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Desolventizing organic solvent-soybean oil miscella using ultrafiltration ceramic membranes



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ABSTRACT

This work reports the use of ceramic membranes with different cut-offs (5, 10 and 20 kDa) for the separation of synthetic mixtures simulating soybean oil industrial miscellas in organic solvents (n-hexane, ethanol and isopropanol). The mass ratios oil/solvent investigated in this work were 1:4, 1:3 and 1:1 (w/w) for the feed pressures of 0.5–4 bar depending on the miscella. It is shown that n-butanol was the best solvent for the proper conditioning of 20 kDa membrane, since it increased permeate flux of n-hexane, up to 314 L/m^2 h at 1 bar of transmembrane pressure. The desolventizing of oil/solvent mixtures was strongly affected by solvent nature, and on the solute–solvent–membrane affinity. The highest retentions were observed for oil/ethanol mixtures, with values usually close to 100%, as a consequence of polarity as well as low solvation power. Crude oil mixture with n-hexane (industrial mixture) yielded greater retention and lower flux than those obtained with refined oil, due to the polarized layer formed by gums and phospholipids. Results reported in this work indicate the potential applicability of this technology in vegetable oil processing and biodiesel industries in the solvent recovery step.

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1. Introduction

The projections of world population growth in addition to the limited area for food production have put forward a maximization scenery and valorization of food chain market. Within such segment, soybean has become undoubtedly one of the major world commodities. Although the production process of soybean oil has reached desirable levels, the soybean oil extraction system has been based on an old process with technical developments mostly focused on production control systems [1].

The conventional extraction process is based on the oil extraction by organic solvent (n-hexane), and its separation through solvent evaporation/distillation involves enormous energy costs. According to Johnson and Lusas [2], the solvent separation from the mixture by distillation is so costly, that if the whole extraction industry is analyzed, only the separation step is responsible for 2/3 of the total energy demand by the industry. Other studies report

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the substantial energy savings achieved by application of membrane separations in oil industry [3]. Then, it seems that a huge energy economy would be possible by making use of membrane technology in this kind of industry.

Despite appearing revolutionary at first glance, membrane technology is commercially used for more than six decades, being born in 1950 for the desalinization of water from the sea. Many advantages of membrane technology can be envisaged, such as its robustness, easy installation with room saving, energy economy, the high selectivity reached, operation and scale-up. Furthermore, low temperature processing may be a great advantage for thermosensitive compounds affording products of higher quality, with lesser sensorial and nutritional changes, and of course significant reduction in energy costs [4].

The first report on membrane separation for oil/solvent mixtures was focused on using polymeric membranes, which present as main disadvantages inherent incrustation, plasticization and swelling when in contact with solutes and organic solvents, hence leading to the reduction of its industrial life time [5–12]. Ceramic membranes, in spite of the higher initial cost, present a great potential application in the separation of solutes from non-aqueous solutions, since the interactions of the solutes and solvents with the

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membrane ceramic material are much smaller when compared with polymeric membranes [13–15]. Such characteristic increases membrane stability and, consequently, provides longer shelf life. However, recent studies have shown that solute–solvent–membrane interactions play an important role even when ceramic membranes are used in non-aqueous permeations [16–18].

The use of alternative solvents to the conventional n-hexane seeks a more economical and sustainable system, since n-hexane comes from fossil reserves and tends to increase prices with the unavoidable depletion of natural petroleum reserves. Besides the price uptrend in coming decades [19], n-hexane is a poisonous solvent, which generates great interest in its substitution for less harmful solvents and eco-friendly compounds, following the tendencies of the concept of Green Chemistry [20].

In this sense, several other solvents can be used for soybean extraction just varying some process parameters. Among some potential solvents, ethanol, isopropanol and their azeotropes have been proposed as alternatives to n-hexane in the vegetable oil extraction industry, in order to reduce the environmental risks while affording a healthier final product [21]. Darvishmanesh et al. [22] and Kwiatkowski and Cheryan [23] proposed the use of renewable solvents for the soybean and corn oil extraction, respectively, and the solvent recovery using commercial nanofiltration polymeric membranes. These authors presented interesting results proving that the membrane technology can be successfully applied in traditional industrial processes for solvent recovery.

Thus, the aim of the present work was to select and explore the application of ceramic membranes in soybean oil separation from conventional solvent (n-hexane) and green solvents (ethanol and isopropanol).

2. Material and methods

2.1. Material and reagents

The commercial refined soybean oil used in the assays was purchased at the local market. The industrial mixture was kindly provided by a soybean oil extraction industry (Erechim, RS, Brazil) and comprised of crude soybean oil in n-hexane (1:4). The solvents used in the experiments had a minimum purity of 95%. The ceramic membrane of 20 kDa and its stainless steel membrane module were purchased from the Jiangsu Jiuwu Hitech Co. (Jiangsu, China). The membrane has 19 internal channels with diameter of 0.004 m and each membrane has 1.016 m length. The ceramic membranes of 5 and 10 kDa were purchased from Pall Corporation (New York, USA). The membranes are single channel with 0.010 m with 0.25 m length. Table 1 shows the specifications of the membranes used in this work. The choice of the membranes investigated in this work was based on previous experience of our research group [5–9].

In this work, retentate and permeate solutions returned to the feed tank for keeping constant feed concentration. Fig. 1 shows the experimental apparatus used in the present work.

For the temperature control a thermostatic bath was used (MA 083, Marconi, São Paulo, Brazil). The rotameter was manufactured by Conaut (Model 440, São Paulo, Brazil). The gear pump was purchased from Micropump-Idex Corporation (Vancouver, WA, USA).

For the evaporation of the solvents, a vacuum oven was employed (Quimis, Q819v2, São Paulo, Brazil). An analytical balance was used for samples weighing (Shimadzu, AY220), with a precision of 0.005 g.

2.2. Mixtures preparation

The minimum volume of feeding solution was defined based on the needed volume for each membrane unit, taking also into account the necessary sampling. Thus, for the 20 kDa membrane, the feed volume stipulated was 1.5 L, and for both 5 and 10 kDa membranes, 0.25 L was adopted. Preparation of the oil/solvent mixtures was made in mass basis, following the mass ratios established in the study. The following mass ratios in the feed were investigated for ethanol, isopropanol and n-hexane: 1:4, 1:4, 1:3, 1:4, 1:3,1:1, respectively.

2.3. Membrane conditioning

Due to the high polarity exhibited by ceramic membranes it is necessary a conditioning step before using, in order to increase the solvent flux and hence make those membranes suitable for industrial applications. A priori, the conditioning, or pretreatment, involves membrane washing in appropriate solvents to either increase or remove preservatives and humectants from membrane surface and its pores [24].

The 20 kDa membrane had its flux checked and it was the only one submitted to tests with n-hexane. In such case, 20 kDa membrane was immersed in several solvents, which were then exchanged to n-hexane. The pre-conditionings that afforded the highest permeate flux of n-hexane was chosen. Some more recent studies show that the conditioning or pretreatment step of hydrophilic membranes can change its hydrophilic character, allowing an increase in organic solvent flux. Basically, the idea is to expose the membrane to a variety of solvents with decreasing polarities [25].

In the case of the 20 kDa membrane, n-butanol was chosen as preconditioning solvent before n-hexane permeation. Briefly, the module was completely filled with n-butanol at room temperature and the membrane was kept soaked in the solvent for 24 h. After removing n-butanol, 3 rinses with n-hexane were carried out, and then the module was maintained filled with n-hexane for 24 h prior to the assays [7].

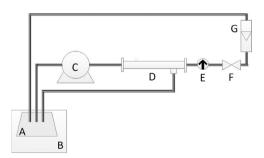


Fig. 1. Schematic diagram of experimental apparatus. A- feed tank, B- thermostatic bath, C- gear pump, D- membrane module, E- manometer, F- valve, G- rotameter.

Membrane description and classification.

Table 1

Class	Membrane material	Manufacturer	MWCO	Permeation area	Model	pH limit
UF	zirconium	Pall Co.	5 kDa	$\begin{array}{c} 0.0055 \ m^2 \\ 0.0055 \ m^2 \\ 0.24 \ m^2 \end{array}$	S700-01446	0-14
UF	zirconium	Pall Co.	10 kDa		S700-01447	0-14
UF	α-aluminia oxide/zirconium	Jiangsu Jiuwu Hitech Co.	20 kDa		CMF 19040-OD30	0-14

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