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Specialty Polymeric Membranes. 13. Separation of Benzene/Cyclohexane Mixtures through Poly(Vinylidene Chloride-*co*-Vinyl Chloride)-*graft*-Poly(Butyl Methacrylate) Membranes

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ABSTRACT

Poly(vinylidene chloride-*co*-vinyl chloride)-*graft*-poly(butyl methacrylate), which was prepared by a radical graft polymerization of butyl methacrylate onto poly(vinylidene chloride-*co*-vinyl chloride) and poly(vinylidene chloride-*co*-vinyl chloride), itself was found to be suitable for the membrane materials for the selective separation of benzene from benzene/cyclohexane mixtures in the whole feed composition range. The permeation rate was enhanced by the introduction of poly(butyl methacrylate) onto poly(vinylidene chloride-*co*-vinyl chloride) membrane.

Key Words. Polymeric membranes; Pervaporation; Graft polymerization; Benzene; Cyclohexane

INTRODUCTION

Separation of aromatics/aliphatics mixtures by pervaporation is one of potential applications of pervaporation separation to industry in connection with petroleum refinery, and these kinds of studies have been investigated since 1967 (1, 2). If the membrane shows permselectivity toward aromatics, aromatics-lean retentate is useful for diesel and an aromatic-rich permeate can be used as high octane number gasoline. Based on this, the authors' research

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group has been studying novel membrane materials for pervaporation separation of aromatics/aliphatics mixtures. As described previously (2), membrane materials should be chosen from commercially available common polymers or chemically modified common ones without complicated modification reaction, because large volumes of mixtures should be treated by the membrane thus prepared. To this end, poly(vinyl chloride) (2), nylon 6 (3–5), and poly(vinyl alcohol) (6) were adopted as membrane materials, and the pervaporation performances of the membranes from modified polymers and unmodified ones were reported. Poly(vinylidene chloride), like poly(vinyl chloride), is also susceptible to a simple chemical modification of radical graft polymerization (7–9).

In the present article, poly(vinylidene chloride-*co*-vinyl chloride) (PVDC/PVC), which can give a more durable membrane than the homopolymer of poly(vinylidene chloride), was adopted as a parent polymer. Novel membrane materials were prepared by a radical graft polymerization of butyl methacrylate onto parent PVDC/PVC. Pervaporation performances of benzene/cyclohexane mixtures through modified PVDC/PVC and unmodified PVDC/PVC membranes were investigated.

EXPERIMENTAL

Materials

Poly(vinylidene chloride-*co*-vinyl chloride) (PVDC/PVC), in which vinylidene chloride unit mole fraction was determined to be 0.902, was kindly provided by Asahi Chemical Industry Co., Ltd. Butyl methacrylate (BMA) (10), benzene for graft polymerization (11), and hexane (11) were purified before being used. Molybdenum hexacarbonyl was sublimed in vacuum (7). Benzene for pervaporation, cyclohexane, tetrahydrofuran (THF), and tetrahydrofuran-d_8 were used without further purification.

Graft Polymerization

Graft polymerization of BMA onto PVDC/PVC was carried out as follows (8, 9): a 300 cm^3, three-necked, round-bottom flask equipped with condenser, overhead stirrer, and thermometer was used. Into 50 cm^3 of polymerization solvent containing PVDC/PVC powder and a prescribed amount of BMA, 40 cm^3 of the same solvent with a prescribed amount of $Mo(CO)_6$ was added, and the reaction mixture was stirred at 80°C. After 24 hours the reaction mixture was filtered, and the precipitate thus obtained was washed with benzene for 48 hours. After that, the precipitate was filtered off and dried in vacuo. Benzene was use as a solvent for the preparation of PVDCBMA-098, PVDCBMA-155, and PVDCBMA-200, and hexane for that of PVDCBMA-614 and PVD-

CBMA-750, where the final last three numbers in the sample code show the ratio of unit mole of grafted PBMA to that of PVDC/PVC.

Spectroscopic Measurement

IR spectra were taken on a JASCO FT/IR-5300 Fourier Transform Infrared Spectrometer.

^{1}H-NMR spectra were obtained with a Bruker ARX-500. The spectra were measured at ambient temperature, using a 100 g dm^{-3} tetrahydrofuran-d_8 solution with tetramethylsilane (TMS) as an internal standard.

Preparation of Membranes

Membranes were prepared from THF solution. The membranes were prepared as follows: 0.300 g of polymer was dissolved in 5 cm^3 of THF. The THF solution thus obtained was poured into a flat laboratory dish (7.0 cm diameter) and the solvent allowed to evaporate at 25°C for 24 hours. The obtained membrane was dried at 50°C for an additional 2 hours. The thickness of the membrane thus obtained was 38–49 μm.

Pervaporation

Permeation of the benzene/cyclohexane mixture was performed by an ordinary pervaporation technique (12). The membrane area in contact with the liquid feed was 17.3 cm^2. The downstream pressure applied was kept at around 267 Pa (2.0 mmHg). Pervaporation experiments were carried out at a constant temperature of 50°C.

Separation analysis was carried out on a Shimadzu GC-8APT gas chromatograph equipped with a 3.0 m long column packed with polyethylene glycol 6000 (Shimalite TPA).

The separation factor, α, is defined as

$$\alpha = (Y_{\mathrm{benzene}}/Y_{\mathrm{cyclohexane}})/(X_{\mathrm{benzene}}/X_{\mathrm{cyclohexane}})$$

where the Y_is are the weight fractions in the permeate and $\mathrm{X_i}$s are those in the feed.

Solubility Measurement

Solubility selectivities of PVDCBMA-200 and PVDC/PVC membranes were measured as described previously (13). The solubility selectivity, S_{S}, is defined as

$$S_{\mathrm{S}} = (Z_{\mathrm{benzene}}/Z_{\mathrm{cyclohexane}})/(X_{\mathrm{benzene}}/X_{\mathrm{cyclohexane}})$$

where Z_is are the weight fractions in the membrane and $\mathrm{X_i}$s are those in the solution.

RESULTS AND DISCUSSION

Radical Graft Polymerization

The introduction of BMA onto PVDC/PVC was confirmed by IR and ^{1}H-NMR spectra of graft polymers. An example of IR spectra is shown in Fig. 1. In the IR spectrum a new absorption peak at 1725 cm^{-1}, which is assigned to the C═O stretching band of PBMA, can be detected. The existence of PBMA in PVDC/PVC was also confirmed by ^{1}H-NMR measurement. The ^{1}H-NMR spectrum of PVDCBMA-200 is shown in Fig. 2. The signal assigned to methyl protons (h) in PBMA is newly observed at around 1.0 ppm and methylene protons (b) in butyl moiety at around 4.0 ppm. The intensity of methylene protons (b, c and d) and the area of methyl protons (h) led to a (PBMA)/(PVDC/PVC) ratio of 0.200. The results of graft polymerization are summarized in Table 1. In the sample code the final three numbers show the ratio of unit mole of grafted PBMA to that of PVDC/PVC. The effect of the amount of initiator for the radical graft polymerization on the composition of grafted polymer is given in Fig. 3. The content of PBMA grafted onto PVDC/PVC increased with an increase in the amount of Mo(CO)$_6$. The relationship between the composition ratio and amount of Mo(CO)$_6$ was not linear. The composition ratio will be asymptotic at higher Mo(CO)$_6$ concentrations. This might be due to the fixed BMA concentration in the present study.

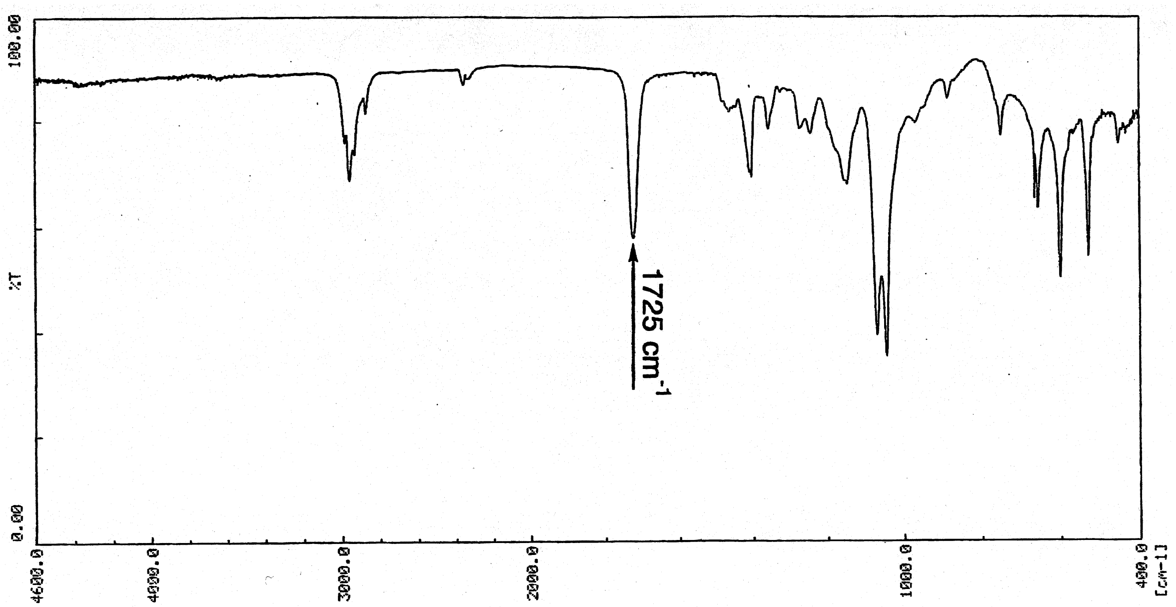

FIG. 1 IR spectrum of PVDCBMA-200. [(PBMA)/(PVDC/PVC) = 0.200.]

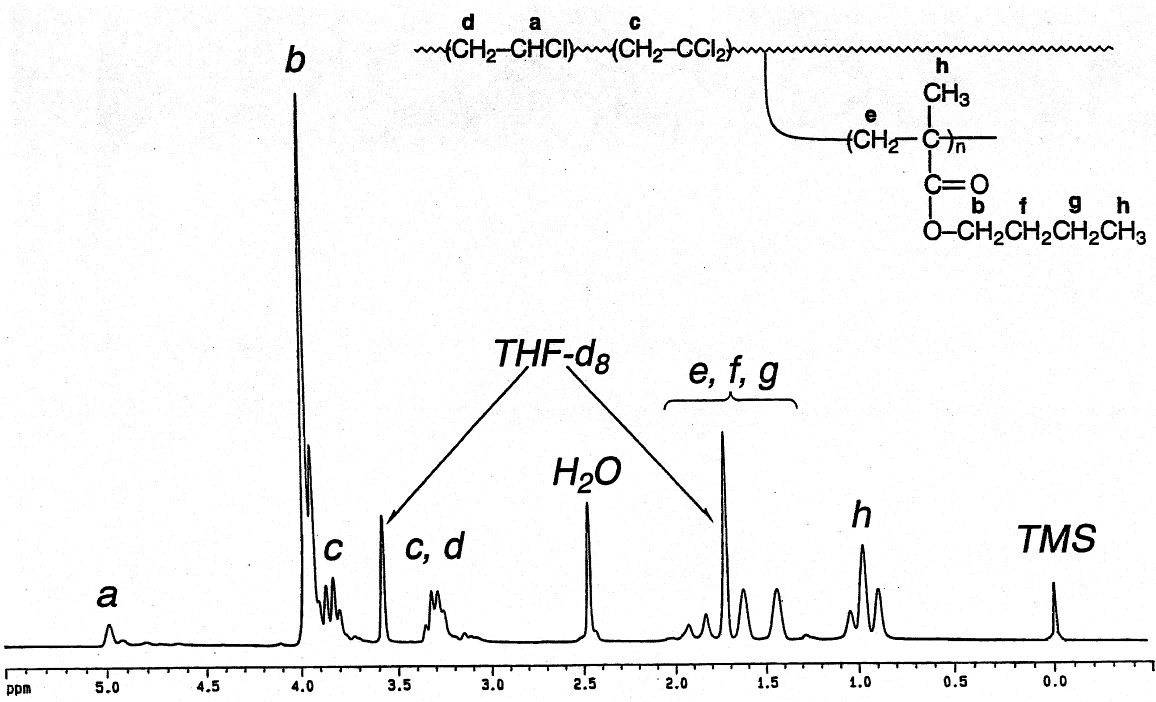

FIG. 2 ^{1}H-NMR spectrum of THF-d_8 solution of PVDCBMA-200. [(PBMA)/(PVDC/PVC) = 0.200.]

TABLE 1

Preparation and Characterization of PVDC/PVC-*graft*-PBMA

	Charged amounts				PVDC/PVD-*graft*-PBMA	
Sample	PVDC/ PVC/g	BMA/g	$Mo(CO)_6$/g	$(Mo(CO)_6)$/ (PVDC/PVC)[a]	Yield/g	(PBMA)/ (PVDC/PVC)[b]
PVDCBMA-098[c]	4.00	1.24×10	1.15×10^{-3}	1.00×10^{-4}	2.85	0.098
PVDCBMA-155[c]	4.00	1.23×10	1.73×10^{-3}	1.51×10^{-4}	2.72	0.155
PVDCBMA-200[c]	4.00	1.24×10	2.30×10^{-3}	2.00×10^{-4}	2.68	0.200
PVDCBMA-614[d]	4.00	1.24×10	1.20×10^{-2}	1.00×10^{-3}	1.45	0.614
PVDCBMA-750[d]	4.00	1.23×10	2.30×10^{-2}	2.00×10^{-3}	1.69	0.750

[a] Ratio for mole of reacted $Mo(CO)_6$ to unit mole of reacted PVDC/PVC.

[b] Ratio for unit mole of PBMA grafted to unit mole of PVDC/PVC.

[c] Graft polymerization was carried out at 80°C for 24 hours in 90 cm^3 of benzene.

[d] Graft polymerization was carried out at 80°C for 24 hours in 90 cm^3 of hexane.

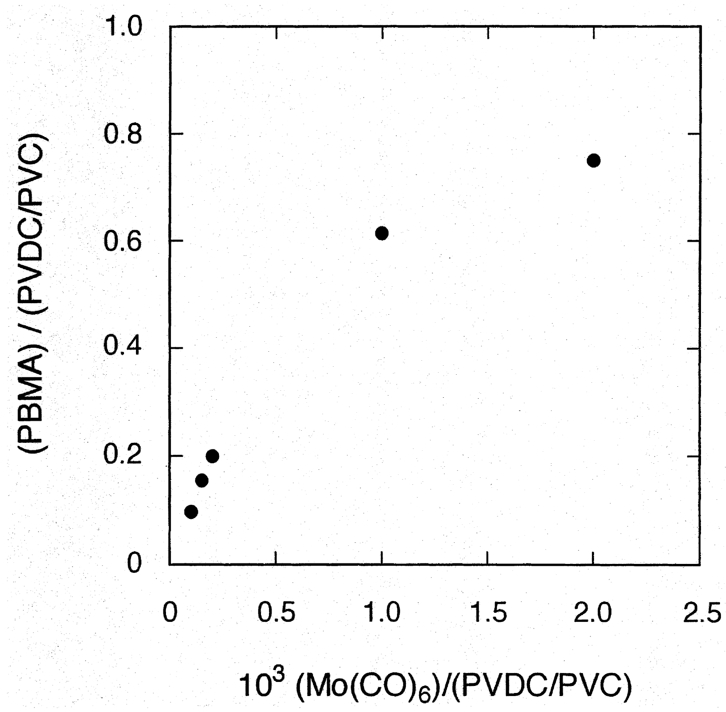

FIG. 3 Relationship between composition of PVDC/PVC-*graft*-PBMA and graft polymerization condition. (Graft polymerization was carried out at 80°C.)

Pervaporation of Benzene/Cyclohexane through PVDCBMA Membranes

Table 2 summarizes the effect of PBMA content on pervaporation of benzene/cyclohexane mixture, where the weight fraction of benzene in the feed was fixed to be around 0.5. The result for the unmodified membrane is also given in the table. In the radical graft polymerization it was difficult to obtain a membrane material with a low PBMA content. The results in Table 2 are also shown visually in Fig. 4. The membrane performance depends on PBMA

TABLE 2
Pervaporation of Benzene/Cyclohexane Mixture through PVDC/PVC-*graft*-PBMA Membranes[a]

Membrane	(PBMA)/(PVDC/PVC)	Thickness/μm	X_{benzene}[b]	Y_{benzene}[b]	α[c]	J/g·m^{-2}·h^{-1}
PVDC/PVC	0	44	0.0991	0.3960	6.0	9.2
PVDCBMA-098	0.098	39	0.0992	0.4485	7.4	4.8
PVDCBMA-155	0.155	38	0.0996	0.3920	5.8	6.9
PVDCBMA-200	0.200	49	0.0992	0.3740	5.4	14.0
PVDCBMA-614	0.614	38	0.0991	0.3240	4.4	33.1
PVDCBMA-750	0.750	40	0.0995	0.2211	2.6	80.3

[a] Pervaporation was carried out at 50°C; downstream pressure, ~267 Pa (2 mmHg).

[b] Weight fraction of benzene.

[c] $\alpha = (Y_{\mathrm{benzene}}/Y_{\mathrm{cyclohexane}})/(X_{\mathrm{benzene}}/X_{\mathrm{cyclohexane}})$.

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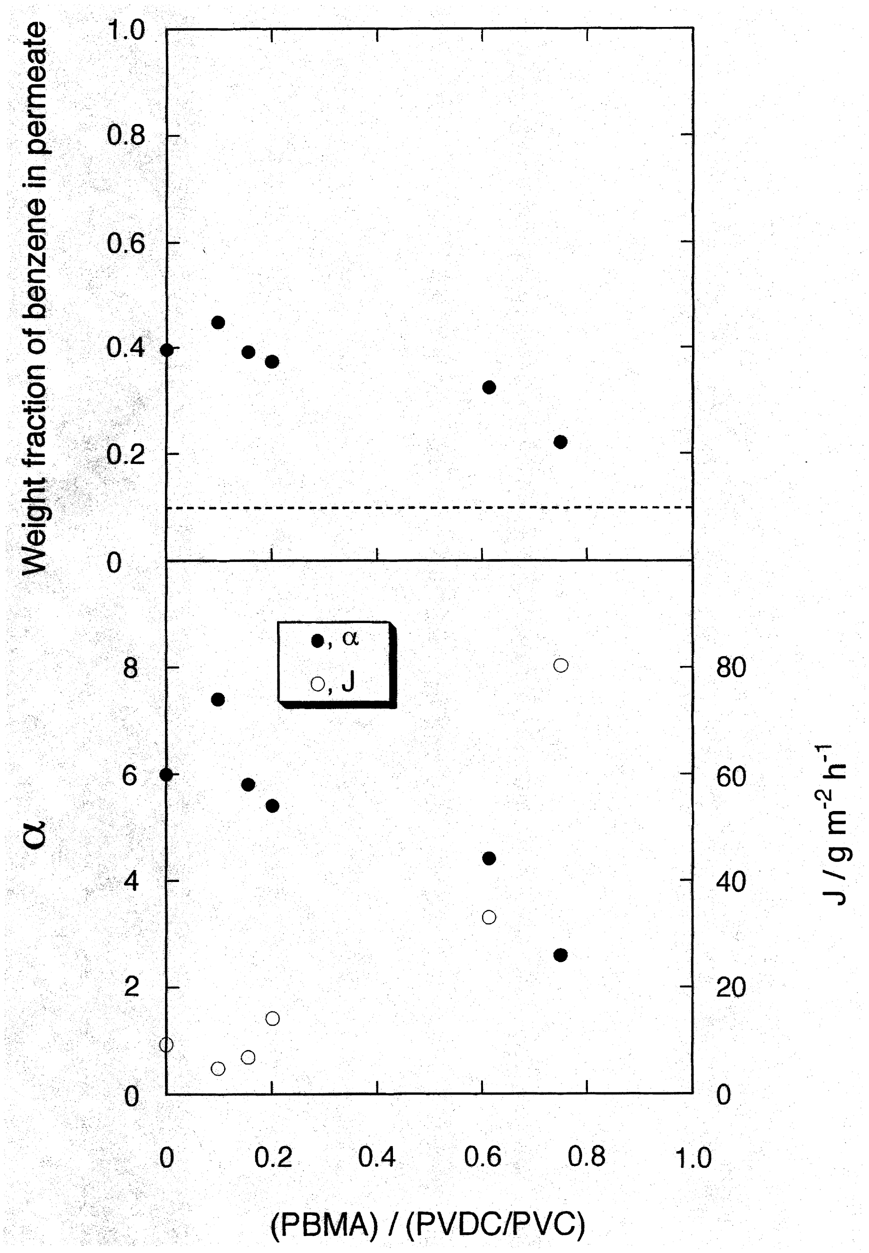

FIG. 4 Effect of composition of PVDC/PVC-*graft*-PBMA on pervaporation of benzene/cyclohexane mixture. [Weight fraction of benzene in feed, ca. 0.1; operating temperature, 50°C; downstream pressure, ca. 267 Pa (2.0 mmHg).]

content, and maximum permselectivity is shown at a (PBMA)/(PVDC/PVC) ratio of 0.098. As reported in the pervaporation of benzene/cyclohexane mixture through poly(vinylidene fluoride) membrane containing 3-methylsulfolene (14), the flux value increased with an increase in the PBMA content in the membrane, while the selectivity toward benzene decreased when the (PBMA)/(PVDC/PVC) ratio exceeded 0.098. Permselectivity toward benzene was still maintained at a (PBMA)/(PVDC/PVC) ratio of 0.750, which is the highest graft ratio in the present study. This is due to the introduction of PBMA onto PVDC/PVC; that is, the introduction of PBMA, which shows affinity toward benzene, onto PVDC/PVC increased the permselectivity toward benzene, while the introduction of PBMA simultaneously led to swelling of the membrane when the membrane was in contact with the feed mixture. As a result, the permselectivity decreased with an increase in PBMA content as shown in Fig. 4.

The dependence of feed composition on pervaporation performance for modified PVDC/PVC and unmodified PVDC/PVC membranes was studied and is summarized in Table 3. PVDCBMA-200 membrane was adopted as a modified PVDC/PVC membrane. Figure 5 shows the results from Table 3 of pervaporation of the benzene/cyclohexane mixture through the PVDCBMA-200 membrane, and those of PVDC/PVC are given in Fig. 6 where the membrane performance is plotted against the weight fraction of benzene in the

TABLE 3

Pervaporation of Benzene/Cyclohexane Mixtures through PVDC/PVDC-*graft*-PBMA (PVDCBMA-200) Membrane[a]

(PBMA)/(PVC)[b]	X_{benzene}[c]	Y_{benzene}[c]	α[d]	$J/\text{g}\cdot\text{m}^{-2}\cdot\text{h}^{-1}$
0.200[e]	0.0992	0.3740	5.4	14.0
0.200[e]	0.2983	0.6073	3.6	39.3
0.200[e]	0.4994	0.7212	2.6	86.1
0.200[e]	0.7002	0.8159	1.9	131.8
0.200[e]	0.9009	0.9324	1.5	165.3
0[f]	0.0991	0.3960	6.0	9.2
0[f]	0.2993	0.6715	4.8	22.2
0[f]	0.5004	0.7846	3.6	47.2
0[f]	0.7004	0.8857	3.3	37.3
0[f]	0.9013	0.9646	3.0	52.6

[a] Pervaporation was carried out at 50°C; downstream pressure, ~267 Pa (2 mmHg).
[b] Ratio for unit mole of PBMA grafted to unit mole of PVDC/PVC.
[c] Weight fraction of benzene.
[d] $\alpha = (Y_{\text{benzene}}/Y_{\text{cyclohexane}})/(X_{\text{benzene}}/X_{\text{cyclohexane}})$.
[e] PVDCBMA-200 membrane; membrane thickness, 49 μm.
[f] PVDC/PVC membrane; membrane thickness, 44 μm.

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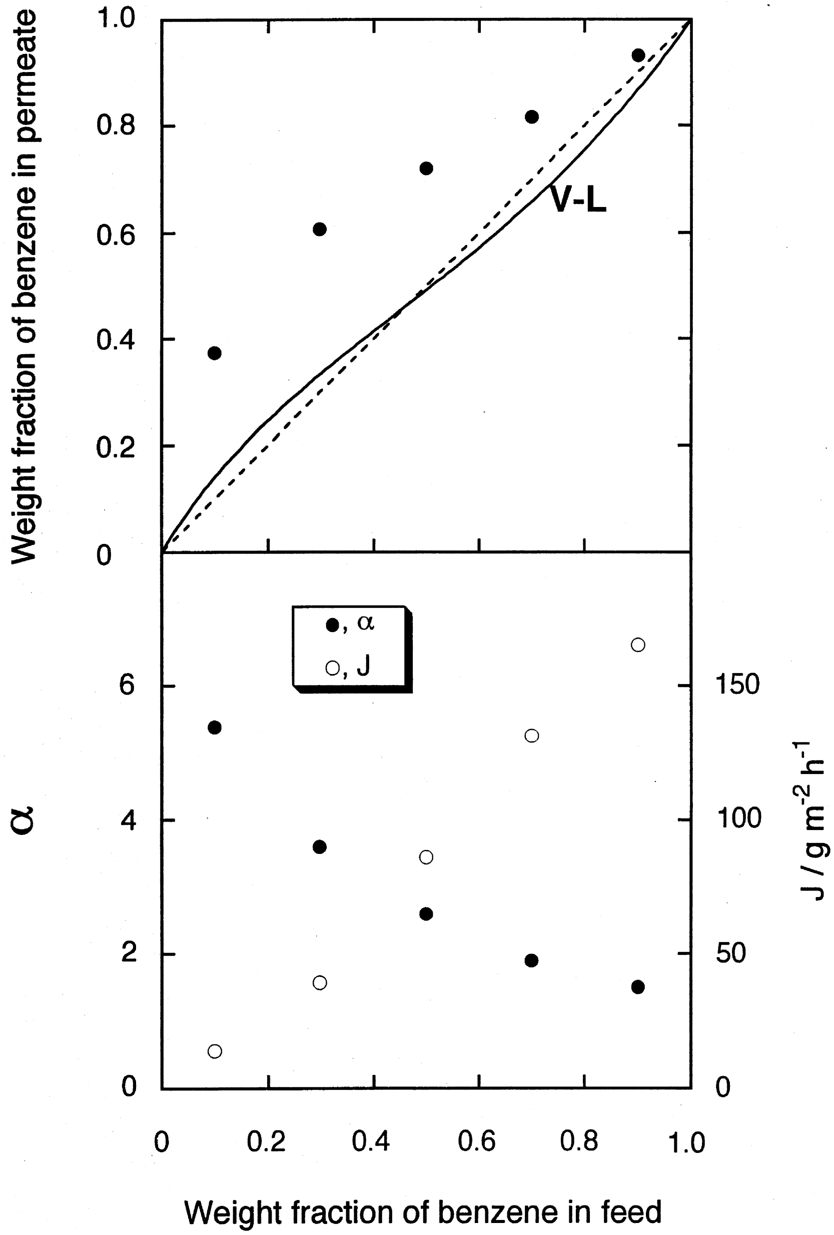

FIG. 5 Effect of feed composition on pervaporation, separation factor (α), and flux (*J*) of benzene/cyclohexane mixtures through PVDC/PVC-*graft*-PBMA (PVDCBMA-200) membrane. [(PBMA)/(PVDC/PVC) = 0.200; operating temperature, 50°C; downstream pressure, ca. 267 Pa (2.0 mmHg).]

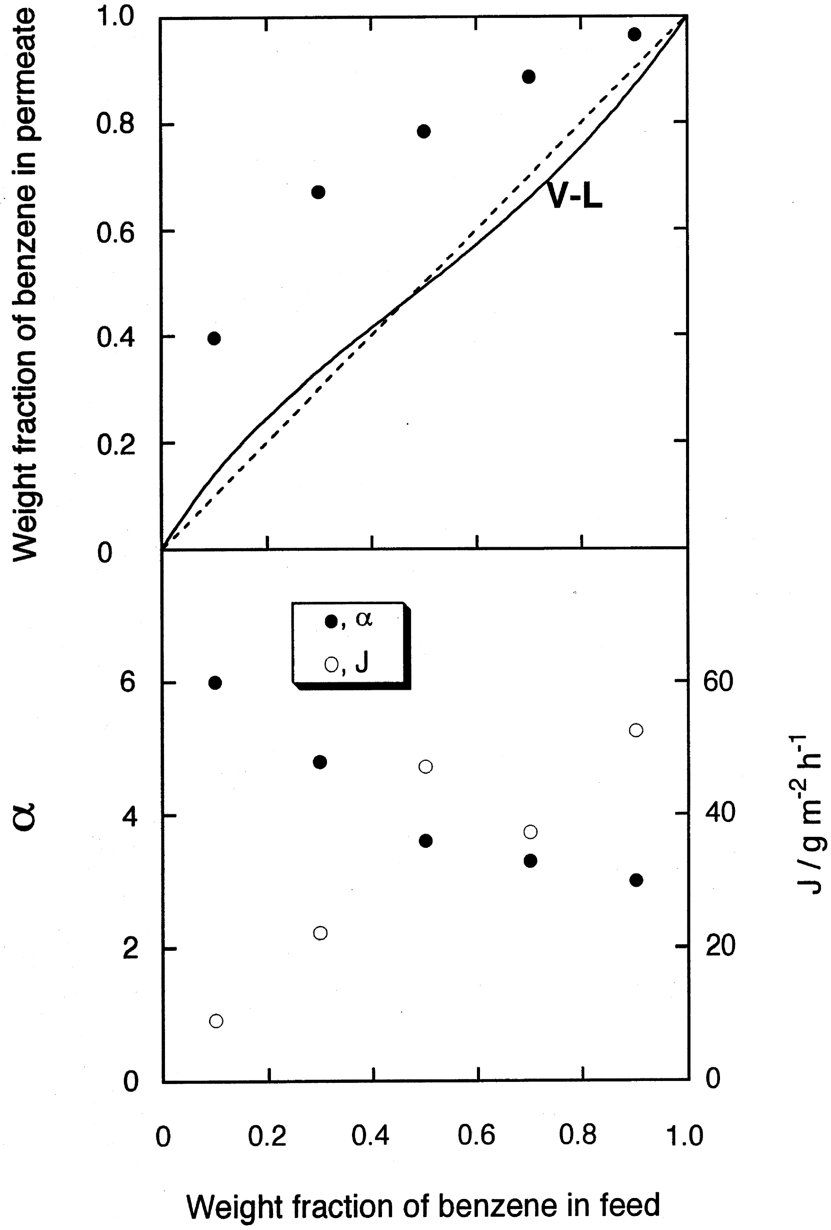

FIG. 6 Effect of feed composition on pervaporation, separation factor (α), and flux (*J*) of benzene/cyclohexane mixtures through PVDC/PVC membrane. [Operating temperature, 50°C; downstream pressure, ca. 267 Pa (2.0 mmHg).]

feed. Both membranes gave a similar performance. The vapor–liquid equilibrium curve for benzene–cyclohexane mixtures at 50°C (15) is also given in these two figures. As expected from the results shown in Table 2 and Fig. 4, the PVDC/PVC membrane gave slightly higher permselectivity toward benzene in whole feed range than did the PVDCBMA-200 membrane. Even in pervaporation with PVDCBMA-200 membrane, the weight fraction of benzene in the permeate was higher than the vapor–liquid equilibrium throughout the whole feed composition. The modified PVDC/PVC membrane gave higher flux values than the PVDC/PVC membrane.

Sorption

The results of sorption of benzene/cyclohexane mixture in PVDCBMA-200 and PVDC/PVC membranes are summarized in Table 4. These results are shown visually in Figs. 7 and 8, respectively. PVDC/PVC membrane showed a slightly higher affinity toward benzene than the modified PVDC/PVC one in the whole solution composition range. This can be explained by the fact that PBMA, which can be swollen by one component of the solution, benzene, was contained in the PVDCBMA-200 membrane.

Using the sorption data, solubility selectivity, S_S, can be obtained. According to the solution-diffusion theory, diffusivity selectivity, S_D (=

TABLE 4

Sorption of Benzene/Cyclohexane Mixtures in PVDC/PVC-*graft*-PBMA (PVDCBMA-200) and PVDC/PVC Membranes at 50°C

(PBMA)/(PVDC)[a]	X_{benzene}[b]	Z_{benzene}[b]	Sorbed benzene g/g-membrane	Sorbed cyclohexane g/g-membrane	S_S[c]	S_D[d]
0.200[e]	0.0972	0.1570	0.021	0.110	1.7	3.2
0.200[e]	0.2910	0.3935	0.068	0.102	1.6	2.3
0.200[e]	0.4988	0.5938	0.099	0.068	1.5	1.7
0.200[e]	0.6988	0.7820	0.163	0.046	1.5	1.3
0.200[e]	0.9012	0.9313	0.209	0.015	1.5	1.0
0[f]	0.0967	0.1692	0.022	0.107	1.9	3.2
0[f]	0.2940	0.4198	0.056	0.077	1.7	2.8
0[f]	0.4991	0.6343	0.084	0.049	1.7	2.1
0[f]	0.6964	0.7981	0.133	0.034	1.7	1.9
0[f]	0.9010	0.9525	0.160	0.008	2.2	1.4

[a] Ratio for unit mole of PBMA grafted to that of PVDC/PVC.
[b] Weight fraction of benzene.
[c] $S_S = (Z_{\text{benzene}}/Z_{\text{cyclohexane}})/(X_{\text{benzene}}/X_{\text{cyclohexane}})$.
[d] $S_D = \alpha/S_S$.
[e] PVDCBMA-200 membrane.
[f] PVDC/PVC membrane.

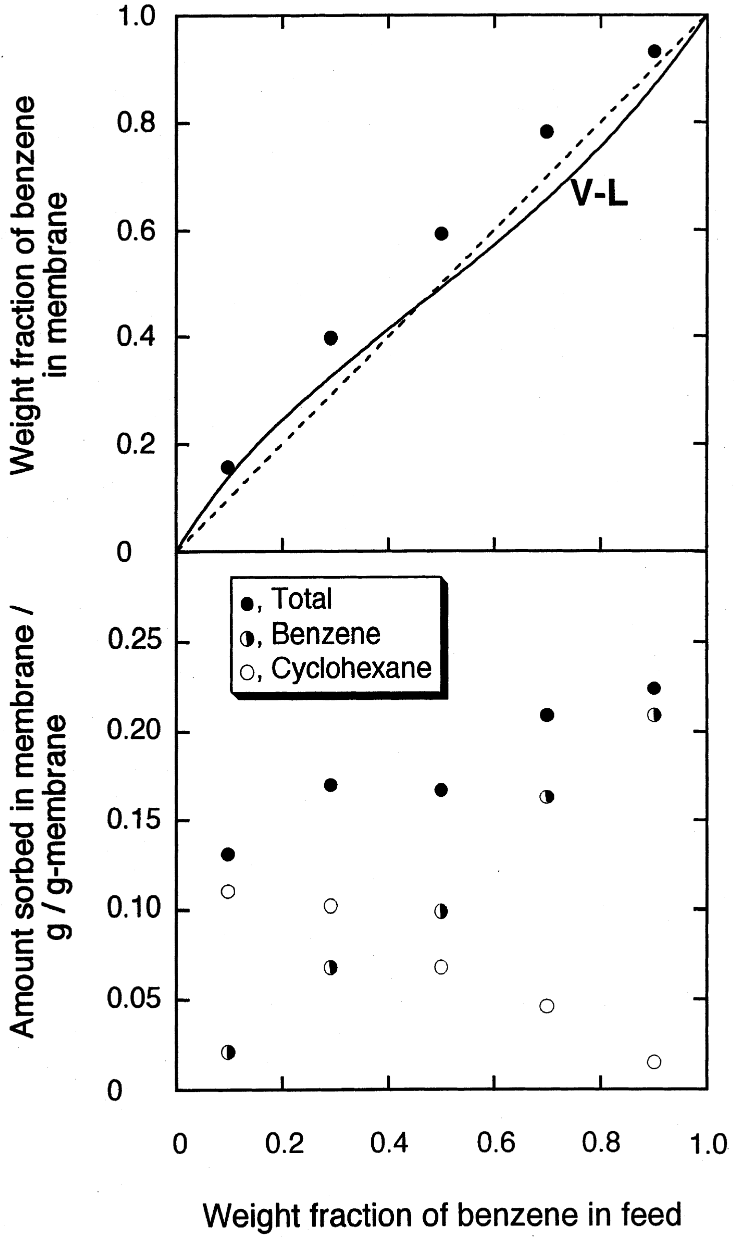

FIG. 7 Effect of feed composition on the composition of solution sorbed in membrane and on total and individual sorptions for PVDC/PVC-*graft*-PBMA (PVDCBMA-200) membrane at 50°C. [(PBMA)/(PVDC/PVC) = 0.200.]

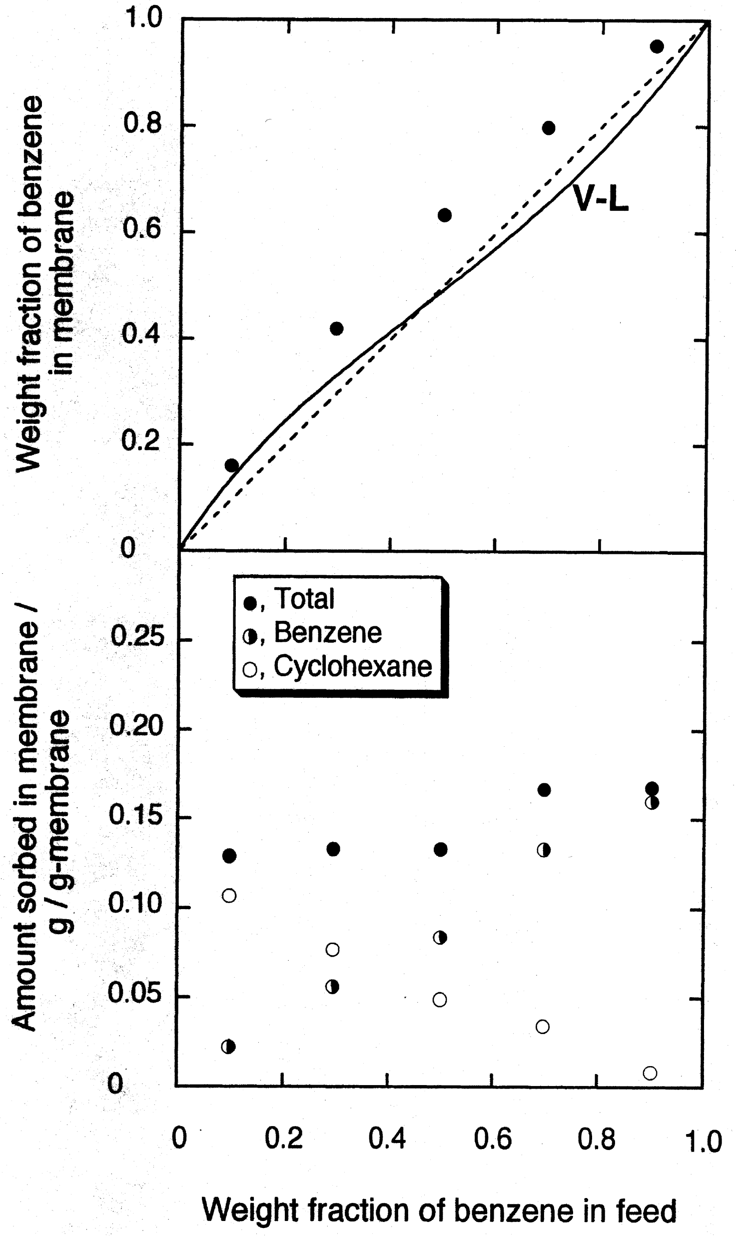

FIG. 8 Effect of feed composition on the composition of solution sorbed in membrane and on total and individual sorptions for PVDC/PVC membrane at 50°C.

$D_{\text{benzene}}/D_{\text{cyclohexane}}$; D_{benzene} and $D_{\text{cyclohexane}}$ are the diffusion coefficients of benzene and cyclohexane, respectively), can be estimated from the separation factor, α, and the solubility selectivity, S_S (= $S_{\text{benzene}}/S_{\text{cyclohexane}}$; S_{benzene} and $S_{\text{cyclohexane}}$ are the solubility coefficients of benzene and cyclohexane, respectively), thus obtained (16). Separation factor, solubility selectivity, and diffusivity selectivity for PVDCBMA-200 and PVDC/PVC membranes are shown in Fig. 9 as a function of the weight fraction of benzene in the feed. Both membranes gave not only benzene solubility selectivity but also benzene diffusivity selectivity. As a result, both membranes showed permselectivity toward benzene in the whole feed composition range.

CONCLUSIONS

From the present study, the following conclusions can be drawn:

1. Poly(vinylidene chloride-*co*-vinyl chloride)-*graft*-poly(butyl methacrylate) and poly(vinylidene chloride-*co*-vinyl chloride) membranes perme-

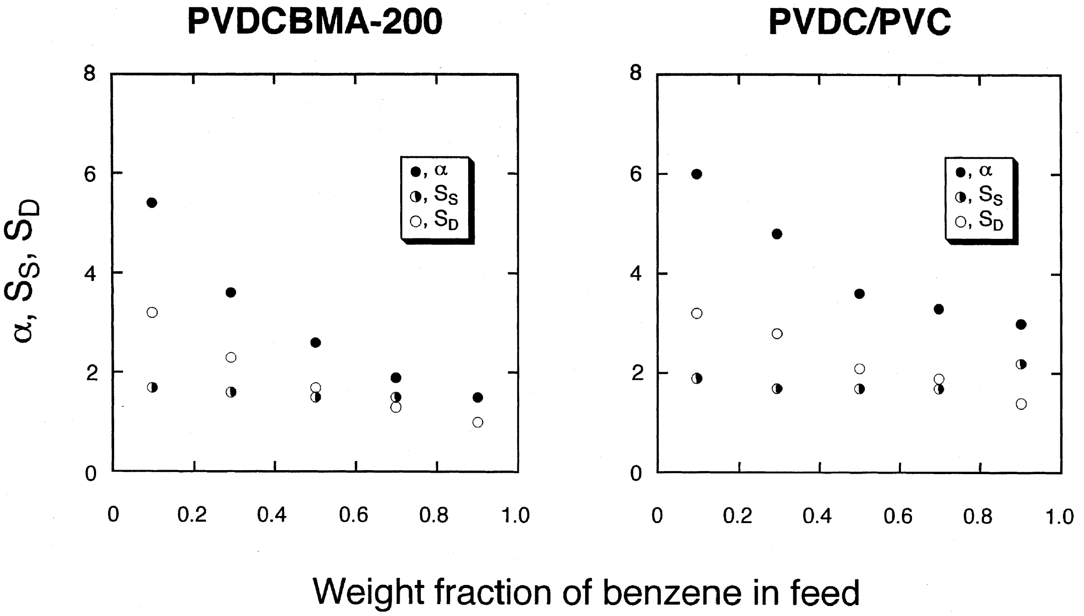

FIG. 9 Effect of feed composition on separation factor (α) solubility selectivity (S_S), and diffusivity selectivity (S_D of PVDC/PVC and PVDC/PVC-*graft*-PBMA (PVDCBMA-200) [(PBMA)/(PVDC/PVC) = 0.200] membranes. [Operating temperature, 50°C; downstream pressure, ca. 267 Pa (2.0 mmHg).]

ated benzene in preference to cyclohexane from benzene/cyclohexane mixtures.

2. Introduction of poly(butyl methacrylate) onto poly(vinylidene chloride-*co*-vinyl chloride) by radical graft polymerization led to about a threefold augmentation in the flux values in comparison with unmodified membranes.
3. Poly(vinylidene chloride-*co*-vinyl chloride)-*graft*-poly(butyl methacrylate) and poly(vinylidene chloride-*co*-vinyl chloride) membranes have potential for application in petroleum refining processes.

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