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Application of Ionic Liquids in Membrane Separation Processes

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1. Introduction

Membrane separation processes are one of the most widely researched and fastest growing separation techniques of our century because of their advantages compared to classical (absorption, liquid-liquid extraction, distillation, etc.) processes, such as simple and compact set-up (Stern, 1994), easy operation at ambient temperature and pressure (Porter, 1990), simple up- and downscaling (Atchariyawut et al., 2006), better energy efficiency (Acharya et al., 2008), high purity products (Jönsson & Mathiasson, 1999) and much lower environmental impact (Soni et al., 2009).

They are successfully used in the food and dairy-, chemical-, beauty-, pharmaceutical- and biotechnology industry, in waste water treatment and in medical applications (Nath, 2008). However efficient these methods are nowadays, their continuous improvement is essential to be suitable for the ever changing requirements of the industries and the environmental laws and regulations.

The unique behaviour of ionic liquids, such as low melting point, negligible vapour pressure and tuneable physicochemical properties (Welton, 1999) make them the ideal candidates for membrane development. In liquid phase they can be used as bulk liquid membranes (BLM), emulsion liquid membranes (ELM) or supported liquid membranes (SLM) and after polymerization as non porous solid membrane films.

In the last decade several researchers have studied the application possibilities of ionic liquid based membranes including the transport and separation of solid matters, liquids and gases. In this chapter the most remarkable scientific results of these studies are reviewed and our latest results on gas separation applying supported ionic liquid membranes and polymerized ionic liquid membranes are described.

2. Membrane separation

The most important element of the membrane separation processes is the membrane itself. According to the international nomenclature and definition the **membrane** is a *permselective barrier between two phases* (Koops, 1995). It separates the influent (feed) stream into two effluent streams, known as permeate and retentate (Strathmann et al., 2006) as it is shown in Figure 1.

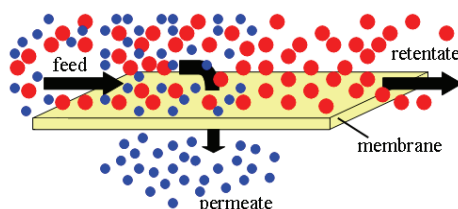


Fig. 1. Theoretical scheme of a membrane process

The separation is based on the difference between the rates of passive transport of the influent molecules. The passive transport occurs as a consequence of a driving force, which is a difference in chemical potential across the membrane (Ulbricht, 2006). It can be the difference in pressure, concentration, temperature or electric potential (Mulder, 1996).

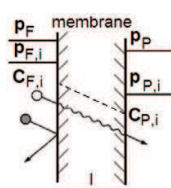


Fig. 2. Scheme of the solution diffusion mechanism

In the case of **non porous solid and liquid membranes** the separation process can be described by the solution diffusion mechanism (Figure 2), which consists of these three independent steps:

1. Molecules dissolve into the membrane material at the high pressure side
2. Molecules diffuse through the membrane according to Fick's law of diffusion
3. Molecules leave the membrane material at the low pressure side (Lashkari et al., 2008).

The rate limiting second step can be described by the following equation:

$$J_i = D_i S_i (p_{i,F} - p_{i,P}) / l = P_i (p_{i,F} - p_{i,P}) / l \quad (1)$$

where: J_i is the diffusive flux; D_i is the diffusion coefficient; S_i is the solubility coefficient; $p_{i,F}$ is the pressure of the component i on the feed side of the membrane; $p_{i,P}$ is the pressure of the component i on the permeate side of the membrane; l is the membrane thickness; and $P_i = D_i S_i$ is the permeability of the membrane (Baker, 2004).

The capability of the membranes to separate components can be efficiently described by the permeability and the selectivity of the membrane. In a simple component process the ideal selectivity (a^*) can be given by this equation (Stern, 1994):

$$a^*_{i/j} = P_i / P_j = (D_i / D_j) (S_i / S_j) \quad (3)$$

Considering a two component mixture, i and j the selectivity (a) is given by the following equation:

$$a_{i/j} = (C_{P,i} / C_{P,j}) / (C_{F,i} / C_{F,j}) \quad (2)$$

where: $C_{P,i}$ and $C_{P,j}$ are the concentrations of the components in permeate; $C_{F,i}$ and $C_{F,j}$ are the concentrations of the components in retentate (Koros & Fleming, 1993).

In general it can be stated that liquid membranes, which are built up of a porous membrane phase and an organic liquid phase held in the pores by capillary forces, have higher selectivity values than the non porous solid membranes because of the higher liquid phase diffusivities

(Chakma, 1995). Due to the incorporation of some carrier (C), which reacts reversibly and selectively with a specific permeate in the liquid membrane, further increase in mass flux and selectivity can be achieved (Ravanchi et al., 2010). This phenomenon is commonly known as facilitated transport (Kocherginsky et al., 2007) and is represented in Figure 3.

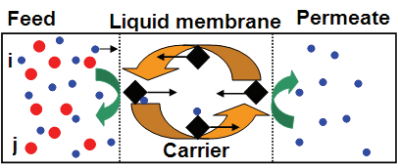


Fig. 3. Facilitated transport of permeants through liquid membranes

3. Ionic liquids

The special properties of ionic liquids, i.e. high thermal and chemical stability, low vapour pressure (Endres et al., 2008), non-flammability (Wasserschied & Welton, 2007) and tuneable physicochemical properties, make them the ideal candidates for the replacement of organic solvents in liquid membranes and hence for the realization of environmentally sound and economical membrane separation processes (Koel, 2008).

Table 1 contains the names, abbreviations and application of the ionic liquids that are most commonly used and investigated in membrane separation processes.

Ionic liquids			
Name	Abbreviation	Compounds to separate	Ref.
MEMBRANE ABSORPTION			
1-butyl-3-methylimidazolium tetrafluoroborate	[bmim][BF ₄]	propylene/propane	Ortiz et al., 2008
MEMBRANE EXTRACTION			
1-butyl-3-methylimidazolium hexafluorophosphate	[bmim][PF ₆]	benzene derivates	Visser et al., 2001
		tyramine and 2-methoxy-phenethylamine	Yung et al., 2005
		octane/ethylbenzene	Birdwell et al., 2006
1-alkyl-3-methylimidazolium hexafluorophosphate	[C _n mim][PF ₆]	short-chain aliphatic carboxylic acids, e.g. acetic acid, butyric acid	Miyako et al., 2003
		penicillin G	Matsumoto et al., 2007
1,3-dialkylimidazolium tetrafluoroborate	[dC _n im][BF ₄]	tetrahydrofurane from water	Jork et al., 2004
1-alkyl-3-methylimidazolium hexafluorophosphate	[C _n mim][PF ₆]	phenol, tyrosol and p-hydroxybenzoic acid	Vidal et al., 2004
1-alkyl-3-methylimidazolium tetrafluoroborate	[C _n mim][BF ₄]		

1-alkyl-3-methyl-imidazolium chloride	[C _n mim][Cl]	taurine from sodium sulfate	Gu et al., 2004
1-alkyl-3-methyl-imidazolium bromide	[C _n mim][Br]		
1-butyl-3-methyl-imidazolium iodide	[bmim][I]		
1-alkyl-3-methyl-imidazolium octylsulfate	[C _n mim][OcSO ₄]	organic sulphur compounds from diesel fuel	Eßer et al., 2004
1-alkyl-3-methyl-imidazolium ethylsulfate	[C _n mim][EtSO ₄]		
1-butyl-3-methyl-imidazolium bis (trifluoromethylsulfonyl) imide	[bmim] [NTf ₂]	toluene, nonan-1-ol, acetic acid and hexanoic acid	McFarlane et al., 2005
1-hexyl-3-methyl-imidazolium bis (trifluoromethylsulfonyl) imide	[hmim] [NTf ₂]		
1-octyl-3-methyl-imidazolium bis (trifluoromethylsulfonyl) imide	[omim] [NTf ₂]		
Trihexyl(tetradecyl)-phosphonium bis (trifluoromethylsulfonyl) imide	[thtdp][NTf ₂]		
Trihexyl(tetradecyl)-phosphonium methanesulfonate	[thtdp][CH ₃ SO ₃]		
Trihexyl(tetradecyl)-phosphonium dodecylbenzenesulfonate	[thtdp][ddbOTf]		
Tributyl(tetradecyl)-phosphonium dodecylbenzenesulfonate	[tbtdp][ddbOTf]		
1-butyl-1-methyl-pyrrolidinium bis(trifluoromethylsulfonyl) imide	[bmpyr][NTf ₂]		
1-butyl-3-methylimidazolium hexafluorophosphate	[bmim][PF ₆]	phenols	Khachatryan et al., 2005
1- <i>n</i> -butylimidazolium hexafluorophosphate	[bim][PF ₆]	aromatic hydrocarbons	Matsumoto et al., 2005
1- <i>n</i> -hexylimidazolium hexafluorophosphate	[him][PF ₆]		
1- <i>n</i> -octyl-3-methylimidazolium hexafluorophosphate	[omim][PF ₆]		
Diethylmethyl (2-methoxyethyl) ammonium bis(trifluoromethane sulfon)imid	[Et ₂ MeMeON][NTf ₂]		
1-butyl-3-methylimidazolium hexafluorophosphate	[bmim][PF ₆]	amino acids, e.g. valine, leucine, tyrosine	Wang et al., 2005

1-hexyl-3-methylimidazolium tetrafluoroborate	[hmim][BF ₄]		
1-octyl-3-methylimidazolium tetrafluoroborate	[omim][BF ₄]	antibiotics, e.g. amoxicillin, ampicillin	Soto et al., 2005
		thiophene from cyclohexane and toluene	Alonso et al., 2007
N-butyl-N-methylpyrrolidinium bis(trifluoromethylsulfonyl) imide	[bmpyr][NTf ₂]	azo dyes	Vijayaraghavan et al., 2006
1-methyl-3-octylimidazolium chloride	[omim][Cl]	toluene	Chakraborty & Bart, 2007
1-ethyl-3-methylimidazolium ethyl sulfate	ECOENG™212		
1-methylimidazole hydrogen sulfate	BASIONIC™ AC39		
1-H-imidazolium ethyl sulfate	TEGO IL IM55		
1-hexyl-3-methylimidazole	hmim	ethanol, butan-1-ol from water	Chapeaux et al., 2008
Tetrahexylammonium dihexylsulfosuccinate	[N ₆₆₆ ⁺][DHS]	phenols, amines	Egorov et al., 2008
Trioctylmethylammonium salicylate	[N ₍₁₎₈₈₈ ⁺][sce]		
1-(2-hydroxyethyl)-3-methylimidazolium tetrafluoroborate	[C ₂ OHmim][BF ₄]	ethanol from ethyl acetate	Hu et al., 2008
N-butyl-3-methylpyridinium thiocyanate	[bmpy][SCN]	organosulfur compounds from refinery products	Holbrey et al., 2008
N-hexylpyridinium tetrafluoroborate	[hpy][BF ₄]	aromatic sulphur compounds from diesel fuel	Gao et al., 2008
N-octylpyridinium tetrafluoroborate	[opy][BF ₄]		
Trihexyl(tetradecyl) phosphonium chloride	Cyphos 101	lactic acid	Matsumoto et al., 2010
Trihexyl(tetradecyl) phosphonium bis(2,2,4-trimethylpentyl-phosphinate	Cyphos 104		Marták et al., 2008
1-alkyl-3-methylimidazolium hexafluorophosphate	[C _n mim][PF ₆]	phenol, bisphenol A, 4-octylphenol and 4-nonylphenol	Fan et al., 2008
1-alkyl-3-methylimidazolium tetrafluoroborate	[C _n mim][BF ₄]		
1-butyl-3-methylimidazolium hexafluorophosphate	[bmim][PF ₆]	transesterification reaction compounds, e.g. vinyl acetate, ethyl decanoate, 1-butanol, 1-propanol, butyric acid	Hernández-Fernández et al., 2007; de los Ríos et al., 2008 a)
1-octyl-3-methylimidazolium hexafluorophosphate	[omim][PF ₆]		

1-butyl-3-methylimidazolium tetrafluoroborate	[bmim][BF ₄]		de los Ríos et al., 2008 a)
1-octyl-3-methylimidazolium tetrafluoroborate	[omim][BF ₄]		
1-butyl-3-methylimidazolium bis(trifluoromethylsulfonyl) imide	[bmim][NTf ₂]		de los Ríos et al., 2010
1-octyl-3-methylimidazolium bis(trifluoromethylsulfonyl) imide	[omim][NTf ₂]		
1,3-dialkylimidazolium bis(trifluoromethylsulfonyl) imide	[dC _n im][NTf ₂]	benzene from hexane	Poole & Poole, 2009
1-ethylpyridinium bis(trifluoromethylsulfonyl) imide	[epy][NTf ₂]		
2-hydroxyethyl-trimethylammonium bis(trifluoromethylsulfonyl) imide	[C ₂ OH <i>Et</i> Me ₃ NO][NTf ₂]		
Trihexyltetradecyl-phosphonium bis(trifluoromethylsulfonyl) imide	[thtdp][NTf ₂]	ethyl benzene from octane	Arce et al., 2009
1-ethyl-3-methylimidazolium bis (trifluoromethylsulfonyl) imide	[emim][NTf ₂]	aromatics from aliphatics	
Tetraalkyl ammonium sulfate	Ammoeng 102		
Tri- <i>n</i> -octylmethyl-ammonium chloride	TOMAC	benzimidazole	Venkantesan & Begum, 2009
3-(trimethoxysilyl)-propylammonium-3-trimethoxysilyl)-propyl carbamate	TMSAC	oil from oil shale and tar sands	Blasucci et al., 2010
3-(triethoxysilyl)-propylammonium 3-(triethoxysilyl)-propyl carbamate	TESAC		
MEMBRANE MICROEXTRACTION			
1-hexadecyl-3-methylimidazolium bromide	[hdmim][Br]	polycyclic aromatic hydrocarbons from sediments	Pino et al., 2008
1-butyl-3-methylimidazolium hexafluorophosphate	[bmim][PF ₆]	organochlorine pesticides from soil	Khodadoust et al., 2006

1-octyl-3-methylimidazolium hexafluorophosphate	[omim][PF ₆]	Sudan dyes, para red from chilli powder	Fan et al., 2009
1-octyl-3-methylimidazolium hexafluorophosphate	[omim][PF ₆]	halomethanes, alkyl aromatics	Aguilera-Herrador et al., 2008
1-butyl-3-methylimidazolium phosphate	[bmim][PO ₄]	aliphatic and aromatic hydrocarbons, e.g. tetradecane, naphthalene	Basheer et al., 2008
1-butyl-3-methylimidazolium methylsulfate	[bmim][MeSO ₄]		
1-butyl-3-methylimidazolium octylsulfate	[bmim][OcSO ₄]		
1-butyl-3-methylimidazolium hexafluorophosphate	[bmim][PF ₆]		
1-hexyl-3-methylimidazolium tetrafluoroborate	[hmim][BF ₄]	pesticides, aromatic amines	Poole & Poole, 2009
MEMBRANE EXTRACTIVE DISTILLATION			
1-ethy-3-methylimidazolium triiodide	[emim][I ₃]	toluene/ <i>n</i> -heptane	Selvan et al., 2000
1-ethyl-3-methylimidazolium acetate	[emim][ace]	water/ethanol	Seller &Hirsch, 2004
1-ethyl-3-methylimidazolium tetrafluoroborate	[emim][BF ₄]		Ge et al., 2008
1-(2-hydroxyethyl)-3-methylimidazolium chloride	[C ₂ OHmim][Cl]	water/tetrahydrofurane	Hu et al., 2006
1-octylquinolin bis(trifluoromethylsulfonyl) imide	[Ocqnl][NTf ₂]	n-hexane/1-hexene	Lei et al., 2006
1-ethyl-3-methylimidazolium bis(trifluoromethylsulfonyl) imide	[emim][NTf ₂]	aromatics/alkanes	Arce et al., 2007
1-hexyl-3-methylimidazolium hexafluorophosphate	[hmim][PF ₆]	ethanol/ <i>n</i> -heptane	Pereiro et al., 2006
1,3-dimethylimidazolium methanesulfonate	[dmim][CH ₃ SO ₃]		Zhang et al., 2009
1-ethyl-3-methylimidazolium acetate	[emim][ace]	water/2-propanol	Zhang et al., 2007
1-ethyl-3-methylimidazolium trtrifluoromethylsulfonate	[emim][CF ₃ SO ₃]	ethanol/chloroform	Orchilies et al., 2008
1-allyl-3-methylimidazolium bromide	[amim][Br]	ethanol/ethyl acetate	Zhang et al., 2008

1-ethyl-3-methylimidazolium tetrafluoroborate	[emim][BF ₄]		Li et al., 2009
1-butyl-3-methylimidazolium methanesulfonate	[bmim][CH ₃ SO ₃]	ethanol/ <i>n</i> -hexane	Pereiro & Rodriguez, 2009 b)
1-hexyl-3-methylimidazolium	[hmim][BTI]	aliphatics/aromatics alkanes/alkenes alcohols/water	Westerholt et al., 2009
1-butyl-3-methyl-pyrrolidinium	[bmpyr][BTI]		
1-ethyl-3-methyl-imidazolium chloride	[emim][Cl]	water/ <i>tert</i> -butanol	Zhang et al. 2009
MEMBRANE PERVAPORATION			
Tetrapropylammonium tetracyanoborate	[N ₃₃₃₃ ⁺][B(CN) ₄]	1,3-propanediol	Izák et al., 2006
1-ethenyl-3-ethylimidazolium hexafluorophosphate	[eeim][PF ₆]	acetone and butan-1-ol from water	Izák et al., 2008
1-ethenyl-3-ethylimidazolium bromide	[eeim][Br]		
MEMBRANE VAPOUR SEPARATION			
1-butyl-3-methyl-imidazolium bis(trifluoromethylsulfonyl) amide	[bmim][BTA]	olefins/paraffins	Krull et al., 2007
MEMBRANE GAS SEPARATON			
1-ethyl-3-methyl-imidazolium bis (trifluoromethyl - sulfonyl)imide	[emim][NTf ₂]	N ₂ , CO ₂ , CH ₄	Scovazzo et al., 2004
1-ethyl-3-methyl-imidazolium triflate	[emim][CF ₃ SO ₃]		
1-ethyl-3-methylimidazolium dicyandiamide	[emim][dca]		
Trihexyl(tetradecyl)-phosphonium chloride	[thtdp][Cl]		
1,3-dimethylimidazolium bis (trifluoromethyl - sulfonyl)imide	[dmim][NTf ₂],	N ₂ , H ₂ , O ₂ , CO	Gan et al., 2006
Methyltrioctylammonium bis (trifluoromethyl - sulfonyl)imide	[N ₍₁₎₈₈₈ ⁺][NTf ₂]		
<i>N</i> -octylpyridinium bis (trifluoromethyl - sulfonyl)imide	[C ₈ Py][NTf ₂]		
1-(3-aminopropyl)-3-methylimidazolium bis(trifluoromethyl - sulfonyl)imide	[H ₂ NC ₃ H ₆ mim][NTf ₂]	H ₂ , CO ₂	Myers et al., 20008
<i>N</i> -aminopropyl-3-methylimidazolium bis	[C ₃ NH ₂ mim][NTf ₂]	CO ₂ , CH ₄	Hanioka et al., 2008

(trifluoromethyl - sulfonyl)imide			
N-aminopropyl-3-methylimidazolium triflate	[C ₃ NH ₂ mim][CF ₃ SO ₃]		
1-methyl-3-(3,3,4,4,4-pentylfluorohexyl)imidazolium bis(trifluoromethyl - sulfonyl)imide	[MpFHiM][NTf ₂],	N ₂ , O ₂ , CO ₂ , CH ₄	Bara et al. 2009
1-methyl-3-(3,3,4,4,5,5,6,6,6-nonafluorohexyl)imidazolium bis (trifluoromethyl - sulfonyl)imide	[MnFHiM][NTf ₂]		
1-methyl-3-(3,3,4,4,5,5,6,6,7,7,8,8,8-tridecyl-fluorohexyl)imidazolium bis (trifluoromethyl - sulfonyl)imide	[MtdFHiM][NTf ₂]		
1-butyl-3-methylimidazolium tetrafluoroborate	[bmim][BF ₄]	N ₂ , CO ₂ , CH ₄ , SO ₂	Jiang et al., 2009
1-ethyl-3-methylimidazolium tetrafluoroborate	[emim][BF ₄]		
1-hexyl-3-methylimidazolium tetrafluoroborate	[hmim][BF ₄]		
1-ethyl-3-methylimidazolium triflate	[emim][CF ₃ SO ₃]		
1-butylimidazolim acetate	[bim][ace]	CO ₂ , SO ₂	Luis et al., 2009
1-methylimidazolium acetate	[mim][ace]		
1-butyl-3-methyl-imidazolium bis(perfluoro-ethylsulfonyl)imide	[bmim][BETI]	N ₂ , CO ₂ , CH ₄	Scovazzo et al., 2009
1-ethyl-3-methylimidazolium dicyandiamide	[emim][dca]		
Trihexyl(tetradecyl) phosphonium bromide	Cyphos 102	N ₂ , H ₂ , CH ₄ , CO ₂	Cserjési et al., 2010
Trihexyl(tetradecyl) phosphonium decanoate	Cyphos 103		
Trihexyl(tetradecyl) phosphonium bis (2,4,4-trimethylpentyl) phosphinate	Cyphos 104		
Triisobutylmethyl phosphonium tosilate	Cyphos 106		

Tetrabutyl phosphonium bromide	Cyphos 163		
Triethylsulfonium bis (trifluoromethyl sulfonyl)imide	[Set ₃][NTf ₂]		
1,3-dimethylimidazolium dimethylphosphate	ECOENG™1111P		
1-decyl-3-methylimidazolium tetrafluoroborate	[dmim][BF ₄]	N ₂ , CH ₄ , CO ₂	Neves et al., 2010
1-butyl-3-methylimidazolium hexafluorophosphate	[bmim][PF ₆]		
1-butyl-3-methylimidazolium tetrafluoroborate	[bmim][BF ₄]		
1-butyl-3-methylimidazolium bis (trifluoromethyl sulfonyl)imide	[bmim][NTf ₂]		
1-octyl-3-methylimidazolium hexafluorophosphate	[omim][PF ₆]		

Table 1. Most commonly used ionic liquids in membrane separations

4. Ionic liquid based membranes

4.1 Bulk ionic liquid membranes (BILMs)

In the bulk liquid membrane an organic membrane phase separates two aqueous or gaseous phases from each other. These non miscible phases can be arranged in several ways, five of the most commonly used possibilities are shown in Figure 4 (Clark et al., 2005; Bélafi-Bakó et al., 2000).

Bulk ionic liquid membranes contain ionic liquids as the membrane phase and have higher selectivity due to the tunable nature of the ionic liquids; for a certain separation task the ionic liquids with the best separation properties can be selected. The extraction of the separated compounds can be easily carried out by a solvent that is totally miscible with the ionic liquid and dissolves the compounds (Gubicza et al., 2003).

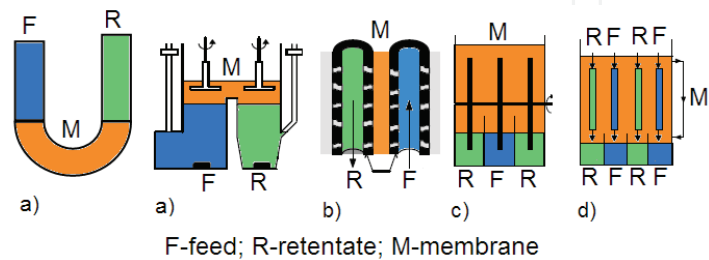


Fig. 4. Most common types of bulk liquid membranes; a) layered BLMs; b) BLM in a rotating disc contactor; c) BLM in a creeping film contactor; d) BLM with immobilised interfaces in a hollow fibre

Several researchers have studied the liquid/liquid extraction properties of bulk ionic liquid membranes, heptane separation from toluene using tetraalkyl ammonium sulfate (Ammonoeng 102) ionic liquids was investigated by Pereiro and Rodriguez (Pereiro & Rodriguez, 2009 a)) and the extraction of oil from oil shale and tar sands with the ionic liquids shown in Table 2., by Blasucci and his co-workers (Blasucci, 2010).

Name	Abbreviation
3-(trimethoxysilyl)-propylammonium 3-(trimethoxysilyl)-propyl carbamate	TMSAC
3-(triethoxysilyl)-propylammonium 3-(triethoxysilyl)-propyl carbamate	TESAC
3-(triethylsilyl)-propylammonium 3-triethylsilyl-propyl carbamate	TETsAC
3-(tripropylsilyl)-propylammonium 3-(tripropylsilyl)-propyl carbamate	TPSAC

Table 2. Ionic liquids used by Blasucci et al.

Chakraborty and Bart carried out toluene transport experiments in bulk ionic liquid membranes containing 1-methyl-3-octyl imidazolium chloride, 1-ethyl-3-methyl imidazolium ethyl sulfate, 1-methylimidazole hydrogen sulfate, 1H-imidazolium ethyl sulfate, 1-ethyl-4,5-dihydro-3-(2-hydroxyethyl)-2-(8-heptadecenyl) ethyl sulfate. They determined that the stirring speed, the feed phase concentration and the temperature had a strong effect on the permeation and that the permeability of toluene is quite low, while the selectivity is relatively high (Chakraborty & Bart, 2007). Gu and fellow researchers reported the extraction of taurine in 1-propyl-3-Methylimidazolium-, 1-butyl-3-methylimidazolium-, 1-amyl-3-methylimidazolium, 1-hexyl-3-methylimidazolium chloride, 1-propyl-3-methylimidazolium-, 1-butyl-3-methylimidazolium-, 1-amyl-3-methylimidazolium-, 1-hexyl-3-methylimidazolium bromide, 1-butyl-3-methylimidazolium iodide, 1-butyl-3-methylimidazolium hexafluorophosphate and 1-butyl-3-methylimidazolium tetrafluoroborate ionic liquids and achieved 96-98,5% separation yield in a single separation step (Gu et al., 2004). Propylene separation from propane by absorption was studied by Ortiz et al. in 1-butyl-3-methylimidazolium tetrafluoroborate bulk ionic liquid membranes (Ortiz et al., 2008) and the extractive distillation of benzene/cyclohexane, 1-hexene/n-hexane, 2-propanol/water binary and the ternary systems applying 1-hexyl-3-methyl-imidazolium- and 1-butyl-1-methylpyrrolidinium bis(trifluoromethylsulfonyl) imide was reported by Westerholt (Westerholt, et al., 2009). They all concluded that the application of ionic liquids increased the efficiency and repeatability and allowed separation for previously unsolved separation problems.

4.2 Emulsion ionic liquid membranes (EILMs)

Emulsion (or surfactant) liquid membranes are prepared by the intensive mixing of two non miscible phases (phase 1 and phase 2) and then the addition of some strong surfactant as it is shown in Figure 5. In this case phase 2 will be the liquid membrane, which can contain facilitating agents that promote separation through formation of chemical complexes from the agents and the components (Kislik, 2009). Since the preparation of emulsion liquid membranes can be complicated and the decomposition of the emulsions after the separation is difficult in the case of relatively strong surfactants, the application of this type is limited to laboratory scale.

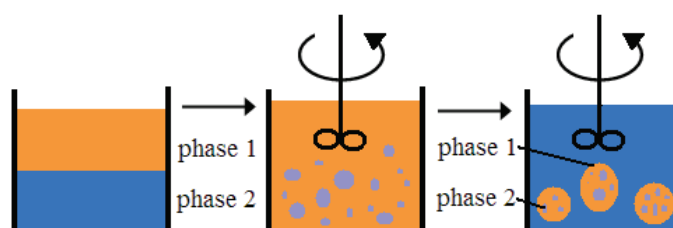


Fig. 5. Formation of emulsion liquid membranes

One of the most important results was achieved by Venkatesan and Begum, who carried out the extraction of benzimidazole by emulsion ionic liquid membrane containing tri-*n*-octyl methyl ammonium chloride as phase 1 and aqueous HCl solution as phase 2 (Venkatesan & Begum, 2009). They studied the effect of surfactant concentration, emulsification time, emulsification speed, carrier concentration, internal phase concentration and the volume ratio of the organic phase (1) to the aqueous phase (2) on emulsion stability and the effect of contact time for extraction, agitation speed, emulsion to external phase ratio, carrier concentration and HCl concentration in the internal aqueous phase on the effect of extraction. It was concluded that this technique is more effective than the conventional liquid-liquid extraction method; more than 97,5% of the benzimidazole present in the aqueous solutions within 12 min.

4.3 Supported ionic liquid membranes (SILMs)

Supported (or immobilized) liquid membranes are also built up of two phases, a supporting porous or non porous membrane and a liquid phase, which is either held in the pores by capillary forces (Jönsson & Mathiasson, 2000) or placed between two membrane sheets (Krull et al., 2008) as it is shown in Figure 6.

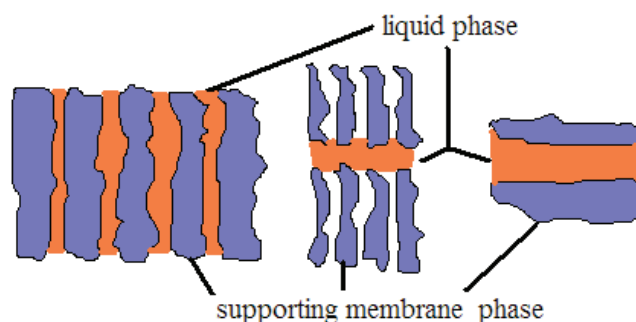


Fig. 6. Types of supported (immobilized) liquid membranes

The separation takes place in the liquid phase, without facilitating agents according to the solution diffusion model or in the presence of such agents through the mechanism of facilitated transport (Kocherginsky et al., 2007).

SLMs offer an elegant solution for separating molecules and have several advantages compared to porous or non porous solid membrane materials (Jönsson & Mathiasson, 1999). With the choice of the right liquid phase high permeability (Ilconich et al., 2007) and selectivity (He, 2008) values can be achieved, expensive, high quality liquids can be used due to the requirement of their small amount (Adebayo & Sarangi, 2008), extraction and stripping can be realized in one step (Poliwoda et al., 2007) and they can be connected to other (analytical) equipments because high pressure is not needed for the separation process (Jönsson & Mathiasson, 1999).

However, SLMs are not stable enough, they suffer from flux decline with time because the liquid phase evaporates or gets easily displaced from the pores due to trans membrane pressure difference (Teramoto et al., 2000). The extension of SLM lifetime was attempted by liquid membrane gelation, surface modification and with the application of ionic liquids (Zhang et al., 2001).

Since ionic liquids are chemically and thermally stable, have non detectable vapour pressure and relatively high viscosity (Keskin et al., 2007) their application as the liquid phase consequently leads to a much more stable membrane, which is often referred to as supported ionic liquid membrane (de los Ríos et al., 2008).

Although the industrial application of supported ionic liquid membranes is still yet to come, laboratory and pilot plant applications have been studied in the last three decades. They were successfully used for liquid-liquid extraction, microextraction, extractive distillation, pervaporation and gas separation.

4.3.1 Liquid-liquid extraction

Miyako et al. successfully realized the transport of organic acids, such as 4-phenoxybutyric acid, 2-phenoxybutyric acid, etc. through 1-alkyl-3-methylimidazolium hexafluorophosphate based SILMs that were stable for 75 hours (Miyako et al., 2003). Marták and co-workers studied lactic acid separation with SILMs prepared with Cyphos 104 and achieved acid purity as high as 95% (Marták et al., 2008). Cassol et al. studied the separation of benzene and alkyl-benzenes from *n*-heptane and *n*-octane (benzene/hexane, toluene/heptane, ethylbenzene/octane and *m*-xylene/*n*-octane) by pyridinium based ionic liquids (Cassol et al., 2007). Matsumoto and co-workers successfully carried out experiments on the separation of lactic acid by Cyphos 101 (Matsumoto et al., 2010), penicillin G by imidazolium based ionic liquids (Matsumoto et al., 2007) and aromatic hydrocarbons i.e. benzene, toluene, *p*-xylene, hexadecane, methanol, acetonitrile and chloroform by 1-butyl-3-methylimidazolium-, 1-hexyl-3-methylimidazolium and 1-octyl-3-methylimidazolium hexafluorophosphate (Matsumoto et al., 2005). De los Ríos and fellow researchers investigated the separation of transesterification products, such as vinyl butyrate, 1-butanol, butyl butyrate, butyric acid (de los Ríos et al., 2008 b)), methanol, vinyl acetate, acetic acid, etc. (de los Ríos et al., 2008 a)) applying 1-butyl-3-methylimidazolium-, 1-oxy-3-methylimidazolium hexafluorophosphate, 1-butyl-3-methylimidazolium-, 1-oxy-3-methylimidazolium tetrafluoroborate, 1-butyl-3-methyl-imidazolium- and 1-oxy-3-methylimidazolium bis(trifluoromethylsulfonyl)imide based SILMs (de los Ríos et al., 2010). They achieved relatively high average selectivity values for the membranes and high permeabilities for methanol, 1-butanol and butyric acid.

4.3.2 Microextraction

Basheer et al. carried out successful experiments on the microextraction of aliphatic and polycyclic aromatic hydrocarbons, such as naphthalene, tridecane, fluorine, heptadecane, pyrene, etc. from water samples by 1-butyl-3-methyl-imidazolium phosphate, 1-butyl-3-methyl-imidazolium methylsulfate, 1-butyl-3-methyl-imidazolium octylsulfate and 1-butyl-3-methylimidazolium hexafluorophosphate based SILMs, which could be directly connected to GC-MS (Basheer et al., 2008).

4.3.3 Pervaporation

Izák and co-workers studied the transport of 1,3-propanediol from aqueous mixture (Izák et al., 2006) and the removal of acetone and butan-1-ol from water by pervaporation (Izák et al., 2008) using SILMs based on tetrapropylammonium tetracyanoborate, 1-ethenyl-3-ethylimidazolium hexafluorophosphate and tetrapropylammonium tetracyano-borate ionic liquids. It was found that the separation factor for propanediol, acetone and butan-1-ol increased from 0.4 to 177, from 2.3 to 7.9 and from 2.2 to 10.9 respectively.

4.3.4 Vapour separation

Krull et al. carried out experiments on the separation of vapourized propylene from propane by 1-butyl-3-methyl-imidazolium bis(trifluoromethylsulfonyl) amide based SILMs and on the stability of the membrane and achieved permeability of 26.5 cm²hbar and selectivity of 1.67. It was found out that the permeability increased while selectivity decreased with the increase in operation temperature and that the membrane was stable up to 5 bar pressure and 72 h reaction time (Krull et al., 2007).

4.3.5 Gas separation

Scovazzo and co-workers investigated the N₂, CO₂ and CH₄ gas separation properties of SILMs prepared with 1-ethyl-3-methyl-imidazolium bis(trifluoromethyl -sulfonyl)imide 1-ethyl-3-methyl-imidazolium triflate, 1-ethyl-3-methylimidazolium dicyandiamide, trihexyl (tetradecyl) phosphonium chloride (Scovazzo et al., 2004) and 1-butyl-3-methylimidazolium bis(perfluoro-ethylsulfonyl)imide and (Scovazzo et al., 2009). The permeability for CO₂, CH₄ and N₂ were 1500, 920 and 350 Barrers relatively and it was determined that the permeabilities decreased with the increase in trans membrane pressure. Gan et al. used 1-butyl-3-methylimidazolium-, 1,3-dimethylimidazolium-, methyltrioctylammonium bis(trifluoromethylsulfonyl)imide and *N*-octylpyridinium bis(trifluoromethylsulfonyl)imide based SILMs for the permeation of H₂, N₂, O₂, CO₂, and CO and it was found that, on the contrary to the results of Scovazzo, the permeabilities decreased with the increase in trans membrane pressure (Gan et al., 2006). Ilconich and co-workers carried out experiments on the permeation of He and CO₂ through 1-hexyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide based SILM between 37°C and 125°C and stated that the permeability for CO₂ and He increased from 744 to 1200 and from 86 to 260 Barrers with the increase in temperature (Ilconich et al., 2007). Hanioka et al. successfully realized the facilitated transport of CO₂ and CH₄ single gases and binary mixture with *N*-aminopropyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide and *N*-aminopropyl-3-methylimidazolium triflate based SILMs. The CO₂/CH₄ selectivity increased with the increase in CO₂ partial pressure and with the increase in temperature from 25°C to 75°C the CO₂ and CH₄ permeability increased from 500 to 1500 and from 100 to 850 Barrers respectively (Hanioka et al., 2008). Jiang and co-workers studied the permeation of N₂, CO₂, CH₄ and SO₂ through SILMs prepared with 1-butyl-3-methylimidazolium-, 1-ethyl-3-methylimidazolium-, 1-hexyl-3-methylimidazolium tetrafluoroborate, 1-ethyl-3-methylimidazolium triflate, 1-butyl-3-methylimidazolium hexafluorophosphate and 1-butyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide and found out that in the case of SO₂ the permeability increased, while in the other cases no significant change was observed with the increase in trans membrane pressure (Jiang et al., 2009). The permeation of CO₂ and SO₂ through 1-

butylimidazolium acetate and 1-methylimidazolium acetate was studied by Luis et al. (Luis et al., 2009) and the permeation of CO_2 , CH_4 , H_2S through 1-butyl-3-methylimidazolium tetrafluoroborate based SILM was studied by Park et al. (Park et al., 2009). In 2009 Scovazzo published a gas solubility and diffusivity model and the permeabilities of SILMs prepared from several imidazolium-, ammonium-, phosphonium based and functionalized ionic liquids for N_2 , O_2 , CO_2 and CH_4 gases (Scovazzo, 2009).

All the scientists concluded that after solving the stability problems of the SILMs, they can perform better or just as well as the polymeric membranes used in the industrial processes.

4.4 Polymerized ionic liquid membranes (PILMs)

The idea of applying polymerized ionic liquids in membrane separation processes is quite young and so far has been mostly used for gas separation (Bara et al., 2007). In this case polymerized ionic liquid solutions can be cast and solid membrane films are achieved (Figure 7), that can be treated as any other commonly used membrane material (Carlisle, 2010).



Fig. 7. Picture of a PILM

PILMs combine the advantageous properties of ionic liquids, such as tuneable physicochemical properties, high thermal and chemical stability, high selectivity, etc. and the advantages of dense membranes, i.e. low membrane thickness, high stability and high permeability values.

Noble and co-workers investigated the N_2 , CO_2 and CH_4 permeability of PILMs prepared from polymerized ionic liquids with varying length n-alkyl substituents (Bara et al., 2007) and from polymerized imidazolium-based ionic liquids containing polar, oligo(ethyl glycol) and alkyl-terminated nitrile substituents (Bara et al., 2008) and the H_2 , N_2 , CO_2 and CH_4 permeability of imidazolium ionene based PILMs and ionic liquid composites (Carlisle et al., 2010). The highest N_2 , H_2 , CO_2 and CH_4 permeability values were as high as 0.83, 5.3, 22 and 1.1 Barrers and the highest CO_2/N_2 , CO_2/CH_4 and H_2/CO_2 ideal selectivities were 44, 37 and 6 respectively. Plotting the performance of these membranes on a "Robeson plot" for CO_2/N_2 showed that PILMs perform as well or better than many other polymers for this separation. The CO_2/CH_4 and H_2/CO_2 separations are less impressive when compared to other polymer membranes on a "Robeson plot", but PILMs perform as well or better than ionic liquids do in bulk fluid gas absorptions for that gas pair.

We have prepared four different PILMs using imidazole-based copolymers and anion-exchanged homopolymers and determined the N_2 , H_2 , O_2 , CO_2 and CH_4 sorption values and the permeabilities and ideal selectivities of the membranes for these gases.

5. Gas sorption and separation properties of polymerized ionic liquid membranes

5.1 Experimental

Materials

High purity nitrogen (N₂), hydrogen (H₂), oxygen (O₂), carbon dioxide (CO₂) and methane (CH₄) gases (99.99%) were provided by Messer Industriegase GmbH (Germany).

To support the ionic liquids Durapore porous hydrophobic polyvinylidene fluoride (PVDF) membrane was used. It has a pore size of 0.22 µm, porosity of 75%, average thickness of 150 µm and was purchased from Millipore Corporation (USA). 1-ethyl-3-methylimidazolium trifluoromethanesulfonate and triethylsulfonium bis(trifluoromethylsulfonyl) imide ionic liquids were purchased from Solvent Innovation GmbH, trihexyltetradecylphosphonium bromide, 2-hydroxyethyl-ammonium formate, 1-allyl-3-methylimidazolium bis(trifluoromethylsulfonyl) imide and 1-allyl-3-methylimidazolium dicyanamide were procured from IoLiTec GmbH & Co. KG. Vinylimidazole, ethyl-bromide, butyl-bromide, trichloro acetic acid, acrylonitrile and ethanol were provided by Sigma Aldrich Co., sodium dicyanamide and sodium thiocyanate by Fluka and dimethyl sulfoxide by LACH-NER, s.r.o. Asobisisobutironitrile and hydroquinone were synthesized at Institute of Macromolecular Chemistry AS CR, v.v.i. (IMC) Prague, Czech Republic.

Preparation of polymerized ionic liquid membranes

Monomer synthesis

The synthesis of the two different monomers, 1-vinyl-3-ethyl-imidazolium bromide and 1-vinyl-3-butyl-imidazolium bromide, was carried out in the following way.

In the case of 1-vinyl-3-ethyl-imidazolium bromide first 1.5 moles of vinylimidazole was poured in a 500 ml flask then few grams of hydroquinone was added in order to prevent polymerization and then it was stirred and heated. When its temperature reached 70°C the previously measured 1.4x1.5 moles of ethyl bromide was slowly added to the vinylimidazole drop by drop so that the temperature of the mixture stayed between 70°C and 110°C. Since it is an exothermic reaction the fast decrease in temperature indicates the decrease in reaction rate and in this case the rate of dripping was increased. When all ethyl bromide was added the mixture was heated up to 150°C and was stirred for another 60 minutes so that the reaction can be completed. In order to crystallize the brownish coloured monomer product acetone was added to the solution and the product crystals were dried in a vacuum oven to constant weight at the temperature of 20°C.

The synthesis of 1-vinyl-3-butyl-imidazolium bromide was similar to the synthesis above described with the exception that 1.2x1.5 moles of butyl bromide was slowly added to 1.5 moles of vinylimidazole.

Copolymerisation and membrane preparation

1-vinyl-3-ethyl-imidazolium bromide and acrylonitrile were used to produce copolymer and to prepare membrane from this copolymer (VEIMBrAN).

5 g 1-vinyl-3-ethyl-imidazolium bromide and 0.1 g asobisisobutironitrile initiator were dissolved in 90 g dimethyl sulfoxide and 5 g acrylonitrile was added with constant stirring. In order to prevent homopolymerization initiated by oxygen carbon-dioxide was bubbling through the produced yellow solution for a few minutes. Then the solution was kept at the temperature of 70°C for 24 hours so that the polymerization reaction can be finished.

After filtration some of the solution was cast onto a flat glass plate and was kept at 70°C for 24 hours and then at 130°C for 1 hour for complete solvent evaporation. The glass plate was placed into water-acetone (50-50 w/w%) solution and the membrane film was removed carefully and dried further in the vacuum oven at 40°C for 24 hours to remove all detectable traces of the casting solvent.

Homopolymerization and membrane preparation

After the anion of 1-vinyl-3-butyl-imidazolium bromide was exchanged using sodium dicyanamide, trichloro acetic acid and sodium thiocyanate, homopolymerization reactions were carried out and membranes were prepared from the polymers.

85 g, 59 g and 88 g crystal 1-vinyl-3-butyl-imidazolium bromide monomer were dissolved in 100-100 ml distilled water with constant stirring, then 8 g sodium dicyanamide, 11 g trichloro acetic acid and 8 g sodium thiocyanate were added to the three solutions respectively. The brownish coloured products were washed with distilled water several times then were dried at 37°C for 24 hours to evaporate all the water and then the polymer products, 1-vinyl-3-butyl-imidazolium dicyanamide (VMIMCN₂), 1-vinyl-3-butyl-imidazolium trichloride (VMIMCl₃) and 1-vinyl-3-butyl-imidazolium thiocyanate (VBIMTCN) were formed.

Two grams of the polymers were dissolved in ethanol and cast onto a flat glass plate then kept at 40°C for 24 hours for membrane film formation.

Gas sorption measurements

The sorption measurements are based on the gravimetric method, which is a well established technique for obtaining sorption of pure gases. In this procedure the total amount of adsorbed and absorbed gas is measured at constant temperature with a microbalance while increasing the gas pressure from zero to a final pressure.

The experimental set-up consists of several units; the most important ones are the sorption balance and the computer used for data storage. The two scale beams, the left one holding a known mass and the right one holding the sample, are situated in glass cases, which can be evacuated using a vacuum pump or filled with gas at given pressure using a gas inlet pipe.

Prior to the determination of gas sorption the sample was degassed for 24 hours at room temperature exposing it to vacuum in order to remove all of the absorbed or adsorbed species from the membrane. After desorption the sorption of N₂, H₂, O₂, CO₂ and CH₄ was measured in the following way. The gas was let into the glass cases at given pressure and gas sorption took place for 2 hours then the glass case including the sample was degassed and the gas was let inside. This was repeated in the case of all membrane samples and gases at the pressure of 8 kPa, 16 kPa and 20 kPa respectively.

Gas permeation measurements

Permeability of all PILMs were determined for H₂, N₂, O₂, CO₂ and CH₄ in a self-designed experimental set-up described in our previous work, with an initial driving force of 2 bar and at constant temperature of 30°C, which was maintained with the help of a thermostat (Cserjési et al., 2010). The whole separation system, was vacuumed before the feed gas entered the membrane module then the gas phase pressure was set and with opening one of the cells the gas permeation started. After eight hours of gas permeation from the pressure data the change in the gas volume was determined and permeability and ideal selectivity values were calculated.

5.2 Results and discussion

The N₂, H₂, O₂, CO₂ and CH₄ sorption results for the four PILMs are shown in Table 3.

membranes	pressure (kPa)	Gas sorption (g/g)				
		nitrogen	hydrogen	oxygen	carbon-dioxide	methane
VEIMBrAN	8	0	0.05	0	0.05	0
	16	0	0.15	0	0.1	0
	20	0.05	0.2	0.35	0.35	0.15
VMIMCN ₂	8	0	0	0	0	0
	16	0	0	0	0	0.08
	20	0	0.03	0.03	0.08	0.23
VMIMCl ₃	8	0	0	0	0	0
	16	0	0	0.007	0	0.14
	20	0	0.14	0.14	0	0.28
VBIMTCN	8	0	0	0	0	0
	16	0.07	0.14	0.07	0.07	0.07
	20	0.14	0.27	0.14	0.21	0.14

Table 3. Gas sorption of the membranes

All of the PILMs had slightly different but extremely low gas sorption, the highest values are measured in the case of O₂ and CO₂ and the lowest ones in the case of N₂ gas. While the VEIMBrAN membrane exhibits the highest sorption followed by the VBIMTCN membrane, the gas sorption increases with the increase in pressure in all cases.

Table 4 contains the N₂, H₂, O₂, CH₄ and CO₂ permeabilities and the ideal selectivities of the investigated PILMs.

	VEIMBrAN	VMIMCN ₂	VMIMCl ₃	VBIMTCN
permeability (Barrer)				
N ₂	0.28	0.18	0.23	0.32
H ₂	3.1	1.9	2.8	3.8
O ₂	4.8	2.3	3.2	4.1
CO ₂	11	10.5	13	17
CH ₄	2.3	1.8	1.5	2.7
ideal selectivity				
H ₂ /N ₂	11	11	12	12
H ₂ /CH ₄	1.3	1.1	1.9	1.4
O ₂ /N ₂	17	13	14	13
O ₂ /CH ₄	2.1	1.3	2.1	1.5
O ₂ /H ₂	1.5	1.2	1.1	1.1
CO ₂ /N ₂	39	58	57	53
CO ₂ /CH ₄	4.8	5.8	8.7	6.3
CO ₂ /H ₂	3.5	5.5	4.6	4.5
CO ₂ /O ₂	2.3	4.6	4.1	4.1
CH ₄ /N ₂	8.2	10	6.5	8.4

Table 4. Permeability and ideal selectivity values of the PILMs

All PILMs have the highest permeability values for CO₂ and the lowest for N₂ and the membrane permeabilities vary in the range of 0.18-0.32 Barrers for N₂, 1.9-3.8 Barrers for H₂, 2.3-4.8 Barrers for O₂, 1.5-2.7 Barrers for CH₄ and 10.5-17 Barrers for CO₂ respectively. VMIMCN₂ membrane has the lowest and VBIMTCN membrane the highest permeability for most of the gases tested. Although, the PILMs show low gas sorption they have relatively high permeability and selectivity values, it can be seen that higher permeabilities belongs to higher gas sorption values. Plotting the performance of the membranes for CO₂/N₂ gas pair on a Robeson plot diagram it can be said that these PILMs perform almost as well as the commercially used polymeric membranes.

In the future PILMs will be prepared based on novel kinds of ionic liquids containing different substituents and their gas permeability and selectivity values will be determined using N₂, H₂, O₂, CO₂, CH₄ and H₂S single gases, binary, tertiary and quaternary gas mixtures.

6. Conclusion

Due to the special features of ionic liquids, such as high thermal and chemical stability, low vapour pressure, non flammability, tunable physicochemical properties, etc. make them perfect candidates for the substitution of organic membrane phase in liquid membranes. Bulk-, emulsion- and supported ionic liquid membranes have higher stability, permeability and selectivity values than conventional liquid membranes and were successfully used in liquid-liquid extraction, microextraction, pervaporation, vapour- and gas separation processes.

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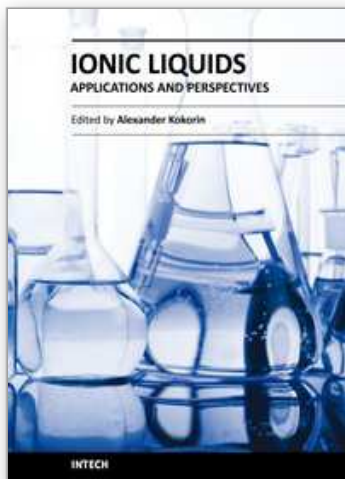
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This book is the second in the series of publications in this field by this publisher, and contains a number of latest research developments on ionic liquids (ILs). This promising new area has received a lot of attention during the last 20 years. Readers will find 30 chapters collected in 6 sections on recent applications of ILs in polymer sciences, material chemistry, catalysis, nanotechnology, biotechnology and electrochemical applications. The authors of each chapter are scientists and technologists from different countries with strong expertise in their respective fields. You will be able to perceive a trend analysis and examine recent developments in different areas of ILs chemistry and technologies. The book should help in systematization of knowledges in ILs science, creation of new approaches in this field and further promotion of ILs technologies for the future.

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