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SOLVENT RECOVERY FROM SOYBEAN OIL/N-BUTANE MIXTURES USING A HOLLOW FIBER ULTRAFILTRATION MEMBRANE

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Abstract - The aim of this work was the study on the separation of soybean oil/n-butane mixtures using a commercial hollow fiber ultrafiltration membrane (50 kDa). Oil/n-butane mixtures with mass ratios of 1:1 and 1:3 (wt), with the feed pressures of 5, 7 and 10 bar and transmembrane pressure of 1 bar were studied. Rejections of oil between 21 to 97.2%, oil fluxes between 0.04 and 0.98 kg/m^2 h and n-butane fluxes between 4 and 46 kg/m^2 h were observed, strongly influenced by the feed concentration. The increase in oil/n-butane mass ratio caused an increase in oil rejection and a decrease in the permeate flux of oil for most assays. The increase in the operating pressure caused an increase in oil flux and a consequent decrease in oil rejection. No degradation was observed in the membrane module during the operation with this non-aqueous feed stream, as confirmed by integrity tests.

Keywords: Soybean oil; n-butane; Solvent recovery; Hollow fiber; Membrane conditioning.

INTRODUCTION

The processes using membranes have been increasingly adopted by biotechnology and food industries, mainly for clarification of beers, juices and wines, and concentration of milk and proteins. The interest in the application of membrane separations in the processing of vegetable oils to replace some traditional steps has also been growing in the last decade (Baker, 2004; Coutinho *et al.,* 2009; Shahidi *et al.*, 2005). Darvishmanesh *et al.* (2011) discussed the potential advantages of application of membrane process in the vegetable oil industries. They showed that a solvent-resistant nanofiltration membrane (SRNF) can be applied to separate triglycerides from the solvent. The concentrated oil should still be further purified by distillation or evaporation, but huge potential savings are obtained when membranes are used due to the reduction of chemicals and the improved quality of the oil.

In conventional processing of vegetable oils, distillation units operated under vacuum and other auxiliary equipment are used in the process. A possible thermal degradation of oil and an incomplete elimination of n-hexane are the major disadvantages of this technology, besides the large amount of energy used in these processing steps (Reverchon and De Marco, 2006).

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Many other solvents have been proposed for soybean oil extraction, with many advantages and drawbacks. Supercritical carbon dioxide and compressed gases such as propane and n-butane are interesting solvents, since they do not leave residues in the oil or in the cake (Seth *et al*., 2007; Hegel *et al*., 2006; Hegel *et al*., 2007). Most of these solvents, compared to the traditional method of extracting with hexane, do not exceed the extraction yields obtained with n-hexane (18 - 20% oil/kg of soybean) (Hegel *et al*., 2007), except n-butane (unpublished results). However, for solvent recovery the recompression of the gas solvent is required.

The proposal of membrane filtration as an alternative for distillation is indeed not optimal. A combination of membrane filtration and distillation as an alternative for the combination of a desolventizer/toaster and classical distillation might be even more advantageous and should be investigated, in view of the suggested advantages of hybrid processes (Darvishmaseh *et al*., 2012; Suk and Matsuura, 2006).

Membrane separation processes have advantages over conventional processes, including energy savings, selectivity, the possibility of separation of thermolabile compounds, ease of system operation and a relatively easy scale up from the lab to the industrial scale (Shahidi *et al*., 2005). When liquefied gases are employed in oil extraction, the use of membrane separation processes will minimize the costs associated with gas recompression.

In addition, since membrane processes can be conducted at low temperatures, the heat sensitive oil components of technological interest, such as natural antioxidants, can be preserved. A more stable product and thus with better quality can be obtained by using membranes (Subramanian *et al*., 2004; Ribeiro *et al*., 2008).

Depending on the kind of mixture to be permeated, a pre-treatment step might be necessary. Among the main effects of pretreatment we can point out the effect of clustering in hydrophobic and hydrophilic sites in the surface layer of the membrane (Hilal *et al*., 2004); removal of preservatives, wetting of the membrane surface and pores (Raman *et al*., 1996; Ribeiro *et al*., 2006); and improvement of the permeate flux, without significantly affecting the rejection (Arora *et al*., 2006; Marenchino *et al*., 2006). Organic solvents can be used to improve the polarity of the membrane (the membrane becomes more polar or nonpolar) (Marenchino *et al*., 2006) or to prevent pore closing in some membranes when they are placed in contact with a mixture of hydrocarbons (García *et al*., 2006). The interactions between the membrane and the solvent can be expected with changes in solvent properties such as dielectric constant, molecular size, dipole moment and Hildebrand solubility parameter (Machado *et al*., 1999).

In this context, the objective of this study was to investigate the separation of soybean oil/n-butane mixtures using a hollow fiber ultrafiltration membrane, testing the effect of pre-treatment on membrane permeability to n-hexane. This study is part of two studies of new technologies for extraction of essential and vegetable oils based on pressurized and liquified gases (Oliveira *et al.,* 2012; Novello *et al.,* 2013; Tres *et al.,* 2009b), and the use of the membrane separation process for recovery of solvents used in such extraction processes (Oliveira *et al.,* 2011; Tres *et al.,* 2009a, 2010, 2012a and 2012b). The final purpose of these works is the application of this technology to aid solvent recovery in oil extraction plants.

MATERIALS AND METHODS

Membrane and Experimental Apparatus

Permeation assays were performed with soybean oil/n-butane mixtures using a hollow fiber UF membrane module. The hollow fiber UF membrane module was supplied by PAM Membranas Seletivas Ltda (Rio de Janeiro, Brazil). The membrane material is poly(ethersulfone)/poly(vinylpyrrolidone) (PES/PVP) with A nominal molecular weight cut off (MWCO) of 50 kDa and a nominal area of 0.043 m^2. Typical water flux at 1 bar of these membranes is 120 $kg/m^2.h$. The n-butane was purchased from White Martins S.A. (99.5% purity in the liquid phase). Commercial refined soybean oil was purchased in the local market (Soya, Bunge Alimentos S.A., Brazil). Phase equilibrium diagrams and physical-chemical characteristics of the n-butane/oil system are presented elsewhere (Ndiyae *et al*., 2006).

Figure 1 presents the schematic diagram of the experimental apparatus designed to carry out the assays with soybean oil/n-butane. The system was operated in continuous mode, in which the streams of soybean oil and n-butane were mixed at the inlet of the module with a static "J" mixer (Valco), and samples were collected at intervals of 10 min. Coriolis mass flow meters (Quantim - Brooks Instrument, model QMBM) with digital display (Brooks Instrument, model 0254) were installed in the permeate and retentate outlets for the mass flow quantification, so as to allow mass balance calculations for the entire system. Throughout this work a feed mixture flow rate of 1 L/min was adopted. Also, 30 min was employed for system stabilization, followed by 60 min for each assay.

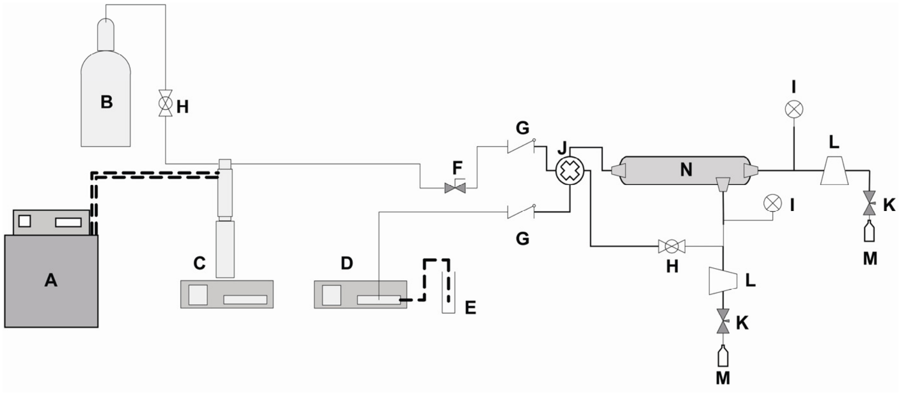

Figure 1: Schematic diagram of the experimental apparatus used in separations of soybean oil/n-butane: A - thermostatic bath, B - n-butane cylinder, C - syringe pump, D - liquid pump, E - sample flask, F - micrometric valve needle, G - check valve, H - ball valve, I - analogue pressure indicators, J - static mixer, K - micrometric needle valves, L - flow meters; M - glass collectors and N - membrane separation module.

Membrane Conditioning

Preliminary tests (n-hexane, Vetec, Rio de Janeiro, Brazil) showed that a membrane-conditioning step was necessary, since the flux obtained was much lower than the nominal water flux of the module. This behavior was attributed to the hydrophilic characteristic of the membrane, due to the presence of PVP on its surface (Simone *et al.*, 2010). Thus, pre-treatments were tested for conditioning the membrane before the permeation assays of oil/solvent mixtures, based on the group's experience and literature data (Darvishmanesh *et al.*, 2009). The solvents tested in the membrane conditioning were ethyl alcohol, n-propyl alcohol, and n-butyl alcohol. All solvents were analytical grade (>99%, Vetec).

First, the ultrapure water (Milli-Q, Millipore, São Paulo, Brazil) flux was compared to the data reported by the manufacturer. For solvent exchange, the excess of the previous solvent was removed from the module, pump and tubing, and the system was washed with 600 mL of the new solvent three times. In the last washing step the solvent was left in contact with the module for 24 h. This latter procedure was not carried out with ethanol, since the manufacturer reported that the epoxy glue was unstable in prolonged contact with this alcohol. All steps were carried out at 25 °C.

Oil/n-Butane Mixtures Separation

After membrane conditioning, the separations of refined soybean oil/n-butane mixtures were carried out with the oil/n-butane mass ratios of 1:1 and 1:3 and at feed pressures of 5, 7 and 10 bar at a transmembrane pressure (TMP) of 1 bar. The assays were started by filling up both sides of the membrane module with liquid n-butane with the “K” valves closed and “H” valve opened. When the module was completed with n-butane, the oil and n-butane flow rates were adjusted in the pumps to achieve the pre-established ratios and the “H” valve then closed. After adjusting the feed pressure and the transmembrane pressure with the help of “K” valves, the system was operated for 30 minutes before sample collection. Samples of the retentate and permeate were periodically withdrawn and placed under vacuum at 65 °C for approximately 24 h up to constant weight to remove the residual n-butane. The permeate flux was calculated by dividing the mass of permeate by the time of sample collection and membrane area. The rejection coefficient was calculated according to:

$$R = \left(1 - \frac{C_p}{C_F}\right).100 \tag{1}$$

where: C_p is the concentration of oil in the permeate and C_F is the concentration of oil in the feed stream.

All separation assays were carried out in duplicate at 25 °C.

Module Integrity Tests

The integrity of the module was measured periodically to check for damage to the fibers, using a procedure suggested by the manufacturer. The solvent was removed from the shell and bore side of the module. The permeate and retentate outlets were then sealed, and nitrogen gas was forced through the feed side (shell) up to a transmembrane pressure of 0.5 bar. The nitrogen cylinder valve was then closed and pressure was monitored for 5 minutes. If the feed pressure drop was higher than 0.1 bar, the module was no longer suitable for use, and then discarded.

RESULTS AND DISCUSSION

Membrane Conditioning

The water permeability determined experimentally, 120 kg/(m^2.h.bar), was close to the value reported by the manufacturer (111 kg/(m^2.h.bar)). The alcohol and n-hexane fluxes obtained during and after membrane conditioning are presented in Figures 2 and 3, respectively.

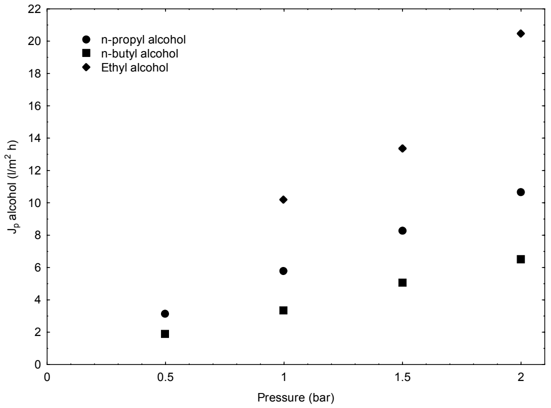

Figure 2: Permeate fluxes of n-propyl alcohol, n-butyl alcohol and ethanol obtained in the membrane-conditioning step.

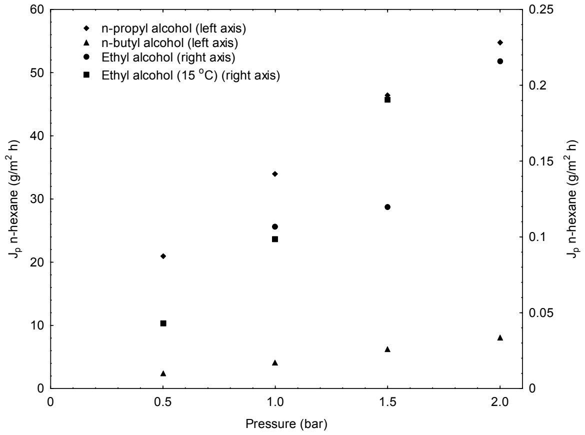

Figure 3: Permeate fluxes of n-hexane obtained after permeation of n-propyl alcohol, n-butyl alcohol and ethanol at room temperature and 15 °C.

The alcohol permeability is inversely proportional to the alcohol chain length. This fact may be explained by the increase in viscosity with the increase in the number of carbons of the alcohol (viscosities of ethyl, n-propyl and n-butyl alcohol are 1.26; 1.94; and 3.0 mPa.s, respectively), in accordance with Darcy's law (Lide, 1999).

Although ethyl alcohol permeability was the highest of the alcohol series, n-hexane fluxes were higher when the membrane was pretreated with n-propyl alcohol. This effect might be due to the modification of the membrane surface polarity caused by the alcohol permeation. n-Propyl alcohol is slightly less polar than ethyl alcohol (dipole moments: ethyl alcohol 1.69 D, n-propyl alcohol 1.68 D, n-butyl alcohol 1.63 D), and immersion in less polar solvents may cause agglomeration of hydrophobic and hydrophilic sites on the membrane surface (van der Bruggen *et al.*, 2002). Consequently, membranes may have their hydrophilicity reduced and the hexane flux could be increased. However, the use of a less polar solvent like n-butyl alcohol does not seem to cause an increase in n-hexane membrane flux, hence demonstrating the complexity of the interfacial phenomena involved in this process.

The use of n-hexane for the flux measurements was due to the fact that this solvent is commonly used in the conventional extraction process and is chemically similar to n-butane.

Oil/n-Butane Mixture Separation and Module Integrity Tests

Figure 4 shows the results of the soybean oil rejection obtained for the separation of refined soybean oil and n-butane. The results for both mass ratios (1:3 and 1:1) are presented, together with those for the feed pressures of 5, 7 and 10 bar, at a transmembrane pressure of 1 bar. Experimental deviations for oil rejection, n-butane and oil permeate fluxes from duplicate assays were smaller than 10% for all experimental conditions investigated.

With the feed mass ratio oil/n-butane of 1:3, the highest oil rejection at 1 h of assay (48.2%) was obtained at the operating pressure of 7 bar, with a slight rejection increase over time. Rejections of oil between 21 and 35.1% were observed for the feed pressures of 5 and 10 bar respectively. The 1:3 mass ratio provided a lower oil rejection compared to the ratio of 1:1 (wt). Under this condition, the oil aggregates are probably smaller, hence promoting the permeation of the mixture. For the mass ratio of 1:1 oil/n-butane, the feed pressure of 10 bar presented the highest oil rejection (97.2%), followed by the pressures of 7 bar (82.4 %) and 5 bar (75.3%), respectively.

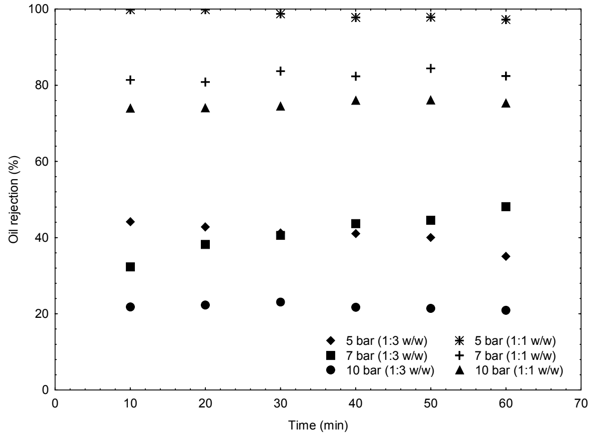

Figure 4: Oil rejections for the hollow fiber membrane at the investigated pressures at a fixed transmembrane pressure of 1 bar.

In a previous work (Tres *et al.*, 2009a) using six flat sheet commercial ultrafiltration membranes in the separation of soybean oil/n-butane, oil rejection and permeate flux results were of the same magnitude as those obtained in this work. The increase in the mixture concentration caused an increase in the oil rejection. A decrease in the total permeate flux with time was observed for the membranes (4 and 5 kDa).

Other authors reported that an increase in rejection is often observed in the separation of vegetable oils containing organic solvents, due to the increase in the polarization layer and fouling on the membrane surface, which increases the resistance to the permeation (Ribeiro *et al.*, 2006; Tres *et al.*, 2009a; Kong *et al.*, 2006; White and Nitsch, 2000; Cheryan, 2005). In this study, the concentration polarization phenomena were a less important issue than the fouling, as described in a previous study (Tres *et al.*, 2010). In that work, regardless of the solvent employed, the isoelectric point of the membrane decreased, as well as the intensity of the negative surface charge. Both changes may be related to membrane fouling by the oil. Characteristic absorption bands of the oil were also found by FT-IR, even after several washes of the membrane with n-hexane, reinforcing the hypothesis of membrane fouling by the oil. The results obtained for oil fluxes are comparable to those reported for polymeric and inorganic ultra- and nanofiltration membranes (Ribeiro *et al.*, 2006; Carvalho *et al.*, 2006).

Moura *et al.* (2007) tested reverse osmosis membranes (BW30 - 4040, DOW) and nanofiltration (DL404 - HL4040 and C - F, Osmonics) poly (amide) (PA) membranes in the permeation of triacylglycerols and free fatty acids/ethyl esters in supercritical CO_2. Feed pressures of 90, 110 and 130 bar were investigated, with TMP's in the range of 7 to 40 bar. All assays were carried out at 40 °C. These authors found that increasing the operating pressure with a TMP fixed at 10 bar led to a reduction in the rejection of triglycerides (between 70 and 100%) assigned to a higher lipid extraction and a loss in selectivity of the extraction. Also, an increase in the pressure caused a reduction in the CO_2 flux (~ 100 to 50 kg/m^2 h) and an increase in the lipid permeation. In our study, the increase in the operating pressure also led to a decrease in membrane rejection and an increase in the oil flux, possibly due to polarization and fouling phenomena.

Results for oil fluxes are shown in the Figure 5. As previously mentioned, the mixtures with the highest amount of solvent (1:3) showed the lowest oil rejection, providing a larger permeate flux of oil when compared with the ratio of 1:1. The feed pressure of 5 bar provided the lower flux of oil, followed by 7 and 10 bar.

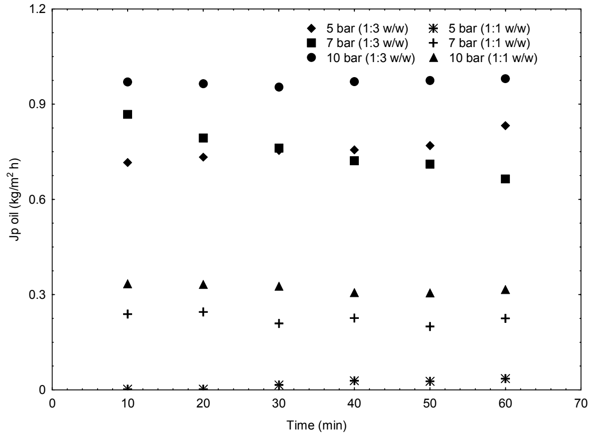

Figure 5: Oil fluxes for the hollow fiber membrane at the investigated pressures and a fixed transmembrane pressure of 1 bar.

In some experimental conditions, with feed pressures of 5 and 10 bar (1:3) and 10 bar (1:1), the oil fluxes increased over time, showing a different behavior when compared with the literature. As noted by Tres *et al.* (2009a), the oil/n-butane system always presented higher rejections of oil when compared with the oil/n-hexane system using the same kind of membranes. This behavior may be related to the coexistence curve of the phase equilibrium of oil/n-butane (Ndiaye *et al.,* 2006). Depending on the feed pressure and the global composition of the system, system operation might be close to the mixture phase coexistence line. Thus, the inherently higher compressibility displayed by the mixture in such a condition, in addition to the possible expansion due to the increased volume of the system due to the presence of pores, may lead to the vaporization of the lighter, much more volatile (compressed) fluid. This might cause the mixture to enter into the biphasic region, causing the separation of phases, dramatically reducing the oil solubility in the light solvent and hence the drag of the solute through the membrane. The membranes used by Tres *et al.* (2009) showed cut offs up to twelve times smaller than the membrane used in this work. However, the results are still similar, reinforcing the hypothesis of gas expansion and pore blocking mentioned previously.

n-Butane permeate fluxes between 4 to 46 kg/m^2 h were obtained for the feed pressures of 5, 7 and 10 bar. The mass balance calculations for the system were performed and deviations less than 8% were obtained for all assays. The module integrity was not affected by the permeation experiments.

CONCLUSIONS

Separation of refined soybean oil/n-butane mixtures using a commercial hollow fiber ultrafiltration membrane (50 kDa) was investigated in this work. Rejections of oil between 21 and 97.2%, oil fluxes between 0.04 and 0.98 kg/m^2 h and n-butane fluxes between 4 and 46 kg/m^2 h were observed, strongly influenced by the feed concentration and feed pressure. The increase in oil/n-butane mass ratio caused an increase in oil rejection and a decrease in the permeate flux of oil in most assays. On the other hand, the increase in the operating pressure caused an increase in oil flux and a consequent decrease in oil rejection. No degradation was observed in the membrane module with operation, as confirmed by integrity tests. It is worth mentioning that the current literature presents just one study regarding the separation of vegetable oil from n-butane by membrane separation processes (Tres *et al.*, 2009a), highlighting the importance of this work in this area.

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