Membrane Processing of Crude Vegetable Oils: Pilot Plant Scale Removal of Solvent from Oil Miscellas

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The recovery of solvents used in the extraction step of edible oil processing is required for economical, environmental, and safety considerations. The miscella (mixture of extracted oil and solvent) exits the extractor at 70 to 75 wt% solvent content. Currently, the solvent is recovered by distillation.

This paper reports the results of a study on separation of vegetable oils from commercial extraction solvents using various types of Reverse Osmosis (RO) and Ultrafiltration (UF) membranes.

Solvent permeation rates and separation performances of various RO and UF membranes were determined by using ethanol, isopropyl alcohol and hexane as the solvents. One membrane exhibited a flux of 200 GFD (ethanol) with 1% oil remaining in the permeate. However, hexane rapidly deteriorated all but one of the membranes tested. The membrane that was compatible with hexane had a low flux and unacceptably low oil retention.

Industrial-scale membranes were also evaluated in pilot plant trials. A hexane separation was attempted with a hollow-fiber membrane unit, and it was noted that the pores of the fibers swelled almost closed. Some of the commercially available membranes selectively removed solvent (ethanol or isopropanol) from the edible oil miscellas with reasonable flow rates.

The research reported has shown that membranes manufactured from polyamide were the least affected by hexane. Fluxes achieved during solvent-oil separations were increased by increases in either temperature or pressure and decreased by increases in oil concentration in the feed. The processing temperature affected the percentage of oil in solution in either ethanol or isopropanol as well as the viscosity of the feed. Both of these factors in turn influenced the flux achieved.

Approximately 2 trillion Btu/yr could be saved using a hybrid membrane system to recover solvents used in the extraction step of crude oil production. Studies to date report marginal success. The development of hexane-resistant membranes may make this application viable.

Edible oil processing. The primary solvent for extracting crude oil from oilseed flakes, expanded collets or presscake is commercial "hexane," a mixture of aliphatic and cyclic hydrocarbons collected over a narrow range of distillation temperatures. "Miscella" from extractors contains 25–30% oil and is typically separated by distillation to reclaim the hexane for reuse (1). Figure 1 illustrates the details of miscella distillation and a solvent recovery system (2). Miscella is pumped from the miscella tank into the evaporator, where a majority of the solvent is removed at this stage, and concentrated miscella (90% or more oil) next flows into the vacuum stripper. Hexane content of the oil is brought to less then 1% by high vacuum at the top of the stripper. The remaining solvent then is stripped by countercurrent live steam during its movement through a series of trays. The solvent and steam are condensed in the oil stripper condenser and the mixture is separated by decanting. Process efficiency is affected by vacuum level, cooling water temperature, configuration of the stripping unit and the temperature of the incoming oil (1).

Economical, environmental and safety reasons make solvent recovery one of the most critical steps in edible oil processing, and it is estimated that more than 2 million tons of hexane are recovered each year in the U.S. alone (3). In a recent study, Mohr *et al.* (4) reported that approximately 2 trillion Btu/yr could be saved using a hybrid membrane system to recover solvent from extraction miscellas.

Separation of nonaqueous mixtures by membrane technology. A principal advantage of removing water by reverse osmosis (RO) is the low energy requirement compared to conventional thermal evaporation processes. By using RO membranes, heating of the liquid to its boiling point and heat of evaporation are avoided, and the only energy required to operate the process is the electricity used to produce the pump pressures that force permeate through the membrane. Figure 2 shows a simplified flow diagram of the membrane separation system.

Investigation of RO membranes for nonaqueous separations has not been as extensive as for aqueous applications. The earliest applications of RO to nonaqueous systems include the work of Sourirajan (5), who attempted to separate various hydrocarbon liquid mixtures in a small high-pressure test cell using commercially-available cellulose acetate (CA), and laboratory-made CA and polyethylene membranes. In trials with xylene-ethyl alcohol and xylene-heptane-ethyl alcohol mixed feedstocks, polyethylene exhibited high separation capability for ethanol-xylene mixtures with an ethanol rejection of 96–98%.

With the introduction of chemically and physically stable membranes in recent years, separation trials have expanded to include aceotropic mixtures (6–11), chemically and physically similar mixtures (7,10–13), and hydrogen-bonded systems (7,8,14–20). The system most studied is benzene-cyclohexane, which forms an azeotrope at 55% benzene. Although membrane composition and processing conditions affect separation efficiency, all systems including aromatic-aliphatic separations (6–9,11,14) showed that the azeotrope can be effectively broken by membrane processing. Separations of physically and chemically similar systems, such as cyclohexane-cyclohexene, styrene-ethyl ben-

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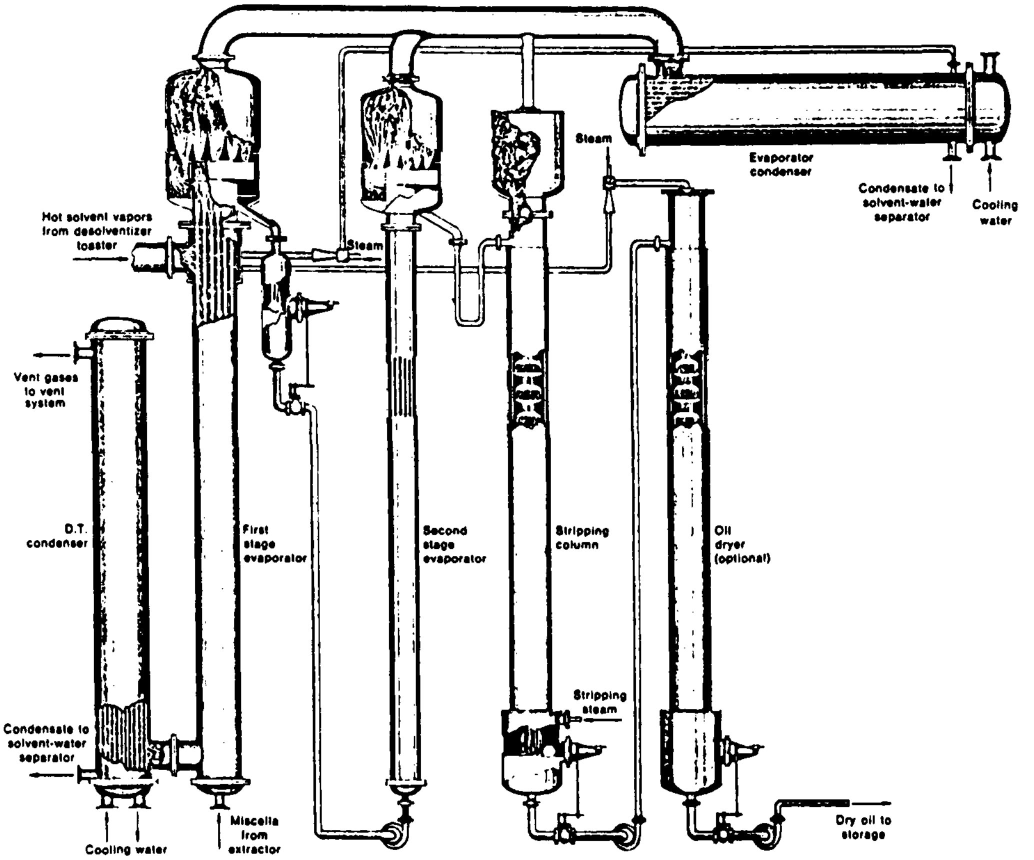

FIG. 1. Distillation system for solvent recovery from edible oil miscellas (2).

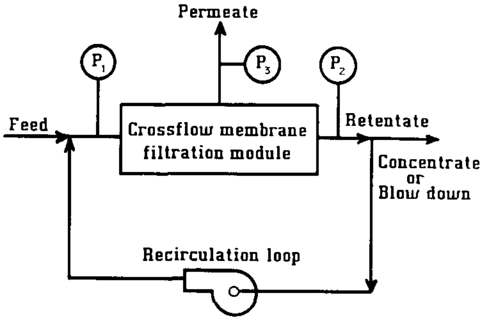

FIG. 2. Simplified schematic of crossflow membrane filtration.

zene and isomeric xylene have been successful, although multistage separation may further improve low selectivities (7,10). Trials on mixtures with very strong hydrogen bonding (including alcohol-alkane, alcohol-ester, alcohol-ketone and alcohol-aromatic mixtures) showed all mixtures to be separable (7,15–20). Mixtures of alcohol with esters, ketones or aromatics had lower selectivities, whereas the other systems had selectivities ranging from 5 to 200.

In other nonaqueous UF applications, Shen and Mir (21) reported superior separation of latex and latex dewatering streams and considerable energy savings over conventional evaporation processes. Hazlett *et al.* (22) evaluated a variety of membranes for processing crude petroleum feedstocks, and obtained a permeate similar to gas oil and a retentate enriched with asphaltene. Modified SEPAREX CA membranes have been shown effective for separating methanol from mixtures of methanol, methyl butyl ether and C-4 mixtures (23,24). Li *et al.* (25) has separated hexane, pentane or heptane from other hydrocarbons obtained from a deasphalting process.

Another promising approach is the Membrane Pervaporization Process, in which the membrane acts as a barrier between feedstock in the liquid phase and the permeate in vapor phase (26). Fluxes are low in pervaporization compared to ultrafiltration (UF) and RO processes, but selectivites are high (approximately 1,000). Figure 3 shows a schematic diagram of the membrane pervaporation system.

With the availability of noncellulosic (NCA) RO membranes with increased temperature and pH versatility, we began studies to determine whether any of the new RO membranes or currently available UF membranes would be compatible with hexane, ethanol and isopropanol for separation of solvent-edible oil miscellas.

EXPERIMENTAL

Oil-solvent mixtures. Oil-solvent mixtures, simulating miscellas, were prepared by mixing unrefined glandless cottonseed oil with commercial hexane in the ratio 1:3 (w/w). Distilled water was permeated through

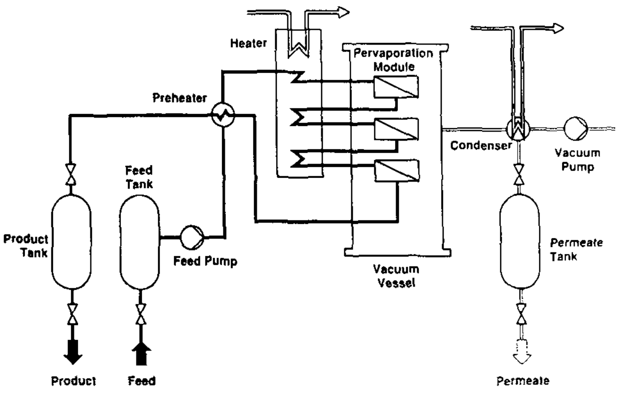

FIG. 3. Simplified diagram of a membrane perevaporation system (26).

each membrane (Table 1) to provide a reference permeation rate.

Membrane separation trials. Twelve membranes (7 RO and 5 small pore UF) were procured from leading membrane manufacturers; 11 of the membranes were of NCA composition. The UF membranes ranged in pore size (molecular weight cut-off, MWCO) from 500 to 1,000 daltons, and the RO membranes had MWCOs of 500 daltons or less. Membranes were obtained as 3.75 inch diameter circular discs, or in flat sheets from which discs were cut.

The flat disc membranes were tested in a RO and UF test cell obtained from Koch Membrane Systems, Inc., Wilmington, MA. This test cell is designed specifically for studying membrane selectivity, flux rate, and stability. It has a capacity of 200 ml and can be operated up to 1,500 psi. The membrane is supported in its 6.25 inch height by 3.75 inch diameter test cell by a sintered porous stainless steel disc covered with filter paper. Continuous agitation is provided just above the membrane surface by a magnetic stirrer bar suspended from a Teflon bearing. An external magnetic laboratory power unit is used to drive the stirrer bar. This test cell is used to identify the most promising membranes. However, because of a small membrane area, it is only used in membrane screening experiments. Therefore their flux rates can not be compared with the data obtained from commercial scale membranes.

The RO/UF cell was operated in batch mode by charging the cell with 200 ml of oil-solvent mixtures and applying pressure from a cylinder of nitrogen gas. The permeated solvent was collected at a port from beneath the membrane support. Pressure, temperature, solvent permeation rate, and percentage of oil in the permeate were determined and recorded. Each trial was repeated two to four times. Membranes evaluated in laboratory experiments are obtained from Abcor Division, Koch Membrane Systems, Wilmington, MA; Film Tec Corporation, Minneapolis, MN; Micro filtration Systems, Dublin, CA; and Osmonics Inc., Minnetonka, MN. The most promising membranes from these runs were evaluated in pilot plant scale and are identified in Table 2.

Pilot plant trials. Nine industrial membrane systems were evaluated for their oil-solvent separation capabilities in explosion-proof facilities meeting NFPA 36 requirements at the Food Protein Research and Development Research Oil Mill. Some of these systems utilized membranes that had shown high potentials in earlier laboratory-scale oil-solvent separations. Seven systems utilized NCA membranes and two had CA membranes. Spiral wound and hollow fiber membrane configurations were included.

Two of the systems—a Romicon PM 1 (PS) hollow fiber UF cartridge (manufactured by Romicon, Inc., Woburn, MA) with 15 square feet of 1,000 MWCO membrane, and an OSMO-52-50 UF Sepralator system (manufactured by Osmonics, Inc. [OSMO], Minnetonka, MN) with 5 square feet of 1,000 MWCO membrane—were evaluated primarily because of ready availability. The pore size of the Romicon PM 1 was recognized from the outset as being slightly oversize. The OSMO-52-50 Sepralator, though not used previously, was more than three years old.

The other seven membranes were chosen (and in some cases manufactured) especially for these tests. Three manufacturers—Osmonics, Inc., Minnetonka, MN; Paterson Candy International Ltd. (PCI), Whitchurch, Hampshire, England; and Romicon, Inc., Woburn, MA—cooperated with this project by providing test units sometimes without charge. Identities of membranes evaluated in pilot plant scale are given in Table 2. Data from the nine larger-sized systems are shown in Table 3.

RESULTS AND DISCUSSION

Laboratory trials. Each membrane then was tested with isopropanol, ethanol, hexane, and then with mixtures of each of the solvents containing 25% glandless cottonseed oils by weight. Permeation rates for membranes A through L with distilled water ranged from a low of 4.85 gfd to a high of 113.00 gfd (Table 1).

Isopropanol. Permeation rates of 91% isopropanol varied from 0 to 17.94 for membranes A through L. Five membranes had fluxes higher than 5.0 gfd, while two membranes gave fluxes of 13.0 gfd or higher. Permeation rates of isopropanol-oil miscella (75:25, w/w) varied from 0 to 19.80 gfd. Oil contents of the permeates ranged from 0 to 20.3%. Three membranes yielded permeates containing less than 1.2% oil. Membrane C had the highest flux (19.80 gfd) and a fairly low percentage of oil in the permeate (3.8%). Membrane F, with a flux of 6.36 gfd and 1.2% oil in the permeate, had the most desirable combination of flux and oil retention and was chosen for further investigation in pilot plant trials.

Ethanol. Permeation of 95% ethanol varied from 4.65 to 351.0 gfd. Five membranes had fluxes higher than 17.0 gfd, and two had fluxes of 70.0 gfd or higher. Ethanol was permeated from ethanol-oil miscella at rates ranging from 0.16 to 202.8 gfd, with oil in the permeates ranging from 0.1 to 1.2%. Ten membranes yielded permeates containing less than 1% oil. One membrane passed neither ethanol nor oil. Membrane H was considered to have the best combination of flux and percentage of oil in the permeate (202.8 gfd and 0.90% oil, respectively).

TABLE 1

Performance of Membranes Separating Solvent from Oil-solvent Mixtures in Laboratory Trials

Membrane (MWCO)[a]	Mixture	Pressure (psi)	Temp. (°F)	Flux (gfd)	% Oil in permeate
A	distilled water	800	81	64.90	0.0
FT-40[b]	IPA	800	77	1.30	0.0
(300)	IPA-oil	800	77	7.31	20.3
	Ethanol	800	73	4.65	0.0
	EtOH-oil	800	73	0.00	0.0
	hexane	800	73	0.00	0.0
	hexane-oil	800	73	0.00	0.0
B	distilled water	800	70	40.30	0.0
FT-30[b]	IPA	800	70	0.00	0.0
(200)	IPA-oil	800	70	0.00	0.0
	ethanol	800	77	5.20	0.0
	EtOH-oil	800	77	1.35	0.5
	hexane	800	77	0.00	0.0
	hexane-oil	800	70	0.00	0.0
C	distilled water	200	73	10.30	0.0
UM05[c]	IPA	200	75	1.80	0.0
(500)	IPA-oil	800	75	19.80	3.8
	ethanol	700	77	9.35	0.0
	EtOH-oil	800	77	1.53	0.6
	hexane	150	Membrane damaged by hexane		
	hexane-oil	200	Membrane damaged by hexane		
D	distilled water	300	75	19.85	0.0
YM2[c]	IPA	300	75	2.14	0.0
(1,000)	IPA-oil	300	75	0.00	0.0
	ethanol	800	77	11.70	0.0
	EtOH-oil	800	77	3.50	0.6
	hexane	200	Membrane damaged by hexane		
	hexane-oil	200	Membrane damaged by hexane		
E	distilled water	800	75	106.60	0.0
Sepa 50 (PA)[d]	IPA	800	75	7.34	0.0
(600)	IPA-oil	800	75	7.72	7.0
	ethanol	800	75	70.98	0.0
	EtOH-oil	600	75	24.83	0.6
	hexane	700	Membrane damaged by hexane		
	hexane-oil	700	Membrane damaged by hexane		
F	distilled water	200	75	14.89	0.0
Sepa S-20	IPA	200	75	17.94	0.0
(VF)[d] (20,000)	IPA-oil	200	75	6.36	1.2
	ethanol	200	75	25.80	0.0
	EtOH-oil	200	75	11.78	0.8
	hexane	700	Membrane damaged by hexane		
	hexane-oil	700	Membrane damaged by hexane		
G	distilled water	800	75	41.60	0.0
Sepa 0 (PA)[d]	IPA	800	75	13.00	0.0
(500–1,000)	IPA-oil	800	75	4.84	8.9
	ethanol	800	77	20.28	0.0
	EtOH-oil	800	75	31.46	1.2
	hexane	800	77	1.56	0.0
	hexane-oil	800	77	4.20	21.6

(Continued)

Hexane. Two membranes permeated hexane without being destroyed, although the rates of 1.56 and 0.81 gfd were very low. Similar damage resulted when attempting to separate hexane-oil miscella. Only Membrane G separated hexane from miscella, with a flux of 4.20 gfd and 21.6% oil in the permeate. Neither the flux nor the percent oil in permeate was considered, but the trial indicated the proper materials for membranes to be used with hexane.

Pilot plant scale trials. The first two trials performed on larger-scale industrial units (Table 3) show that neither of these membranes was sufficiently tight to retain the oil. Essentially all of the oil came through the Romicon PM-1 membrane; the fibers expanded in

TABLE 1 (Continued)

Membrane (MWCO)[a]	Mixture	Pressure (psi)	Temp. (°F)	Flux (gfd)	% Oil in permeate
H					
YC05[c]	distilled water	500	77	113.10	0.0
(300–400)	IPA	500	77	8.39	0.0
	IPA-oil	500	77	1.30	2.9
	Ethanol	500	77	351.00	0.0
	EtOH-oil	500	77	202.80	0.9
	hexane	800	Membrane damaged by hexane		
	hexane-oil	800	Membrane damaged by hexane		
I					
Non-CA (RO)[c]	distilled water	800	77	11.70	0.0
(200–1,000)	IPA	800	77	0.00	0.0
	IPA-oil	800	77	0.00	0.0
	ethanol	800	77	17.16	0.0
	EtOH-oil	800	77	1.81	0.9
	hexane	800	Membrane damaged by hexane		
	hexane-oil	800	Membrane damaged by hexane		
J					
ICO6[e]	distilled water	300	77	15.60	0.0
(500)	IPA	500	77	1.87	0.0
	IPA-oil	500	77	0.24	1.1
	ethanol	300	77	7.80	0.0
	EtOH-oil	300	77	1.94	0.1
	hexane	800	Membrane damaged by hexane		
	hexane-oil	800	Membrane damaged by hexane		
K					
(NA)[f]	distilled water	800	77	7.80	0.0
	IPA	800	77	0.00	0.0
	IPA-oil	800	77	0.13	1.5
	ethanol	500	77	1.51	0.0
	EtOH-oil	500	77	4.86	0.3
	hexane	500	77	negligible	0.0
	hexane-oil	800	77	negligible	0.0
L					
Sepa 97 (CA)[d]	distilled water	500	77	4.85	0.0
(150–200)	IPA	500	77	5.09	0.0
	IPA-oil	500	77	1.02	0.6
	ethanol	500	77	15.21	0.0
	EtOH-oil	800	71	0.16	0.1
	hexane	800	77	0.81	0.0
	hexane-oil	800	77	0.00	0.0
	hexane-oil	500	100	4.94	0.0

[a] Nominal molecular weight cut off.
[b] Film Tec Corporation, Minneapolis, MN.
[c] Abcor Division, Koch Membrane Systems, Wilmington, MA.
[d] Osmonics Inc., Minnetonka, MN.
[e] Micro Filtration Systems, Dublin, CA.
[f] Membrane can not be identified due to the confidentiality agreement.

TABLE 2

Membranes and Their Specifications

Company	Membrane	Type	Estimated MWCO	Polymer
Romicon, Inc.	A	PM 1	1,000	polysulfone
Osmonics, Inc.	B	OSMO 52-50 (VF)	600	fluorinated polymer
	C	OSMO Sepa 0 (PA)	500	polyamide
	D	OSMO Sepa S-20 (VF)	20,000	fluorinated polymer
	G	OSMO 192T-89 (PA)	300–400	polyamide
	H	OSMO 192T-0 (PA)	500	polyamide
	I	OSMO 192T-97 (CA)	150–200	cellulose acetate
Paterson Candy	E	PCI ZF-99	<400	composite
International	F	PCI RO (CA)	<400	cellulose acetate

TABLE 3

Separation of Cottonseed Oil from Solvent-Oil Mixtures Using Industrial Membrane Systems

Membrane	Mixture	P (psi)	T (°F)	Flux (gfd)	% Oil in feed	% Oil in permeate
PM1 (PS)[a] (1,000)	Hexane-oil	10	73	5.21	21.50	21.54
		15	73	7.24	21.50	20.98
		20	73	9.14	21.50	20.55
		25	73	11.18	21.50	20.50
OSMO 52-50 (VF)[b] (1,000)	IPA-oil	200	100	13.46	17.90	15.59
	hexane-oil	200	79	5.83	16.90	17.33
Sepa 0 (PA)[b] (500)	EtOH-oil	200	83	4.29	45.93	1.03
		200	84	3.81	43.16	1.20
	hexane-oil	200	85	2.53	27.59	10.71
Sepa S-20 (VF)[b] (300–1,000)	IPA-oil	200	106	5.27	25.20	3.41
		200	106	7.00	27.29	12.61
		200	106	6.45	34.18	14.16
	EtOH-oil	250	106	12.89	32.30	1.72
		250	106	13.69	40.17	1.69
PCI ZF-99[c] (<400)	EtOH-oil	500	82	1.91	22.38	0.07
		600	114	28.50	35.56	0.07
		600	144	2.23	63.86	0.28
	EtOH-oil	400	85	3.75	23.15	—
		600	88	6.66	—	—
		800	93	9.59	—	—
	hexane-oil	600	93	0.57	24.34	0.14
		600	105	0.34	24.59	0.67
PCI RO (CA)[c] (<400)	EtOH-oil	800	90	12.26	25.08	0.15
		800	91	8.04	36.05	0.15
		800	94	7.08	51.05	0.12
		800	94	0.55	64.31	0.14
	EtOH-oil	200	89	6.54	25.08	0.00
		400	90	11.85	25.08	0.00
		600	90	15.27	25.08	0.00
		800	91	18.52	25.08	0.00
	EtOH-oil	800	80	—	—	0.22
		800	90	—	—	0.28
		800	100	—	—	0.35
		800	110	—	—	0.39
	IPA-oil	800	82	4.10	24.39	0.00
		800	92	4.10	24.39	2.48
		800	101	5.45	24.39	2.22
		800	110	6.27	24.39	2.14
		810	116	6.27	25.58	0.00
		800	108	5.04	29.81	0.00
		800	116	4.36	41.07	0.00
		800	110	2.04	55.17	0.00

(Continued)

the presence of hexane, causing them to buckle in the holder, and to almost close the pores by swelling. The OSMO-52-50 unit did not retain any oil from the miscella. As mentioned earlier, these units were not selected on the basis of laboratory prescreening.

OSMO Sepa O (PA) membrane. The Sepa O (PA) membrane (500 MWCO) was identified during laboratory trials as a membrane that would not deteriorate in hexane. The data from tests with larger units are shown in Table 3. The larger unit matched the laboratory trials closely in percentage of oil in permeate in ethanol separation tests. However, flux achieved in the larger unit was lower (4.29 gfd compared to 31.46 gfd in the laboratory). One reason for this was that the Sepa membranes were housed in PVC tubes that would withstand pressures of only 250 psi in pilot plant tests, while the laboratory test cell was subjected to 800 psi. A second cause of lower flux from the larger unit may have been the higher concentration of oil in the ethanol-oil miscella fed to the larger membrane. When processing a hexane-oil mixture, the larger Sepa O membrane unit also passed less oil in the permeate than the laboratory-sized membrane, but retention still was low and a tighter membrane of the same composition was procured for additional trials.

OSMO 192T-O (PA) membrane. The 192T-O (PA)

TABLE 3 (Continued)

Membrane	Mixture	P (psi)	T (°F)	Flux (gfd)	% Oil in feed	% Oil in permeate
OSMO 192T-89 (PA)[b] (300–400)	EtOH-oil	225	109	1.39	24.43	0.41[d]
		225	109	0.28	23.89	
	IPA-oil	250	115	1.63	25.02	0.36[d]
		250	115	1.45	25.95	
		250	113	1.14	27.21	
		250	115	1.04	29.16	
OSMO 192T-0 (PA)[b] (500)	IPA-oil	250	109	2.08	26.57	7.02[d]
		250	111	1.97	27.49	
		250	111	1.90	28.22	
		250	109	1.70	28.98	
	EtOH-oil	250	109	0.54	24.50	0.32[d]
OSMO 192T-97 (CA)[b] (150–200)	IPA-oil	250	109	1.00	27.01	1.52[d]
		250	113	1.31	27.49	
		250	111	1.31	28.62	
		250	111	0.90	28.49	
	EtOH-oil	250	113	1.87	23.61	0.27[d]
		250	113	1.49	25.32	
		250	109	1.52	25.57	
		250	111	1.59	26.19	

[a] Romicon Inc., Woburn, MA.
[b] Osmonics Inc., Minnetonka, MN.
[c] Paterson Candy International Ltd. (PCI), Whitchurch, Hampshire, England.
[d] Composite permeate.

membrane (500 MWCO) supposedly was of the same composition and pore size as the Sepa O (PA) membrane, but differed in that it was contained in a stainless steel housing. This membrane was first tested with an isopropanol-oil mixture, where oil was undesirably high in the permeate and the isopropanol flux unacceptably low. In ethanol-oil separations, the 192T-O (PA) membrane effectively separated ethanol (0.32% oil in permeate) but the flux was unacceptably low. Hardly any hexane permeated in hexane-oil trials. Evaluation of the membrane was discontinued.

OSMO Sepa S-20 (VF) membrane. The Sepa S-20 (VF) membrane was deteriorated by hexane in laboratory trials, and was not tested with hexane in pilot plant trials. When processing an isopropanol-oil mixture, the larger unit passed more oil as permeate than in the laboratory test cell. Fluxes of the two sizes of units were undesirably low in both cases. The flux rate for separating ethanol by the larger unit was only about 13 gfd, but the pressure was limited to 250 psi.

PCI ZF-99 NCA RO membrane. The PCI ZF-99 membrane separated ethanol well while concentrating oil in the feedstock miscella from 22.38% to 63.86%. Average oil content in the permeate was only 0.082%. The flux varied from 1.91 to 28.5 gfd and back down to 2.23 gfd as temperature increased from 82 to 144°F, and oil concentration in the feed rose to 63.86%. Rising temperatures decrease viscosity of the feed and raise the oil solution capacity of ethanol. The ratio of oil in solution in the feed to undissolved oil appeared to affect the flux. In a second test processing ethanol-oil miscella, the pressure was varied from 400 to 800 psi while the temperature remained at 93°F or below. Under these conditions, flux rose from 3.75 to 9.59 gfd; and again, the feed contained a larger percentage of nonsolubilized oil at 93°F.

The PCI ZF-99 membrane permeated hexane from a hexane-oil miscella at a very low rate, and the low percentage of oil in the permeate indicated that a membrane of the same composition, but with larger pores, might be satisfactory. However, this membrane began to show signs of failure when allowed to sit in contact with alcohol for several days. It was not determined whether failure was due to deterioration of the membrane or that of the support materials (i.e., adhesives, seals, etc.). The unit continued to leak although the visible seals were changed.

PCI CA RO membrane. This membrane showed good oil retention in a series of tests with ethanol-oil mixtures. In the initial test, system pressure was held constant at 800 psi with the temperature rising only slightly and oil content of the retentate increased from 25.08 to 64.31%. A mean flux of only 6.88 gfd was obtained; however, the percentage of oil in the composite permeate was desirably low at 0.15%. In a second test of the same system with an ethanol-oil mixture, the temperature was held constant and pressure was varied from 200 psi to 800 psi. Increased pressure caused an increase in flux from 6.54 gfd to 18.52 gfd at the original oil concentration of 25.08%. The relationship between pressure and flux is readily apparent from the second test (Table 3).

A third test was conducted to determine effects of temperature on oil content in the permeate of the CA RO membrane. As temperature increased from 80 to 110°F with pressure held constant, the percentage of oil in the permeate increased from 0.22% to 0.39%. Despite increased oil passage, the oil retention was still highly desirable. When tested with an isopropanol-oil mixture, the CA RO membrane passed 2.48% of the oil with the permeate. The flux at a constant pressure of 800 psi changed from 4.1 gfd to 6.27 gfd as the temperature was increased from 82 to 110°F. Oil in the feedstock was held constant at 24.39% in a second test, in which oil in the retentate was concentrated from 25.58% to a final concentration of 55.17%, while the flux dropped from 6.27 to 2.04 gfd. Attempts to separate hexane from hexane-oil mixtures with the PCI CA RO were unsuccessful, with only a few drops of hexane permeating through the membranes at temperatures ranging from 95 to 110°F with the pressure at 800 psi.

OSMO 192T-89 PA membrane. The 192T-89 PA membrane (200 MWCO) was first tested with an ethanol-oil mixture and the percentage of oil permeating through the membrane was desirably low (0.41%). However, fluxes achieved were unacceptably low. If it had been possible to increase the pressure to 800 psi, the flux would have probably increased several fold.

The 89 PA membrane showed similar results when tested with an isopropanol-oil mixture. Again, rejection characteristics for oil were good, but the flux was unacceptable. After the first two tests, the 89 PA membrane was allowed to sit overnight in ethanol, and then was flushed with clean hexane and tested with a hexane-oil mixture. No hexane permeated through the membrane at 94°F and 250 psi. The trial was stopped when hardly any hexane permeated after the temperature was raised to 117°F.

OSMO 192T-97 CA membrane. The final separations tests were conducted using a 192T-97 membrane (150 MWCO) manufactured from cellulose acetate. Results of the tests with ethanol-oil and isopropanol-oil at a constant pressure of 250 psi and with the temperature at 111°F were very similar. Each alcohol was separated with a low percentage of oil in the permeate, 1.52% and 0.27%, for isopropanol and ethanol, respectively, with the flux being unacceptably low in each instance. In attempts to separate a hexane-oil mixture, no hexane permeated through the membrane.

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