



## Separation of soybean oil/n-hexane and soybean oil/n-butane mixtures using ceramic membranes



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### ABSTRACT

This study aimed to investigate the separations of mixtures of refined soybean oil/n-hexane, crude soybean oil/n-hexane (industrial miscella) and refined soybean oil/pressurized n-butane using membrane technology. Commercial ceramic membranes with molecular weight cut-offs between 5 and 10 kDa were employed, varying the mass ratios of the oil/solvent, from 1:1 to 1:3 (w/w). Oil rejections up to 100%, total permeate fluxes (oil + solvent) up to 42.97 kg/m<sup>2</sup> h with oil permeate fluxes up to 1.4 kg/m<sup>2</sup> h were obtained. The industrial miscella showed the same behavior observed for the synthetic mixtures presenting an increase in the oil rejection with a decrease in total permeate flux. In the separation of the oil/n-butane mixtures, higher oil rejections were obtained when compared to the system oil/n-hexane. The results presented in this work indicate the potential applicability of membrane technology in vegetable oil processing and biodiesel industries in the solvent recovery step.

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### 1. Introduction

Soybean is an oil seed originating from northern and central China (Missão, 2006). Brazil and the United States of America are the major producers of soybeans in the world, but the introduction of this oilseed in these countries was recent (18th and 19th centuries). Soybean is primarily a seed grown for industrial extraction of oil and protein (Erickson & Wiedermann, 1991; Liu, 2000).

In conventional extraction plants of vegetable oils, distillation units, and vacuum-operated and other auxiliary equipment using steam or some other form of heat are employed in the processing of oilseeds. A possible thermal degradation of the oil and an incomplete elimination of the solvent, n-hexane, are the major drawbacks of this technology, compared to the technology that uses pressurized fluids as solvents, in addition to the large amount of energy used in these processing steps (Reverchon & Marco, 2006).

The n-hexane is a relatively inexpensive solvent and is very effective for extracting nonpolar lipids. However, it is highly volatile and is considered to be toxic to animals and humans, even at low concentrations. Vapors of n-hexane should be monitored during industrial operation of extraction, since it can cause explosions, due to the high flammability. Additionally, if the oil and defatted meal are used in foods, complete

removal of the solvent is required (Sparks, Hernandez, Zappi, Blackwell, & Fleming, 2006).

The extraction of oilseeds with high added value using compressed gas may present many advantages over conventional solvent (liquid) solvents. Compared with oil extraction using n-hexane, extraction technology using compressed fluids (gases at ambient conditions) presents as the main characteristics the low temperature used and cost reduction in desolventizing. The advantages of using this technology can be summarized in three main aspects: 1) maximum preservation of oil quality due to low temperature used, and therefore it is more suitable for extraction of oil or high-value crops with modified lipid composition, 2) maximum preservation of the quality of the cake, with high protein content, and 3) reduction of the total investment and utility cost (Xuede & Liu, 2005).

The membrane separation processes often have advantages over conventional separation procedures, which include energy savings in processing, the selectivity of the compounds obtained, the possibility of separation of thermolabile compounds, simplicity of operation of the system and ease of scaling a prototype from laboratory scale to industrial large scale (Habert, Borges, & Nobrega, 2006). The basic parameters for a successful implementation of this technology in oil and fat industry require special attention to the characteristics of feeding the oil/solvent aging/degradation of the membrane, pre-treatment (conditioning), and providing the process conditions, sanitary design, cleaning and disinfection of the membrane (Subramanian, Nakajima,

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Raghavarao, & Kimura, 2004). The energy savings in the application of membrane processes can occur at different stages of the process. The use of steam can be minimized, since the operating temperatures for membrane processes are usually much lower than those required in conventional separations. The greatest potential for reducing power consumption in the oil industry, through the use of membrane technology, is the complete or partial replacement of traditional degumming, refining and bleaching steps. Bleaching and degumming can be combined into a single step, which is efficient in terms of energy expenditure. Reducing the loss of oil and solvents used in the bleaching are other advantages of applying the membrane processing in the manufacture of edible oils. A membrane ideal for solvent recovery should combine specific properties such as high oil retention and permeate streams suitable for industrial scale, as well as heat resistance, and mechanical and chemical compatibility with the process (Köseoğlu & Engelgau, 1990).

Nowadays, companies extracting vegetable oils and producing biodiesel are seeking to maximize energy efficiency in its plants, as well as alternative processes to obtain higher quality oils. Thus, considering these factors is essential to develop new processes for this industrial sector. When pressurized fluids are considered for oil extraction, membrane separation could be advantageously used for minimizing the need of solvent recompression. One of the major limitations for the implementation of this technology in the vegetable oil processing is to find membranes that are stable to oil and organic solvents, with high oil retention, high permeate flux and low tendency to fouling. Thus, the aim of this work was to select and explore the application of ceramic membranes for the separation of soybean oil from mixtures of this oil in n-hexane and in pressurized (liquefied) n-butane.

## 2. Materials and methods

### 2.1. Experimental apparatus for soybean oil/n-hexane separations

Fig. 1 shows the schematic diagram of the experimental apparatus developed for the assays using synthetic and industrial soybean oil/n-hexane miscellas. The membrane technical information and solvents investigated in the conditioning step are presented in Tables 1 and 2, respectively. Transmembrane pressures from 3 to 5 bar and soybean oil/n-hexane mass ratios of 1:1 and 1:3 (w/w) were studied. The

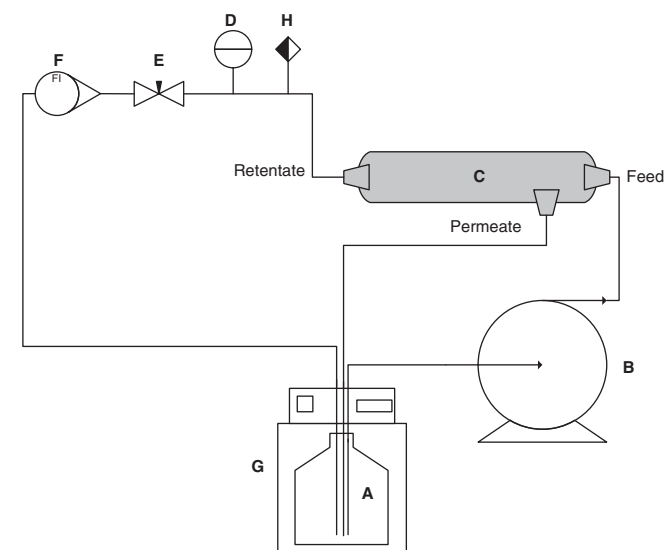


Fig. 1. Schematic diagram of the experimental apparatus used in the separation assays of soybean oil/n-hexane. A – Feed bottle containing the miscella (300 mL), B – gear pump (Micropump, Cole Parmer, Model 72211-15), C – membrane module, D – manometer (Salvi, 0–16 bar), E – micrometric valve (NuPro, model SS-4BK), F – rotameter (Conaut, model 440), G – thermostatic bath (Marconi), H – temperature sensor (PT-100).

soybean oil was purchased in the local market and a biodiesel producer in the region of Erechim, RS, Brazil, supplied the industrial miscella.

### 2.2. Experimental apparatus for soybean oil/n-butane separations

Fig. 2 presents the schematic diagram of the experimental apparatus designed to carry out the assays. The system was operated in continuous mode, where the streams of soybean oil and n-butane were mixed at the entrance of the module with a static mixer (Valco), and the samples were collected at intervals of 10 min. All assays lasted an hour. Coriolis mass flow meters (Quantim – Brooks Instrument, model QMBM) together with its digital display (Brooks Instrument, model 0254AA1B11A) were installed in the permeate and retentate outlets for the mass flow quantification of these two streams. Phase equilibrium diagrams and physical-chemical characteristics of n-butane/oil system are presented elsewhere (Ndiaye et al., 2006; Oliveira, Di Luccio, & Tres, 2011; Oliveira, Di Luccio, & Tres, 2012; Tres, Mohr, Corazza, Di Luccio, & Oliveira, 2009).

### 2.3. Membrane conditioning

Preliminary assays have shown the need of a conditioning step of the ceramic membranes prior to use, since the permeate flux of n-hexane of the untreated membranes was very low (data not shown). This behavior was attributed to the hydrophilic characteristic of membranes. Thus, pre-treatments have been investigated for proper conditioning before separations of oil/solvents.

The experimental procedure for conditioning was started by the permeation of a homologous series of alcohols of different chain lengths (ethyl, n-propyl and n-butyl alcohols). Membrane conditioning was started letting the membrane in contact with the respective alcohol for 24 h. The alcohol flux was measured at transmembrane pressures from 2 to 6 bar. The alcohol was then exchanged for n-hexane and after 24 h the hexane flux was measured.

### 2.4. Assays for separation of soybean oil from n-hexane

After membrane conditioning, separation assays using industrial miscella and synthetic miscellas of refined soybean oil/n-hexane were carried out for oil/n-hexane mass ratios of 1:3 and 1:1 (w/w) and the transmembrane pressures (TMP) of 3, 4 and 5 bar. The pressurization was performed restricting the valve E of the system (Fig. 1). The feed flow rate was 120 kg/h. The flow rate was measured with a flow meter placed at the exit of retentate and converted to mass flux using the density of the miscella measured at the operating temperature in a digital densimeter (Anton Paar, model DMA 4500). All separation tests were performed in duplicate and at 25 °C. Variations in the results were below 10%.

The permeate samples were collected at intervals of 10 min in test tubes. During sample collection, the sample-collecting flask was immersed in an ice bath and immediately after sampling the tube was stoppered to avoid n-hexane losses by evaporation. Samples were then weighed on an analytical balance (Shimadzu, model AY220). The permeate flux was calculated according to the Eq. (1).

$$J_p = \frac{M_p}{T \cdot A} \quad (1)$$

where:  $M_p$  is the mass of permeate,  $T$  is the sampling time and  $A$  is the area of the membrane.

The concentrations of the feed and permeate streams were estimated by measuring the density of the miscella in a digital densimeter at 25 °C using a calibration curve. The rejection coefficient (retention) was calculated according the Eq. (2).

$$R = \left(1 - \frac{C_p}{C_f}\right) \cdot 100 \quad (2)$$

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