formulated and published. The experimental results together with the mathematical description were also published in high impacted journals. Concerning new materials, we prepared large set of membranes based on

derivatives of saccharides and polyimides with covalently bonded chiral selectors or chiral secondary structure and membranes with chiral ionic liquid incorporated into the membrane.

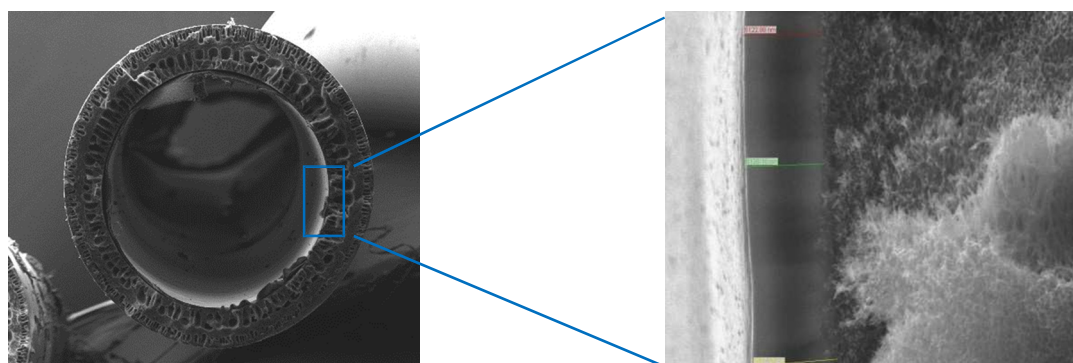
By our study, it was demonstrated that the supported chiral liquid membranes offer high selective solute transport which makes them particularly suitable for the recovery of minority solutes from complex mixtures, enantiomeric resolution and for the removal of specific contaminants from liquid or gas streams. We showed that these processes may be favored by the use supported ionic liquid membranes systems, since they allow better control of solute permeability and improved membrane stability by non-invasively modulation of magnetic ionic liquids viscosity and solubility by an external magnetic field. This aspect represents an important advantage for the optimization of small-scale processes commonly used in pharmaceutical industry. Our molecular simulations predicted a significant enhancement of the cation and anion densities at the interface in comparison with the liquid densities. In addition, the charge and atomic density profiles indicated that the vapor sides of interfaces are primarily populated by cation alkyl or chiral chains.

We considered possible patenting based on our results obtained within this basic research project. Also this project undoubtedly brought important findings in other related fields as physical and polymer chemistry, chemical engineering, and pharmacy. [Refs. 1, 2, 24, 36]

### Separation of polar and non-polar gasses by membrane processes

(P. Izák, [izak@icpf.cas.cz](mailto:izak@icpf.cas.cz); supported by GACR, project No. GA14-12695S)

Biogas upgrading is a widely studied and discussed topic. Many different technologies have been employed to obtain biomethane from biogas. Poly(ether-b-amide) (PEBAX 1657)/polyacrylonitrile copolymer (PAN) composite hollow fiber membranes for potential use in CO<sub>2</sub>/CH<sub>4</sub> separation were prepared by a new continuous coating method, referred to as cross-flow filtration. This technique allows obtaining the simultaneous coating of a large number of fibers, facilitating the scale-up. The dense layer was deposited in the lumen of the fibers allowing the coating of all the fibers in a single step. The coating on the inner surface of the fibers avoids the negative effects such as sticking or accidental mechanical damages occurring in the case of external coating. The membrane preparation was optimized by modulating different parameters. The optimal range of viscosity and concentration of the polymer solution to obtain a selective homogeneous PEBAX layer was identified. The presence of the PEBAX 1657 dense layer was confirmed by IR spectroscopy and the morphology of the composite membranes was observed by SEM analysis (see Fig.). Gas separation performance of the membrane modules was evaluated by single gas permeation measurements. A preliminary optimization yielded membranes with  $P(\text{CO}_2) = 5 \cdot 10^{-3} \text{ [m}^3 \text{ m}^{-2} \text{ h}^{-1} \text{ bar}^{-1}]$ ,  $\alpha(\text{CO}_2/\text{CH}_4) = 18$  equal to that of the neat dense polymer. The PEBAX/PAN hollow fibers modules are potentially useful for application in the biogas purification. [Refs. 5, 37, 39]

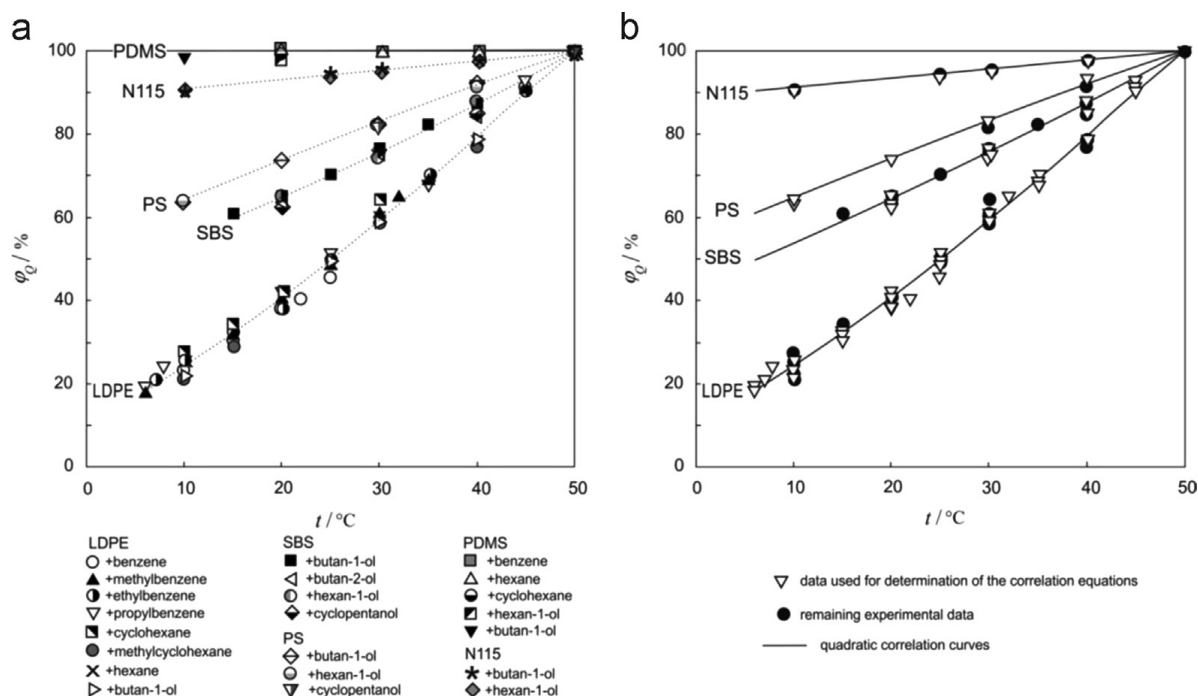


SEM images of the cross section of: composite hollow fiber membrane (left side); detail of the PEBAX coating layer on the inner surface (right side) [Ref. 5]

## Supported ionic liquid membrane for separation of volatile organic compounds and pollutants from flue gases

(P. Izák, [izak@icpf.cas.cz](mailto:izak@icpf.cas.cz); supported by MEYS, project No. LD14094)

The aim of the project is the development of ionic-liquid containing membranes for the separation of volatile organic compounds and pollutants from flue gases. A new prediction method for sorption of low-molecular organic liquids (non-solvents) in polymers is presented (see Fig.). It was derived from the gravimetric data for sorption of linear and cyclic alkanes, aromatics and alcohols into various kinds of polymers (glassy polystyrene, rubbery polydimethylsiloxane, semi-crystalline low-density polyethylene, ion exchange Nafion, and copolymer poly(styrene-butadiene-styrene)) in temperature range 5-50 °C. [Refs. 21, 25, 35]



The normalized sorption values  $\phi_Q$  as a function of temperature, (a) all experimental results, (b) quadratic polynomial correlation curves and experimental points divided into two groups: experimental data used for determination of the correlation equations ( $\nabla$ ) and remaining experimental data for verification ( $\bullet$ ). Solid lines represent a prediction model while dashed lines serve as an eye guide only

## Permeation of condensable gases through asymmetric membranes

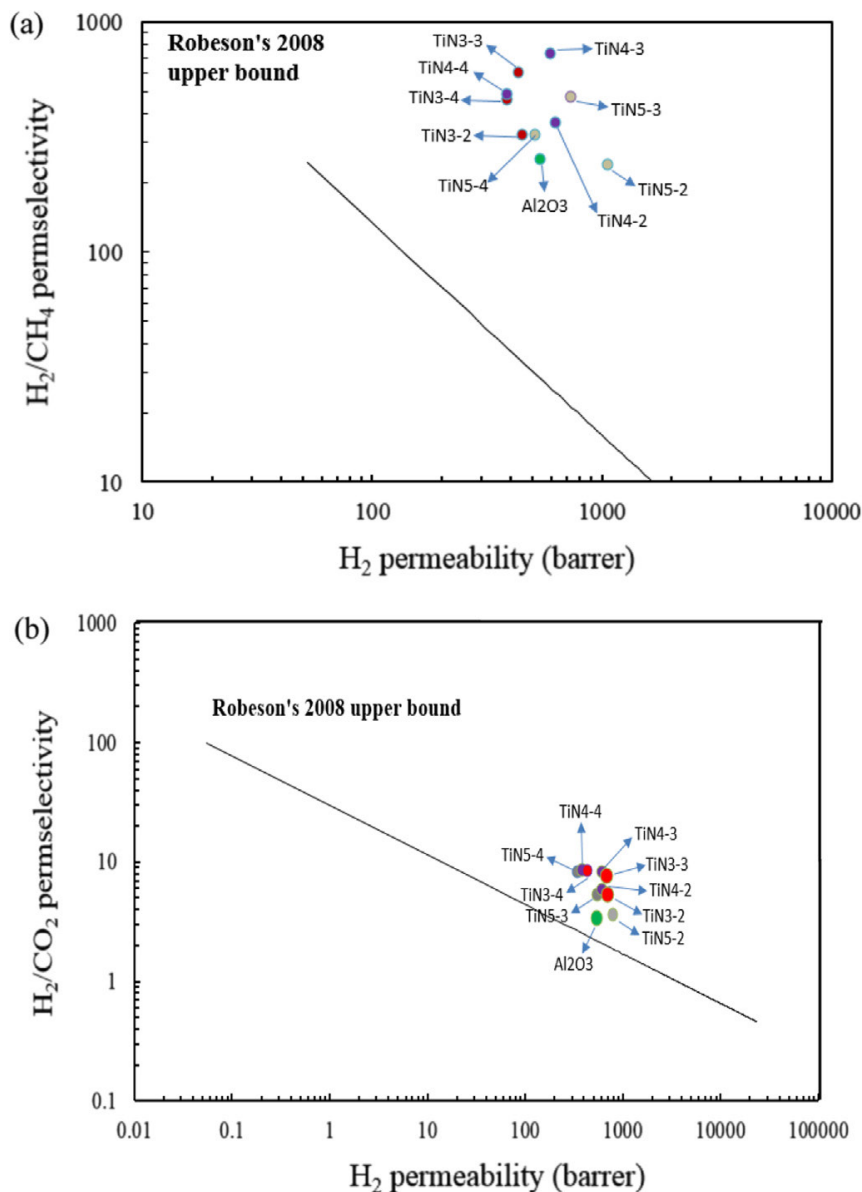
(J. Řezníčková, [reznickova@icpf.cas.cz](mailto:reznickova@icpf.cas.cz); joint project with Institut für Strömungsmechanik und Wärmeübertragung, Technische Universität Wien; supported by MEYS, MOBILITY, project No. 7AMB14AT011)

The aim of our collaboration is to carry out a series of experiments to obtain the pressure and temperature distributions within asymmetric ceramic membranes. Experimental data will improve our understanding of permeation process especially under condition of condensation. It is difficult to set and maintain the correct experimental conditions. Furthermore, the direct measurement of the temperature and pressure distributions is not possible. Experimental difficulties are one of the reasons for the lack of sufficient experimental data. Therefore, a special apparatus was designed and constructed. The use of this apparatus will help at obtaining at least in an indirect way the desired pressure and temperature distributions. Data gleaned from our experiments will help at understanding the flow process. By condensation it may be possible to enhance the production in similar processes. [Ref. 17]

### Preparation and characterization of mixed matrix membrane for gas separation

(P. Uchytíl, [uchytíl@icpf.cas.cz](mailto:uchytíl@icpf.cas.cz); joint project with Department of Occupational Safety and Health, Chung Medical University, Taiwan, supported by CAS, PPP project MOST/14/02)

Carbon/TiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> sandwich molecular sieving membrane synthesized via a sol-gel spin coating method followed by a carbonization process was proposed for the first time. Significantly influence on the adhesion mechanisms is observed from the TiO<sub>2</sub> intermediate layer coverage on an Al<sub>2</sub>O<sub>3</sub> support and the microstructure and surface roughness of the TiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> composite support, whose fractions strongly depended on the sol-gel conditions, i.e., the molar ratio of HNO<sub>3</sub>/TTIP and the number of coatings. Furthermore, to the best of our knowledge, the adhesion mechanisms of the CMS layer on the TiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> composite support were evaluated for the first time using EDX-line scanning, FTIR, and contact angle to determine the contribution from mechanical interlocking, chemical bonding, and adsorption, respectively. The results show that the three adhesion mechanisms might simultaneously contribute to the intrinsic adhesion depending on the preparation variables of the TiO<sub>2</sub> nano-network.



**Performance of the TiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> supported CMS for the separation of (a) H<sub>2</sub>/CH<sub>4</sub> and (b) CO<sub>2</sub>/N<sub>2</sub> with respect to the Robeson trade-offline**

### CO<sub>2</sub>/CH<sub>4</sub> separation by ionic liquid/polymer or composite membranes

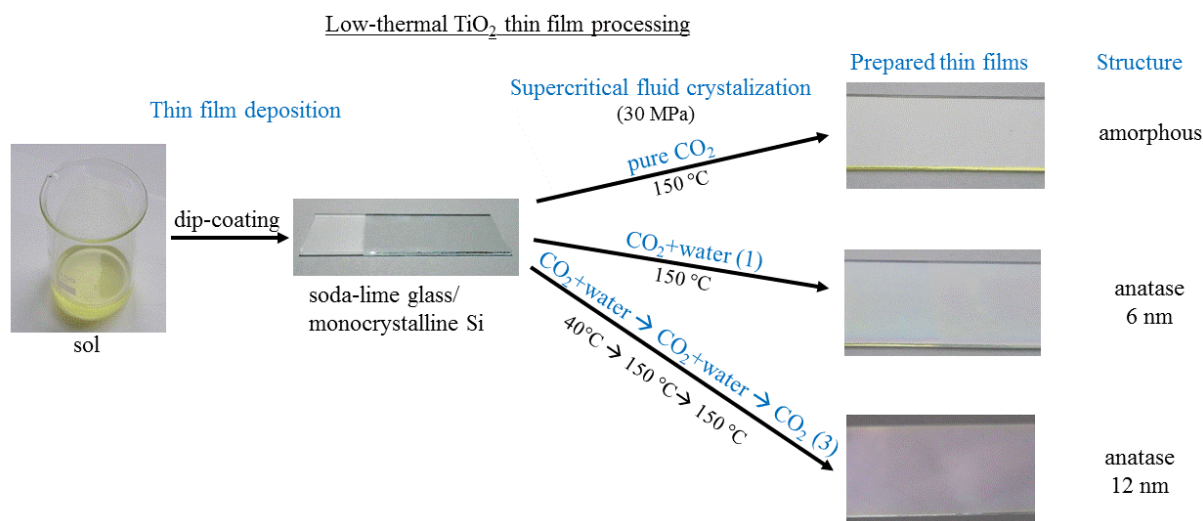
(K. Setničková, [setnickova@icpf.cas.cz](mailto:setnickova@icpf.cas.cz); joint project with Department of Chemical Engineering, National Chung Hsing University, Taiwan, supported by CAS, PPP project MOST/15/05)

Carbon dioxide is a major contributor to global warming. Significant efforts on decreasing CO<sub>2</sub> emission and increasing its recovery are undertaken all over the world. Membrane process is considered a promising technology for CO<sub>2</sub> recovery from gas mixture, and developing membranes with high CO<sub>2</sub> permeability and high separation factor becomes very important. Ionic liquid membrane is one of the candidates to achieve this goal. In most studies, ionic liquid is filled into the pores of a solid support. Gas permeation is accordingly affected by both the solid material and ionic liquid. It is suggested in this project by coating an ionic liquid layer upon a dense polymeric membrane with high CO<sub>2</sub> permeability to reduce the resistance on CO<sub>2</sub> permeation. Moreover, the combination of ionic liquid layer and dense membrane may enhance the overall gas selectivity. [Ref. 27]

### Unconventional preparation of nanostructured metal oxides by using pressurized and supercritical fluids

(M. Sajfřtová, [sajfřtova@icpf.cas.cz](mailto:sajfřtova@icpf.cas.cz); joint project with Nanotechnology Centre, VSB-TU Ostrava and X-ray group of Faculty of Mathematics and Physics, CU, Prague; supported by GACR, project No. GA14-23274S)

The project concerns the direct preparation of highly pure nanocrystalline titanium thin films and aerogels by using supercritical fluids without any subsequent thermal treatment. The crystallization with pure and modified scCO<sub>2</sub> by water (5-30 wt. %) and/or organic solvent (10 wt. %) was tested. The temperature (40-150 °C), pressure (10-30 MPa) and the volume of CO<sub>2</sub> (50-200 g) passed through the extractor were optimized with respect to the microstructure and purity of TiO<sub>2</sub> thin films and aerogels. The purity and crystallinity of prepared TiO<sub>2</sub> thin films and aerogels were estimated by Raman spectroscopy. The crystallite size and the phase composition were determined by X-ray diffraction. Textural properties of aerogels such as the surface area and the pore-size distribution were characterized by nitrogen physisorption and helium pycnometry measurements.



### Morphology evolution of nano- and micro-cellular polymeric foams

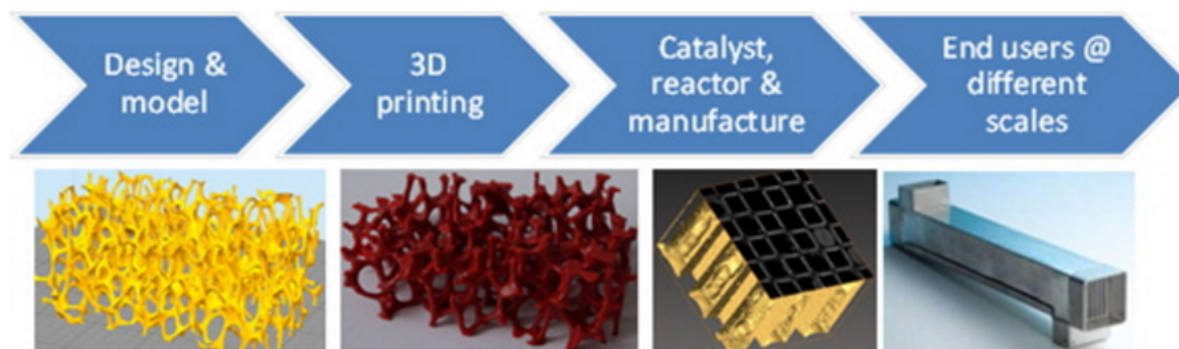
(H. Sovová, [sovova@icpf.cas.cz](mailto:sovova@icpf.cas.cz); joint project with Faculty of Chemical Engineering, UCT Prague and New Technology – Research Centre, University of West Bohemia, supported by GACR, project No. GA14-18938S)

The project is aimed at first-principles understanding of early stages of polymer foam evolution including nucleation or phase separation by spinodal decomposition and coalescence of cells. Experimental methods include, among others, the foaming of polystyrene with supercritical CO<sub>2</sub>. Based on the improved understanding of nucleation and coalescence, the methods of nano-/micro-cellular foam preparation will be assessed and optimized.

### **Process intensification through adaptable catalytic reactors made by 3D printing (PRINTCR3DIT)**

(P. Stavárek, [stavarek@icpf.cas.cz](mailto:stavarek@icpf.cas.cz); EC Horizon 2020, project: 680414, [www.printcr3dit.eu](http://www.printcr3dit.eu))

PRINTCR3DIT is a joint effort between world-leading industries (4), innovative SMEs (4), R&D institutes (4) and a university that aim to accelerate deployment of a set of products to the market. The project is coordinated by SINTEF Materials & Chemistry, Norway. The main objective of the PRINTCR3DIT project is to implement a methodology to integrate 3D printing in the advanced design, modelling and manufacture of structured catalysts and catalytic reactors with significant cost reductions, access to new design strategies and faster lead times.



The principal target of the project is to increase the efficiency through process intensification in reactions that present heat, mass and momentum transfer limitations, with targeted goals to significantly reduce the energy consumption, increased selectivity and longer lifetimes. The utility and flexibility of the methodology will be demonstrated through three industrial processes that span different chemical sectors, scales of production and catalytic reactors. The selected examples will target the production of fine chemicals, commodity chemicals and fertilizers.

### **Development of advanced separation methods for liquid-liquid systems**

(P. Stavárek, [stavarek@icpf.cas.cz](mailto:stavarek@icpf.cas.cz); research contract with Modelarna LIAZ s.r.o.)

The research activities on the topic of advanced separation methods for mixtures of miscible liquids continued well on basis of results gained from previous cooperation. The efforts were focused on experimental validation and optimization of the methods with a constructed laboratory apparatus. The collected results were documented and critically evaluated in three confidential research reports.

### **Application of microreactors for gas phase catalytic reactions**

(P. Stavárek, [stavarek@icpf.cas.cz](mailto:stavarek@icpf.cas.cz); research contract with Hexion a.s.)

Well established cooperation with Hexion a.s. continued with systematic research of microreactors and their application to gas phase catalytic reactions. As the microreactor design was completed in previous project stages the efforts were now focused on the microreactor construction and its assembling, laboratory apparatus design and analytical

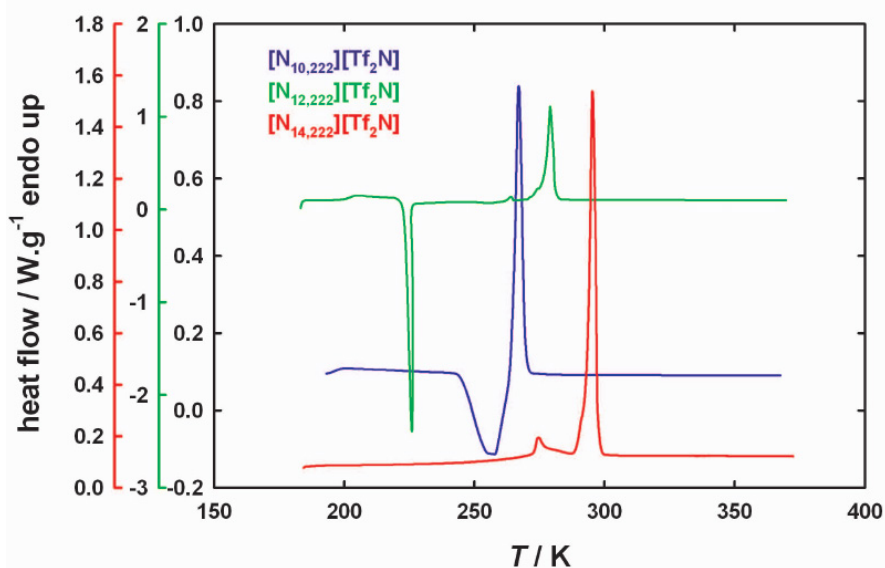


method development to analyze products of a case study reaction. The analytical method was successfully developed, experimentally validated and quantified with participation of one diploma thesis student. Next project stage is the laboratory testing with a case study reaction.

### Thermal properties of alkyltriethylammonium bis(trifluoromethyl)sulfonylimide ionic liquids

(M. Bendová, [bendova@icpf.cas.cz](mailto:bendova@icpf.cas.cz); joint project with J. Jacquemin of QUILL Belfast, UK)

Thermal properties, phase and glass transition behavior, of selected members of a homologous series of alkyltriethylammonium bis(trifluoromethyl)sulfonylimide, i.e.  $[N_{R,222}][Tf_2N]$ , ionic liquids were studied using differential scanning calorimetry (DSC). Decomposition temperature was also determined in the present series by thermal gravimetry analysis. Furthermore, isobaric molar heat capacity as a function of temperature for hexyltriethylammonium, octyltriethylammonium, and dodecyltriethylammonium bis(trifluoromethyl)sulfonylimide ionic liquids. Based on the data obtained, we discuss the influence of the alkyl chain length of the cation on the studied ionic liquids on the measured properties. Using viscosity data obtained in a previous work, the liquid fragility of the ionic liquids is then discussed. Viscosity data were correlated by the VTF equation using a robust regression along a gnostic influence function. In this way, more reliable VTF model parameters were obtained than in our previous work and a good estimate of liquid fragility of ionic liquids was made.

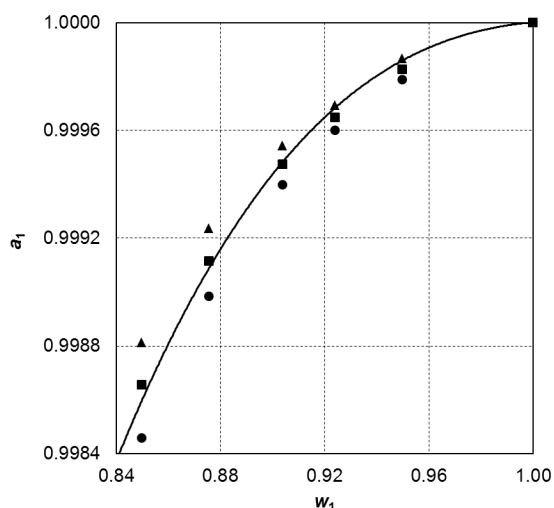


### Comparison of solid-liquid and solid-solid phase transitions for $[N_{R,222}][Tf_2N]$ ionic liquids where R stands for decyl, dodecyl, and tetradecyl [19]

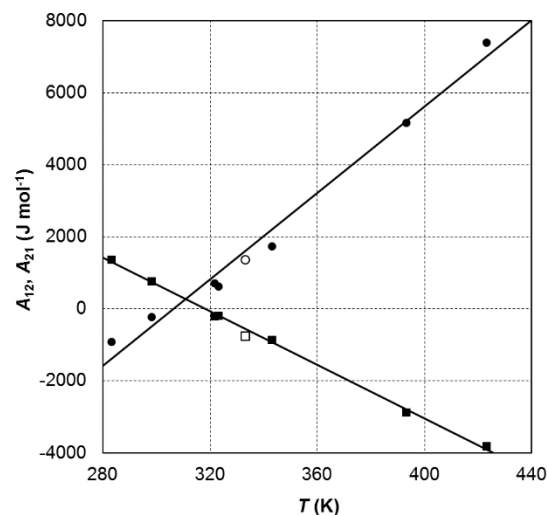
### Thermodynamics of polymer solutions: experiments meet theory and vice versa

(I. Wichterle, [wi@icpf.cas.cz](mailto:wi@icpf.cas.cz); supported by GACR, project No. 15-19542S)

*Experiments:* Vapor-liquid equilibria have been determined in systems composed of polystyrene + toluene [Ref. 22], and polystyrene + butan-2-one [Fluid Phase Equilibria, in press] by ebulliometric method, i.e. by total pressure measurement. The ebulliometer has been redesigned in 2014 and experimental procedure has been upgraded. Experiments have been carried out isothermally, the measured data were correlated by the UNIQUAC-FV model, and compared with available literature data. It should be stressed that this type of measurements, i.e. ebulliometry in the high-concentration region of solvent, is unique and is presently carried out only at the ICPF. Results were presented at two international conferences, too.



**Activity  $a_1$  of butan-2-one in PS as a function of butan-2-one weight fraction  $w_1$ . Experimental data at (●) 323.15 K, (■) 333.15 K and (▲) 343.15 K. Solid line represents predicted activities at 333.15 K using the UNIQUAC-FV parameters estimated using all data within temperature range 323.15–343.15 K.**



**Temperature dependence of UNIQUAC-FV  $A_{12}$  (■) and  $A_{21}$  (●) parameters. Open points represent UNIQUAC-FV parameters evaluated from our data at 323.15–343.15 K and plotted at mean temperature 333.15 K.**

*Theory:* The first year study was aimed to electrolytes since ions are present in every solution. The complete description of solution is at hand if chemical potential is known. The calculation of chemical potential via molecular simulation is complicated and moreover existing data are not in mutual agreement even for simple systems. However, all the proofs have testified that our simulations are well working. Two papers by other scientists presented in 2015 have confirmed correctness of our (the best in the world) computations. This result has been published at 5 international meeting and prof. Nezbeda was invited to prepare the perspective review dedicated to this project for Molecular Physics journal, which was submitted in December 2015.

The problems of polymer self-assembly in relation to phase equilibria (solid/liquid and liquid/vapor interfaces) were studied. Experimental investigation of the co-assembly of block copolymers with surfactants has been performed. The extensive manuscript deals with the electrostatic co-assembly of polymeric micelles with surfactants by a combination of a number of experimental techniques. Even though it is aimed mainly on the structure of formed nanoparticles and on the mechanisms of underlying processes, it shows that some fluids components of the studied polymer–colloid–solvent mixtures are engaged in non-volatile complexes and do not contribute to the vapor–liquid equilibria.

*Other activities:* High-pressure phase equilibrium data in the carbon dioxide + 1-chloropropane binary system were published [Ref. 11]. As guest editors, we prepared a special issue of the Chemical and Biochemical Engineering Quarterly with contributions from 21<sup>st</sup> CHISA Congress [Ref. 40].

## International co-operations

- Institut für Strömungsmechanik und Wärmeübertragung Technische Universität Wien: Flow of saturated vapors through porous membranes
- Institute of Chemical Engineering, Sofia, BAS: High-pressure phase equilibria
- Institute of Macromolecules, St. Petersburg, RAS, Russia: Membrane separation
- Institute on Membrane Technology, CNR, Italy: Novel composite membranes containing ionic liquid and selected polymers for specific gas/gas, gas/vapor and vapor/vapor separations
- Institute of Chemical Engineering, Sofia, BAS: High-pressure phase equilibria
- KIT Karlsruhe, Germany: Design of pilot plant size microreactor for sulfur dioxide catalytic heterogeneous oxidation
- Momentive Specialty Chemicals, Czech Republic: Application of microreactors for gas phase catalytic reactions
- National Chung Hsing University, Taiwan: Preparation of Dense Homogeneous Polymeric Membranes and Study on Their Gas Permeation Properties
- New University of Lisbon, Portugal: Membrane separation processes
- Otto von Guericke University of Magdeburg, Max-Planck-Institut für Dynamik-komplexer technischer Systeme, Magdeburg, Germany: Mass transport through porous membranes
- Procter&Gamble, Belgium: Research and developments of microapparatus characteristics
- Procter&Gamble, Belgium: Hydrodynamics of micro reactor for sulfonation
- Procter&Gamble, Belgium: Hydrodynamics of annular gas-liquid flow
- Queen's University Belfast, QUILL: Database on liquid-liquid equilibria of binary mixtures of ionic and molecular compounds, IUPAC Project #2011-065-3-500
- Swiss Federal Institute, Switzerland: Chiral ionic liquids and membrane separation
- Technische Universität Wien, Institut für Strömungslehre und Wärmeübertragung, Austria: Flow of saturated vapors through porous membranes
- University of Colorado, Boulder, CO, USA: Mass transport during vapor permeation and pervaporation, ionic liquids
- University of Burgos, Spain: Enzymatic reactions of oil in supercritical CO<sub>2</sub> medium
- University of KwaZulu-Natal, Republic of South Africa: Liquid-liquid extraction processes with fluorinated hydrocarbons, recovery of lanthanides
- Wrocław University of Technology, Faculty of Chemistry: Physicochemical characterization of chiral ionic liquids based on a natural terpene substituent
- J. Hanika: UCT, Faculty of Chemical Technology, postgradual course “Multiphase Reactors”
- H. Sovová: UT Maribor and Graz UT, The European Summer School in High Pressure Technology
- M. Bendová: UCT, Faculty of Chemical Engineering, postgraduate course “Physical Chemistry for Technological Practice”
- M. Bendová: UCT, Faculty of Chemical Engineering, postgraduate course “Introduction into irreversible thermodynamics: theory and practice”

Z. Sedláková: UJEP, Faculty of Science, courses for under-graduated students “Membrane Separations”, “Basic of Chemistry”, “Physical chemistry”, “Experimental and Nonexperimental methods in Physical Chemistry”

## Awards

Hanika J. – Hanuš Memorial Medal, 2015, for the merits for the development of chemistry, from the Czech Chemical Society

Hanika J. – Annual Prize 2015, for the merits for the development of chemical reaction engineering, from the Czech Society of Chemical Engineering

## Publications

### Original papers

- [1] Brožová L., Žitka J., Sysel P., Hovorka Š., Randová A., Storch J., Kačírková M., Izák P.: Sorption of Single Enantiomers and Racemic Mixture of (+/-)- $\alpha$ -Pinene into Nafion Membranes. *Desalin. Water Treat.* 55(11), 2967-2972 (2015).
- [2] Brožová L., Žitka J., Sysel P., Hovorka Š., Randová A., Storch J., Kačírková M., Izák P.: Stereoselective Behavior of Nafion<sup>®</sup> Membranes towards (+)- $\alpha$ -Pinene and (-)- $\alpha$ -Pinene. *Chem. Eng. Technol.* 38(9), 1617-1624 (2015).
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- [8] Hanika J.: 2. Chemická technologie a lidské společenství. (Czech) Chemical Technology and Human Community. *Valašský chemik* 48(11), 4 (2015).
- [9] Hanika J.: 3. Chemická technologie a lidské společenství. (Czech) Chemical Technology and Human Community. *Valašský chemik* 48(12), 2 (2015).
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