

Department of Separation Processes

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TECHNICAL STAFF

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Fields of research

- Hydrodynamics of two phase flow in narrow channel
- Sulfur dioxide oxidation, sulfation and sulfonation
- Utilization of microreactors for enzymatic reactions
- Fluorinated hydrocarbons as potential solvents in liquid-liquid extraction processes
- Supercritical fluid extraction of biologically active substances
- Chemical and enzymatic reactions in supercritical CO₂
- Mass transport through ionic liquid membranes
- Membrane separation of methane and CO₂
- Separation of racemic mixtures
- Separation of gasoline vapors from air by supported ionic liquids membranes

Applied research

- Research and development of new methods of emulsification using microtechnology
- Hydrodynamic characterization of micromixers
- Heat and mass transfer of liquid flow in microreactors
- 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 liquid membrane

Research projects

Flexible, fast and future production processes (F³ Factory)

(V. Jiříčný, jiricny@icpf.cas.cz; 7th FP collaborative large integrated project, Theme NMP-2008-3.2-1; supported by EU under Contract No. CP-IP 228867-2 F³ Factory)

The goals of the projects are in improvements of EU chemical industry's competitive position by development modular continuous plant (F³ Plant) which combines world scale continuous plant efficiency, consistency and scalability with the versatility of batch operation. Project will 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. Simulations with the model were used for development and design of new microreactor. New pilot plant size microreactor for SO₂ oxidation has been manufactured by KIT Karlsruhe. ICPF designed and manufactured for sulfonation and sulfation new microreactor and conducted hydrodynamic tests. [Refs. 14, 35, 36]

Research and developments of new methods of emulsification using microtechnology

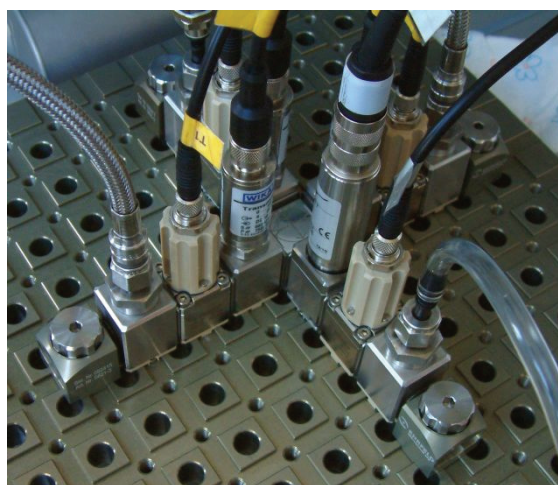
(J. Křišťál, kristal@icpf.cas.cz; contract with Procter&Gamble)

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 published in confidential Procter&Gamble research reports. [Ref. 27]

Research and developments of various microapparatus characteristics

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

The contract deals with experimental measurement of various microapparatuses (preferably mixers) and determination of their hydrodynamic characteristics with respect to various physical-chemical properties selected liquids. The collected data and developed methodology of micromixer selection will form databasis for design and development of new chemical processes. Results published in confidential Procter&Gamble research reports. [Refs. 22, 23, 45, 46]

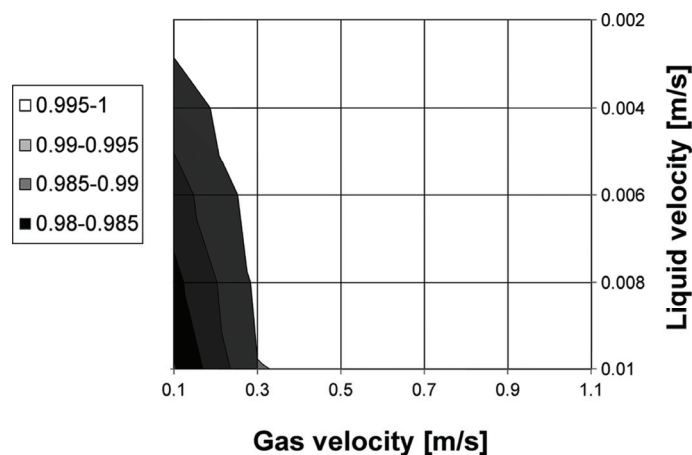


Ehrfeld micro system experimental set up for testing of micro mixers

Minimum energy dissipation under cocurrent flow in packed beds

(V. Staněk, stanek@icpf.cas.cz; supported by the GACR, grant No. 104/09/0880)

Functional analysis of mass balances on gas and liquid, and pressure drop equations describing the cocurrent flow in packed beds has yielded two criteria. The positive values of these criteria has been shown sufficient to achieve energy savings by synchronized periodic pulsations of inlet gas and liquid velocities or by the liquid velocity only compared to the situation under the same mean steady inlet phase velocities. This, however, is not the case of pulsation by gas inlet velocity alone. [Refs. 1]

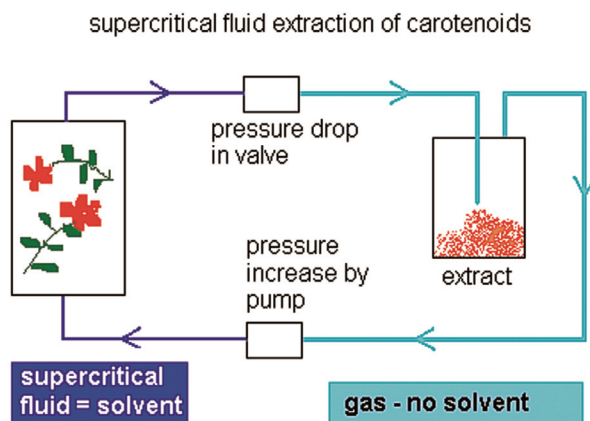


Contours of constant relative rate of energy dissipation under synchronized pulsations of liquid and gas inlet velocities for toluene/air system

Supercritical fluid extraction from vegetable materials

(H. Sovová, sovova@icpf.cas.cz; joint project with ICT Prague, Institute of Experimental Botany ASCR, Institute of Chemical Engineering of BAS, Sofia, Bulgaria; supported by MEYS, project No. 2B06024)

The sustainable use of renewable resources, complying with consumer health and environmental requirements, motivates the design, optimization, and application of green benign processes. Supercritical fluid extraction is a typical example of a novel green technology. The review offers an enumeration of extracted plant materials, discusses the mathematical modeling of the process, and advocates a choice for the appropriate model that is based on characteristic times of individual extraction steps. Finally, the attention is focused on the elements of a thermodynamic modeling framework designed to predict and model robustly and efficiently the complex phase equilibria of the systems solute+supercritical fluid. [Refs. 12, 32]



Formulation for protection of plants against insects and use thereof

(H. Sovová, sovova@icpf.cas.cz; joint project with ICT Prague, Crop Research Institute, Agra Group, a.s.; supported by MEYS, project No. 2B06049)

The solution concerns a means for the plant protection that contains a concentrate of extract from flowering aerial parts of summer savory (*Satureja hortensis* L.) in the concentration of 0.5-99.5 % wt. and at least two carriers, while at least one carrier is based on a surface active agent and/or emulsifier in the amount of 0.5-99.5 % wt. The solution concerns further the application of the insecticidal means for plant protection against insects, when the means contains at least 0.5 % wt. of the effective insecticidal substance. [Refs. 28, 30, 31, 34]

Research and development of new products for complex plant protection

(M. Sajfrtová, sajfrtova@icpf.cas.cz; joint project with Matoušek CZ a.s., Crop Research Institute; supported by TACR, project No. 01010578)

New preparations for eco-agriculture are being developed on the basis of hydrodistillates and supercritical extracts from tropical plants with high content of the biologically active substances.

Mass transport during membrane permeation and pervaporation

(P. Uchytíl, uchyt@icpf.cas.cz; joint project with University of Colorado, Boulder, USA; supported by MEYS, KONTAKT project No. ME 889)

Solubility, diffusivity and permeability of toluene vapors in a low-density polyethylene (LDPE) membrane of various thicknesses (approximately 48, 93, 138 and 187 μm) at different temperatures 30, 40 and 50 $^{\circ}\text{C}$ in the range of vapor relative pressure $p/p^0 = (0.05; 0.95)$ were measured using new type of permeation apparatus. Moreover, special construction of the new cell enables determination of the permeant amount sorbed in the membrane in the steady state of the vapor permeation process. [Ref. 6]

The simple semi-empirical model of toluene transport in a polyethylene membrane based on relation between experimentally obtained effective diffusion coefficients and concentration dependent diffusion coefficients evaluated from experiments on new permeation apparatus was proposed. The model enables estimation of toluene fluxes, sorption in the steady state of vapor permeation and concentration profiles in a polyethylene membrane from equilibrium sorption isotherms and effective diffusion coefficients. Very good agreement between experimental and calculated values from the proposed model was obtained. [Ref. 11, 33]

The flow of vapors through Vycor glass membranes is investigated theoretically and experimentally. The mass flow is measured for a range of pore diameters between 20 and 200 nm and for a range of upstream conditions and pressure differences. Attention is paid to accurately describe the flow if condensation occurs. An isothermal description of the flow is found to be not sufficient. The flow of a fluid through a porous membrane constitutes a Joule-Thomson process. A vapor is cooler at the downstream side of the membrane than at the upstream side. Hence, the vapor may not only condense due to capillary condensation, but also due to heat conduction in downstream direction. The balances of mass, momentum and energy are used to describe the flow as a one-dimensional, adiabatic throttling process. The porous medium is modeled as a bundle of equivalent capillaries. Momentum transport by viscous and by molecular flow is taken into account. An expression for a characteristic pore size is given. It is found that condensation does not have a noticeable influence on the mass flow for membranes with pores larger than the characteristic pore size. The agreement between the present description and experimental data is qualitatively good and quantitatively moderate. [Ref. 5]

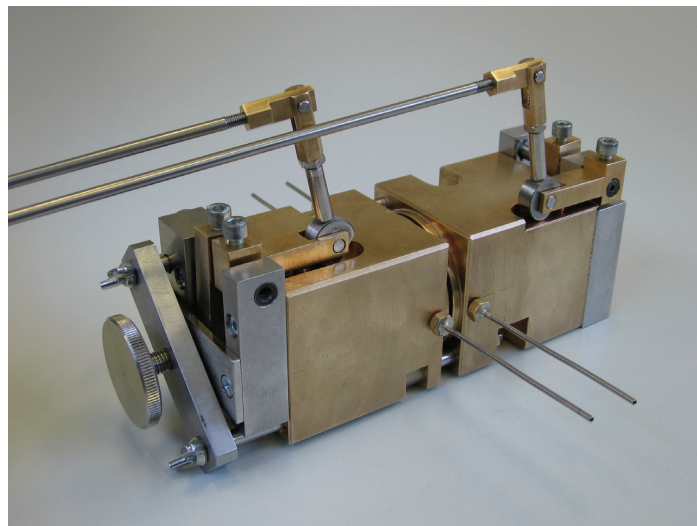


Photo of a new type of the permeation cell for determination of transport parameters in polymeric membranes

Liquid layers immobilized between nanoparticles-filled membranes for gas separation

(P. Uchytíl, uchyt@icpf.cas.cz; joint project with National Chung Hsing University, Taiwan; supported by ASCR and by National Science Council of Taiwan, project No. 106/10/J038)

Polyethersulfone (PES)-based mixed matrix membranes (MMMs) with the incorporation of inorganic fillers of different shapes (Na-montmorillonite (MMT) clays and TiO₂ nanoparticles) were prepared in this study, and separation of carbone dioxide and methane was studied. It was observed that gas permeabilities increased significantly with the increasing filler content and consequently the gas selectivity was greatly reduced. At high MMT loadings (10 and 20 wt%), Knudsen diffusion became the predominant gas transport mechanism. A different trend was achieved in the case of PES/TiO₂ MMMs. The CO₂/CH₄ separation factor increased from 24.5 (pure PES membrane) to 38.5 for 4 wt% TiO₂ MMM and then decreased with a further increase in TiO₂ content (17.3 for 20 wt%). The formation of interface voids and the enlarged layer spacing of MMT clays in MMMs should have contributed to the high gas permeabilities and low gas selectivity. Moreover, inorganic filler agglomeration became serious at high loading cases and resulted in worse gas separation performance. We used also these membranes for testing transport properties of ionic liquids. [Refs. 13, 24, 40]

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

(P. Uchytíl, uchyt@icpf.cas.cz; joint project with IMC, supported by the GACR, grant No. 104/09/1165)

Pervaporation transport of binary mixtures of four butanol isomers (1-butanol, 2-butanol, isobutanol and *tert*-butanol) with water through polyethylene membrane was studied. The pervaporation experiments were performed with binary mixtures of the large concentration range of butanol in water which is limited by their mutual miscibility except of *tert*-butanol. The effect of temperature, feed concentration and the shape of the permeating species on pervaporation characteristics is investigated. In spite of the ability of butanols to form hydrogen-bonds through their hydroxyl groups and therefore to form clusters in liquid feed, with respect to hydrophobic character of polyethylene membrane the strong influence of the water on the butanol isomers transport was not expected. [Ref. 25]

The obtained results are interesting—diffusion coefficients with increasing of water concentration in the binary mixtures were increasing. Permeation fluxes were decreasing strongly for 10 wt. % of water in comparison with pure butanols – around 40 wt. % but for higher content of water in mixtures till 90 wt. % they remained practically constant. By comparing permeance of butanols, we found that the permeance of studied butanol isomers followed the following order: 2-butanol > 1-butanol > isobutanol > *tert*-butanol. To better understand these results it will be necessary to do additional experiments (i.e. with mass spectrometer) with the study of water transport. Study of transport properties of pervaporation membranes on the basis of poly- γ -benzyl-L-glutamate was published. [Ref. 4]

Ionic membranes for selective separation of liquid mixtures by pervaporation

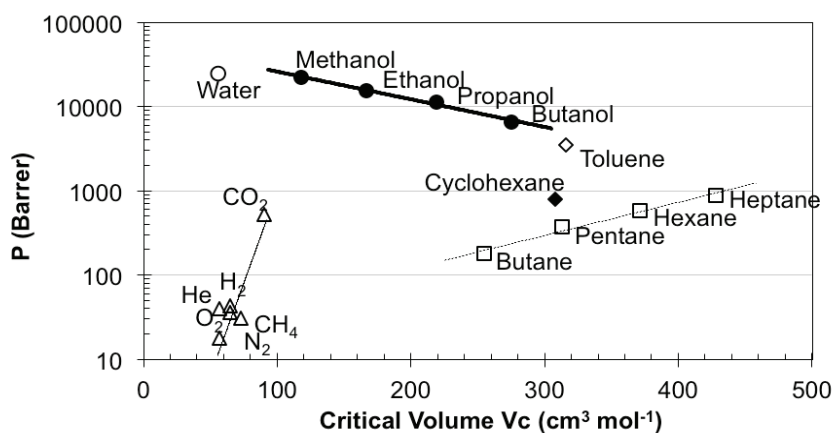
(P. Izák, izak@icpf.cas.cz; joint project with ICT Prague; supported by GACR, grant No. 104/08/0600)

Ionic liquids seem to have a large potential in downstream processing, especially when applied in a form that requires only a small amount of them, e.g. in supported liquid membranes. The special property of ILs is their non-measurable vapor pressure that makes their application in liquid membranes attractive for pervaporation. The objective is to investigate the development and application of supported liquid membranes on the base of ionic liquids. The project will be focused on products of biotransformation, waste water treatment and other valuable products, where practical application in industry is ensured. [Ref. 8, 19, 20]

Separation of volatile organic compounds (VOCs) from air

(P. Izák, izak@icpf.cas.cz; joint project with ICT Prague; supported by GACR, grant No. 106/10/1194)

The aim of this project is a development and application of membrane techniques to increase the affectivity of classical separation processes with help of computer simulations and theoretical modeling. In most of processes the goal of separation techniques is to recycle vapor phase which was lost in a sweeping gas. The optimization of polymeric membrane for specific separation task is time consuming and financial demanding. Ionic liquids have a great potential for membrane processes especially if only a small amount of ionic liquid is necessary e.g. supported ionic liquid membranes (SILMs). Characteristic property of ionic liquid is their very low vapor pressure which makes them attractive for gas and vapor permeation. [Ref. 3, 16, 18]



Permeability of supported ionic liquid membrane on critical volume of permeating molecules

Effective purification of biogas by condensing-liquid membrane

(P. Izák, izak@icpf.cas.cz; joint project with Czech head)

Currently, the biogas upgrading is widely discussed topic and because of shortening resources of fossil fuels it will remain. Biogas is easily accessible renewable fuel. Today biogas is mostly used in combined heat and power production plants however the electricity from these plants will not be so advantageous in the future because of limited capacity of the system. Biomethane as the substitute for natural gas will soon be necessary to keep our level of living. Much more progress is necessary for development of new and effective processes for biogas upgrading.

Impurities and CO₂ in raw biogas are separated by a “condensing-liquid membrane”, based on the different solubility of components in a very thin continuously refreshed water layer in a hydrophilic porous membrane. The permeation flux of each component of biogas depends on the feed flow rate of the gases and pressure and temperature differences between the upstream and downstream side of the condensing-liquid membrane. Selectivity increases with a lower feed flow rate. The molar balance based on 43 linear equations confirmed the high potential of this method to upgrade raw biogas. The condensing-liquid membrane can also be used under unfavorable conditions in which other polymeric membranes could be contaminated or destroyed by aggressive substances. [Refs. 7, 15, 21, 26]

International co-operations

CNRS Toulouse, France: Characterization of two phase flow in microchannels

CNRS Lyon: Hydrogenation in falling film microreactor

KIT Karlsruhe: Design of pilot plant size microreactor for sulfur dioxide catalytic heterogeneous oxidation

Procter&Gamble: Hydrodynamics of micro reactor for sulfonation

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

Institute of Chemical Engineering, Sofia, BAS: 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/vapor and vapor/vapor separations

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 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 CO₂ medium

University of KwaZulu-Natal, Republic of South Africa: Liquid-liquid extraction processes with fluorinated hydrocarbons

Visits abroad

J. Křišťál: Procter & Gamble, Brussels, Belgium (3 months)

P. Zálaha, CNRS, Toulouse, France (2 weeks)

P. Uchytíl, National Chung Hsing University, Taichung, Taiwan (3 weeks)

Visitors

J. Aubin, CNRS Toulouse, France

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

V. Villar, ENSIASET, Toulouse, France

V. Rochatte, 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

- [1] Akramov T.A., Stavárek P., Jiříčný V., Staněk V.: Minimum Energy Dissipation under Cocurrent Flow in Packed Beds. *Ind. Eng. Chem. Res.* 50(18), 10824-10832 (2011).
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- [12] Sovová H., Stateva R.P.: Supercritical Fluid Extraction from Vegetable Materials. *Rev. Chem. Eng.* 27(3-4), 79-156 (2011).
- [13] Uchytíl P., Schauer J., Petričkovič R., Setničková K., Suen S.-Y.: Ionic Liquid Membranes for Carbon Dioxide-Methane Separation. *J. Membr. Sci.* 383(1-2), 262-271 (2011).

Chapters in books

- [14] Evans J.W., Jiříčný V.: Chapter 16: Spouted Bed Electrochemical Reactors. (Eng) In: Spouted and Spout-Fluid Beds: Fundamentals and Applications. (Epstein N. - Grace J.R., Ed.), pp. 269-282, Cambridge University Press, New York 2011.
- [15] Kárászová M., Friess K., Šípek M., Jansen J.C., Izák P.: Biogas: Production, consumption and applications. In *Biogas upgrading for the 21st century*. (Litonjua R., Cvetkovski I., Ed.), Nova Science Publishers, New York 2011.
- [16] Friess K., Izák P., Šípek M., Jansen J.C.: Transport of VOCs in Polymers. In: *Volatile Organic Compounds*. (Columbus F., Ed.), Nova Science Publishers, New York 2011.

Patents

- [17] Gruber V., Rousková M., Heyberger A., Staf M.: Způsob získávání extraktů s obsahem europia a yttria. (Czech) Method for Reclaiming of Organic Extracts Containing Europium and Yttrium Ions. Pat. No. CZ302854/PV 2010-928. Applied: 10.12.14, patented: 11.12.14.

International conferences

- [18] Friess K., Jansen J.C., Clarizia G., Bernardo G.P., Bazzarelli G.F., Schauer J., Jarmarová V., Kačírková M., Izák P.: Separace plynů a par pomocí fluoropolymerních membrán s vysokým obsahem iontové kapaliny. (Czech) Separation of Gases and Vapours by High Ionic Liquid Content Fluoropolymer Membranes. 58. Konference chemického a procesního inženýrství CHISA 2011, Sborník, p. 40 (A2.3), Srní, Šumava, Czech Republic, 24-27 October 2011.
- [19] Kačírková M., Randová A., Hovorka Š., Schauer J., Tisma M., Izák P.: How Ionic Liquid Changes Properties of Dense Membrane in Pervaporation Separation Process? 25th European Symposium on Applied Thermodynamics, Book of Abstracts, p. 40, Saint Petersburg, Russia, 24-27 June 2011.
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- [22] Křišťál J., Drhová M., Jiříčný V., Kuncová G.: Microreactor Flow System for Enzymatic Reactions. CAMURE-8 & ISMR-7, Book of Abstracts, p. 149, Naantali, Finland, 22-25 May 2011.
- [23] Křišťál J., Jiříčný V., Hanika J.: Modulární mikrosystémy pro výzkum nových procesů. (Czech) Modular Microsystems for Novel Processes Research. 58. Konference chemického a procesního inženýrství CHISA 2011, Sborník, 7 pp., full text on CD-ROM, p. 51 (C2.1), Srní, Šumava, Czech Republic, 24-27 October 2011.
- [24] Liang C.-Y., Petříčkovič R., Uchytíl P., Suen S.-Y.: Gas Separation Using PES-MMT Membrane/Ionic Liquid/PES-MMT Membrane in a Sandwich Form. International Congress on Membranes and Membrane Processes ICOM 2011, Poster Session 1, ICOM672, Amsterdam, Netherlands, 23-29 July 2011.
- [25] Petříčkovič R., von Langemann M., Setničková K., Uchytíl P.: The Influence of Water on Butanols Isomers Pervaporation Transport through Polyethylene Membrane. 21st Annual Meeting of the North American Membrane Society, Program Book, p. Skyview 4, Las Vegas, Nevada, USA, 4-8 June 2011.
- [26] Poloncarzová M., Vejražka J., Veselý V., Izák P.: Effective Purification of Biogas by a Condensing-Liquid Membrane. 38th International Conference of Slovak Society of Chemical Engineering, p. 102, Tatranské Matliare, Slovakia, 23-27 May 2011.
- [27] Preziosi V., Křišťál J., Simoncelli A., Guido S.: Microstructure of Model Emulsion in Process Flow. 12th International Conference on Multiphase Flow in Industrial Plants, Conference Program, p. 43, Ischia, Napoli, Italy, 21-23 September 2011.
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- [29] Rousková M., Heyberger A.: Extrakce tálového mýdla vybranými organickými rozpouštědly. (Czech) Extraction of Tall Soap by Use of Selected Organic Solvents. 20. Chemicko-technologická konference s mezinárodní účastí APROCHEM 2011, Sborník přednášek, p. 736-743, Kouty nad Desnou, Czech Republic, 11-13 April 2011.
- [30] Sajfrtová M., Pavela R., Karban J.: Vliv podmínek superkritické extrakce na insekticidní účinky výtažků z routy vonné. (Czech) Effect of Conditions of Supercritical Fluid Extraction on Insecticidal Activity of Isolates from Rue. 58. Konference chemického a procesního inženýrství CHISA 2011, Sborník, p. 166 (V39), Srní, Šumava, Czech Republic, 24-27 October 2011.
- [31] Sajfrtová M., Rochová K., Karban J., Sovová H., Pavela R.: Supercritical Fluid Extraction of Botanical Insecticides from Rue. 11th International Conference on Carbon Dioxide Utilization, Program and Abstracts, p. 207, Dijon, France, 27-30 June 2011.
- [32] Sajfrtová M., Sovová H.: High-Pressure CO₂ as a Green Solvent for Extraction, Reaction, Particle Formation and Others Processes. Biotech 2011 and 5th Czech-Swiss Symposium with Exhibition, Book of Abstracts, pp. 61-62, Prague, Czech Republic, 15-17 June 2011.
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