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SEPARATION OF METHANOL AND ISOPENTANE BY PERVAPORATION AND TAME PRODUCTION APPLICATION

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ABSTRACT

Separation of methanol (MeOH) and isopentane (iC_5) is a problematic process in the production of tertiary-amyle methyl ether (TAME). The removal of methanol from the product stream and its subsequent recycling to the reactor requires breakage of its azeotrope with iC_5. While the conventional separation processes are costly and cumbersome, pervaporation offers a simple and inexpensive alternative to traditional techniques.

In the present study, pervaporation was used to examine the performance of two commercial rubbery membranes as well as Nafion-170 to separate methanol and isopentane. The results indicate that all three membranes were highly selective and permeable toward methanol. Both the selectivity and permeability were strongly feed-composition dependent. Selectivity decreased and

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permeability increased as methanol concentration in the feed increased. With few exceptions, methanol was collected at 95% to 99.8% purity from the permeate side at a wide range of feed compositions varying from 1% to 25% methanol. Average selectivities of 170 to 370, and fluxes up to 2.93 $kg/m^2 \cdot h$ were observed. We also found that preconditioning of the membranes enhances their performances.

INTRODUCTION

The quest for replacement of octane enhancers for gasoline started in the late 1970s and early 1980s with the phase-out of lead and lead-based compounds. Since then, oxygen-containing compounds, known as oxygenates, became attractive as replacements for lead in automobile fuel. A 15% reduction in hydrocarbon emissions from the exhaust of automobiles has been realized by using reformulated gasoline containing 2.0 to 2.7% (wt) oxygen (1).

Tertiary-amyl methyl ether, commonly known as TAME, is among a family of oxygenates suitable for replacing lead in gasoline. Other major compounds include ethanol and methyl tertiary-butyl ether (MTBE). The advantage of TAME over MTBE is the use of C_5 hydrocarbons as feedstock, which are more widely available and can be readily used for the production of oxygenates. In addition, cases of groundwater contamination by MTBE in the recent years has caused a great pressure to phase out this substance in California and a number of other states. Also, compared with ethanol, TAME has a higher caloric value and lower sensitivity to water.

TAME is produced by the catalytic reaction of iso-amylene (IA) and methanol (MeOH). Iso-amylenes include three isomers of methyl butene, namely, 2-methyl-1-butene (2M1B), 2-methyl-2-butene (2M2B), and 3-methyl 1-butene (3M1B). In an etherification process only 2M1B and 2M2B react (2–4). The reaction also includes isomerization of 2M1B to 2M2B:

$$\left.\begin{array}{c} \mathrm{CH_3{-}CH_2{-}C(CH_3){=}CH_2} \\ \uparrow\downarrow \\ \mathrm{CH_3{-}CH{=}C(CH_3)_2} \end{array}\right\} + \mathrm{CH_3OH} \rightarrow \mathrm{CH_3{-}CH_2{-}C(CH_3)_2{-}O{-}CH_3}$$

The conversion of IA to TAME also has the advantage of removing this atmospherically reactive substance from the gasoline pool (2). IA is obtained through steam cracking and fluidized catalytic cracking (FCC) operations containing an average of 25% (wt) reactive olefins (5,6). A typical feed-stream composition of a TAME reactor is shown in Table 1. Such a stream mainly contains isopentane and IA, and only IA participates in the reaction when the reaction is

Table 1. Composition of the Feed Stream to the TAME Reactor

Components	Mole Fraction	Mass Fraction	Boiling Point (°C)
Isopentane	0.4821	0.488	27.8
n-pentane	0.085	0.086	36
1-pentene	0.0366	0.036	29.9
2-pentene (cis)	0.1555	0.153	36.9
2-methyl-1-butene	0.0823	0.081	31.1
2-methyl-2-butene	0.1585	0.156	38.5

catalyzed with a sulfonic acid ion-exchange resin (3). The conversion rate reduces as a function of temperature (7), and because of the reversible nature of the etherification reaction, an excess amount of methanol is required for enhanced conversion; as much as a 25% excess MeOH is commonly added to the reaction (7). The reactor effluent includes TAME, methanol, unreacted IA, and other C_5s.

The addition of excess methanol creates a separation problem because it forms azeotropic mixtures with almost all of the reaction products. Table 2 shows the azeotropic composition of methanol with other constituents of the reactor effluent at three pressures (7). Methanol forms azeotropic mixtures with TAME at high concentrations of methanol. The high concentration of methanol occur when a significant portion of TAME has been recovered in the effluent. The formation of azeotropes with the raffinate presents a difficult separation problem due to the relatively low methanol concentration in the mixture, which makes the recovery difficult through a secondary distillation process.

Table 2. Composition of the Azeotropic Mixtures of Methanol and Other Components Present in the TAME Reaction Product Stream

Components		$p = 2.5$ bar		$p = 4.0$ bar		$p = 5.5$ bar	
1	2	x_1	T (°C)	x_1	T (°C)	x_1	T (°C)
Methanol	2M1B	0.21	53.76	0.243	69.24	0.268	80.07
Methanol	2M2B	0.28	58.69	0.31	73.78	0.331	84.69
Methanol	*n*-pentane	0.295	58.64	0.328	73.96	0.347	85.2
Methanol	i-pentane	0.21	51.22	0.252	66.61	0.28	77.85
Methanol	1-pentene	0.22	53.64	0.267	69.06	0.283	80.75
Methanol	2-pentene	0.265	56.73	0.301	72.29	0.322	82.72
Methanol	TAME	0.763	87.56	0.793	102.45	0.802	113.2

Data from Subawalla and Fair (7).

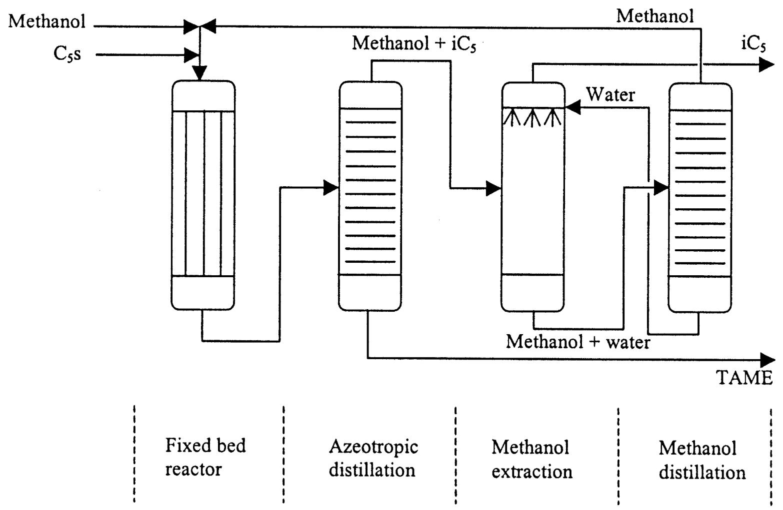

Figure 1. A conventional distillation/extraction hybrid system for TAME production.

The conventional technology to recover TAME and excess methanol is a distillation/extraction hybrid process. In this process, shown in Fig. 1, the reactor effluent is fed into an azeotropic distillation column where pure TAME is collected at the bottom and an azeotropic mixture of methanol and C_5 hydrocarbons is produced as the raffinate. This raffinate stream consists mainly of isopentane; it comprises almost one half of the feed stream to the reactor (Table 1). The azeotropic raffinate is transferred to a scrubber where the methanol content is washed out by water and iC_5 and other hydrocarbons are recovered. Finally, a methanol/water mixture is distilled in a secondary distillation unit. The recovered methanol is recycled to the reactor and the water is recycled to the scrubber for reuse (7–11).

While conventional technologies are costly and cumbersome, pervaporation (PV) can offer a simple and economical alternative. Because PV is not limited by the relative volatility of the feed components forming azeotropic mixtures, it has become a major application for the separation process. A distillation/PV hybrid system can be implemented into a TAME production process in a variety of configurations. For example, placing a PV system between the reactor and the first distillation column will remove most of MeOH from the feed stream and direct it to the distillation column thereby significantly increasing the capacity of the existing process. Alternatively, the MeOH/TAME azeotrope can be withdrawn from the distillation tower and fed into a pervaporation system to remove MeOH and return a TAME-rich stream to the distillation column. In this arrangement, the formation of an azeotrope is retarded and the need for costly azeotropic distillation processing is eliminated. Furthermore, pervaporation technology can be used to remove methanol from the raffinate stream. This application eliminates the need for a methanol extraction stage, which consists of a water wash tower and a secondary distillation tower (Fig. 1).

In this study, we focused on the PV system via the removal of methanol from the raffinate stream of the first distillation tower. We believe that this study is the first of its kind. The predominant C_5 compound contained in the raffinate stream is isopentane. Therefore, in the laboratory experiments we focused our study on MeOH/iC_5 separation by pervaporation. A suitable membrane should demonstrate affinity for polar compounds, such as alcohols. In the present work, two polydimethylsyloxane-based membranes from GKSS (Geesthact, Germany) and Sulzer (Neunkirchen, Germany), were tested for their ability to separate MeOH and iC_5. Also, the commercial Nafion-170 membrane (DuPont) was demonstrated to possess the characteristics required for such a separation.

EXPERIMENTAL

A schematic of the apparatus is shown in Fig. 2. The system consisted of a separation cell, permeate traps, and a vacuum pump. The cell, tubing, and traps were all made of glass.

The membrane sample with an effective surface area of 11.3 cm^2 was mounted on top of a plastic porous support. The feed mixtures were present at atmospheric pressure and room temperature (approximately 23°C). The permeate side was maintained at a pressure of 1 torr absolute. Two liquid nitrogen cooled traps were used to condense the permeate. The permeate is defined as amount of material transferred through a membrane and collected downstream of the mem-

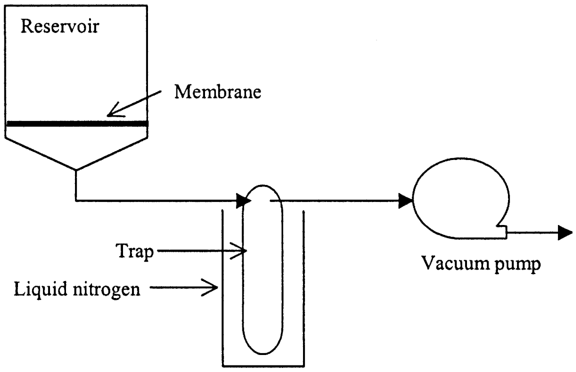

Figure 2. Laboratory pervaporation apparatus.

brane system. Noncondensable gases were evacuated from the permeate stream by the vacuum pump. Compositions of the feed, permeate, and residue (the feed remaining after completion of the experiment) were analyzed with a Shimadzu model 8A gas chromatograph (GC).

At the end of each experiment, the trap was sealed and removed from the liquid nitrogen and allowed to equilibrate to room temperature. The final permeate sample was weighed and its composition was determined. The flux was calculated by dividing the weight of permeate by membrane area and permeation time. The flux was reported in the units of kmh ($\text{kg/m}^2\cdot\text{h}$), which are commonly used in industry.

With few exceptions, the residue from one experiment was used as the feed for the next. We accomplished this by switching the permeate stream from one trap to another after each experiment was completed. While the temperature of the first trap was equilibrating, the permeate was collected inside the second. At each transition, the feed composition was determined and was used as the residue for the completed experiment as well as the feed for the following one. The experiment was stopped when the methanol content of the residue reached the target value of <1000 wppm (part per million by weight).

RESULTS AND DISCUSSIONS

Table 3 and Figs. 3 and 4 summarize the results obtained using rubbery membrane A. The experiments were performed in 5 series, shown as experiment numbers 1–5 in Table 3. In experiments No. 1 and No. 4, the tests were interrupted to determine the composition of the permeate and residue. The residue composi-

Table 3. Results of MeOH/iC5 Separation Using Rubbery Membrane A

Experiment No.	MeOH% (wt) Feed	Residue	Permeate	Selectivity	Flux kmh	Stage Cut % (overall)	Recovery %
1	13	3.25	95.5	140	0.742	10.6 (10.6)	77.6
	3.25	0.085	97	960	0.218	3.3 (13.5)	97.5
2	0.085	0	—	—	—	—	—
3	12.3	4.2	98	350	0.841	9.6	69.1
4	12.1	6.8	98	240	0.509	5.8	47.1
	8	0.24	92.6	144	0.619	8.4 (8.4)	97.3
5	0.24	0	82	1900	—	0.3 (8.7)	100
	1.03	0.108	94	1500	0.061	1	89.6

— Data were unmeasurable.
The stage cuts shown in the parentheses represent the overall values.

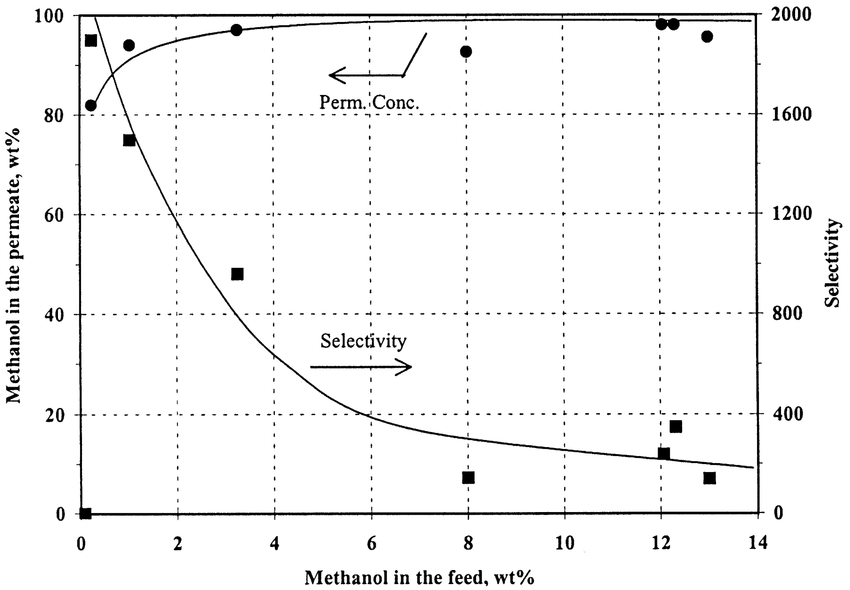

Figure 3. Results of the pervaporation experiments in which rubbery membrane A was used. Permeate composition and selectivity versus methanol concentration in the feed.

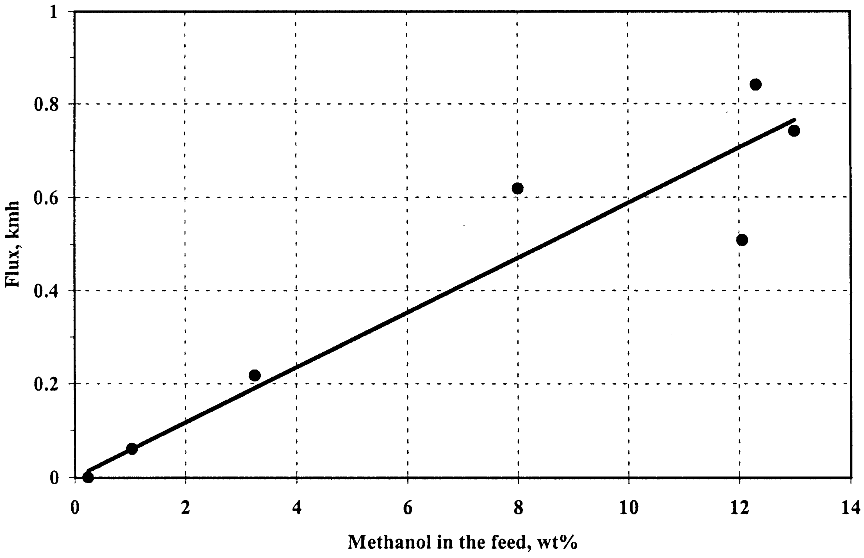

Figure 4. Results of the pervaporation experiments in which rubbery membrane A was used. Flux versus methanol concentration in the feed.

tion from one experiment was used as the feed composition for the next. An alternative way to look at the interrupted results is to consider them as the membrane performance at different stage cuts. The stage-cut and recovery values are shown in Table 3. Recovery is defined as the ratio of amount of desired component (methanol in this study) in the permeate to that in the feed. A stage cut is percentage of feed permeating through a membrane. In a batch process (such as the present study), stage cut is calculated as

$$\frac{\text{feed \% (wt)} - \text{residue \% (wt)}}{\text{permeate \% (wt)} - \text{residue \% (wt)}}$$

Comparisons of overall stage-cut values and the initial methanol concentration in the feed indicate that these parameters differ negligibly. In other words, due to the high selectivity of the membrane, methanol can be removed from the stream in 1 stage with a minimum loss of iC_5. Selectivity of a mixture is defined as ratio of concentration of desired component to nondesired component(s) in the permeate over those respective components in the feed:

$$\alpha = \frac{y}{1-y} \Big/ \frac{x}{1-x}$$

where, x is the concentration of desired component (methanol in this case) in the feed, and y is the concentration of desired components in the permeate.

Feed concentrations varied from 13.0% (wt) to 0.085% (wt) (850 wppm) methanol in iC_5. The resulting permeate concentration ranged from 98% (wt) to 82% (wt) methanol, and the residue concentrations were seen to decrease to as low as 0.00% (wt). This degree of methanol depletion fulfils the common safety-requirement level of 1000 wppm MeOH in the residue for most industrial applications. Figure 3 shows the relation between both permeate concentration and membrane selectivity versus feed concentration. This figure indicates that at feed concentrations higher than 1% (wt) the permeate was collected at concentrations greater than 92% (wt) methanol.

In experiment No. 1 the third-stage permeate composition and flux could not be determined due to an insufficient sample collected for analysis. However, no methanol was detected in the residue, indicating a complete removal of methanol.

The effect of membrane preconditioning was observed. The selectivity of the membrane samples reached optimum values only after they were wetted in the feed mixture for 1 or 2 hours. As a result, few experiments were done without preconditioning, which resulted in significantly lower values of selectivity. For example, an experiment done with a 12.2% (wt) feed concentration resulted in 88.5% (wt) methanol in the permeate. Similar experiments with preconditioned membranes, as reported in Table 3, produced 98.0% (wt) methanol.

Membrane selectivity increased sharply as MeOH was depleted in the feed. Figure 3 shows that the selectivity dropped from 1900 to around 200 and passed through a plateau as the feed concentration increased. The average selectivity at the plateau was around 200. This trend could be due to the plasticization effect of methanol. Increased selectivities at small feed concentrations of methanol indicate that isopentane losses to the permeate were minimal. In other words, the overall methanol recovery remained high and relatively constant for a wide range of feed compositions.

The relationship of permeate flux with feed concentration is shown in Fig. 4. The flux increase was nearly linear ($R^2 = 90\%$) with increasing feed concentration. Generally, the flux was high and comparable to other well-established applications of pervaporation technology.

Table 4 and Figs. 5–7 show the results of the experiments in which rubbery membrane B was used. The experiments were conducted in 4 series of tests shown as experiments numbered 1–4 in Table 4.

Feed concentrations of 1.27–25.0% (wt) MeOH in iC_5 were used. The permeate concentration varied between 87.8 and 98.3% (wt) methanol. The separation experiments were continued until the target value of 1000 wppm methanol in the residue was met.

Figure 5 shows the relationships between both methanol concentration in the permeate and membrane selectivity versus a changing feed concentration. Over the range studied, permeate concentration remained constant, independent of the feed concentration. Such a result is an indication of the opposite and compet-

Table 4. Results of MeOH/iC5 Separation with Membrane B

Experiment No.	MeOH% (wt)			Selectivity	Flux kmh	Stage Cut % (overall)	Recovery %
	Feed	Residue	Permeate				
1	25	22	98	147	2.934	4	15.5
	22	19	97.5	138	2.757	3.8	16.9
	19	12	97.3	154	2.346	8.2	42
	12	3.1	96.7	215	1.416	9.5	76.6
	3.1	0.15	97.6	1300	0.219	3.0 (25.6)	95.3 (99.4)
2	14.9	5.3	97.8	254	2.212	10.4	68.1
	5.3	1.4	96.5	493	0.664	4.1	74.7
	1.4	0	97.2	2400	0.088	1.4 (15.3)	100 (100)
3	12.8	5.7	97.8	300	1.77	7.7	58.9
	5.7	1.9	97.7	700	0.885	4	68
	1.9	0.07	96.8	1560	0.1	1.9 (13.0)	96.4 (99.5)
4	11.3	7.2	95.7	175	1.77	4.6	39.2
	7.2	1.27	97	420	0.793	6.2	83.5
	1.27	0.08	97.3	2800	0.066	1.2 (11.6)	93.8 (99.3)

The stage cuts shown in the parentheses represent the overall values.

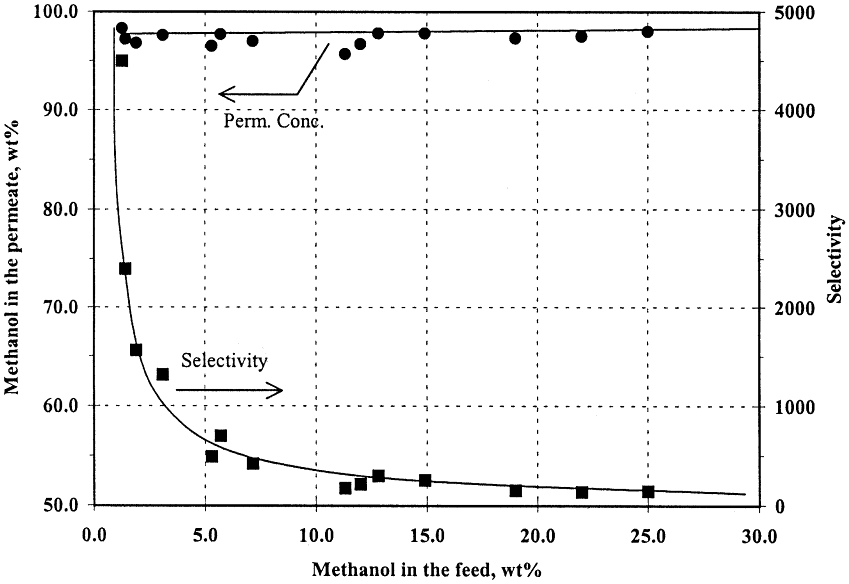

Figure 5. Results of the pervaporation experiments in which rubbery membrane B was used. Permeate composition and selectivity versus methanol concentration in the feed.

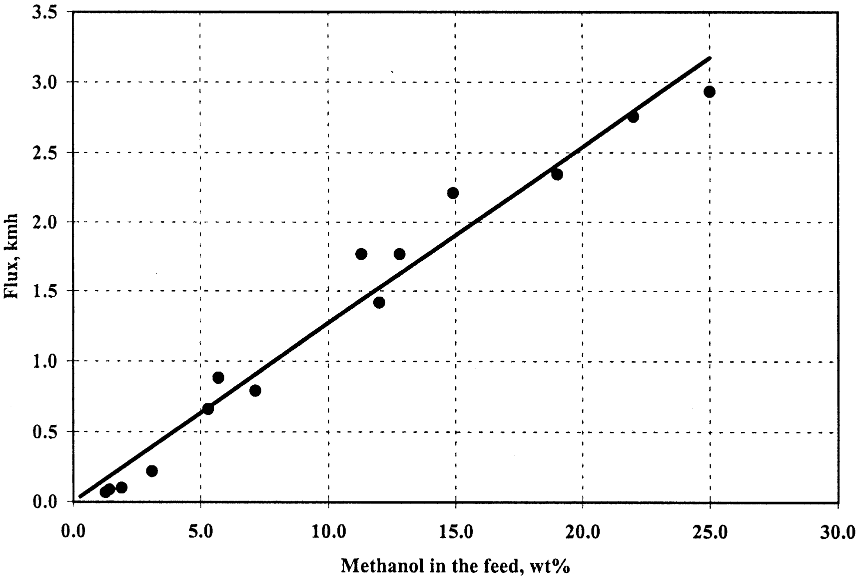

Figure 6. Results of the pervaporation experiments in which rubbery membrane B was used. Flux versus methanol concentration in the feed.

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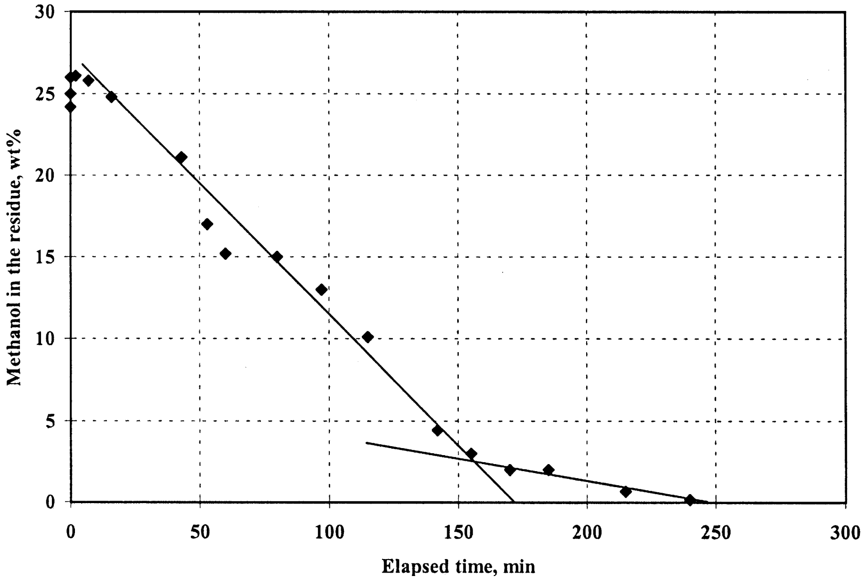

Figure 7. Depletion of methanol in the feed with time. Membrane B was used.

ing effects of high selectivity of the membrane toward methanol, on one hand, and the plasticization effect of this compound on the other. That is say, while the membrane is highly methanol selective, it is plasticized at higher feed concentrations, allowing more isopentane to permeate. As a result of the balance between these two effects, the methanol concentration in the permeate remains relatively independent of the feed concentration. This effect is manifested by a sharp decrease in membrane selectivity as the methanol feed concentration is increased as shown in Table 4 and Fig. 5. The selectivity ranged from around 150 to 2800 with a sharp initial decline at low feed concentrations followed by a plateau at higher concentrations. The presence of a plateau in the curve of selectivity is evident in this figure. The asymptotic value of selectivity was approximately 170.

Figure 6 shows the influence of methanol feed concentration on permeate flux. The dependance was linear ($R^2 = 97\%$) and flux increased with increasing feed methanol concentration. Membrane B demonstrated a larger permeability toward methanol than did membrane A. Permeability is defined as rate of material transferred from the feed side to the permeate side through a unit area of a membrane, under unit pressure, and at unit time.The enhanced flux value with membrane B was associated with a loss in the selectivity.

In addition to the above tests, one single continuous experiment was performed to determine the depletion rate of methanol in the feed with time. The results are shown in Table 4 as experiment No. 1 and in Fig. 7. In this experiment, 30.00 g of feed at 25.0% (wt) methanol was pervaporated until its concentration decreased to 0.15% (wt) (1500 wppm). The composition of the residue was determined at time intervals during the experiment and the values were plotted against time as shown in Fig. 7.

Two distinct regions are observed in the figure. The initial region is that indicated from the starting time to around 150 minutes, which corresponds to a residue concentration ranging from 25% (wt) to approximately 3% (wt). The second region is that defined by an elapsed time from 150 minutes to 250 minutes and corresponds to a residue concentration of 3% (wt) to 0.15% (wt). In both regions the dependency of residue methanol concentration on time was approximated with linear relationships defined by distinctly different slopes. The slope of the line defining the first region was larger than that of the second line indicating a fast depletion of methanol. The slope at this region was approximately −0.15% (wt)/min, which corresponds to a flux of −0.045 g/min or −2.4 kmh. In the second region, the slope decreased significantly, indicating a slower rate of methanol transfer through the membrane. The slope of the line defining this region was approximately −0.04% (wt)/min or a flux of −0.012 g/min (−0.62 kmh). The change in the slope can be explained by the increased resistance of the boundary layer introduced at low concentrations of methanol. At the end of the experiment, a total of 6.25 g of permeate at an average purity of 97.4% (wt) methanol was collected. The overall isopentane recovery was over 99% at a purity of 99.85% (wt).

Table 5 and Figs. 8 and 9 show the results obtained using Nafion-170 membrane. Feed concentration was varied between 20.3% (wt) to 2.0% (wt) methanol in isopentane. Over this range, the permeate concentration was relatively constant between 96.2% (wt) and 99.8% (wt) methanol, and the concentration of methanol in the residue decreased to 0.055% (wt) (550 wppm). Both the permeate concentration and the membrane selectivity showed similar trends discovered through the use of membranes A and B. That is, the permeate concentration remained relatively independent of the feed composition. Also, the high selectivity of the Nafion-170 membrane declined sharply as the feed concentration increased and reached an average asymptotic value of 370.

Table 5. Results of MeOH/iC5 Separation with Nafion-170 Membrane

Experiment No.	MeOH% (wt) Feed	Residue	Permeate	Selectivity	Flux kmh
1	20.3	18.4	99.0	410	0.776
	18.4	12.3	98.0	350	0.597
	12.3	6.5	98.0	350	0.554
	6.5	2.0	96.2	360	0.413
	2.0	0.055	99.8	24 500	0.099

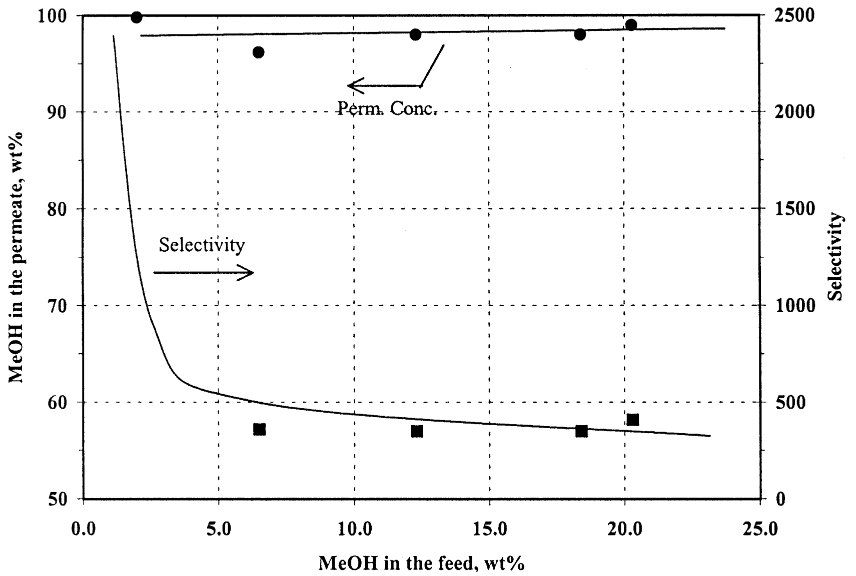

Figure 8. Results of the pervaporation experiments in which Nafion-70 membrane was used. Permeate composition and the selectivity versus methanol concentration in the feed.

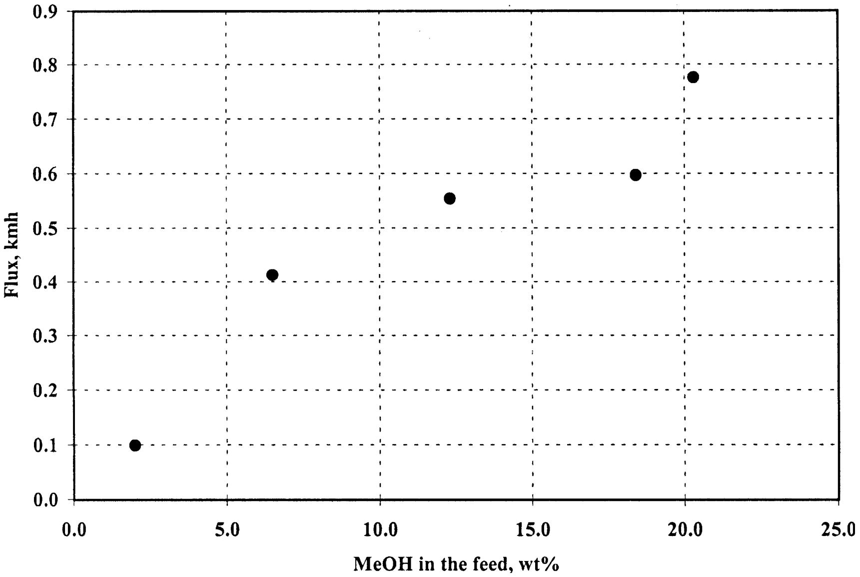

Figure 9. Results of the pervaporation experiments in which Nafion-170 membrane was used. Flux versus methanol concentration in the feed.

The variation of flux with feed concentration is shown in Fig. 9. Although a linear increase of the flux was expected with increasing feed concentration, a deviation from linearity was observed: $R^2 = 82\%$. Therefore, no attempt was made to fit a curve to the data points. However, the flux demonstrated by the Nafion-170 membrane was in the same range as that of membrane A and was smaller than that of membrane B.

CONCLUSIONS

All three membranes tested demonstrated effective flux and selectivity values for the methanol/isopentane system and were able to strip the methanol component from the methanol/iC_5 mixture, and hence, exceeding the target value of <1000 wppm. A common characteristic among the 3 membranes was an elevated methanol concentration in the permeate that was independent of the feed concentration. Therefore, a single-stage pervaporation system can efficiently recover the methanol content of the raffinate stream in a distillation/pervaporation hybrid. Such a system would eliminate the need for a water wash and the secondary distillation stage necessary in conventional MeOH/iC_5 separation processes. The differences found for the permeability and selectivity values of the tested membranes provide a design flexibility that will allow operators to chose based on the purity and flux requirements of the process. Also, membrane preconditioning was found to positively affect membrane selectivities.

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