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Separation of fermentation products from ABE mixtures by perstraction using hydrophobic ionic liquids as extractants



G. Merlet, F. Uribe, C. Aravena, M. Rodríguez, R. Cabezas, E. Quijada-Maldonado, J. Romero*

Laboratory of Membrane Separation Processes (LabProSeM), Department of Chemical Engineering, University of Santiago de Chile (U. de Santiago), Chile

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ABSTRACT

In this work, the perstraction process was implemented to separate butanol, acetone and ethanol from aqueous solutions using four different commercial hydrophobic ionic liquids (ILs) as receiving phase: $[bmin][PF_6]$, $[bmim][Tf_2N]$, $[omim][Tf_2N]$ and $[P_{6,6,6,14}][DCA]$. These ILs constituted by different cations and anions were selected in terms of its hydrophobicity, good solubility of butanol and commercial availability.

Perstraction assays were carried out using a symmetric membrane made in polydimethylsiloxane (PDMS) in order to quantify the extraction percentage and transmembrane fluxes of butanol, acetone, ethanol and water. The results indicate that the transmembrane fluxes of butanol were particularly high considering that the PDMS membrane used in the experiments was relatively thick (160 μ m). The highest average flux of butanol was obtained using [P_{6,6,6,14}] [DCA] as the extractant reaching a value of 5.5×10^{-3} kg h⁻¹ m⁻². Nevertheless, the IL with the best separation performance was [omim] [Tf₂N] with a low flux of butanol (4.3×10^{-3} kg h⁻¹ m⁻²) but with the highest butanol/water selectivity value equal to 64.25.

This perstraction technique combined with ILs could allow to design a wide range of separation processes to purify a large variety of molecules. Besides that, the perstraction process could be considered a good alternative for the selective separation of fermentation or reaction products with high commercial value.

1. Introduction

Biobutanol is a second generation biofuel with chemical properties very similar to gasoline, and better combustion properties than other low-carbon alcohols like ethanol. Among these specific properties, butanol stands out for its high energy content, which is similar to gasoline and higher than ethanol; meanwhile, its vapor pressure is lower than ethanol, being less volatile and easier to handle. Moreover, butanol is less hygroscopic and less corrosive than ethanol. Thus, biobutanol can be considered as a better candidate for distribution through the existing infrastructure and its use in gasoline engines [1]. ABE (Acetone-Butanol-Etanol) fermentation is a well-known process, which has been mainly used to produce butanol and acetone since the beginning of the 20th century [2]. This bioprocess uses a strain of bacteria of *Clostridium* family for the fermentation of different types of biomass feedstocks, e.g. starch rich, sugar rich of lignocellulosic materials [3].

The ABE fermentation produces mainly three products in a 3:6:1 ratio which are acetone, butanol and ethanol, respectively [4].

One of the main drawbacks in the production of butanol from a biotechnological process is the low concentration obtained in the

fermentation broths, since the generated compounds are toxic for the most common *Clostridium* bacteria. In this way, the presence of these products in relatively low concentrations (< 40,000 ppm) inhibits the fermentation process [5]. An additional problem associated to this low concentration of the ABE products in the fermentation broth is related to the great energy consumption required for the selective separation of each compound from the aqueous solution.

There is a vast body of literature on different separation technologies to recover alcohols from fermentation broths such as adsorption [6], membrane distillation [7], vacuum flash distillation [8,9], gas stripping [10], pervaporation [11,12], or liquid-liquid extraction [13]. However, these techniques are not free of several drawbacks such as high energy requirements, toxicity of solvents, loss of extractant agents, need of sterilization, adsorption of microorganisms on the surfaces, among others. From these separation technologies, perstraction arises as a viable process because of a moderate energy requirement and the indirect contact between the feed solution and the extractant phase.

This work deals with the use of a perstraction process for the selective separation of butanol from aqueous mixtures containing the three main ABE fermentation products where a polymeric membrane separates two streams: the first, an aqueous feed solution containing

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^{*} Corresponding author. E-mail address: julio.romero@usach.cl (J. Romero).

acetone, butanol and ethanol in the same concentration ratio of typical ABE fermentation broths and the second, a hydrophobic ionic liquid [14]. The principle of this operation combines the permeation with the extraction process. Thus, butanol as the compound of interest is transferred from an aqueous phase through a dense polymeric membrane to an organic phase or so called extractant where the butanol is absorbed. Perstraction has been already studied as an alternative separation technique for fermentation products where the coupling of this membrane technique to a bioreactor could increase the yield because of the constant removal of products [15]. In this case, ionic liquids could be considered as alternative solvents, which show negligible vapor pressure, are non-flammable and can be highly hydrophobic [16]. Several authors have already reported the use of ionic liquids as solvents in liquid-liquid extraction operations [17-19]. Ha and coworkers [20] performed these tests to extract butanol from aqueous solutions finding the influence of the anion of the IL on its hydrophobic character. Thus, an extraction efficiency equal to 74% was obtained when [omim][Tf₂N] was used as extractant.

In this study, the selectivity of the membrane is combined with the use of a hydrophobic ionic liquid in order to recover butanol from a fermentation broth. This separation process could be proposed as a first separation step for the purification of biobutanol in a process with a low energy requirement.

2. Experimental

2.1. Materials and reagents

Acetone, n-butanol and ethanol were supplied by Merck[®]. ABE solutions were prepared by mixing these compounds in ultrapure water obtained from a PureLab[®] classic system.

1–butyl–3–methylimidazolium hexafluorophosphate ([bmim] [PF₆]), synthesis grade, and 1–butyl–3–methylimidazolium bis(trifluoromethylsulfonyl)imide ([bmim][Tf₂N]), synthesis grade, were supplied by Merck[®]; meanwhile, 1-octyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide ([omim][Tf₂N]), purity 99%, and trihexyltetradecylphosphonium dicyanamide ([P_{6,6,6,14}][DCA]), purity 95%, were supplied by IoLiTec[®]. All these ionic liquids were used without further purification.

Table 1 summarizes the chemical structure of the chosen ILs. These compounds were chosen because of their commercial availability, high purity and well-known properties, which have been widely reported in literature. Furthermore, these ILs are hydrophobic in order to reduce



Fig. 1. Experimental perstraction setup used in this work.

the extraction of water, which represents the main component of the feed solution. Finally, from previous works it has been demonstrated a high solubility of butanol in the selected ionic liquids [1].

On the other hand, PDMS membranes were purchased from Kolm[®], Chile. These membranes were selected from their organophilic character and as one of the most used materials in perstraction membranes [21].

2.2. Perstraction assays

Perstraction tests were carried out in an experimental setup specially designed for this purpose. This perstraction setup is described in Fig. 1 where two streams: an aqueous feed containing acetone, n-butanol and ethanol and other an ionic liquid playing the role of receiving phase of the organic solutes, are separated by a polydimethyl-siloxane (PDMS) membrane with a thickness of 160 μ m, an effective surface area available for mass transfer equal to 152 cm² and a density of 1200 kg m⁻³. This flat sheet membrane was placed in module made of poly(methyl methacrylate) (PMMA). The length, width and depth of the circulation channels on each side of the membrane module are 19 cm, 8 cm and 0.3 cm, respectively.

In this work, four different hydrophobic ionic liquids described in Table 1 were tested as receiving phase to assess the effect of different cations and anions on the extraction capacity of this process. The main

Table	1
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Ionic liquids used as extractant phase in this work: nomenclature and structural formula.

Name	Nomenclature	Cation	Anion
1-butyl-3-methylimidazolium hexafluorophosphate	[bmim][PF ₆]	R _ N _ CH ₃ R=C ₄ H ₉	F F F F F F
1-butyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide	[bmim][Tf ₂ N]	R R = C ₄ H ₉	F ₃ C CF ₃
1-octyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide	[omim][Tf ₂ N]	R _ N _ CH ₃ R=C ₈ H ₁₇	F ₃ C CF ₃
trihexyl(tetradecyl)phosphonium dicyanamide	[P _{6,6,6,14}][DCA]	C ₆ H ₁₃ H ₁₃ C ₆	N-CN

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