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Separation of CO₂ from CO₂/N₂ Mixture Using Supported Polymeric Liquid Membranes at Elevated Temperatures

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ABSTRACT

Novel supported polymeric liquid (SPL) membranes have been prepared and shown to be applicable for the separation of CO₂ from mixtures with N₂ at the elevated temperature encountered in flue gas. The membranes were fabricated by immobilizing polystyrene, polyethylene, and polydimethylsiloxane into the pores of borosilicate glass supports. At 250°C, the CO₂ permeability and CO₂/N₂ separation factors were 3000–9000 barrer and 1.7–3.7, respectively. It was shown that polymers which have a lower T_g or melting temperature than the operating temperature can be used as SPL membrane materials at elevated temperatures.

Key Words. Carbon dioxide; Gas separation; Supported polymeric liquid membranes; Separation factor; Permeability

INTRODUCTION

The increased concentration of CO₂ in the atmosphere is considered as the main reason for global warming. Separation of CO₂ from hot flue gas is thought to be a key technology for the protection of the global environment from the greenhouse effect (1). In recent years, increasing attention has been given to CO₂ separation using membranes due to the low energy consumption and simplicity of the process.

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Polymer membranes show considerable selectivity of CO_2 to N_2 at room temperature (2–5), but their thermal stability is insufficient for use above the glass transition temperature (T_g). Several types of polyimide membranes which have relatively high T_g s have been used to separate CO_2 at elevated temperatures (6, 7).

Carbon membranes produced from polymeric precursors have also been investigated as a promising alternative (8–12). Hayashi et al. (9) coated 3,3'-4,4'-biphenyltetracarboxylic dianhydride-4,4'-oxidianiline (BPDA-ODA) polyimide on a porous alumina support tube and carbonized the polyimide to form a thin carbon layer. CO_2/N_2 permselectivity was reported as 15 at 100°C. However, no permeation experiment was carried out above 100°C although the temperature of flue gas is typically 150°C or higher.

Inorganic membranes which are thermally stable at high temperatures have been used to separate CO_2 from mixed gases. CO_2 permeability of inorganic membranes is in the 10–10⁵ barrer range. These values are higher than those of polymer membranes. Because the separation factor of CO_2/N_2 by Knudsen diffusion is only 0.8, modification of inorganic membranes should be introduced to improve selectivity (13–18). Silica membranes (17) and zeolite membranes (18) show especially good performance at elevated temperatures. Kusakabe et al. (18) prepared a Y-type zeolite membrane on a porous α -alumina support; its CO_2/N_2 separation factor was reported to be 20 at 130°C.

Since diffusion of gases is much faster in liquids than in solids, liquid membranes are candidates for CO_2 separation provided their thermal stability is guaranteed. Liquid membranes typically take advantage of a reversible reaction with CO_2 to achieve high selectivity (19–21). However, they suffer from both the loss of membrane materials during extended period of operation and the vaporization of active materials at elevated temperatures.

In the present study we developed novel composite membranes, called supported polymeric liquid (SPL) membranes by impregnating polymers into the pores of a porous inorganic support to separate CO_2 at elevated temperatures. In our approach, polymers which have high selectivity for CO_2 were used as active membrane materials in the liquid state at elevated temperatures. Polymeric liquid is trapped in the pores of the inorganic support by capillary force. The main idea is to separate two functions of conventional polymeric membranes—mechanical stability and selectivity—and then to assign the former to the inorganic support and the latter to polymers in the liquid state. Therefore, polymers which have a lower T_g or melting temperature (T_m) than the operating temperature can be used as SPL membrane materials. In what follows, the permeability and CO_2/N_2 separation



factor of SPL membranes are reported, and the thermal stability of SPL membranes is discussed.

EXPERIMENTAL

Materials

A disk-type porous borosilicate glass (Millipore) was used as the support of the SPL membranes. The physical properties of the support are summarized in Table 1. Carbon dioxide (high purity, >99.95%), nitrogen (ultrahigh purity, >99.99%) and helium (ultrahigh purity, >99.99%) were supplied by BOC Gases Korea Co. Ltd.. Polyethylene (PE), polystyrene (PS), and tetrahydrofuran (THF, 99.5%) were supplied by Aldrich Chemical Inc. 1-Phenylazo-2-naphthalenamine (Yellow AB) and polydimethylsiloxane (PDMS) were supplied by Tokyo Kasei Kokyo Co. and Shin-Etsu Chemical Co. Ltd., respectively. Table 1 also shows the properties of the SPL membrane materials. All materials in this study were used as received without further treatment. T_g and T_m of polymers were measured by using a differential scanning calorimeter (TA instrument, DSC 3000).

TABLE 1
Physical Properties of Selected Materials and the Support

Support		
Manufacturer	Millipore	
Model number	APFF04700	
Material	Borosilicate glass	
Shape	Disk	
Diameter	47 mm	
Average pore diameter	2.7–7 μ m	
Thickness	500 μ m	
Thermal stability	Maximum: 550°C	
Membrane material		
Material	T_g/T_m	Molecular weight (viscosity at 25°C)
PE	110°C (T_m)	M_w : 13,000
PS	13°C (T_g)	M_w : 760
PDMS 1	Liquid at room temperature	Viscosity: 50 cs ^a
PDMS 2	Liquid at room temperature	Viscosity: 10^3 cs
PDMS 3	Liquid at room temperature	Viscosity: 10^5 cs
PDMS 4	Liquid at room temperature	Viscosity: 3×10^5 cs
Yellow AB	103°C	247

^a1 cs = 10^{-2} cm²/s.



Preparation of SPL Membranes

The SPL membranes were fabricated by putting the polymeric materials into the pores of the support. Three different methods were used for the fabrication of the SPL membranes. The first is to wet the support with drops of a polymeric liquid at room temperature. PDMSs are viscous liquids at room temperature. The support readily absorbed PDMS and turned translucent when its pores were filled with PDMS. Excess PDMS on the surface of the support was removed by wiping it off with tissue paper.

The second method is to heat a polymer above its T_m and to wet the support. PE beads were heated to 110–115°C, and the support was wetted to be translucent. Yellow AB was impregnated by the same method.

The third method is to dissolve the membrane material in a solvent, wet the support, and evaporate the solvent. PS was dissolved in THF, and the support was soaked with this solution. THF was later removed at 90°C by vaporizing it in an oven. This procedure was repeated several times to obtain maximum pore filling.

The morphology of the SPL membranes was observed by using a scanning electron microscope (Phillips, SEM 515).

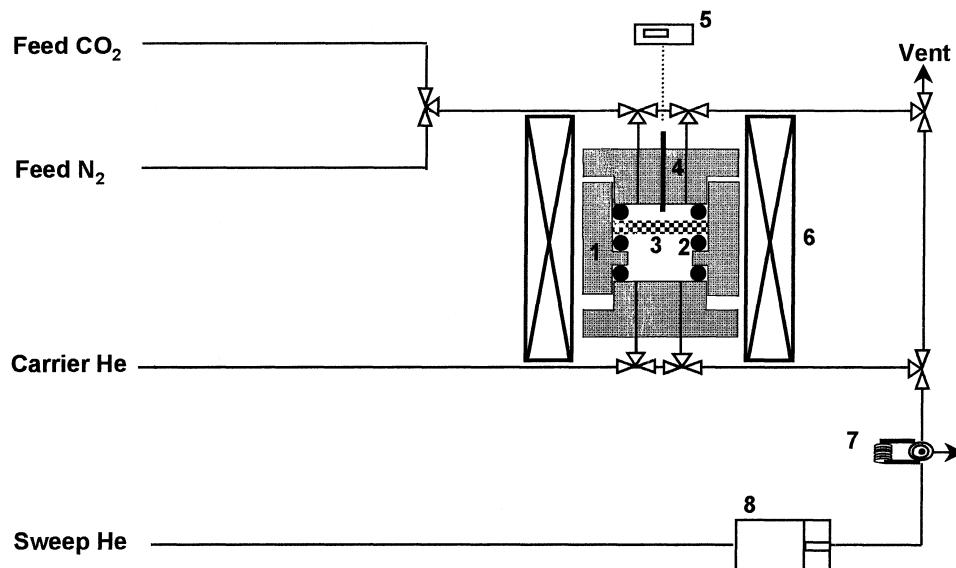
Gas Permeation

The apparatus used to measure the permeability of SPL membranes at elevated temperatures is similar to that described by Pez et al. (22). Figure 1 schematically depicts the complete setup for the permeation experiment. The membrane test cell consists of an SPL membrane, three Teflon O-rings which are stable up to 320°C, a thermocouple located 1–2 mm above the SPL membrane, $\frac{1}{8}$ inch stainless steel feed lines, and $\frac{1}{8}$ inch stainless steel permeate lines. The prepared SPL membrane was fitted firmly by Teflon O-rings in the gas permeation cell. The membrane area was 8.84 cm² for all gas permeation experiments. The membrane test cell was located in the tube furnace (Thermolyne, 21100) which was held at constant temperature with a precision of $\pm 1^\circ\text{C}$.

Mixed gases were produced by blending CO₂ and N₂ using mass flow controllers (MKS 247C). All gases were streamed through bypass lines. The system was heated to the test temperature, and helium gas was used to sweep the air in the lines and the gas permeation cell. After confirming that no nitrogen peak appeared in gas chromatography (Young-In 680D), all gases which had been streamed through bypass lines were switched to the feed line and the permeate line. A mixture of CO₂ and N₂ was fed to the feed side of the membrane test cell.

The membrane test cell consisting of the SPL membrane was brought to a steady state with a CO₂ and N₂ feed gas mixture whose total flow rate was 30





1. Membrane cell 2. O-ring 3. SPL Membrane 4. Thermocouple
5. Temperature reader 6. Furnace 7. Sampling valve 8. Gas chromatography

FIG. 1 Schematic diagram of gas permeation apparatus.

sccm [sccm = cm³(STP)/min]. The flow rates of helium sweep streams were 25 sccm on both sides of the membrane at 76 cmHg. In this study the mole ratio of CO₂ over N₂ was fixed at 0.2, which is the typical composition of flue gas (23). The permeate side was swept with helium gas which flowed to the gas chromatograph. The gases were separated in the packed column (Porapak, 80/100 mesh) and analyzed with a thermal conductivity detector (TCD).

RESULTS AND DISCUSSIONS

SPL Membranes

PDMS, PS, and PE were selected as SPL membrane materials. PDMS and PS are amorphous polymers and PE is a crystalline polymer. Four kinds of PDMS were used to study the effect of molecular weight (evidenced as different viscosities in Table 1) on the permeation of carbon dioxide. Thermal stabilities of the SPL membranes were demonstrated by comparing them with the Yellow AB liquid membrane.

Figure 2 shows the top and the fractured surfaces of the supports and the SPL membranes. Polymeric materials are uniformly distributed throughout the support and form liquid membranes. Some cracks were found on the fractured sur-



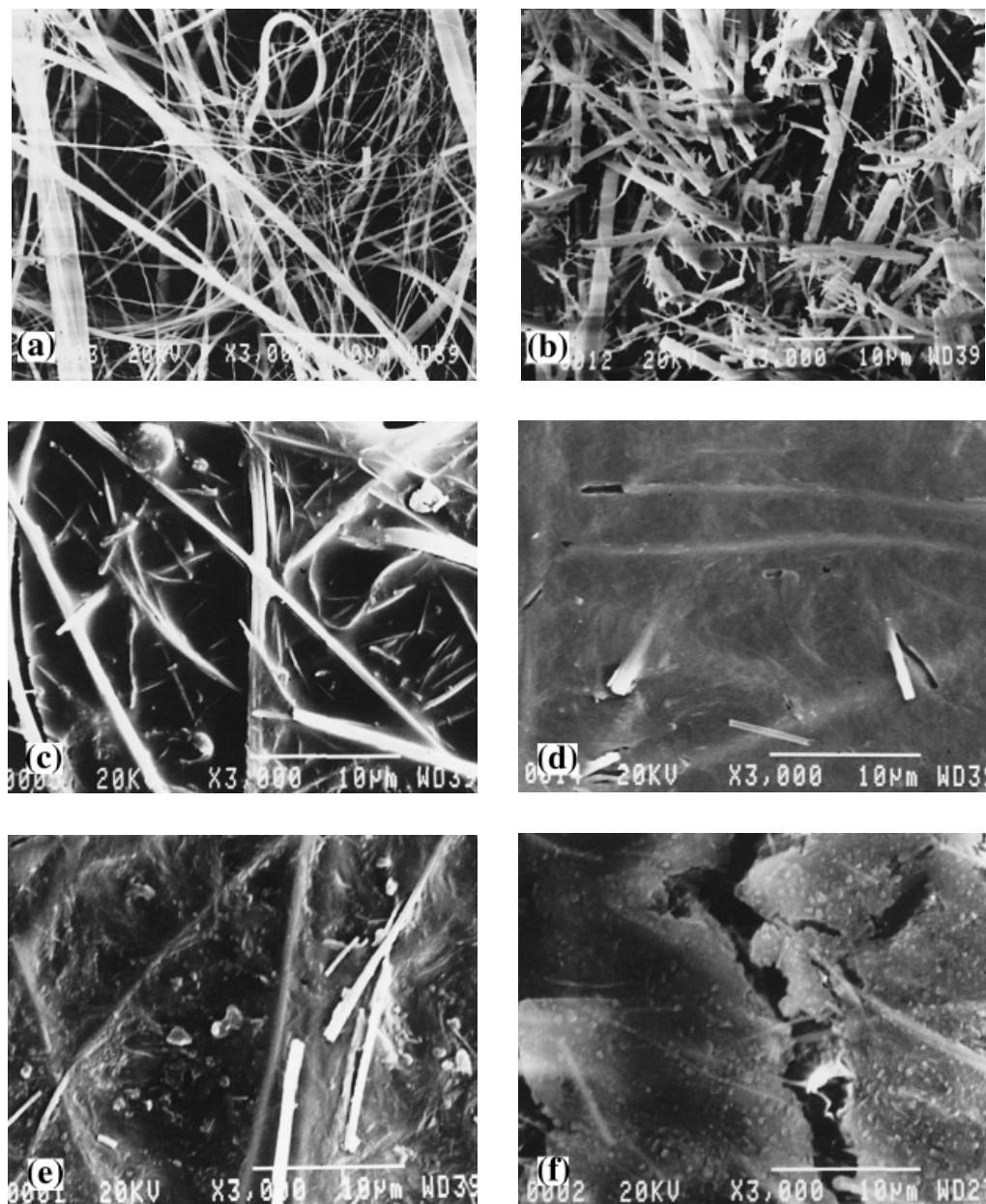


FIG. 2 SEM image of support and SPL membranes ($\times 3000$): (a) Surface of support, (b) fracture surface of support, (c) surface of PS SPL membrane, (d) fracture surface of PS SPL membrane, (e) surface of PE SPL membrane, (f) fracture surface of PE SPL membrane.

face in Fig. 2(f). This crack disappeared during gas permeation experiments at 120–250°C because T_m of PE is only 110°C and PE became a low viscosity liquid at these temperatures. Since PDMSs exist in a liquid state at room temperature, the SEM images are not included for the PDMS membranes.



Gas Permeation

Permeation characteristics of the SPL membranes are described using a one-dimensional Fickian diffusion model. The steady-state flux of permeate gas is converted into the permeability coefficient by using

$$P = Ql/\Delta pA$$

where Q is the steady-state flux of permeate gas, Δp is the pressure difference of a particular gas across the SPL membrane, A is the membrane area (11 cm²) and l is the membrane thickness (0.05 cm). The permeability defined by Eq. (1) is an experimental permeability subject to the experimental conditions as described by Pez et al. (24).

Separation factor α (CO₂/N₂) is calculated from Eq. (2):

$$\alpha = \frac{y/(1-y)}{x/(1-x)} \quad (2)$$

where x and y are the mole fractions of CO₂ at the permeate and the feed sides, respectively. Permeability and separation factors of PS SPL membranes are given in Table 2. This table shows that CO₂ and N₂ permeability both increase with increasing temperature while the separation factor decreases from 11.1 at 100°C to 3.7 at 250°C. On the other hand, the PE SPL membranes show a

TABLE 2
Permeability (P , barrer) and Separation Factor (α , CO₂/N₂) of the SPL Membranes

Material	P (CO ₂ /N ₂)	Temperature (°C)					
		50	100	120 (115)	150	200	250
PDMS 1	P (CO ₂ /N ₂)	10,890/1832	9473/2534		8431/3186	8558/8810	8811/5573
	α	6.2	4.1		2.7	1.9	1.6
PDMS 2	P (CO ₂ /N ₂)	8730/1376	8021/1793		7300/2226	6824/3653	6671/4150
	α	6.6	4.6		3.4	2.0	1.7
PDMS 3	P (CO ₂ /N ₂)	7849/1016	6143/1304		5724/1749	5280/2415	5744/2720
	α	8.0	4.9		3.5	2.4	2.2
PDMS 4	P (CO ₂ /N ₂)	7256/926	5883/1262		5624/1541	5058/1918	5032/2179
	α	8.2	5.1		3.8	2.8	2.4
PS	P (CO ₂ /N ₂)		1271/118	1697/263		2121/387	2357/662
	α		11.1		6.7	5.7	3.7
PE	P (CO ₂ /N ₂)			2012/269	1985/414	1856/528	1816/613
	α			6.6	4.6	3.3	2.7
Yellow AB	P (CO ₂ /N ₂)			(1171/118)	1495/232	1831/392	2395/686
	α			(10.5)	6.5	4.8	3.6



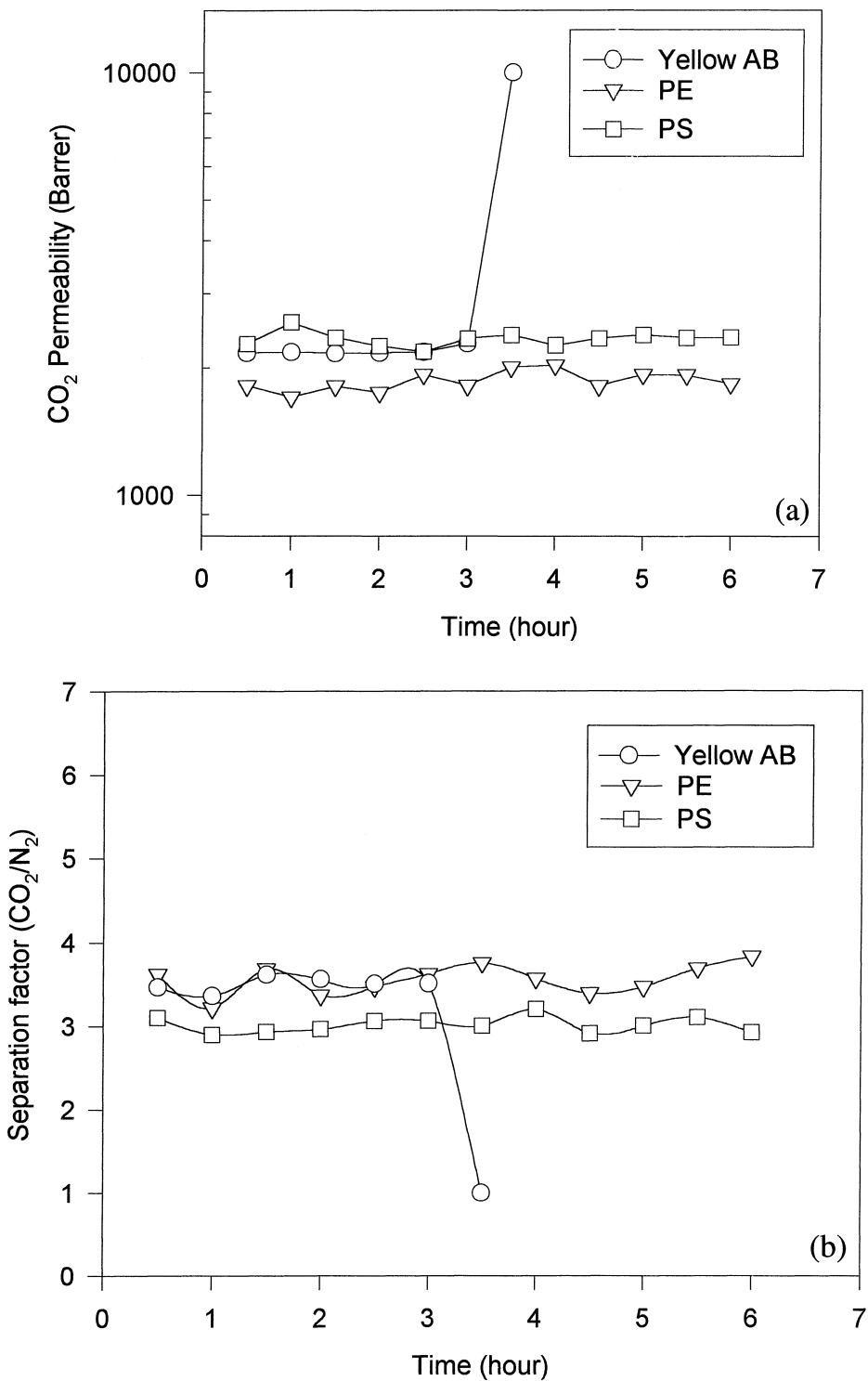


FIG. 3 Thermal stability of SPL membranes at 250°C. (a) CO₂ permeability, (b) Separation factor (CO₂/N₂).



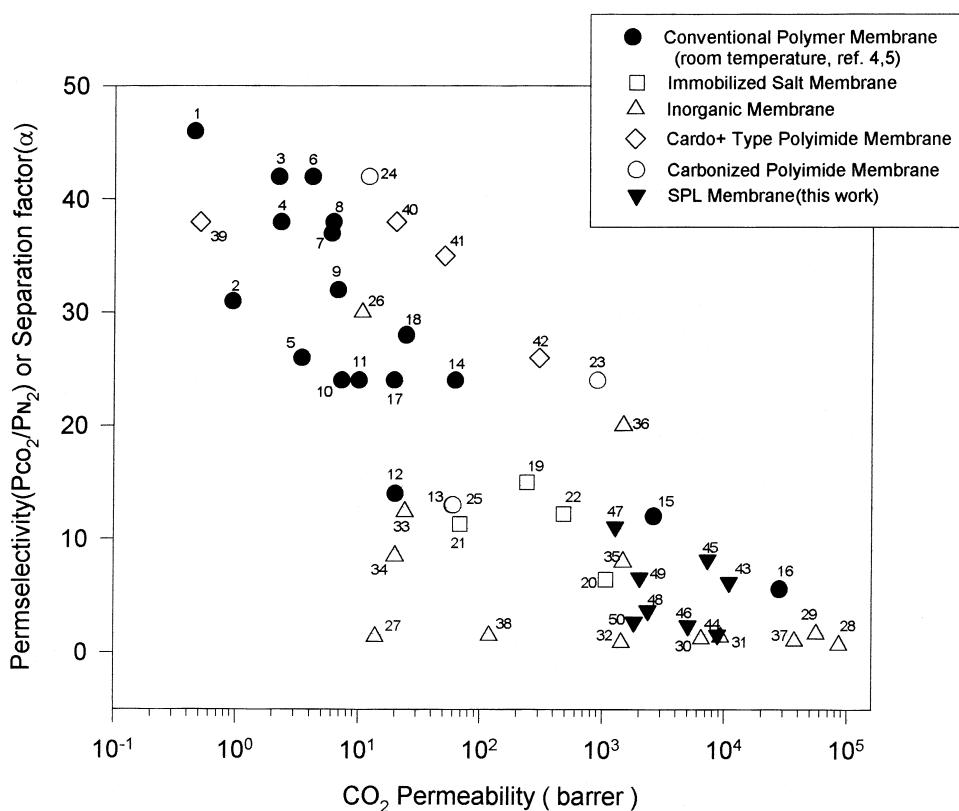


FIG. 4 Performance of various membranes for CO₂/N₂ separation. Cardo polymer means a series of polymers which have loop-like moieties in a principal chain. In the case of inorganic membranes, flux rates through the support material are high enough to be compared with those of an effective top layer. So the permeance unit (mol/m²·s·Pa) was converted to the permeability unit (cm³·cm/cm²·s·cmHg) by multiplying the approximate thickness of an effective top layer in permeance units and converting the units in order to make comparisons with polymeric membrane systems. 1: Polyimide; 2: Cellulose nitrate; 3: Polyvinyl acetate; 4: Cellulose acetate; 5: Polyvinyl butyral; 6: Polyethersulfone; 7: Polysulfone; 8: Cellulose triacetate; 9: Polycarbonate; 10: Polyethyl methacrylate; 11: Polystyrene; 12: Polyethylene; 13: Poly(4-methyl-1-pentene); 14: Polyphenylene oxide; 15: Silicone rubber; 16: Poly(1-trimethylsilyl-1-propyne); 17: 6FDA-DAF; 18: 6FDA-IPDA; 19: Tetrahexylammonium benzoate (40°C, Ref. 24); 20: Tetrahexylammonium benzoate (120°C, Ref. 24); 21: Tetrahexylammonium sarcosinate (40°C, Ref. 24); 22: Tetrahexylammonium sarcosinate (75°C, Ref. 24); 23: Carbonized polyimide (100°C, Ref. 8); 24: Carbonized polyimide (25°C, Ref. 9); 25: Carbonized polyimide (100°C, Ref. 9); 26: Silica-polyimide composite (30°C, Ref. 1); 27: Silica-polyimide composite (300 °C, Ref. 1); 28: γ -Al₂O₃ (20°C Ref. 16); 29: Iron-oxide-modified γ -Al₂O₃ (20°C, Ref. 16); 30: Ceramic zeolite composite (20°C, Ref. 25); 31: Silica-modified γ -Al₂O₃ (25°C, Ref. 13); 32: Silica-modified γ -Al₂O₃ (400°C, Ref. 13); 33: Silica membrane (150°C, Ref. 17); 34: Silica membrane (200°C, Ref. 17); 35: Y-type zeolite (130°C, Ref. 18); 36: Y-type zeolite (130°C, Ref. 18); 37: MgO-modified γ -Al₂O₃ (20°C, Ref. 15); 38: Si-O-C composite (400°C, Ref. 26); 39: Cardo polyimide (room temperature, Ref. 27); 40: Cardo polyimide (room temperature, Ref. 27); 41: Cardo polyimide (room temperature, Ref. 27); 42: Cardo polyimide (room temperature, Ref. 27); 43: PDMS 1 SPL membrane (50°C); 44: PDMS 1 SPL membrane (250°C); 45 PDMS 4 SPL membrane (50°C); 46: PDMS 4 SPL membrane (250°C); 47: PS SPL membrane (100°C); 48: PS SPL membrane (250°C); 49: PE SPL membrane (120°C); 50: PE SPL membrane (250°C).



slight decrease of CO_2 permeability with increasing temperature. This behavior can be understood by interpreting the permeability as a product of diffusivity and solubility. Solubility of CO_2 in PE at the polymeric liquid state (above T_m) decreases with increasing temperature. However, the diffusivity of CO_2 through PE increases with increasing temperature and the product of solubility and diffusivity results in a net decrease of permeability with increasing temperature. The experiments to verify this interpretation are ongoing.

To observe the behavior of the permeability and the separation factor with respect to viscosity (thus molecular weight), four kinds of PDMSs which have different viscosities were selected. All of the PDMS SPL membranes show a tendency for the CO_2/N_2 separation factor and CO_2 permeability to decrease with temperature while N_2 permeability increases. At the same temperature the CO_2/N_2 separation factor increases and permeability decreases with viscosity (thus molecular weight). This result can be attributed to the higher mobility of the polymer chain at a higher temperature and a lower viscosity. Compared with the PE and the PS SPL membranes, the PDMS SPL membranes show a higher permeability and a lower separation factor at the same temperature. The high flexibility of the Si-O linkage in PDMS may be responsible for this result.

The stability of the SPL membrane is demonstrated in Fig 3 by comparing it with a supported organic liquid membrane at the same experimental conditions. Yellow AB impregnated into the borosilicate support was stable for only 3 hours while SPL membranes showed no sign of degradation at 250°C.

In Fig. 4 the CO_2/N_2 permselectivity or separation factor is plotted as a function of CO_2 permeability for various membranes. In this figure the permeance unit ($\text{mol}/(\text{m}^2 \cdot \text{s} \cdot \text{Pa})$) of inorganic membranes was converted to the permeability unit ($\text{cm}^3 \cdot \text{cm}/(\text{cm}^2 \cdot \text{s} \cdot \text{cmHg})$) by multiplying by the approximate thickness of the effective top layer.

Inorganic membranes which are readily applicable to elevated temperature show higher CO_2 permeabilities than those of other membrane systems and a considerable separation factor even at elevated temperature. CO_2 permeabilities of the SPL membranes are much larger than those of conventional polymer membranes partly due to the liquid state transport and partly due to the elevated temperature of operation.

CONCLUSIONS

We have developed a novel composite membrane, called a supported polymeric liquid (SPL) membrane, to separate CO_2 at elevated temperatures. Polymeric liquid is trapped in the pores of the inorganic support by capillary force. The key idea is to separate two functions of conventional polymeric membranes—mechanical stability and selectivity—and then to assign the former to the inorganic support and the latter to polymers in the liquid state. Therefore,



polymers which have a lower T_g or melting temperature (T_m) than the operating temperature can be used as SPL membranes.

PE, PS, and PDMS were selected as the impregnation materials. SPL membranes with these polymers can separate CO₂ from CO₂/N₂ mixtures at 250°C. CO₂ permeability was much higher than for conventional polymeric membranes although the separation factor followed the general trend of separation factor–permeability diagrams. Proper selection of polymer materials can possibly result in higher separation factors.

SYMBOLS

A	surface area of membrane (cm ²)
Q	permeation flow rate (sccm)
l	membrane thickness (cm)
Δp	pressure difference across the membrane (cmHg)
P	permeability (cm ³ (STP)cm/cm ² ·s·cmHg)
D	diffusivity coefficient through membrane (cm ² /s)
S	solubility coefficient (cm ³ /cm ³)
α	separation factor
x	mole fraction of CO ₂ at the permeate side
y	mole fraction of CO ₂ at the feed side

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