

Separation of Sunflower Oil from Hexane by Use of Composite Polymeric Membranes

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Abstract Vegetable oil extraction, as performed today by the oilseed-crushing industry, usually involves solvent extraction with commercial hexane. After this step, the vegetable oil–hexane mixture (miscella) must be treated to separate its components by distillation. If solvent-resistant membranes with good permeation properties can be obtained, membrane separation may replace, or be used in combination with, conventional evaporation. Two tailor-made flat composite membranes, poly(vinylidene fluoride) (PVDF–Si and PVDF–CA) and a commercially available composite membrane (MPF-50), were used to separate a crude sunflower oil–hexane mixture. The effects of temperature, cross-flow velocity (v), transmembrane pressure (Δp), and feed oil concentration (C_f) on membrane selectivity and permeation flux were determined. The PVDF–Si membrane achieved the best results, being stable in commercial hexane and having promising permselectivity properties for separation of vegetable oil–hexane miscella. Improved separation performance was obtained at $C_f = 25\%$, $\Delta p = 7.8$ bar, $T = 30$ °C, and $v = 0.8$ m s⁻¹; a limiting permeate flux of 12 Lm⁻² h⁻¹ and 46.2% oil retention were achieved. Low membrane fouling was observed under all the experimental conditions studied.

Keywords Vegetable oil · Solvent recovery · Membrane separation · Nanofiltration

Introduction

The extraction of vegetable oil from oilseeds involves solvent extraction with an organic solvent, normally commercial hexane. As a result of the extraction process, a mixture (miscella) of oil and hexane (usually 25–35% w/w of oil) is obtained. The miscella undergoes a complex process of distillation and condensation to recover the solvent, which is re-used. Solvent is removed from the miscella by the double effect evaporation and steam stripping [1]. The equipment used in the first stage, called an economizer, is designed to remove most of the solvent and concentrate the miscella as much as possible (70–90% oil). The concentrated miscella is then pumped to the second-stage evaporator (under partial vacuum), where the oil concentration is increased to >99% (<1% hexane). Solvent recovery is of great importance because it affects the global economy of the process, industrial safety, and environmental protection. During the last three decades membrane technology has been accepted in various food processing applications in which its use leads to increased product yield and quality, and with substantial energy savings. It has been estimated that in the USA about two trillion of Btu per year could be saved by using a hybrid membrane system to recover solvents in the extraction of crude oils [2].

Fundamental aspects of membrane separation processes related to degumming, dewaxing, deacidification, solvent recovery, etc., have been recently reviewed [3]. Use of membranes in the vegetable oil extraction industry is currently limited compared with other food industries. Several researchers [4, 5] have reported on the permeation of different families of organic solvents using nanofiltration (NF) membranes. Much work has been reported on the use of membranes in the degumming process and in the separation of undesirable compounds present in vegetable oils

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(phosphatides, waxes, and free fatty acids). Iwama [6] reported the degumming of oil–hexane miscella with a commercial tubular ultrafiltration (UF) membrane. Pagliero et al. [7] evaluated the efficiency of membrane technology for degumming crude soybean oil–hexane miscella by use of a commercial polyamide membrane and a laboratory-made PVDF-based membrane. Raman et al. [8] described a process for solvent recovery and partial solvent (methanol) deacidification from miscella by use of different commercial membranes. A process combining solvent extraction with membrane technology to recover the oil was studied by Kwiatkowski et al. [9]. Separation of oil and solvent from the miscella by the direct use of a membrane process has been the subject of a few studies. The use of different commercial RO/NF membranes for separating cotton oil from hexane, ethanol, and isopropanol was reported by Koseoglu et al. [2]. Only one of these membranes (polyamide) was permeated by hexane without being destroyed. Wu and Lee [10] investigated removal of hexane from a crude soybean oil–hexane mixture by use of porous UF ceramic membranes, and achieved nearly 20% oil retention. Ribeiro et al. [11] evaluated solvent recovery from soybean oil–hexane miscellas, on the bench-scale, by use of flat sheet polymeric commercially available polysulfone and polysulfone–polyamide membranes under different operating conditions. The main objective of this study was to evaluate the efficiency of membrane technology for partial recovery of hexane from a mixture of crude sunflower oil and hexane (miscella).

Experimental

Two tailor-made flat composite membranes, poly(vinylidene fluoride) (PVDF) as support and silicone rubber or cellulose acetate as selective top layer, and a commercially available polyimide membrane were used in different flow arrangements to perform the experimental tests. Membrane permeability to hexane at different temperatures was measured in a dead-end permeation cell. The permeation characteristics of these membranes with crude sunflower oil and hexane solutions were studied in a cross-flow permeation cell. The effects of solute concentration, cross flow velocity, transmembrane pressure, and temperature on membrane flux and oil rejection were analyzed. Performances of the membranes were evaluated in terms of their permeability and oil-retention capability.

Materials

Non-woven Viledon 2431 support was provided by Carl Freudenberg (Weinheim, Germany). PVDF high-viscosity Solef 1015 supplied by Solvay (Brussels, Belgium) and dimethylformamide (DMF) purchased from Aldrich (Buenos Aires, Argentina) were used for preparing an asymmetric

membrane. Analytical reagent-grade chloroform and *n*-hexane were used as supplied. The coating materials were commercial polydimethylsiloxane (Siloc PU-55; Anaeróbico, Buenos Aires, Argentina) and cellulose acetate (Aldrich, Argentina).

Crude sunflower oil was obtained from a local company (Olca Saic, General Cabrera, Córdoba, Argentina) and was used to prepare synthetic miscella solutions. The oil concentration in the oil–hexane miscella samples varied from 15 to 35% (*w/w*).

Membranes

Asymmetric Membrane Preparation

The flat asymmetric membrane was prepared by the phase-inversion process. Polymer solution, 23% (*w/w*) PVDF in dimethylformamide (DMF), was cast on to a non-woven Viledon polymeric flat support, at 25 °C in air (45–50% relative humidity), by using a film extensor with a 400 μm knife gap. After solvent evaporation for 20 s the nascent membrane was immersed in a bi-distilled water coagulation bath ($T = 25$ °C) for 1 h and then stored in fresh water. The asymmetric PVDF membrane was dried at room temperature for 48 h before use. Mean pore radius, $r_p = 0.15$ μm, was determined by the liquid–liquid displacement porosimetry technique (LLDP).

Composite Membrane Preparation

The composite membrane, identified as PVDF–Si, was prepared by coating the surface of the dried asymmetric PVDF membrane twice with a polysiloxane solution (film extensor gap 200 μm, $T = 25$ °C). The coating solution was prepared by dissolving 5% (*w/w*) polydimethylsiloxane in hexane. The cross-linking reaction (acetic cure) of the polydimethylsiloxane coating was accomplished in an oven at 60 °C for 4 h.

The composite PVDF–cellulose acetate membrane (PVDF–CA) was prepared by coating the asymmetric PVDF support surface with a 0.45% (*w/w*) solution of cellulose acetate in chloroform (film extensor gap 200 μm, $T = 25$ °C). The chloroform solvent was eliminated by evaporation under ambient conditions.

Commercial Composite Membrane

The commercial membrane selected for this study was Selro MPF-50, a composite solvent-resistant nanofiltration flat membrane made of polyimide (PI) coated with polydimethylsiloxane (PDMS). It was purchased from Koch Membrane Systems (Wilmington, MA, USA). The

membrane's molecular weight cut-off (MWCO), measured by using water as solvent and based on a 95% solute rejection, was 700 Da. The membrane was supplied preserved in a 50% ethanol solution. This commercial membrane has been widely used in previous research as a solvent-resistant membrane for solvent recovery and solvent partial deacidification from miscella.

The hydrophobic character of the asymmetric PVDF support and the composite membranes was determined by measuring the water-membrane contact angle (θ) by the sessile-drop technique using a contact-angle device (Micromeritics Instrument Corporation, Norcross, GA, USA). Three drops of water were measured for each surface and the average contact-angle values were calculated (Table 1).

Methods

Pure Solvent Permeation

The flux of hexane through the membranes was determined in a dead-end filtration set-up described elsewhere [6].

Table 1 Contact angle (θ) and hexane permeability (L_h)

Membrane	θ	T (°C)	L_h (L m ⁻² h ⁻¹ bar ⁻¹)
PVDF (support)	62 ± 5	30	9.80 ± 0.8
		40	10.70 ± 0.8
		50	12.90 ± 0.9
PVDF-Si	114 ± 8	30	5.02 ± 0.5
		40	5.55 ± 0.4
		50	6.21 ± 0.5
PVDF-CA	56 ± 4	30	0.09 ± 0.01
		40	0.10 ± 0.01
		50	0.15 ± 0.01
MPF-50	116 ± 8	30	3.41 ± 0.2
		40	3.94 ± 0.3
		50	4.22 ± 0.3

Each experiment was carried out in triplicate. The cell reservoir had a capacity of 400 mL and an effective area $A = 3.17 \times 10^{-3}$ m². The membrane was supported on a sintered porous stainless-steel disc. Transmembrane pressure, Δp , was supplied by a nitrogen cylinder connected to the top of the cell. The unit was operated in batch mode by charging the reservoir cell with pure organic solvent, and solvent flux through the membrane was measured as a function of transmembrane pressure ($\Delta p = 4$ –10 bar) at different temperatures ($T = 30, 40,$ and 50 °C). Permeate flux (J) was determined by measuring the permeate solvent volume accumulated during the operation time under steady-state conditions ($\Delta V/\Delta t$) and calculated by use of the equation:

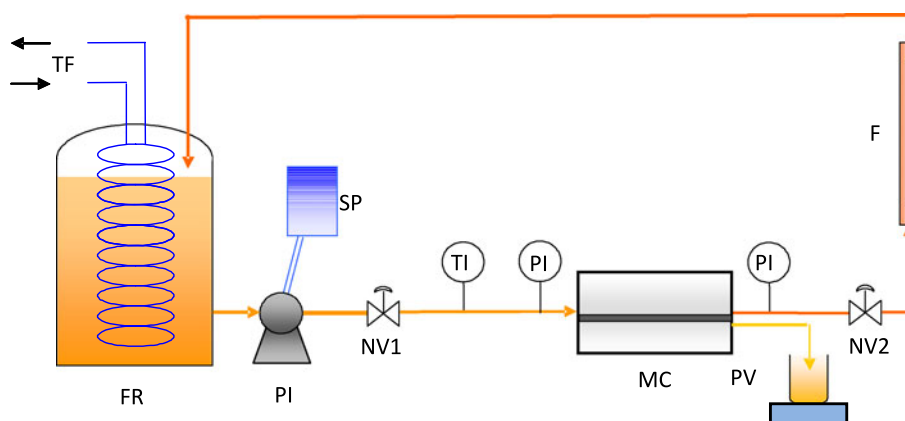
$$J = \frac{1}{A} \frac{\Delta V}{\Delta t} \quad (1)$$

Oil–Hexane Permeation

Permeation of sunflower oil–hexane miscella was performed in a laboratory-scale cross-flow filtration cell (Osmonics/Sepa CF II; Minnetonka, MN, USA). A diagram of the setup is given in Fig. 1. A single piece of rectangular membrane was placed on the permeation cell with an effective transfer area of $A = 0.014$ m². The original miscella solution was stored in a 15-L feed tank with a cooling/heating jacket connected to a temperature-controlled water bath. The feed tank was sealed to prevent solvent evaporation and the feed solution was pumped continuously through the cross-flow cell. Each experimental run was carried out for approximately 1 h with total retentate recycling by means of a centrifugal pump (Grundfos CRN2-220; Fresno, CA, USA). Permeate flux (J) data were determined from permeate volume accumulated versus time and by use of Eq. 1.

The variable conditions investigated in the oil–hexane separation-performance experiments were feed cross flow ($v = 0.8$ and 1.3 m s⁻¹), transmembrane pressure (5.8 and 7.8 bar), operating temperature ($T = 30$ – 50 °C), and oil

Fig. 1 Cross-flow filtration arrangement. *F* flowmeter; *FR* feed reservoir; *MC* membrane cell; *NV1* *NV2* needle valves; *P* pump; *PI* pressure indicator; *SP* speed controller; *TI* temperature indicator; *TF* thermostatic fluid; *PV* permeate volume



feed miscella concentration ($C_f = 15\text{--}35\%$ w/w). The maximum feed velocity and pressure used in the experimental runs were restricted by the operating capacity of the pump. The separation performance was evaluated from permeate flux data and by use of the oil retention factor, %R, defined as:

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

where C_p and C_f are the oil concentrations in the permeate and feed, respectively.

Oil concentrations in the feed and permeate were measured gravimetrically at 75 °C. A known amount of miscella sample was placed in a glass beaker and kept in an oven at 75 °C for 1 h under vacuum. The glass was cooled in a desiccator for at least 1 h to room temperature and weighed with a precision balance (± 0.1 mg). The heating, cooling, and weighing were repeated until constant weight was achieved (difference between two weighings approx. ± 2 mg).

All permeation trials were carried out in triplicate. Membranes were reused after each permeation experiment. Before measurement of initial membrane solvent flux, membranes were cleaned in situ by pumping pure ethanol through the membrane surface for 30 min; pure hexane was then impelled for 1 h.

Results and Discussion

Membrane Solvent Stability and Permeability

Membrane instability can result in unusually high or low fluxes because of crack-like openings on its surface and swelling or shrinking of its structural matrix. To gain a better idea of the stability and durability of the membranes in hexane, their geometry was observed visually after exposure to hexane during 48 h. No significant structural change was observed for any of the membranes; there was slight swelling/shrinkage of the PVDF–Si and MPF-50 membranes (slightly rolled).

Most of the NF membranes were designed for aqueous systems, so, when tested with organic solvents, their permeation behavior is different. Solvent J values were used to evaluate permeability for *n*-hexane, L_h , by use of Darcy's law, as follows;

$$L_h = \frac{J}{\Delta p} = \frac{1}{\eta R_m} \quad (3)$$

where R_m is intrinsic membrane resistance, and η is solvent viscosity. Table 1 shows the solvent permeability data for the membranes obtained from the slope when J is plotted against Δp . This permeability is strongly dependent on both

the hydrophilicity of the membrane and the solvent used. The hydrophilicity/hydrophobicity of the membrane surface and the solvent polarity determine, to a great extent, the flux through the membrane. From the contact angle measurements (Table 1) it can be observed that the PVDF–CA composite membrane was the most hydrophilic, because of the cellulose acetate coating; the PVDF–Si and MPF-50 membranes were more hydrophobic (higher contact angle) because of their active layers prepared from silicone rubber material, favoring permeation of non-polar solvents. This is shown in Table 1—MPF-50 and PVDF–Si permeability to hexane (low-polarity solvent) were

Table 2 Hexane permeability (L_h) of MPF-50 membrane

Ref.	T (°C)	L_h ($\text{L m}^{-2} \text{h}^{-1} \text{bar}^{-1}$)
Raman et al. [12]	25	1.52
Machado et al. [13]	30	28.1
Van der Bruggen et al. [3]	25	18.5–96.0
Darvishmanesh et al. [14]		18.2
This work	30	3.41

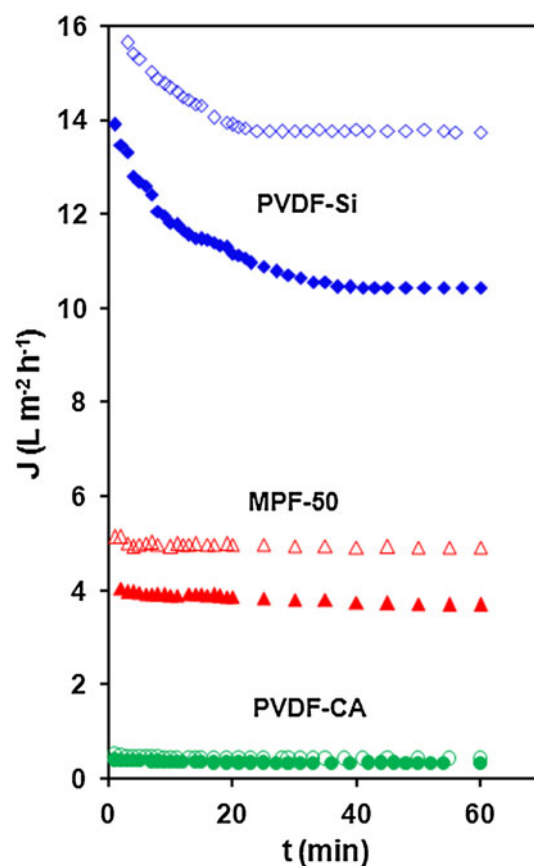


Fig. 2 Effect of transmembrane pressure (Δp) on permeate flux (J) (temperature $T = 50$ °C, feed velocity $v = 0.8$ m s $^{-1}$, and feed oil concentration $C_f = 25\%$ w/w). Open symbols $\Delta p = 7.8$ bar; solid symbols $\Delta p = 5.8$ bar

Table 3 Membrane performance for sunflower oil–hexane miscella: limiting flux (J^*) and oil retention (% R)

Membrane	C_f (% w/w)	T (°C)	Δp (bar)	v (m s ⁻¹)	J^* (L m ⁻² h ⁻¹)	R (%)			
PVDF–Si	25	30	5.8	1.3	9.43 ± 0.32	37.8 ± 1.38			
			5.8	0.8	8.15 ± 0.46	42.5 ± 0.78			
			7.8	0.8	12.0 ± 1.27	46.2 ± 1.20			
		40	5.8	1.3	10.5 ± 1.04	30.8 ± 1.45			
			5.8	0.8	9.5 ± 0.63	36.2 ± 1.06			
			7.8	0.8	12.5 ± 1.35	42.1 ± 1.49			
	15	40	5.8	1.3	12.9 ± 1.13	31.5 ± 1.35			
			5.8	0.8	10.8 ± 0.89	33.2 ± 1.26			
			7.8	0.8	14.3 ± 1.27	36.3 ± 2.26			
		35	40	7.8	0.8	16.5 ± 1.65	39.9 ± 1.65		
			MPF-50	25	30	5.8	1.3	2.20 ± 0.21	30.8 ± 1.45
						5.8	0.8	2.05 ± 0.17	32.5 ± 0.68
7.8	0.8	2.45 ± 0.24				33.8 ± 1.00			
40	5.8	1.3		2.52 ± 0.19	32.9 ± 1.36				
	5.8	0.8		2.23 ± 0.24	35.2 ± 0.82				
	7.8	0.8		3.34 ± 0.38	36.1 ± 1.58				
50	5.8	1.3	3.57 ± 0.56	30.3 ± 0.71					
	5.8	0.8	3.24 ± 0.33	32.1 ± 1.48					
	7.8	0.8	4.34 ± 0.57	32.9 ± 1.78					
15	40	7.8	0.8	6.97 ± 0.70	35.3 ± 0.87				
		PVDF-CA	25	30	5.8	1.3	0.29 ± 0.03	100	
					5.8	0.8	0.35 ± 0.02	100	
	7.8				0.8	0.38 ± 0.04	100		
	40	5.8	1.3	0.31 ± 0.03	100				
		5.8	0.8	0.23 ± 0.02	100				
7.8		0.8	0.39 ± 0.06	100					
50	5.8	1.3	0.25 ± 0.02	100					
	5.8	0.8	0.18 ± 0.03	100					
	7.8	0.8	0.43 ± 0.04	100					
15	40	7.8	0.8	0.52 ± 0.05	100				
		35	40	7.8	0.8	0.11 ± 0.01	100		

between 28 and 56 times higher than that of PVDF–CA. The L_h value of PVDF–Si was 50% higher than that of the MPF-50 composite membrane. The MPF-50 and PVDF–Si membranes have almost identical contact angles (these membranes are chemically very similar). The different fluxes were attributed to the specific characteristics of the supports and possibly to the fact that the surface pores of the PVDF support were not totally covered with coating material. Higher temperatures increased membrane hexane permeability, most likely because of the reduction of solvent viscosity with temperature.

Table 2 compares data on MPF-50 permeability to hexane previously reported in the literature and obtained in our research. Discrepancies can be observed in the

permeability values for the same membrane. These dissimilar values could be because of different pretreatment procedures applied to the membranes before the test and the time it had been exposed to the solvent.

Oil–Hexane Membrane Permselectivity

The experimental runs were specifically designed to study the effects of transmembrane pressure, temperature, feed concentration, and cross-flow velocity on permeate flux. Figure 2 presents the effect of pressure on sunflower oil–hexane miscella permeation flux through the composite membranes at $T = 50$ °C, $v = 0.8$ m s⁻¹, and $C_f = 25\%$ (w/w). It shows the variation of permeate flux with time

during the permeation experiments. There was a moderate drop in permeate flux (around 12–35%) during the first 40 min; it then remained essentially constant during the rest of the test reaching pseudo-steady state (limiting flux, J^*). This behavior implies that the membrane resistance changes during the initial NF process, possibly because of concentration polarization phenomenon and development of an oil gel-layer on the membrane surface. Increasing the pressure from 5.8 to 7.8 bar increased permeate flux. This behavior is typical when permeate flux is controlled by pressure (low pressures, low feed concentrations, and high feed velocities). Similar flux decline behavior was observed in other experimental runs. The limiting flux values at $t = 50$ min, J^* , were used as reference fluxes to evaluate the effect of pressure, temperature, cross flow velocity, and oil concentration on membrane permselectivity properties. Table 3 summarizes the J^* and oil rejection average values obtained under different operational conditions for the PVDF–Si, MPF-50, and PVDF–CA composite membranes.

Representative effects of feed velocity, temperature, and oil–hexane feed concentration on permeate flux are shown in Figs. 3, 4, 5. At a given pressure and temperature, increased cross-flow velocity increases permeate flux

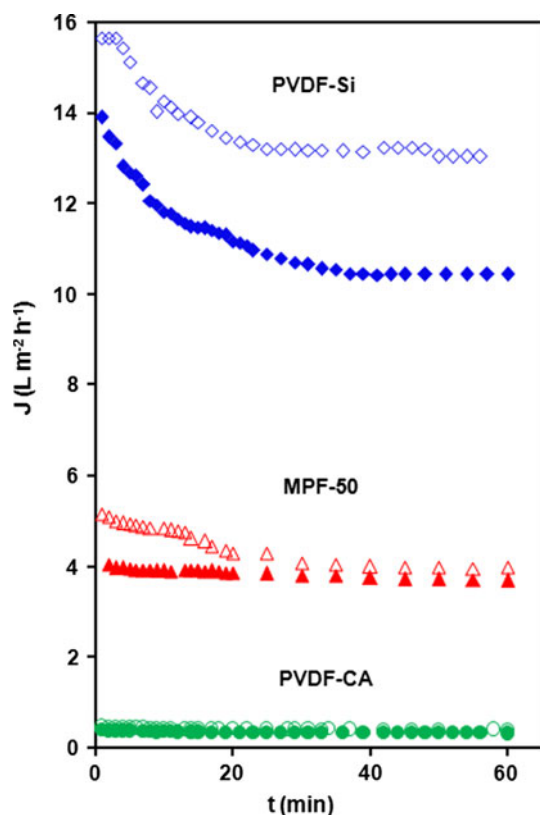


Fig. 3 Effect of feed velocity on permeate flux ($T = 50$ °C, $\Delta p = 5.8$ bar, and $C_f = 25\%$ w/w). Open symbols $v = 1.3$ m s⁻¹; solid symbols $v = 0.8$ m s⁻¹

(Fig. 3). This effect is explained by the increase in shear stress on the membrane surface leading to a reduction in the concentration polarization layer. The temperature affects J^* as shown in Fig. 4. Permeate flux rises when temperature increases. This behavior can be explained by the decrease of feed viscosity and the increase of oil diffusivity with temperature. These results show the convenience of using high temperatures in this application, taking into account the limits imposed by membrane stability and feed evaporation. Figure 5 shows the effect of feed oil concentration on permeate flux. When the concentration of oil in the miscella increases, permeate flux is greatly reduced because of the increasing viscosity of the miscella and the higher fouling and concentration polarization effects.

Although the retention values of the PVDF–Si and MPF-50 varied with pressure and velocity conditions, these variations were within experimental error (± 10 – 15%). General trends indicated that the oil rejection factor increased as the transmembrane pressure increased and both temperature and feed velocity were reduced. Reduction in cross-flow velocity causes an increase of the dynamic membrane resistance increasing the retention coefficient.

The membrane PVDF–Si flux of miscella solutions were fourfold to fivefold higher than those of the MPF-50 membrane, with PVDF–Si oil retention ($R = 31$ – 45%) being higher than for the MPF-50 membrane ($R = 30$ – 34%). Although the PVDF–CA membrane rejected all the oil ($R = 100\%$) under all the experimental conditions studied, the flux was a factor of 30–70 less than those obtained with PVDF–Si. This can be explained by analyzing the hydrophobic properties of composite membrane coatings. The more hydrophobic character of polydimethylsiloxane favors

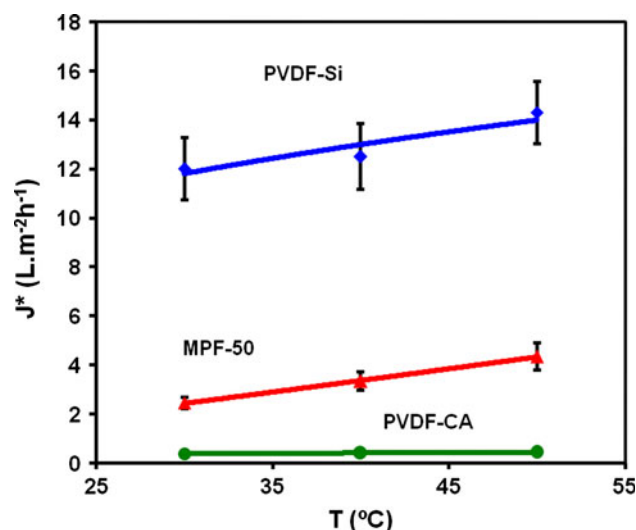


Fig. 4 Effect of temperature on permeate limiting flux (J^*) ($\Delta p = 7.8$ bar, $v = 0.8$ m s⁻¹, $C_f = 25\%$ w/w)

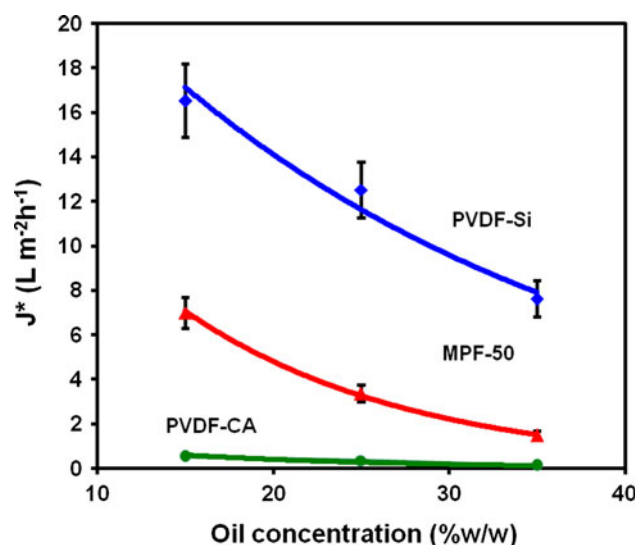


Fig. 5 Effect of feed oil concentration on permeate limiting flux ($\Delta p = 7.8$ bar, $v = 0.8$ m s⁻¹, and $T = 40$ °C)

flux of both the hexane (non polar solvent) and the practically non-polar oil molecules. In contrast, the small interaction between the cellulose acetate coating (more hydrophilic material) and the hexane and oil molecules results in low solvent flux and high solute rejection.

Conclusions

In this work, two composite membranes prepared in our laboratory, PVDF–Si and PVDF–CA, and a commercially available MPF-50 membrane were used to treat a crude sunflower oil–hexane miscella. Permeate flux depended on applied transmembrane pressure, temperature, feed concentration, and cross-flow velocity. Higher pressures, temperatures, and cross-flow velocities yielded better permeate flux. On the other hand, lower cross-flow velocities led to greater retention.

Of the three membranes studied, the PVDF–Si membrane had the best permselectivity. The PVDF–CA membrane resulted in the best oil rejection ($R = 100\%$); its limiting flux was, however, a factor of 25–57 less than those obtained with the PVDF–Si membrane. The commercial MPF-50 membrane had less oil retention and lower limiting flux values than when using the PVDF–Si membrane. The main features of the PVDF–Si membrane were:

- 1 good chemical and physical stability after long-term permeation experiments;
- 2 the best oil–hexane limiting permeate flux ($J^* = 7.6$ – 16.5 L m⁻² h⁻¹) with rejection factors between 30.8 and 46.2%; and

- 3 moderate initial flux decay (around 12–15%) during the first minutes of operation (low fouling phenomena).

The PVDF–Si synthesized membrane has promising permselectivity properties for separation of vegetable oil–hexane miscella using membrane technology.

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