Department of Separation Processes

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Fields of research

- Research and development of electrochemical bipolar microreactor for alkoxylation
- Hydrodynamics of two phase flow in narrow channel
- Sulfur dioxide oxidation, sulfation and sulfonation
- Integrated multiscale process units with locally structured elements
- Liquid-liquid extraction of tall oil from wastewaters of paper industry
- Fluorinated hydrocarbons as potential solvents in liquid-liquid extraction processes
- Supercritical fluid extraction of biologically active substances
- Enzymatic reactions in supercritical CO_2
- Mass transport through ionic liquid membranes
- Membrane separation of methane and CO_2
- Separation of racemic mixtures
- Separation of gasoline vapours from air by supported ionic liquids membranes

Applied research

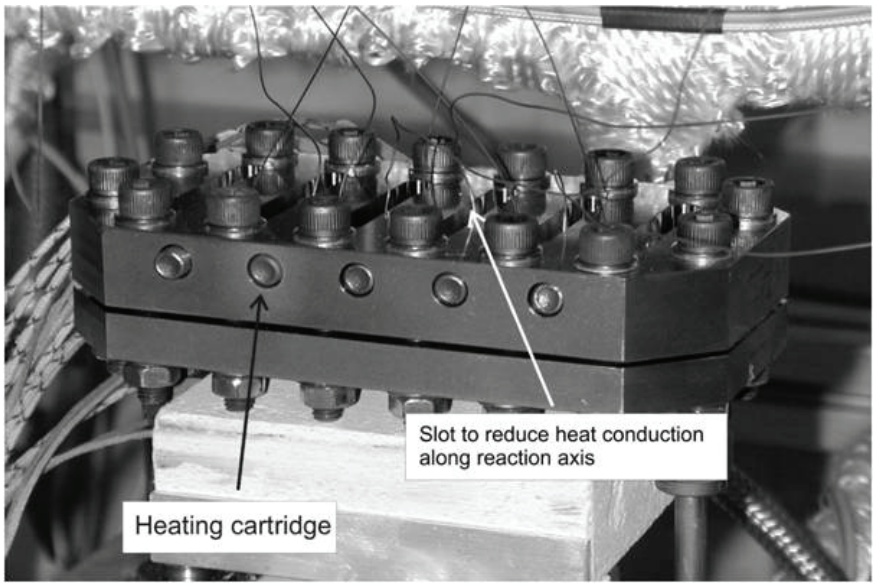
- Research and development of new methods of emulsification using microtechnology
- Hydrodynamic characterization of micromixers
- Extraction and production of plastic modifiers for production of tyres
- Liquid extraction of luminophores, recycling of Y and Eu
- Liquid and supercritical fluid extraction and refining of plant extracts
- Purification of biogas by supported ionic liquid membrane
- Reclaiming of phytosterols and other valuable compounds from tall soap/oil

Research projects

Flexible, fast and future production processes (F^3 Factory)

(V. Jiřičný, 7th FP collaborative large integrated project, Theme NMP-2008-3.2-1, supported by EU under Contract No. CP-IP 228867-2 F^3 Factory)

The goals of the projects are in improvements of EU chemical industry's competitive position by development modular continuous plant (F^3 Plant) which combines world scale continuous plant efficiency, consistency and scalability with the versatility of batch operation. Project is deliver new production mode based on plug-and-play modular production technology and holistic process design methodology applying intensification concepts and innovative decision tools. ICPF in cooperation with Procter&Gamble (leader of subtask) and KIT Karlsruhe are involved in research and developments of sulfur dioxide oxidation, sulfation and sulfonation. The mathematical model for sulfur dioxide oxidation has been developed in ICPF. Model was validated with experimental data conducted on microreactor developed and manufactured by KIT Karlsruhe. Simulations with the model were used for development and design of new microreactor. [Refs. 1, 25, 37, 62, 63, 70].

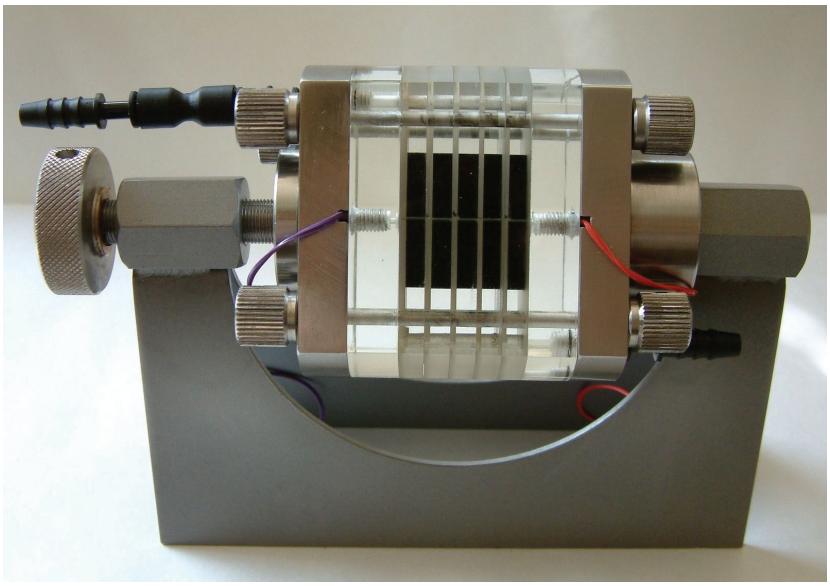


Photograph of the lab scale reactor for SO_2 oxidation with slotted holes in the cover plate and holes for the heat cartridges designed and manufactured in KIT

Chemical degradation of polybrominated diphenyl ethers

(V. Jiřičný, supported by GACR, project No. GA104/09/0880)

Polybrominated diphenyl ethers (PBDEs) are widely used as flame retardants, mainly for polymers and textiles. PBDEs have an ideal property for these matrices because they are decomposed at temperature 50 °C lower than point of flammability of matrix. PBDEs are pollutants of environment. They are lipophilic, potential carcinogenic and neurotoxic compounds, which accumulate in environment and they can migrate by food chain into human organisms. The various methods of degradation of PBDEs have been experimentally verified. The best and original results have been conducted with electrochemical micro-reactor. The reliable GC analytical method has been developed. [Refs. 26, 43, 46, 67-69].



Filter-press type bipolar electrochemical microreactor designed and manufactured in ICPF

Research and developments of new methods of emulsification using microtechnology

(J. Křišťál, contract with Procter&Gamble, ICPF Contract No. 171020)

The contract is focused on the development of new methods for production of very stable emulsions. Microtechnology equipments are used in this research to reach the desired goals. Results are published in confidential Procter&Gamble research reports.

Research and developments of various microapparatus characteristics

(J. Křišťál, contract with Procter&Gamble, ICPF Contract No. 171050)

The contract deals with experimental measurement of various microapparatuses (preferably mixers) and determination of their hydrodynamic characteristics with respect to various physical-chemical properties of selected liquids. The collected data and developed methodology of micromixer selection are forming databasis for design and development of new chemical processes. Results are published in confidential Procter&Gamble research reports.

Integrated multiscale process units with locally structured elements

(J. Hanika, IMPULSE, 6FP integrated project)

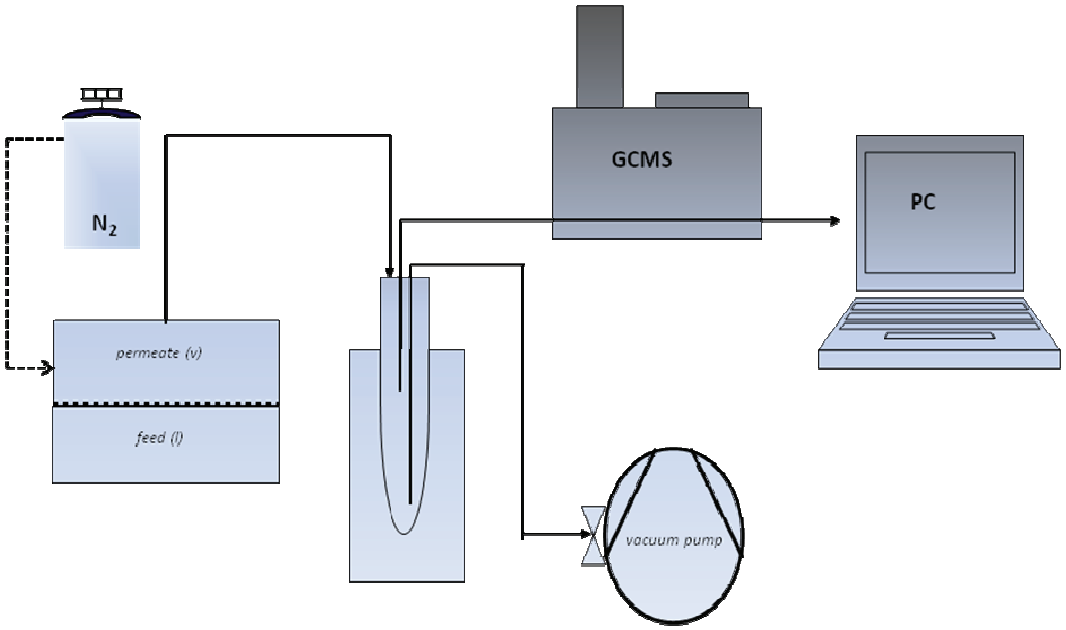
The objective of project is effective, targeted integration of innovative process equipment such as microreactors, heat exchangers, thin-film devices and other micro components to attain radical performance enhancement for whole process systems in chemical production. We are involved in the application research of electroorganic synthesis in electrochemical micro-reactor. Bipolar electrochemical microreactors proved advantages of microtechnology. While conversion and selectivity of the process are comparable with conventional process, the main profit of applied microtechnology is in less expensive separation of the product from reaction mixture. [Refs. 3-5, 27, 38-40, 64-65]

Ionic membranes for selective separation of liquid mixtures by pervaporation

(P. Izák, joint project with ICT, supported by GACR, grant No. GA104/08/0600)

In this project, we evaluated the effect of ionic liquid to the equilibrium solvent (butan-1-ol) vapour sorption in PDMS and effects of the ionic liquid content on the sorption and desorption kinetics of butan-1-ol, all at 37 °C. The generalized Fick's second law, solved at

time dependent boundary conditions occurring in the apparatus, provided a satisfactory approximation of the data on sorption and desorption kinetics of butan-1-ol vapour in PDMS and in the PDMS–ionic liquid blends; the resulting values of diffusion coefficient depended on the choice of the equilibrium vapour sorption model. The significant decrease of diffusion butan-1-ol diffusion coefficients at equilibrium activities above approx. 0.6 indicate anomalous sorption and desorption of butan-1-ol at higher concentrations in the membranes investigated. The increased content of the benzyl-3-butylimidazolium tetrafluoroborate ionic liquid in the PDMS based membranes positively influenced the equilibrium concentration of butan-1-ol in such membranes over the whole butan-1-ol vapour activity interval and, at the same time, reduced the butan-1-ol diffusion coefficients. [Refs. 7, 12-14, 18, 28, 30, 41, 42, 44, 49].

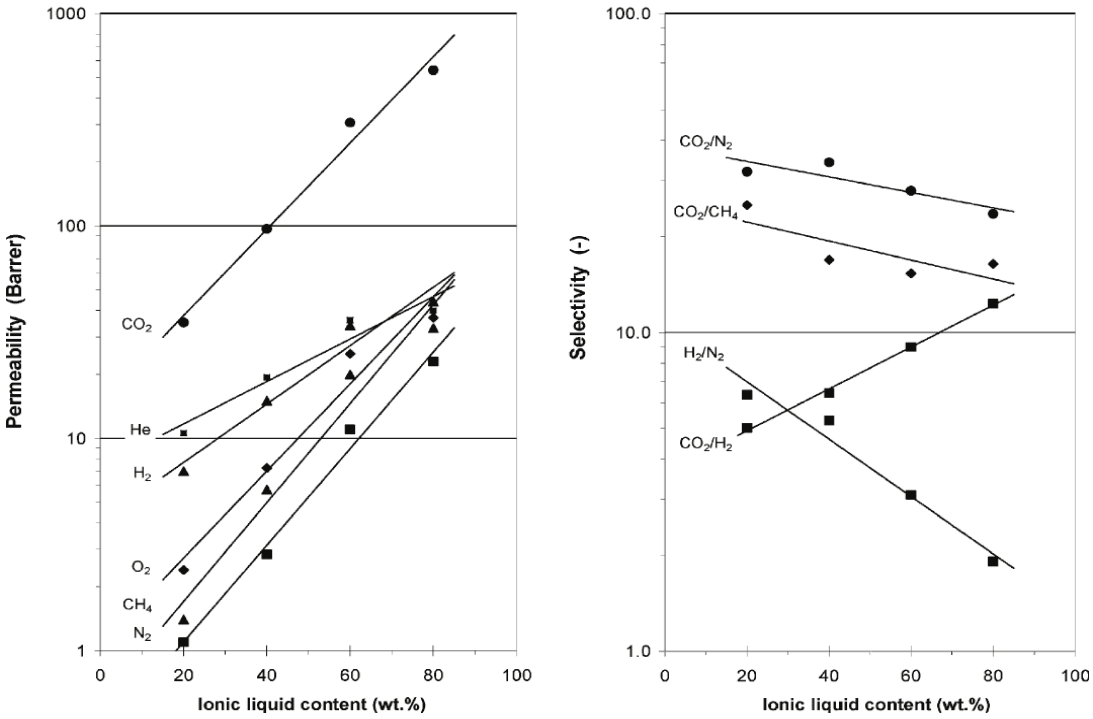


Scheme of pervaporation apparatus

Separation of volatile organic compounds from air

(P. Izák, joint project with ICT, supported by GACR, grant No. P106/10/1194)

Outflow of vapours of volatile organic compounds (VOCs) leaking from gasoline during its storage, transportation and handling is a serious ecological problem due to the toxicity of these compounds (e.g. some of them can cause a cancer). It is also an economical problem connected with the loss of a valuable industrial product. Although amounts of gasoline vapours lost during common operations like handling and storage may seem negligible, they reach hundreds of tons per year. All above mentioned facts are the reason, why the separation of these compounds from air and their recycling is critically important. The aim of this project is finding of a convenient separation method of VOCs from the air based on new approach to the problem (using of room temperature ionic liquid (RTIL) or modified polymer membrane). [Refs. 6, 29].

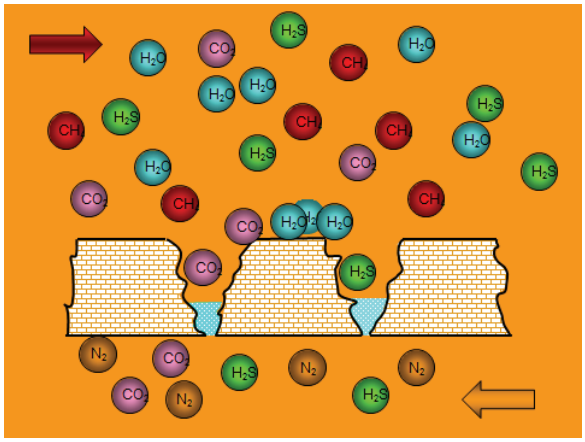


Permeability of six permanent gases as a function of the IL content (left) and corresponding selectivity between selected gas pairs (right)

Purification of biogas by supported ionic liquid membrane

(P. Izák, joint project with Česká hlava s.r.o., supported by MIT, project No. ER-TI1/245)

A new method for raw biogas purification from impurities and carbon dioxide separation by "condensing liquid membrane" was verified. The hydrophilic porous membrane helped to form under condensing condition (below dew point of the raw biogas feed) a very thin selective water layer. Major difference in permeability of certain raw biogas components (carbon dioxide, hydrogen sulfide) and methane through water layer is responsible for high upgrading of raw biogas to biomethane quality. Condensing liquid membrane is also environmentally friendly because it produces no toxic waste and the separation may become a part of waste water treatment. It represents a new approach to production of biofuel from biogas. [Refs. 6, 11, 19, 33, 34, 47, 48].

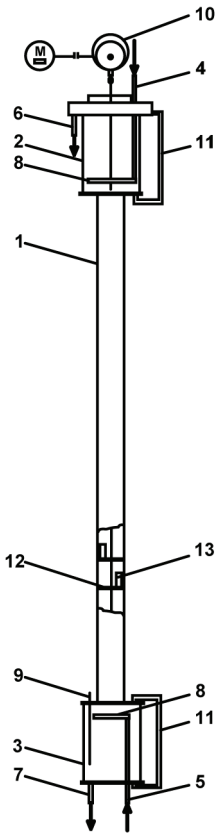


Impurities and CO_2 in raw biogas are separated by a "condensing-liquid membrane", based on the different solubility of components in a very thin layer in a porous membrane

Reclaiming of phytosterols and other valuable compounds from tall soap/oil

(M. Rousková, A. Heyberger, joint project with Technological Park in Chomutov and Institute of Systems Biology and Ecology ASCR)

The aim of the project is to study the tall soap composition and to develop methods of extractive separation of the valuable compounds. Besides of working out the necessary analytical methods, the extraction equilibria in systems with various solvents were measured, and the separation processes were simulated in a laboratory vibrating plate extraction column. A novel extraction processes and equipment have been designed for recovering phytosterols and unsaturated fatty acids from tall soap. [Refs. 15, 21-23, 31, 32, 50, 54]



Symbols used

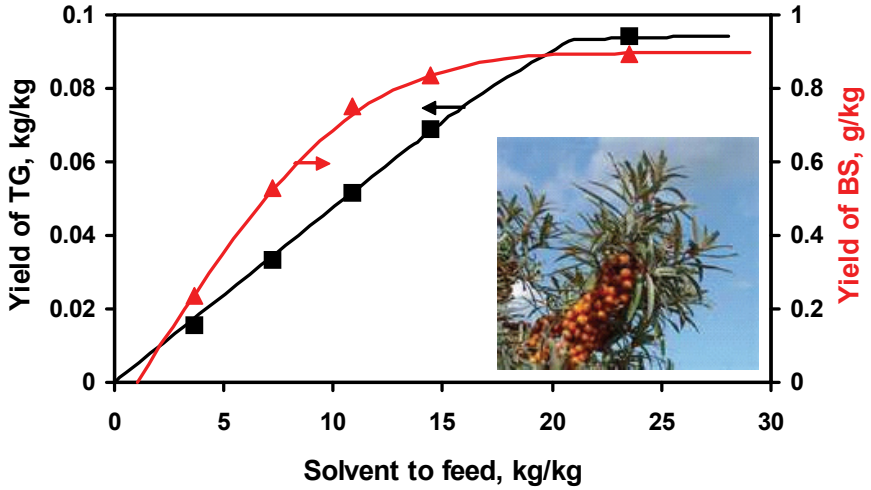
1 column coat
2 upper settler
3 lower settler
4 inlet of heavier phase (soln. of tall soap)
5 inlet of lighter phase (solvent)
6 outlet of extract
7 outlet of raffinate
8 liquid distributor
9 control of interphase boundary
10 eccentric drive
11 liquid level control
12 perforated plates
13 downcomers for continuous phase

Scheme of the vibrating plate extractor (VPE)

Supramolecular materials based on natural phytosterols for applications in biology

(H. Sovová, joint project with IOCB, ICT, and Chemispol s.r.o., supported by MEYS, project No. 2B06024)

Supercritical fluid extraction of β-sitosterol as a component of sea buckthorn and sunflower oils was studied in detail. The experimental data on the effect of extraction conditions on the yield and extract composition were interpreted in relation to phase equilibrium. This approach was verified on literature data on the extraction of carotenoids with oil. A sitosterol-enriched fraction was obtained using two separators. An alternative resource of β-sitosterol was stinging nettle root. The potential of enzymatic reactions of vegetable oils in supercritical carbon dioxide to enhance the concentration of minor components was studied. 20-hydroxyecdysone enrichment up to 12 wt. % in a fraction of *Leuzea carthamoides* CO_2 extract was achieved using ethanol as entrainer [Refs. 16, 17, 52, 53, 55, 56, 61].

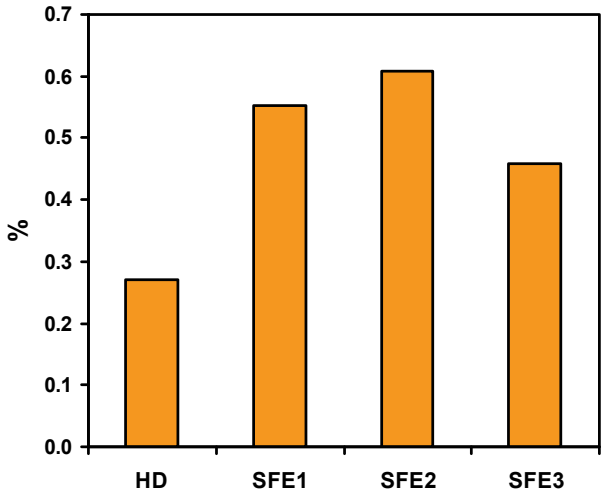


Extraction of oil from sea buckthorn seeds with CO_2 at 60 °C and 28 MPa: β-sitosterol is extracted faster than triglycerides, the major component

Optimization of supercritical fluid extraction for maximal yield of biologically active substances from plants

(H. Sovová, joint project with Research Institute of Plant Production and Agra Group, supported by MEYS, project No. 2B06049)

The kinetics of supercritical fluid extraction of different essential oils was described with a phenomenological model. Strong insecticidal activities of *Tanacetum parthenium* extracts were observed and quantified in dependence on extraction conditions. Applications for a patent on *Ruta graveolens* based means for plant protection against pests were submitted. The operational conditions of savory extraction with respect to thymoquinone and other volatile oil components in the extract were optimized. The composition and insecticidal activity of extracts from four *Lamiaceae* plants were compared with the aim to find the active extract components [Refs. 2, 9, 35, 36, 51, 56, 57].

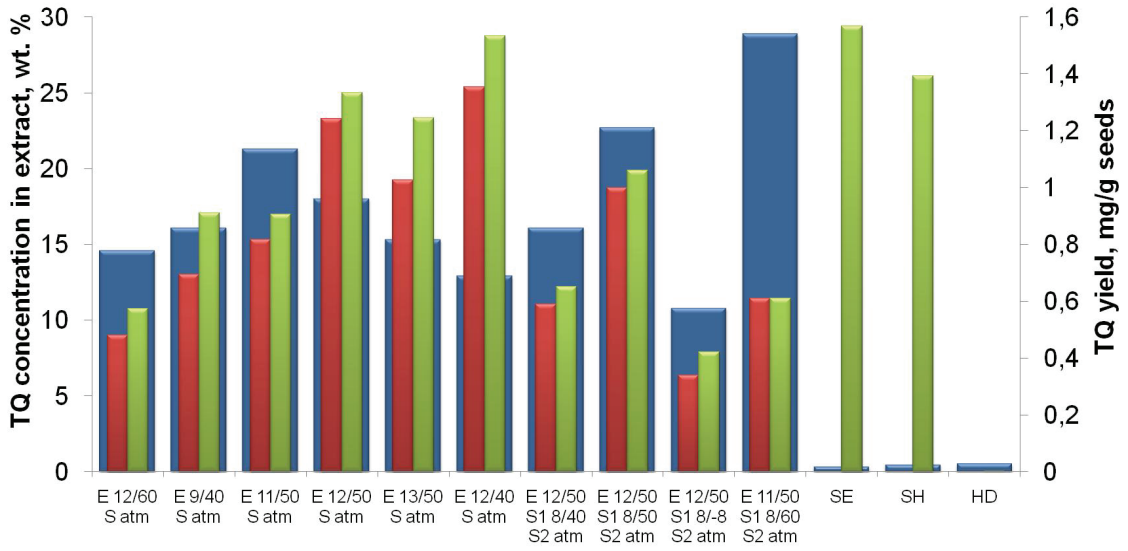


The yield of identified essential oil components (%, w/w) isolated from *Tanacetum parthenium* by hydrodistillation (HD) and supercritical CO_2 extraction (SFE1-3)

Determination of biological activity and chemical composition of selected tropical and subtropical Ranunculaceae species

(H. Sovová, joint project with Czech University of Life Sciences, and IOCB, supported by GACR, grant No. GA525/08/1179)

The research was focused on the enhancement of thymoquinone (TQ) concentration in CO_2 extracts of black cumin (*Nigella sativa* L.) seeds using two separators. With respect the TQ yield and its concentration in extract, the best results were obtained at 12 MPa and 50 °C in the extractor and 8 MPa and 50 °C in the first separator. The yield was 1.0 mg TQ/g seeds and 22.7 wt. % TQ in the extract [Ref. 58].



Supercritical fluid extraction of thymoquinone from *Nigella sativa* seeds (E) is more efficient and selective than Soxhlet extraction with ethanol (SE) or hexane (SH) and than hydrodistillation (HD): blue: TQ concentration in 1st fr. of extract, red: yield of TQ in 1st fr., green: total yield of TQ

Study of polymeric membrane swelling and make use of this effect for increasing its permeability

(P. Uchytil, joint project with IMC, supported by the GACR, Grant No. GA104/09/1165)

The relation between polymeric structure of pervaporation membranes on the basis of poly-γ-benzyl-L-glutamate and their transport properties was studied with the group of Dr. S. Kononova (IMC). The pervaporation of binary mixture toluene and heptane was measured. High separation was achieved. Paper with the results was accepted in Crystallography Reports [Ref. 8, 45].

Preparation of dense homogeneous polymeric membranes and study on their gas permeation properties

(P. Uchytil, joint project with National Chung Hsing University, Taiwan, supported by ASCR and by National Science Council of Taiwan, project No. P106/10/J038)

The project is focused on the study of carbon dioxide and methane transport in a poly(vinylidene fluoride-co-hexafluoropropylene) polymeric membrane (Viton) containing ionic liquid. Two ionic liquids 1-hexyl-3-methylimidazolium bis (trifluoromethylsulfonyl) imide ([HMIM][TFSI]) and 1-ethyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide ([EMIM][TFSI]) were used for preparation of ionic liquid membranes. The transport properties of these membranes were tested using dynamic permeation method [Ref. 66].

Mass transport during membrane permeation and pervaporation

(P. Uchytil, joint project with University of Colorado, Boulder, USA, supported by MEYS, KONTAKT project No. ME 889)

A new apparatus for gas permeability, diffusivity and solubility assessing in dense polymeric membranes was constructed and his function was verified. Toluene transport model based on data from the new permeation apparatus was proposed. The theoretical and experimental study of butane and isobutane vapour flow through porous Vycor glass membrane with pore size between 20 and 200 nm under different conditions was performed. The experiment results and predicted results were compared with an adiabatic model [Refs. 20, 10, 24, 59, 60].

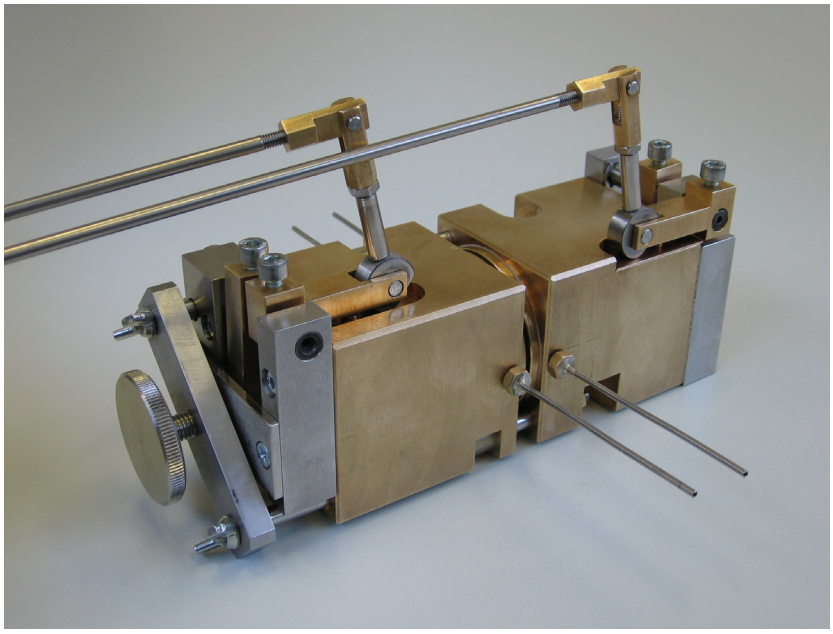


Photo of a new type of the permeation cell for determination of transport parameters in polymeric membranes

International co-operations

CNRS Toulouse, France: Characterization of two phase flow in microchannels
CNRS Lyon: Hydrogenation in falling film microreactor
CSIR of Pretoria and Johannesburg, Republic of South Africa: Extraction of essential oils from plant raw materials
Institute of Chemical Engineering, Sofia, Bulgarian AS: High-pressure phase equilibria
Institute of Macromolecules, St. Petersburg, Russian Academy of Science, Russia: Membrane separation
Institute on Membrane Technology, ITM-CNR, Italy: Novel composite membranes containing ionic liquid and selected polymers for specific gas/gas, gas/vapour and vapour/vapour separations
KIT Karlsruhe: Sulfur dioxide oxidation using heterogeneous catalytic microreactor
National Chung Hsing University, Taiwan: Preparation of Dense Homogeneous Polymeric Membranes and Study on Their Gas Permeation Properties
Otto von Guericke University of Magdeburg, Magdeburg, Germany: Mass transport through porous membranes

Procter&Gamble, Belgium: Research and development of new methods of emulsification using microtechnology
Slovak University of Technology in Bratislava, Slovakia: Processing of tall soap/oil extraction products
Technische Universität Wien, Institut für Strömungslehre und Wärmeübertragung, Austria: Flow of saturated vapours 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 vapour 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

Visits abroad

A. Heyberger: CSIR of Johannesburg, University of KwaZulu-Natal, Durban, South Africa (1 month)
J. Křišťál: Procter & Gamble, Brussels, Belgium (3 months)

Visitors

J. Aubin, CNRS Toulouse, France
M. Botha, University of KwaZulu-Natal, Durban, Republic of South Africa
A. Bucić-Kojić, Faculty of Food Technology, Osijek University, Croatia
M. Čársky, University of KwaZulu-Natal, Durban, Republic of South Africa
A. Palavra, Technical University of Lisbon, Portugal
S. Tigri, ENSIASET, Toulouse, France

Teaching

P. Izák: ICT, Faculty of Chemical Engineering, postgraduate course “Physical chemistry for technological practice”
J. Hanika: ICT, Faculty of Chemical Technology, postgradual course “Multiphase reactors”
J. Hanika: ICT, Faculty of Chemical Technology, course “Pharmaceutical engineering”
H. Sovová: ICT, Faculty of Chemical Engineering, postgraduate course “Properties and application of supercritical fluids”

Publications

Original papers

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[11] Poloncarzová M., Vejrazka J., Veselý V., Izák P.: Effective Purification of Biogas by Condensing-Liquid Membrane. (Eng) Angew. Chem. Int. Ed. 50(3) 669-671 (2011).
[12] Randová A., Bartovská L., Hovorka Š., Bartovský T., Izák P., Poloncarzová M, Friess K.: Diffusion Coefficients in Systems LDPE + Cyclohexane and LDPE + Benzene. (Eng) E-Polymers 068 (2010).
[13] Randová A., Bartovská L., Hovorka Š., Izák P., Poloncarzová M., Bartovský T.: Low-Density Polyethylene in Mixtures of Hexane and Benzene Derivates. (Eng) Chem. Pap. 65(5), 652-656 (2010).
[14] Randová A., Bartovská L., Hovorka S., Izák P., Friess K., Janků J.: Sorption of Binary Mixtures of Toluene + Lower Aliphatic Alcohols C_1-C_6 in Low-Density Polyethylene. (Eng) J. Appl. Polym. Sci. 119(3) 1781-1787 (2011).
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[22] Rathilal S., Čárský M., Heyberger A., Rousková M.: Difference of Hydrodynamics for a VPE with and without Mass Transfer and Effect of Agitation Level on Extent of Mass Transfer. (Eng) Sep. Purif. Technol., submitted.
[23] Rousková M., Heyberger A., Čárský M., Tříska J., Krtička M.: The Effect of pH on Liquid-Liquid Extraction Efficiency of Phytosterols from Tall Soap. (Eng) Sep. Purif. Technol., submitted.
[24] Setničková K., Wagner Z., Noble R., Uchytil P.: Semi-Empirical Model of Toluene Transport in Polyethylene Membrane Based on the Data of New Type of Apparatus for Gas Permeability, Diffusivity and Solubility Grant. (Eng) Eur. Polym. J., submitted.

Review papers

[25] Aubin J., Ferrando M., Jiřičný V.: Current Methods for Characterising Mixing and Flow in Microchannels. (Eng) Chem. Eng. Sci. 65(6), 2065-2093 (2010).

Chapters in books

[26] Evans J.W., Jiřičný V.: Chapter 16, Spouted Bed Electrochemical Reactors (Eng) In: Spouted and Spout-Fluid Beds Fundamentals and Applications, Edited by N. Epstein and J.R. Grace, Cambridge University Press 2010.
[27] Gogová Z., Hanika J., Markoš J.: Optimal Design of a Multifunctional Reactor for Catalytic Oxidation of Glucose with Fast Catalyst Deactivation. (Eng) (Brito A.V., Ed.), pp. 209-232, Intech, Vukovar 2010.
[28] Izák P., Friess K., Šípek M.: Chapter 12: Pervaporation and Permeation Taking Advantage of Ionic Liquids. (Eng) In: Handbook of Membrane Research: Properties, Performance and Applications. (Gorley, S.V., Ed.), pp. 387-402, Nova Science Publishers, New York 2010.

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Patents

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