

# Separation of Organic Vapor from Air by Aromatic Polyimide Membranes

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## SYNOPSIS

An attempt was made to prepare dry asymmetric membranes of aromatic polyimide material for the separation of air-organic vapor mixtures. Membranes with the permeability of organic vapor two orders of magnitude higher than that of air could be obtained. The membrane performance depends strongly on the conditions of membrane preparation. It was found that the permeability of organic vapors exhibits a strong correlation with their cohesive energy data.

## INTRODUCTION

Many industrial processes handling organic solvents produce solvent-containing air-exhaust streams. These streams cause not only severe air pollution problems but also a significant economic loss. The industries producing exhaust air streams contaminated by organic solvents have been under increasing economic and regulatory pressure. The sources of organic solvent emissions are quite fragmented. According to the U.S. Environmental Protection Agency, approximately 2 million tons of organic solvents are emitted to the atmosphere annually from solvent-based coating facilities,<sup>1</sup> and the synthetic organic chemical industry is first on the list as the single most significant contributor to the volatile organic compound emissions.<sup>2</sup> In spite of various emission sources, naphtha, toluene, xylene, perchloroethylene, trichloroethane, acetone, ethanol, and methanol together share almost 80% of the total emissions.<sup>3</sup> Most existing techniques to control organic vapor emissions such as adsorption, absorption, and condensation have so far proved to be unsatisfactory.<sup>4</sup> The separation and recovery of organic vapors from waste air streams using membrane

technology is expected to provide an attractive alternative.

The separation of organic vapor from air by means of a membrane is different from membrane gas-gas separation. Besides permeability and selectivity requirements, the ability of the polymer membrane to withstand organic vapor attack is also important. Although there is a great amount of data in the literature concerning gas permeation through polymeric membranes, there are much fewer data on the permeation of organic vapors.<sup>5</sup>

Based on the consideration of the contribution of partition coefficient and diffusion coefficient to the permeability of a membrane, Strathmann et al. concluded that elastomeric polymers such as silicone rubber should be used for the removal of organic solvents from air, where the higher molecular weight organic vapors should permeate preferentially.<sup>6,7</sup> Since then, most of the experimental work reported have been done using silicone rubber membranes. Strathmann et al.,<sup>6</sup> Kimmerle et al.,<sup>8</sup> and Paul et al.<sup>9</sup> tested silicone rubber membranes coated on a porous polysulfone substrate. Behling chose polyetherimide as a support material because it is much more stable against organic vapors than is polysulfone.<sup>10</sup> To reduce the resistance of nonselective support to vapor permeation, the substrate has to be highly porous. However, the pores on the membrane surface must be sufficiently small so that they can be bridged by the coating material. The experimental

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data of Pinnau et al. indicate that the resistance of the support layer is not negligible, and sometimes it is of the same order of magnitude as the resistance of the selective silicone rubber layer.<sup>11</sup> Therefore, the reported performance data of silicone rubber thin film composite membranes are quite different, depending on the properties of the support layer.

The appropriate membrane materials will be different for different organic vapors since a number of factors enter into material selection. For instance, the resistance of silicone rubber to gasoline is poor.<sup>12</sup> Hence, silicone rubber is not expected necessarily to be a good choice as the membrane material for the permeation of C<sub>6</sub>–C<sub>8</sub> hydrocarbons, which are the main components of gasoline.

In all the above work, the basic concept of the membrane design is to coat a thin layer of silicone rubber on the top of the porous sublayer made of a glassy polymeric material that is sufficiently resistant to the organic vapor. In this study, a new attempt is made to prepare a membrane using one glassy polymeric material alone without any silicone coating. Two requirements have to be fulfilled to develop a successful membrane for the permeation of the organic vapor. One is to introduce asymmetry to the membrane structure in order to increase the permeation rate. The other is to use a polymeric material that is sufficiently resistant in the organic vapor environment. Aromatic polyimide material was chosen for this purpose, since it is known that asymmetric reverse osmosis membranes with small pore sizes on the membrane surface can be produced from this polymeric material and also that the polymer can withstand the organic liquid during reverse osmosis and ultrafiltration experiments. Membranes are prepared under different conditions, tested for the permeation of air and organic vapors, and the results reported.

## EXPERIMENTAL

### Materials

Aromatic polyimide (PI 2080) was supplied by the Upjohn Company. The polymer powder was dried at 120°C for 16 h before use. A reagent grade *N,N*-dimethylacetamide (DMAc), supplied by BDH Chemicals, served as solvent for the preparation of membrane casting solutions. Lithium chloride (LiCl) from Fisher Scientific was dried at 140°C for 4 h before being used as an additive. Ethanol, acetone, *p*-xylene, 1,2-dichloroethane, benzene, chloroform, and carbon tetrachloride were supplied by

BDH Chemicals; methanol and hexane, by Fisher Scientific; 1,2-dichloropropane, by Matheson & Bell; and *m*-xylene, by Phillips Petroleum Company. Industrial-grade hydrogen, nitrogen, and air gases were obtained from Air Products. The purities of hydrogen and nitrogen were specified as 99.95% and 99.997%, respectively. The total content of hydrocarbon impurities in the air was stated by the supplier to be less than 25 ppm. The gases were used without further purification.

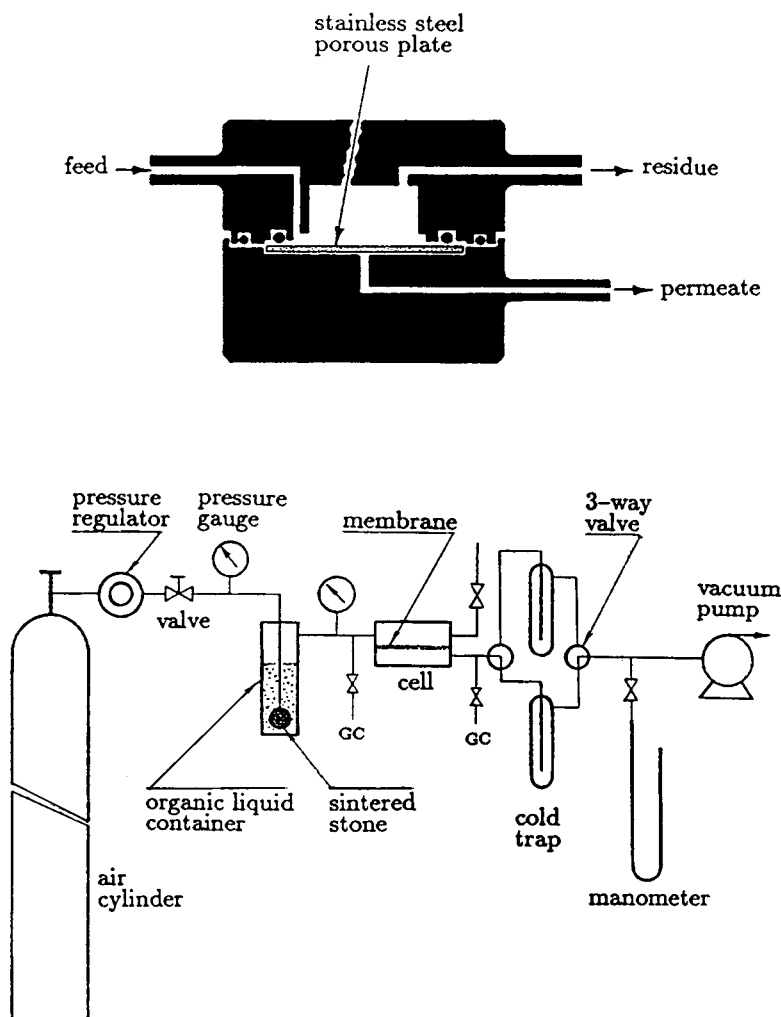
### Membrane Preparation

LiCl was dissolved in DMAc and then mixed with polyimide powder in various proportions. The slurry was stirred at 40°C for at least 30 h and subsequently centrifuged at 5000 rpm for 45 min to remove any undissolved residual solid. The polymer solution was cast on a clean Pyrex glass plate to a thickness of 250 μm. The casting temperature and atmosphere were ambient (23°C, relative humidity ≤ 60%). The cast film, together with the glass plate, was placed in an oven at a specific temperature for a specific period of time to partially evaporate the solvent on the film surface. Then, the film, still on a glass plate, was immersed into ice-cold water for 20 min, during which time gelation took place and the film stood apart from the glass plate. The wet film was then placed into an ethanol bath so that the water in the film was replaced by ethanol. After 24 h of the solvent exchange, the film was air dried.

### Apparatus and Procedures

The permeability of air might be affected by organic vapors. But the literature source indicates that the effect of organic vapors on nitrogen permeability is very small.<sup>3,13</sup> In this study, for convenience, membrane selectivities will be characterized in terms of the ratio of the organic vapor permeation rate obtained in the presence of air over the permeation rate of air obtained by a separate test run in the absence of organic vapor. The permeation rates of hydrogen and nitrogen will also be tested. Their ratio gives, to some extent, an indication of the pore size on the membrane surface.

The experimental setup shown schematically in Figure 1 was used to measure the organic vapor permeation rates. The permeation cell consisted of two detachable stainless steel parts. A porous stainless steel plate was embedded in one of the parts to support the membrane. The two parts of the cell were set in proper alignment with two rubber O-rings that were larger in diameter than was the porous plate.



**Figure 1** The test cell and the schematic flow diagram of equipment used for organic vapor permeation experiments.

A pressure-tight seal was obtained by clamping the two parts tightly between two end plates. The effective area of the cell for permeation was  $22.06 \text{ cm}^2$ .

Air went into a sintered stone ball immersed in an organic liquid to produce fine air bubbles that rose through the liquid. A mixture of air and organic vapor was obtained, and the vapor concentration was analyzed using a gas chromatograph. In the experimental range of air-flow rates ( $50\text{--}80 \text{ mL/min}$ ), a vapor saturation level of 98–99% was achieved. The feed air saturated with organic vapor was introduced to the feed side of the membrane at a sufficiently high flow rate so that the permeation stage cut was very small and the concentration variation along the membrane surface was negligible. This means that the primary test data are local properties of the membrane.

A vacuum pump was applied on the permeate side to provide the necessary driving force for the permeation and to withdraw the permeated sample to a cold trap immersed in liquid nitrogen. The permeation rate of an organic vapor was determined by weighing the sample condensed in a cold trap over a known period of time. The study was concerned with the steady-state permeation. The approach to the steady state was monitored by measuring the permeation rate. Approximately 2–3 h were required to reach the steady state.

Gas permeation rate measurement was performed in a traditional permeation cell, whose structure was the same as that of the static reverse osmosis cell.<sup>14</sup> The permeate side of the membrane was open to atmosphere, and the permeation rate was determined using a bubble flowmeter. The effective per-

meation area was 9.6 cm<sup>2</sup>. All the tests were carried out at laboratory temperature (23°C).

### Determination of Permeation Rates

The permeation rates of gases through the membrane,  $J_{\text{gas}}$  (mol/m<sup>2</sup> s Pa), is obtained by

$$J_{\text{gas}} = \frac{V}{A\Delta P} \frac{273.15}{273.15 + 23} \frac{1}{22400 \times 60} \quad (1)$$

where  $V$  is the volumetric flow rate of the permeated gas measured by a bubble flowmeter (mL/min);  $A$ , the effective film area (m<sup>2</sup>); and  $\Delta P$ , the pressure difference across membrane (Pa).

Assuming that the permeation rate of air is unaffected by the presence of an organic vapor, the organic vapor permeation rate,  $J_{\text{vap}}$  (mol/m<sup>2</sup> s Pa), can be obtained by solving the following set of equations using the air permeation rate value  $J_{\text{air}}$  given by eq. (1):

$$Q_{\text{vap}} = J_{\text{vap}}(P_1 Y_1 - P_3 Y_3) \quad (2)$$

$$Q_{\text{air}} = J_{\text{air}}[P_1(1 - Y_1) - P_3(1 - Y_3)] \quad (3)$$

$$Y_3 = Q_{\text{vap}} / (Q_{\text{vap}} + Q_{\text{air}}) \quad (4)$$

where  $Q$ 's are permeation fluxes (mol/m<sup>2</sup> s);  $P$ 's, pressures (Pa); and  $Y$ 's, the mole fractions of organic vapor. Subscripts 1 and 3 represent the feed side and the permeate side, respectively. Note that

**Table I** Permselectivities of Polyimide and Silicone Membranes

Organic Vapor	Polyimide		Silicone
	$J_{\text{vap}}^a$	$J_{\text{vap}}/J_{\text{air}}$	$J_{\text{vap}}/J_{\text{air}}^b$
Methanol	$5.02 \times 10^{-8}$	221.2	38.0
Ethanol	$6.73 \times 10^{-8}$	296.9	—
Acetone	$1.07 \times 10^{-8}$	47.4	16.1
Benzene	$1.15 \times 10^{-8}$	50.5	29.5
Toluene	$4.09 \times 10^{-8}$	179.9	24.9
<i>p</i> -Xylene	$1.04 \times 10^{-7}$	460.2	—
<i>m</i> -Xylene	$1.16 \times 10^{-7}$	513.3	—
1,2-Dichloroethane	$1.18 \times 10^{-8}$	52.1	—
1,2-Dichloropropane	$1.30 \times 10^{-8}$	57.2	—
Chloroform	$4.79 \times 10^{-9}$	21.1	—
Carbon tetrachloride	$5.56 \times 10^{-9}$	24.4	191.3
Hexane	$7.33 \times 10^{-9}$	32.4	25.7

<sup>a</sup> In mol/m<sup>2</sup> s Pa.

<sup>b</sup> Calculated from Ref. 15.

**Table II** Summary of Membrane Preparation Parameters

Casting solution composition
Polymer: aromatic polyimide, 25 wt %
Additive: LiCl, 3.6 wt %
Solvent: DMAc, 71.4 wt %
Temperature of casting solution: 40°C
Thickness of cast film: 250 μm
Film casting atmosphere: air
Temperature of casting atmosphere: 23°C
Solvent evaporation temperature: 85°C
Solvent evaporation period: 20 min
Gelation medium: ice-cold water, 2°C
Gelation period: 20 min
Solvent for replacing water in the film: ethanol
Solvent exchange period: 24 h
Drying of membrane: air dried

$J_{\text{air}}$  was measured in the absence of organic vapor, whereas  $Q_{\text{air}}$  represents the air flux occurring in the presence of organic vapor.

In the above equations,  $Q_{\text{vap}}$ ,  $P_1$ ,  $P_3$ ,  $Y_1$ , and  $J_{\text{air}}$  are known quantities from air and organic vapor permeation experiments, and therefore three unknowns,  $J_{\text{vap}}$ ,  $Q_{\text{air}}$ , and  $Y_3$ , are obtainable from the above three equations.

## RESULTS AND DISCUSSION

### General Observations

When the polymer content was less than 20 wt % and the salt content ranged from zero to 6.2 wt %, the resultant membranes were netlike. On the other hand, when polymer content was beyond 30 wt % and salt content was 4.4 wt %, the membrane broke into pieces during either the evaporation step or the gelation step. The polymer content of 25 wt % could be used to obtain useful membranes. The latter polymer content was used throughout this study to test the effects of the membrane preparation parameters.

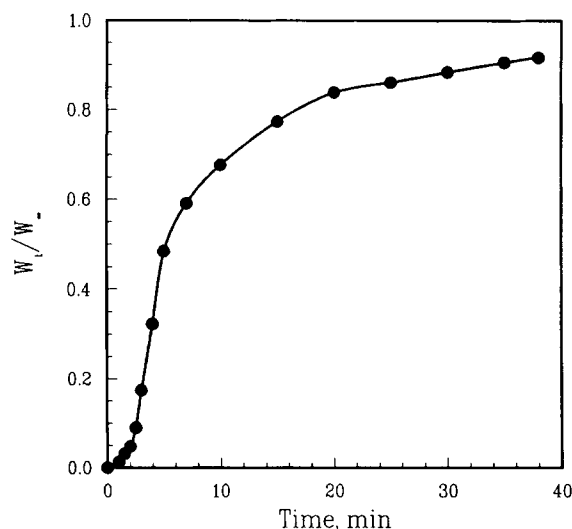
Table I shows the typical permeation data for one of the polyimide membranes studied in this work. The conditions of the preparation of this particular membrane are given in Table II. For comparison, the reported selectivity data of silicone rubber membranes of General Electric Co.<sup>15</sup> are also included. Table I shows that the organic vapor/air permeation rate ratios are greater than those for the pinhole-free dimethyl silicone polymer membrane reported by General Electric except for carbon tetrachloride. Furthermore, the permeation rates of

polyimide membranes are able to be controlled by changing the membrane-making conditions. This will be shown in the following parts of the paper.

When the conditions of the membrane preparation are changed, the permeation rate for each organic vapor changes for different membranes, but a common pattern in the permeation rate is observed, i.e., (*m*-xylene, *p*-xylene) > (toluene, methanol, ethanol) > (benzene, 1,2-dichloroethane, 1,2-dichloropropane) > (acetone, hexane) > (chloroform, carbon tetrachloride)  $\gg$  air. Acetone and *m*-xylene are to be chosen in this paper to show the effects of membrane preparation variables. The conditions of the membrane preparation will be the same as those given in Table II unless otherwise specified. It has to be noted that acetone and *m*-xylene represent a less permeable and a more permeable organic vapor, respectively, among the organic vapors under study. Moreover, they are two of the eight organic solvents producing most of the organic emissions into the atmosphere.<sup>3</sup>

#### Effect of Salt Content on Membrane Performance

Figure 2 illustrates the amount of LiCl transferred from the cast film into the gelation medium as a function of gelation period, which was monitored using a conductivity meter (CDM80 model). As gelation went on, the rate of salt leaving the film decreased. It is reasonable because the salt within the cast film has to diffuse through the gelled polymer



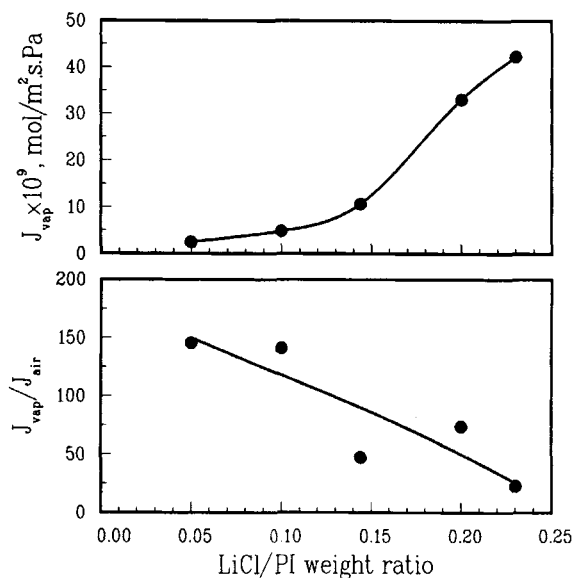
**Figure 2** A rate curve for LiCl leaching out of the cast film into the gelation bath. Film casting and gelation conditions are given in Table II.  $W_t$ ,  $W_\infty$  = amount of LiCl leached into gelation bath at time  $t$  and at time infinity, respectively.

layer formed at the interface between the polymer and the gelation medium. The gelled polymer layer acts as a barrier to the salt leaching, and the rate of salt leaching gives some information concerning the thickness and porosity of the gelled polymer layer that is mainly responsible for the selective permeability of a membrane. It is shown that after 20 min of gelation, most of the salt has come out of the cast film for the particular case.

When considering the effect of LiCl on the porous property of the membrane, it is better to use the relative quantities of LiCl and polymer, instead of the absolute quantity of LiCl. Therefore, in the following study, the ratio of LiCl to polymer was changed while fixing the wt % of the polymer in the casting solution at 25%. Figures 3 and 4 show that the weight ratio of salt and polymer in the casting solution has a significant effect on the permeation rate of the membrane ultimately obtained. As the salt concentration increases, the size and the effective number of pores on the membrane surface increase. Consequently, permeabilities of the membrane to organic vapors increase. The organic vapor permeation rate changes smoothly with the salt/polymer weight ratio, but the data for the permeation rate of air scatters. Therefore, the selectivity data expressed in terms of organic vapor/air permeation rate ratio scatters. The above results may be explained from the difference between gases and organic vapors. Organic vapors are believed to condense in the membrane pore or they form multilayers on the surface of the membrane pore by adsorption, as discussed later. On the other hand, gas molecules are believed to be transported through the pore by the slip or viscous flow because of their lesser condensibility and weaker interaction with the polymeric material. Because of the difference in the transport mechanism, the effect of the pore size on the membrane permeability is different for organic vapors and gases.

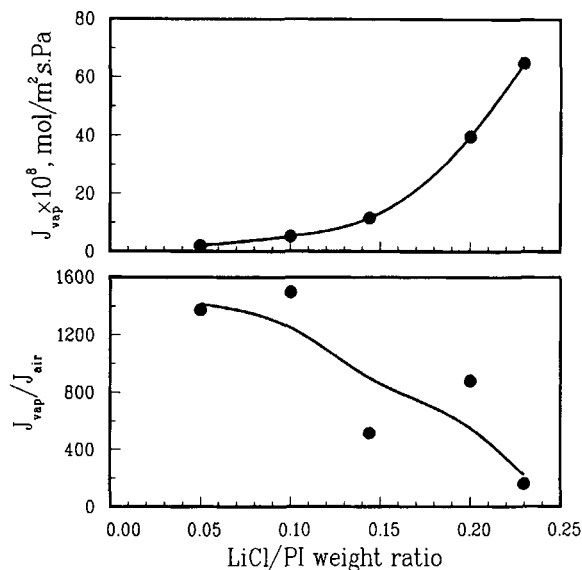
Though the selectivity data in Figures 3 and 4 scatter significantly, there is a tendency that a higher salt concentration in the casting solution leads to a less selective membrane. When the salt/polymer weight ratio in the casting solution increases from 0.05 to 0.23, the permeation rates of acetone and *m*-xylene increase by about 10-fold, and the permeation rate ratio of organic vapor to air decreases by nearly one order of magnitude. This means that as the salt content increases the air permeation rate increases 10 times faster than does the organic vapor.

The salt content is a very effective parameter to control the membrane performance. But, when the salt/polymer weight ratio is above 0.25, it is difficult



**Figure 3** Effect of LiCl/polymer ratio in the casting solution on the permeability and selectivity of the membrane with respect to acetone. Polymer concentration in the casting solution was maintained at 25 wt % while changing the LiCl/polymer ratio. Other conditions of the membrane preparation are the same as those given in Table II.

to make the casting solution homogeneous. On the other hand, if no salt is added, it is necessary to use a high concentration of polymer in preparing the casting solution. Otherwise, the casting solution is

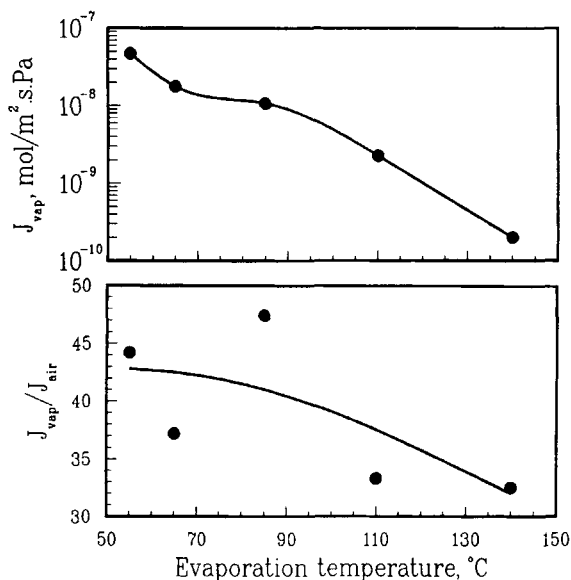


**Figure 4** Effect of LiCl/polymer ratio in the casting solution on the permeability and selectivity of the membrane with respect to *m*-xylene. Conditions of the membrane preparation are the same as those in Figure 3.

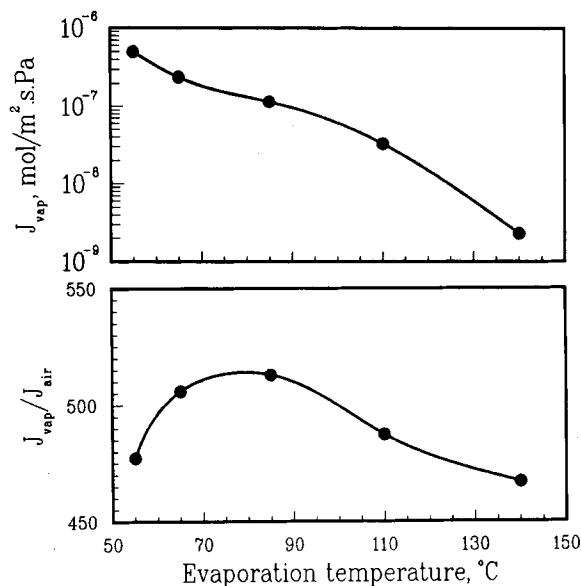
too fluid to form a membrane by the phase inversion process. A high polymer concentration will significantly reduce the permeation rate. Therefore, the permeation rate of organic vapors, the organic vapor/air permeation rates ratio, and the ability to form a membrane must be considered simultaneously before deciding the amount of salt added to the casting solution.

### Effect of Solvent Evaporation Temperature on Membrane Performance

The data presented in Figures 5 and 6 indicate that an increase in solvent evaporation temperature significantly reduces the permeation rate of organic vapors. The data for the permeation rate ratio of organic vapor to air are scattered but less affected by solvent evaporation temperature. When the evaporation temperature changes from 55°C to 140°C, the permeation rate of organic vapor undergoes a decrease of nearly two orders of magnitude. However, the vapor/air permeation rate ratio decreases by only 40% for acetone and 10% for *m*-xylene. The solvent evaporation from the casting solution is supposed to have the following two effects on the membrane structure and performance: (1) During the solvent evaporation, the local polymer concentration on the surface of the cast polymer increases due to the loss of solvent, which tends to



**Figure 5** Effect of solvent evaporation temperature on the permeability and selectivity of the membrane with respect to acetone. Conditions of the membrane preparation are the same as those given in Table II except for evaporation temperature.



**Figure 6** Effect of solvent evaporation temperature on the permeability and selectivity of the membrane with respect to *m*-xylene. Conditions of the membrane preparation are the same as those in Figure 5.

cause a low porosity of the membrane and, hence, a high permeation resistance. (2) During the solvent evaporation, the salt concentration on the cast film surface increases, which tends to increase the membrane porosity. These two effects are apparently opposing against each other. Of course, the higher the evaporation temperature, the higher the evaporation rate. At a high evaporation temperature, the solvent evaporation rate rapidly rises to such a level that solvent loss on the surface of the cast film cannot be compensated by solvent diffusion from the interior to the film surface. The polymer soon separates from the casting solution and forms a skin layer on the membrane surface. Therefore, for a given period of evaporation, the thickness of the dense skin layer increases as the evaporation temperature increases, resulting in an increase in the permeation resistance. This is the reason for a significant decrease in the vapor permeation rate with an increase in the evaporation temperature. As for the membrane pore size, the above two effects seem to be balanced and there is no significant change in the pore size, leading to little change in the vapor/air permeation rate ratio. The results illustrated in Figures 5 and 6 also suggest that a lower evaporation temperature is preferable to prepare a membrane of high permeability and high selectivity. But, because of the relatively high boiling point (165°C) of solvent DMAc, a low evaporation temperature leads to

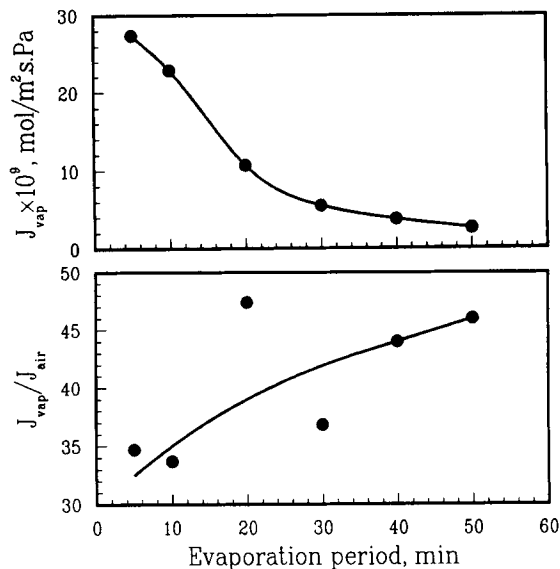
a high porosity on the membrane, resulting in insufficient mechanical strength of the membrane.

#### Effect of Solvent Evaporation Period on Membrane Performance

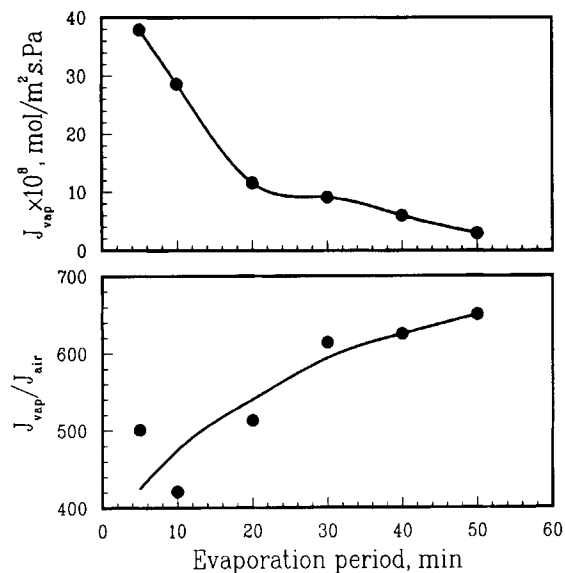
As indicated in Figures 7 and 8, the membrane performance is influenced by the solvent evaporation period. Increasing evaporation time prior to immersion of the cast film in the gelation bath causes a decrease in permeation rate. This result is consistent with the observation of Kesting,<sup>16</sup> who tested the water permeation rate through cellulose acetate membranes. It is understandable since the skin layer becomes thicker and the possibility of existence of big pores on the membrane surface becomes smaller as the evaporation period increases. The effect of the evaporation period on the vapor/air permeation rate ratio is much less. It is interesting to note that the ratio increases with an increase in the evaporation period, whereas the ratio has a decreasing tendency with an increase of the evaporation temperature.

#### Correlation between Permeation Rate and Cohesive Energy of Organic Vapors

Figure 9 shows that there is a linear correlation between  $\log J_{\text{vap}}$  and the cohesive energy of the organic

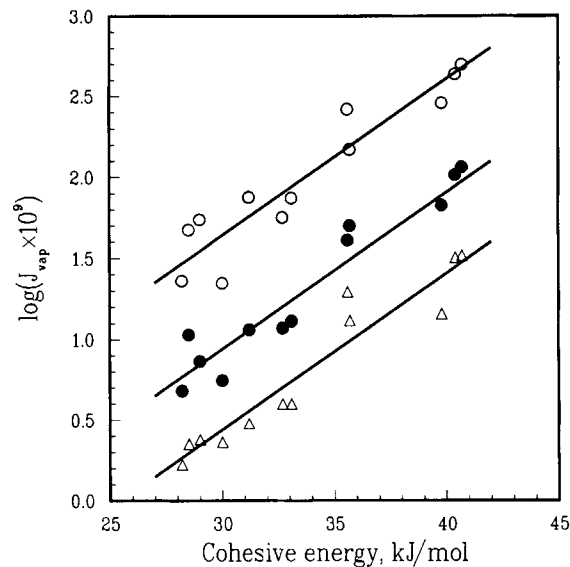


**Figure 7** Effect of solvent evaporation period on the permeability and selectivity of the membrane with respect to acetone. Conditions of the membrane preparation are the same as those given in Table II except for evaporation period.



**Figure 8** Effect of solvent evaporation period on the permeability and selectivity of the membrane with respect to *m*-xylene. Conditions of the membrane preparation are the same as those in Figure 7.

vapor. The slope of the straight line is almost constant for three membranes of different permeabilities. The cohesive energy data used are listed in Table III. Note that cohesive energy is equal to vaporization energy calculated from the data on solubility parameter and molar volume. Note also that they are characteristic to the organic liquid. For all the membranes tested, on the other hand, the permeation rate ratio of hydrogen to nitrogen is in the range of 1.5 to 1.8. These values are less than 3.74, which value is obtained by calculation using the Knudsen flow mechanism. This indicates that some



**Figure 9**  $\log J_{\text{vap}}$  vs. cohesive energy. Conditions of the membrane preparation: (●) same as those given in Table II; (○, △) same as those given in Table II except evaporation temperatures are 55°C and 110°C, respectively.

large pores exist on the membrane surface that permit slip flow and/or viscous flow. Furthermore, the molar permeation rate of the organic vapor is much greater than that of the gas.

The above two experimental observations—one that logarithm of the permeability is correlated linearly to the vaporization energy of the organic compound, and the other that the permeation rate ratio of hydrogen to nitrogen is less than 3.74—indicate that the organic vapor is condensed or forms a multilayer in the membrane pore. In both cases, the formation of the condensed phase of the organic liq-

**Table III** Cohesive Energy Data for Organic Compounds

Organic Compound	Molar Volume $\times 10^6$ (m <sup>3</sup> /mol)	Solubility Parameter <sup>a</sup> (MPa) <sup>0.5</sup>	Cohesive Energy (kJ/mol)
Methanol	40.5	29.7	35.7
Ethanol	58.4	26.1	39.8
Acetone	73.5	19.7	28.5
Benzene	89.1	18.7	31.2
Toluene	106.3	18.3	35.6
<i>p</i> -Xylene	123.3	18.1	40.4
<i>m</i> -Xylene	122.9	18.2	40.7
1,2-Dichloroethane	80.1	20.2	32.7
1,2-Dichloropropane	97.7	18.4	33.1
Chloroform	80.5	18.7	28.2
Carbon tetrachloride	96.9	17.6	30.0
Hexane	130.5	14.9	29.0

<sup>a</sup> From Ref. 17.



uid in the pore is governed by the interaction between organic vapor molecules themselves rather than that between the organic molecule and the membrane. Hence, it is natural that a strong correlation exists between the vaporization energy and the permeability of the organic vapor. Judging from the constancy of the slope, it can be concluded that the mechanism of vapor permeation did not depend on the pore size at least for those three membranes that were tested in this work.

## CONCLUSIONS

The following conclusions can be drawn from the observations made in this work:

1. The dry asymmetric aromatic polyimide membrane is preferentially permeable to organic vapors when feed air-organic vapor mixtures are treated.
2. The membrane performance is affected strongly by the conditions of membrane preparation.
3. For a given membrane, the permeability of organic vapor increases with an increase in its cohesive energy.

The interaction between the permeant and the membrane, and the membrane morphology, govern the membrane transport. A continuous effort to express these two effects explicitly is necessary to have better understanding of the membrane transport.

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