Eduard Hála Laboratory of Separation Processes

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Fields of research

- Thermophysical properties of pure ionic liquids and their liquid phase behaviour in mixtures with molecular solvents
- Experimental determination of vapour-liquid equilibria in mixtures containing components of low and high molecular mass
- Data processing using activity coefficient models and equations of state
- Prediction of phase behaviour by means of models based on group contribution methods
- Mass transport in polymeric membranes, mutual influence of permeating substances
- Membrane separation of CH_4 and CO_2 mixtures
- Separation of gases by ionic liquids membranes
- Condensation in porous membranes during vapor permeation
- Gas transport in asymmetric porous membranes
- Pervaporation - dehydration of alcohols, separation of azeotropic mixture, etc.
- Separation of volatile organic compound from air
- Pertraction – separation of enantiomers
- Evaluation of transport properties such as permeability, activation energy of permeation and selectivity of process
- 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_2 followed by rapid depressurization
- Design and construction of extraction units based on Vibrating Plate Extractor (VPE)
- Extractive separation of biologically active substances from plants and microalgae
- Kinetic studies of hydrogenation reactions in a packed-bed microreactor
- Utilization of microreactors as an efficient tool for photosensitive reactions

Applied research

- Technology for the preparation of molecularly imprinted polymeric materials
- Separation of unwanted components from raw biogas
- Separation of volatile organic compound from air
- Separation of racemic mixtures
- CO_2 extracts of insecticidal substances from plants for botanical pesticides
- Recovery of precious and special metals from waste electrical and electronic equipment using Vibrating Plate Extractor (VPE)
- Microtechnology application for kinetic studies and process intensification

Research projects

Enrichment of raw biogas by methane

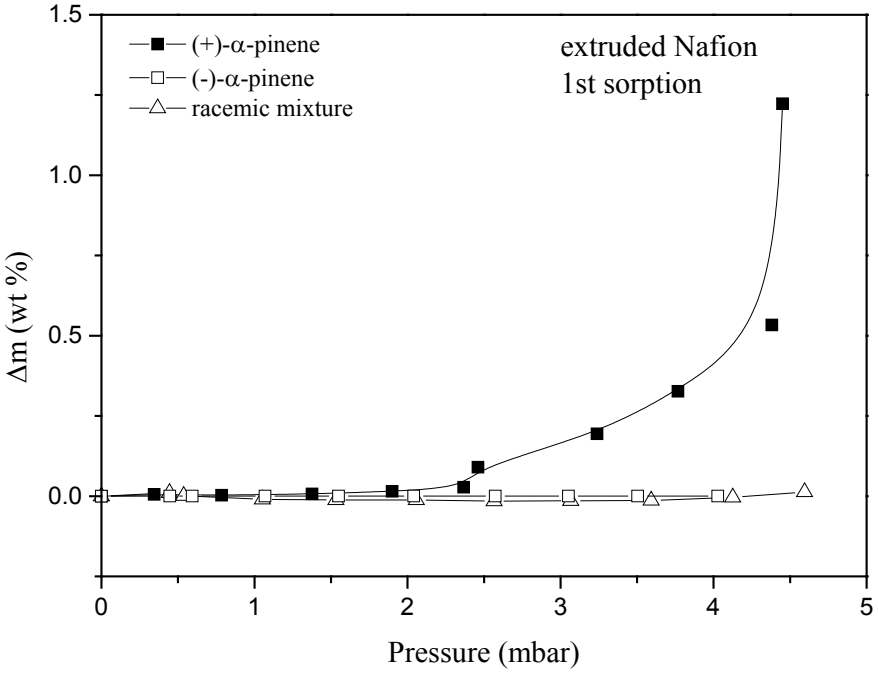
(P. Izák, izak@icpf.cas.cz; joint project with UCT, Prague; supported by MEYS, project No. LH14006)

The principal aim of this project is preparation, characterization and testing of advanced separation polymeric membranes with enhanced performance containing anchored ionic liquids in a suitable non-porous polymeric membrane using (i) Van der Waals forces in coated (composite) porous asymmetric polymeric membrane or (ii) a covalent bond or (iii) high ionic liquid content cross-linked polymer gels. Preparation of such novel separation membranes is targeted for an efficient methane separation from biogas. It will also be developed a model enabling prediction the transport and separation characteristics of these membranes intended for separation of gas mixtures. [Refs. 2, 21]

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

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

The aim of this work was to elucidate the sorption of α-pinene in the membranes of solution-cast and melt-extruded Nafion. The extruded membrane was Nafion® 115 in H^+ form (DuPont). The cast Nafion membrane was prepared by casting from a 20 wt.% solution of Nafion® (H^+ form) in lower aliphatic alcohols and water. (+)-α-pinene, (−)-α-pinene and racemic mixture sorption isotherms were determined by a gravimetric method using the sorption balance. It was found that the sorption of α-pinene in the Nafion membrane is a stereoselective process. The sorption of α-pinene in cast Nafion is much higher than in extruded Nafion. The sorption of (+)-α-pinene in the extruded Nafion membrane is quite low while the sorption of (−)-α-pinene approaches zero. The sorption of (−)-α-pinene in the cast Nafion membrane is high while the sorption of (+)-α-pinene is low. [Refs. 8, 12]

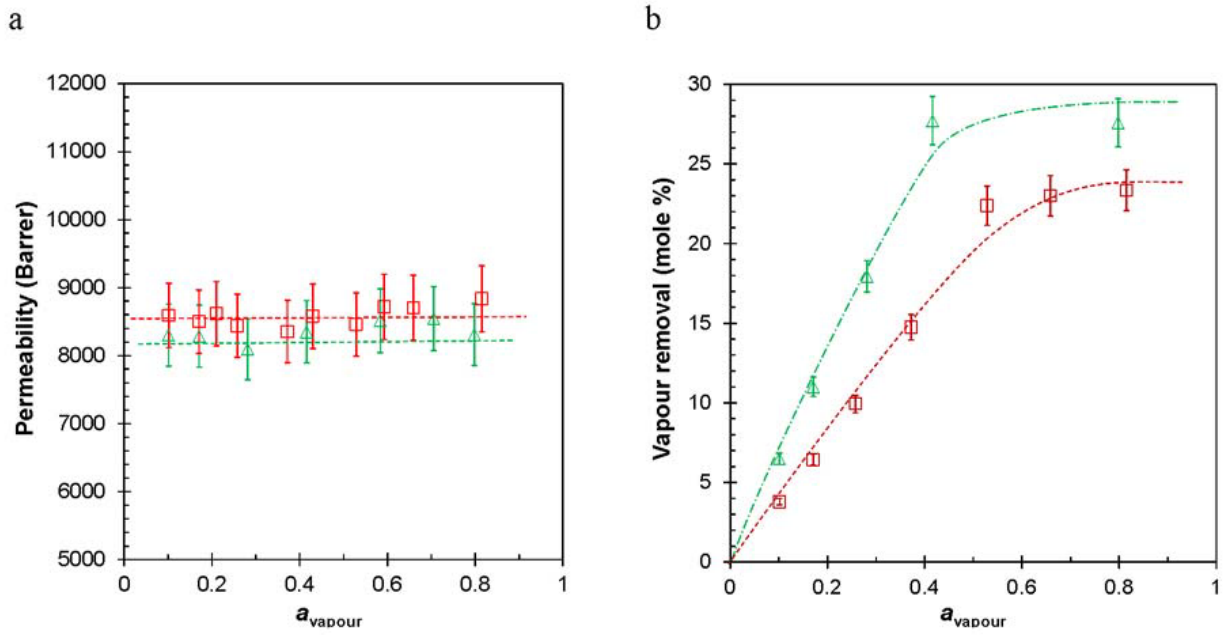


Sorption isotherms of (+)-α-pinene, (-)-α-pinene and their racemic mixture in extruded Nafion - the first sorption run

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

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

The aim of proposed project is the development of ionic-liquid containing membranes for the separation of volatile organic compounds and pollutants from flue gases. Also development of the model, which allows testing hypotheses about non-constancy of membrane characteristics along its surface and its possible dependency on various parameters, is part of the project. By comparison of experimental data with the model will also be validated. The validated model will be then used for the supported room temperature ionic liquid membrane optimization of its operating parameters and geometry. [Refs. 10, 13]

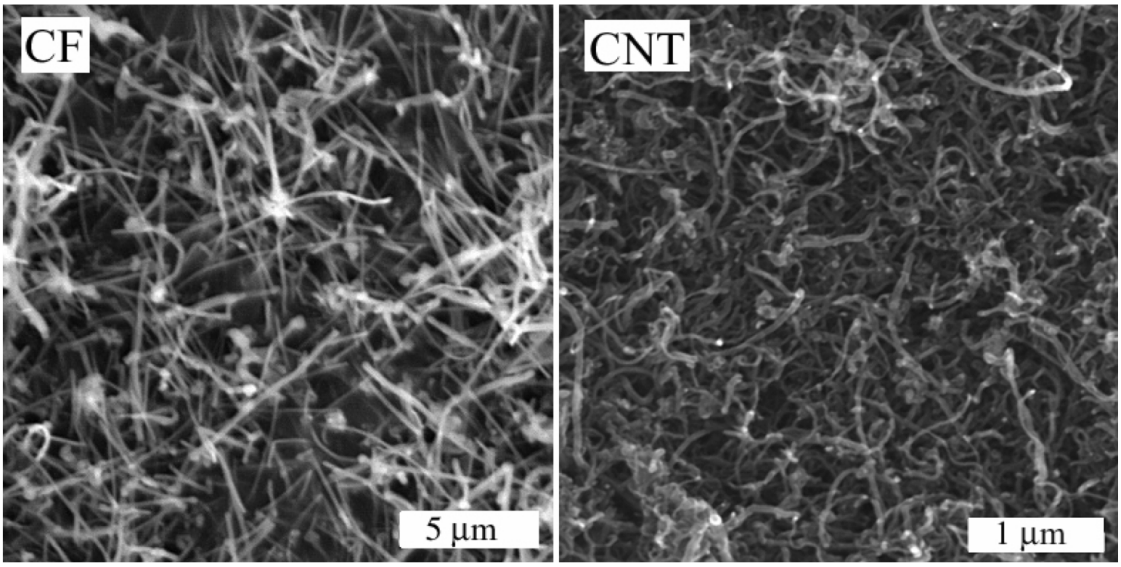


Comparison of permeabilities (a) and corresponding vapour removal (b) for hexane (Δ) and isooctane (□) at 35°C (lines are plotted to guide the eye)

Separation of polar and non-polar gasses by membrane processes

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

The use of upgraded biogas is considered as one of the most efficient means of utilizing renewable and sustainable energy. Replacing of conventional methods by low-costs, membrane separations is therefore of a great interest. The aim of this project is to develop innovative highly selective membranes with improved performance for efficient biogas upgrading. Two parallel approaches of unique membrane preparation will be used: (i) the supported ionic liquid membranes and (ii) the water-swollen thin film composite membranes. A complementary and multidisciplinary approach to laboratory experiments for gas separation can be provided by modeling, which can pre-select the best ionic liquid and safe a lot of experimental work. The aspect connected with designing, preparation, characterization and evaluation of membranes for efficient biogas upgrading will be addressed to achieve the most successful membrane. The major issues are achieving high selectivity towards the target gas (methane) and testing this new separation method also for other polar gasses. [Refs. 7, 15, 19]



Scanning electron microscopy analyses of used fillers: carbon fibers (CFs) and carbon nanotubes (CNTs) [Ref. 15]

Permeation of condensable gases trough asymmetric membranes

(J. Řezníčková, 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. The experimental data will improve our understanding of the 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. The 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. Uchytil, uchytil@icpf.cas.cz; joint project with Department of Occupational Safety and Health, Chung Medical University, Taiwan, supported by CAS, PPP project, project No. MOST/14/02)

A nano-network TiO_2 intermediate layer was synthesized via a sol-gel method for fabricating a sandwich carbon molecular sieving (CMS) membrane with high separation performances for the H_2/CH_4 and H_2/CO_2 gas pairs. The effects of the TiO_2 nano-network preparation variables, including acid catalyst amounts and number of coats, on the structure of alumina support were evaluated. By adjusting the hydrolysis-condensation rate of the titania precursor using acid catalysts, control of the pore structure and roughness of TiO_2/Al_2O_3 composite supports could be achieved. Incorporation of a TiO_2 intermediate layer can also improve adhesion between the CMS layer and the support. Three techniques (field emission-scanning electron microscopy line scanning, Fourier transform infrared spectroscopy, and contact angle) were used to determine the individual contributions from mechanical

interlocking, from chemical bonding, and from the adsorption to adhesion mechanism. The sandwich CMS membrane follows the molecular sieving mechanism and exhibits the best values of 725.9, 8.3, and 87.9 for H_2/CH_4, H_2/CO_2, and CO_2/CH_4, respectively, with a H_2 permeability of 600.7 Barrer.

Applications of liquid-liquid extraction in recovery of precious and special metals

(A. Heyberger, heyberger@icpf.cas.cz; joint project with University of KwaZulu-Natal, Durban, Republic of South Africa)

On the basis of pilot plant experiments applied in recycling of luminophores from waste compact fluorescent light bulbs design of extraction unit with counter-current vibrating plate extractor (VPE) was proposed. Unit was constructed and operated at cooperating University. The pilot plant unit was designed universally for further adaptation to other similar hydro-metallurgical processes, e.g. recovery and purification of individual fractions of lanthanides from permanent magnets.

Pilot plant unit for recovery of luminophores in Durban

Research and development of new products for complex plant protection

(M. Sajfrtová, sajfrtova@icpf.cas.cz; joint project with Matoušek CZ a.s. and Crop Research Institute; supported by TACR, project No. TA01010578)

Supercritical extracts and hydrodistillates from tropical plants supplied from South Africa were produced to develop new protective preparations for eco-agriculture. The relationship extraction conditions - extract composition - pesticidal activity of extracts was examined for the supercritical fluid extraction and hydrodistillation and the optimal method and its conditions were determined for different effects against as the acute mortality, chronic mortality, antifeedance, and others. Several fractionation methods for dividing of the extract and increasing the concentration of different groups of active substances have been tested in laboratory scale [Ref. 14]. Mathematical description for scale up based on experiments conducted in laboratory and pilot plant apparatus was derived for the eucalyptus extraction.

Schema of development of new protective preparations for eco-agriculture

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

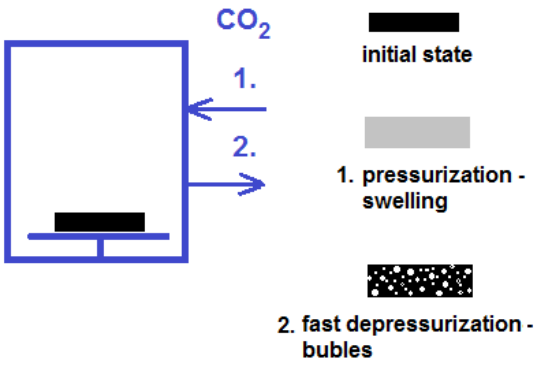
(M. Sajfrtová, sajfrtova@icpf.cas.cz; joint project with Nanotechnology Centre, VSB-TU Ostrava and X-ray group of Faculty of Mathematics and Physics, CU, Prague; supported by GACR, project No. GA14-23274S)

The project deals with investigation of preparation of various macroscopic forms (powders, thin films, monoliths) of nanostructured materials based on TiO_2, CeO_2, ZrO_2 and ZnO using sol-gel processes, thermal hydrolysis and extraction techniques developed for purpose of oxides purification and direct crystallization. Besides extraction by pressurized water and/or by super/subcritical methanol, also extraction by supercritical carbon dioxide with modifier/s is tested and optimized in a broad range of experimental conditions. The influence of individual types of extraction approaches and experimental conditions on microstructure is examined.

Morphology evolution of nano- and micro-cellular polymeric foams

(H. Sovová, 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)

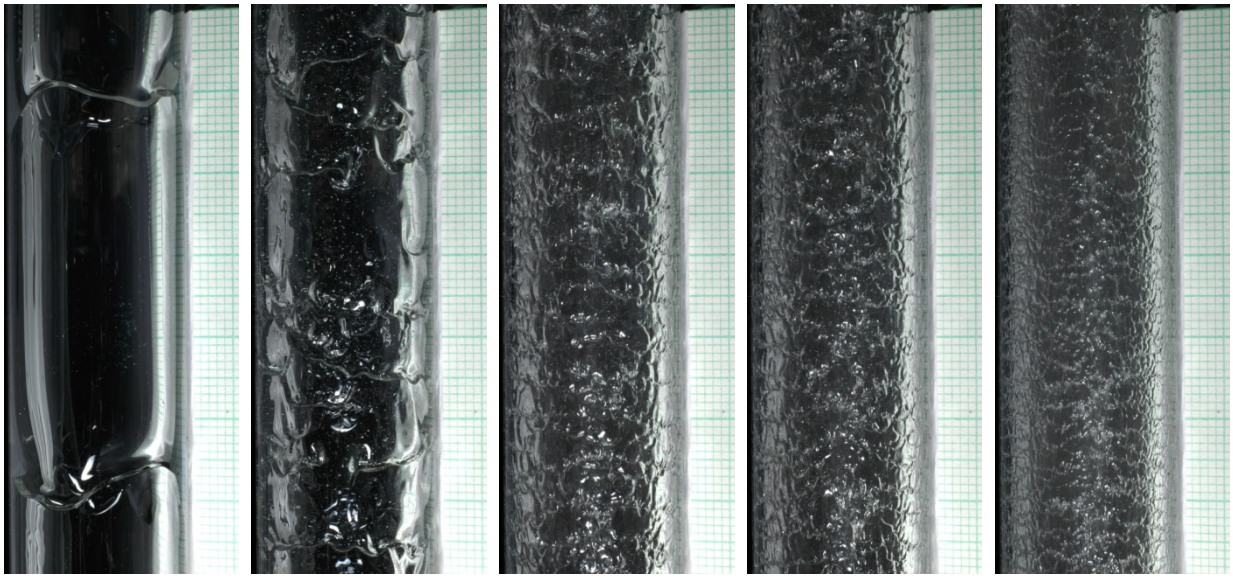
The project is aimed at first-principles understanding of early stages of polymer foam evolution including nucleation or phase separation by spinodal decomposition and coalescence of cells. Experimental methods include, among others, the foaming of polystyrene with supercritical CO_2. Based on the improved understanding of nucleation and coalescence, the methods of nano-/micro-cellular foam preparation will be assessed and optimized.



Pressure drop during the annular gas-liquid flow

(J. Křišťál, kristal@icpf.cas.cz; research contract with Procter&Gamble)

This project followed our cooperation with Procter&Gamble established during previous EU projects (IMPULSE, F^3 Factory) and research contracts. The objective of the project was a hydrodynamic study of annular gas-liquid flow. Range of operation of the existing experimental unit was extended to accommodate the reactors with different inner diameter. Wide range of experiments was carried out in order to correlate a pressure drop and a flow regime with gas and liquid velocities.



v_G = 6.4 m/s v_G = 19.1 m/s v_G = 50.6 m/s v_G = 63.3 m/s v_G = 96.1 m/s

Flow regime visualization of annular gas-liquid flow

Consulting and support on microProcessing capabilities

(J. Křišťál, kristal@icpf.cas.cz; research contract with Procter&Gamble)

Procter&Gamble was interested in our know-how accumulated about the microreactor technology application for process intensification. The work consisted in an efficient information exchange and support in various fields of microprocessing.

Application of microreactors for gas phase catalytic reactions

(P. Stavárek, stavarek@icpf.cas.cz; research contract with Momentive Specialty Chemicals a.s.)

The feasibility of application of microreactors was assessed and evaluated for the gas phase catalytic oxidation reaction. Based on this feasibility study, the preliminary design of an experimental laboratory apparatus was prepared. The work followed with a detailed design of a laboratory microreactor and the whole apparatus (under construction now) to create a basis for future laboratory testing, which will enable even widening of our current cooperation.

Development of advanced separation methods for liquid-liquid systems

(J. Křišťál, kristal@icpf.cas.cz; research contract with: Modelarna LIAZ s. r. o.)

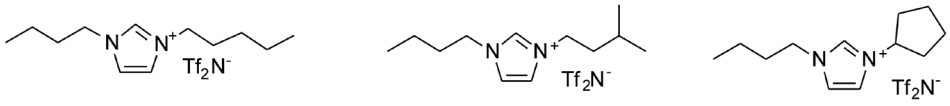
The objective of this project was the development of advanced separation methods for mixtures of miscible liquids and its experimental validation. For this purpose a laboratory apparatus was constructed and a wide range of tests were performed. The collected results provided a basis for a common patent application. [Ref. 3]

Branched and cyclic alkyl groups in imidazolium-based ionic liquids: Molecular organization and physico-chemical properties

(M. Bendová, bendova@icpf.cas.cz; joint project with Equipe Thermodynamique des Intéractions Moléculaires, Institut de Chimie de Clermont-Ferrand, Université Blaise Pascal, France)

Novel $[C_4C_5Im][Tf_2N]$ ionic liquids with a variation in structure of a C5 alkyl group were synthesized for the first time and characterized as to their thermophysical and transport properties. Branching of the alkyl chain has no influence on the ionic liquid density. Isopentyl

and cyclopentyl groups increase the ionic liquid viscosity. Surprisingly the cyclopentyl substituted ionic liquid shows the highest conductivity in spite of its high viscosity. This however is in agreement with a higher degree of its ion dissociation found from the experimental self-diffusion coefficients. [Refs. 1, 9]

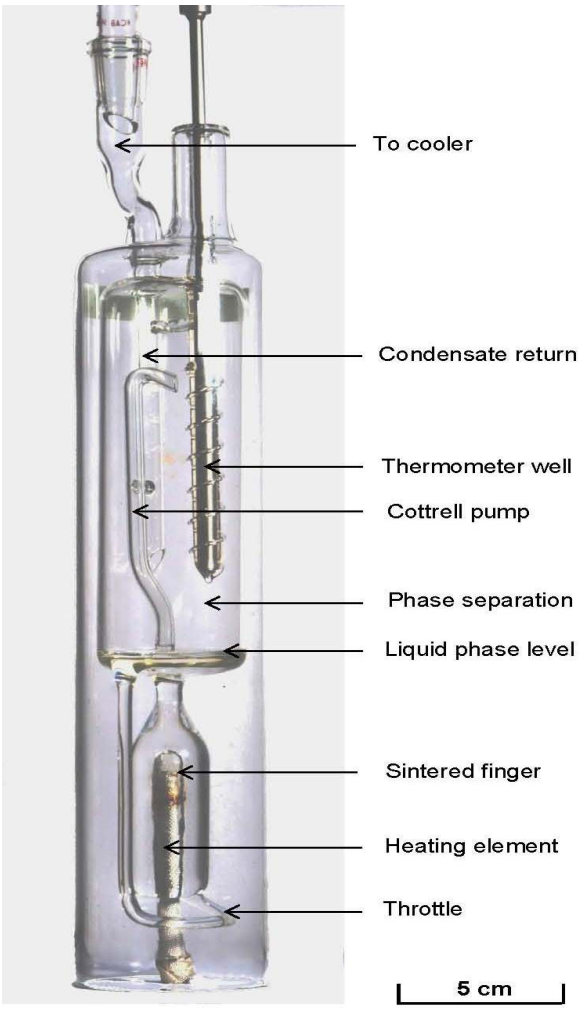


Structures of the 1-alkyl-3-butylimidazolium bis(trifluoromethylsulfonyl)imide ionic liquids

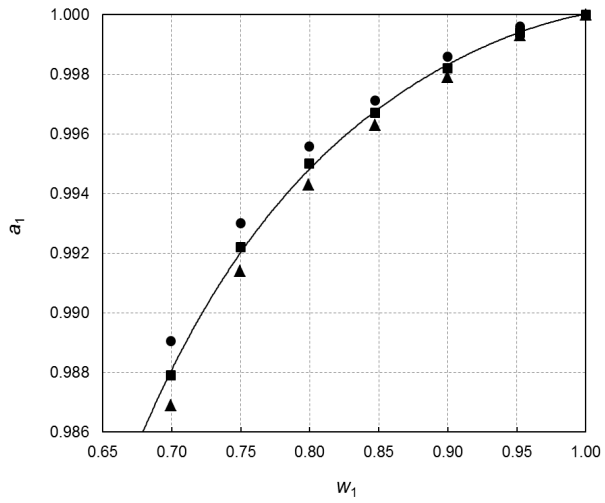
Vapour-liquid equilibrium in systems containing polymers - measurement and data processing

(I. Wichterle, wichterle@icpf.cas.cz; within the project "Group of phase equilibria" supported by ICPF)

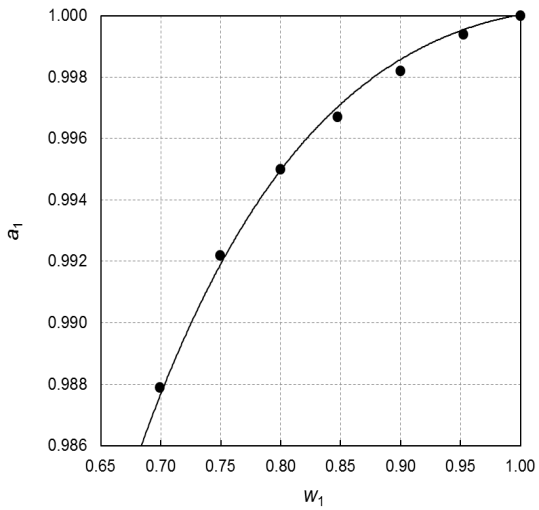
Vapour-liquid equilibria have been determined in systems composed of poly-(acrylic acid) with water, and poly(methyl methacrylate) with 2-butanone by ebullio-metric (total pressure measurement) method. Ebulliometer has been redesigned (see the Figure) and experimental procedure has been upgraded. Experiments 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. The polystyrene + toluene system has been measured, too (paper published in 2015). Results were published in journal [Ref. 11] and at two international confe-rences.



Microebulliometer – new design



Activity a1 of 2-butanone in PMMA as a function of 2-butanone mass fraction w1. Experimental data at (•) 333.15 K, (■) 343.15 K and (▲) 353.15 K. Solid line represents predicted activities at 343.15 K using the UNIQUAC-FV parameters estimated using all data within temperature range 333.15–353.15 K



Activity a1 of 2-butanone in PMMA as a function of 2-butanone mass fraction w1. Experimental data at (•) 343.15 K. Solid line represents predicted activities using UNIFAC-vdw-FV model

International co-operations

CSIR of Pretoria and Johannesburg, Republic of South Africa: Extraction of essential oils from plant raw materials

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

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, Germany, Max-Planck-Institut für Dynamikkomplexer technischer Systeme. Magdeburg: 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

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

Technical University of Lisbon, Portugal: Supercritical extraction of biological compounds from aromatic plants
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_2 medium
University of KwaZulu-Natal, Republic of South Africa: Liquid-liquid extraction processes with fluorinated hydrocarbons, recovery of luminophores

Visitors

Prof. A. Seidel-Morgenstern, University of Magdeburg, Max-Planck Institute, Germany
Dr. Thomas Loimer, Technische Universität Wien
Prof. H-H. Tseng, Chung Medical University, Taichung, Taiwan
Dr. S. Kononovou, Institute of Macromolecular Compounds, Russian Academy of Sciences, Saint-Petersburg, Russia

Teaching

J. Hanika: UCT, Faculty of Chemical Technology, postgradual course “Multiphase Reactors”
J. Hanika: UCT, Faculty of Chemical Technology, course “Pharmaceutical Engineering”
H. Sovová: TU Darmstadt, Life Long Intensive Program “Process Intensification by High Pressure Technologies – Actual Strategies for Energy and Resources Conservation”
M. Bendová: UCT, Faculty of Chemical Engineering, postgraduate course "Physical Chemistry for Technological Practice"
Z. Sedláková: UJEP, Faculty of Science, courses "Membrane Separations", "Basic of Chemistry", "Laboratory of Analytical Chemistry"

Publications

Original papers

[1] Andresová A., Storch J., Traïkia M., Wagner Z., Bendová M., Husson P.: Branched and Cyclic Alkyl Groups in Imidazolium-Based Ionic Liquids: Molecular Organization and Physico-chemical Properties. *Fluid Phase Equilib.* 371, 41-49 (2014).

[2] Dolejš P., Poštulka V., Sedláková Z., Jandová V., Vejražka J., Esposito E., Jansen J.C., Izák P.: Simultaneous Hydrogen Sulphide and Carbon Dioxide Removal from Biogas by Water-Swollen Reverse Osmosis Membrane. *Sep. Purif. Technol.* 131, 108–116 (2014).

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[11] Pavlíček J., Bogdanić G., Wichterle I.: Vapour–Liquid Equilibria in the Poly(methyl methacrylate) + 2-Butanone System Containing Lower Concentrations of Solute at Normal or Reduced Pressures. *Chem. Biochem. Eng. Q.* 28(4), 447-450 (2014).

[12] Randová A., Bartovská L., Friess K., Hovorka Š., Izák P.: Fundamental Study of Sorption of Pure Liquids and Liquid Mixtures into Polymeric Membrane. *Eur. Polymer J.* 61, 64-71 (2014).

[13] Randová A., Bartovská L., Izák P., Friess K.: A New Prediction Method for Organic Liquids Sorption into Polymers. *J. Membrane Sci.* 475, 545-551 (2014).

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Books and monographs

[18] Bendová M.: Eduard Hála. 16pp., Nakladatelství Academia, SSČ AV ČR, v. v. i., Praha 2014.

Chapters in books

[19] Bobák M., Dolejš P., Izák P., Sedláková Z.: Kapitola 5: Průmyslové aplikace dělení plynů a par. Chapter 5: Industry Application of Separation Gas and Vapors. In: *Membránové dělení plynů a par*. (Šípek, M., Ed.), pp. 103-131, Vydavatelství VŠCHT, Praha 2014.

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