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NOTE
Pressure-Driven Pervaporation

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INTRODUCTION

Pervaporation is a promising method for the fractionation, purification, and recovery of volatile liquids (*1*). Current interest in ethanol as a fuel and the possibility to obtain this fuel by biomass fermentation, which yields dilute (8–12% w/w) aqueous ethanol solutions, led to considerable research effort in ethanol purification and dehydration by pervaporation (*2–4*).

Some membranes are more permeable to ethanol than to water, but others are permeated by water at higher rates (*4–10*). By using membranes of the last type, results were good enough to allow the construction and operation of a pilot-scale ethanol dehydration plant (*11*).

Standard pervaporation procedures do not offer any major advantage over distillation as far as energy consumption is concerned. This is because pervaporation involves a net phase change from liquid to vapor phase. Some pervaporation arrangements use vacuum chambers and cooled traps to condense vapors. This requires additional and important energy inputs (*12*).

Pervaporation and condensation of liquids at constant temperature would be a phase change at nearly reversible conditions, under which maximum thermodynamic efficiency can be attained. In this paper we give theoretical arguments and experimental results showing that iosthermal, pressure-driven pervaporation of liquids is feasible.

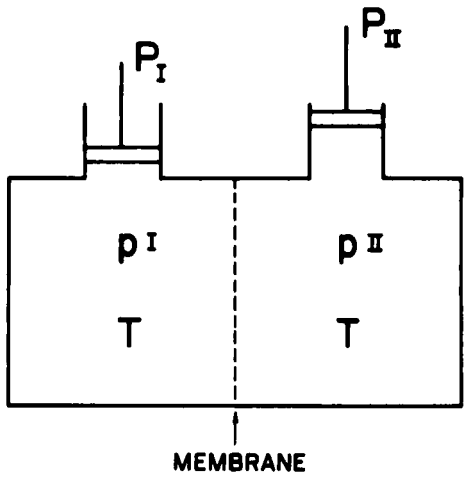

FIG. 1. Schematic description of pressure-driven pervaporation.

THEORETICAL

The vapor pressure of a liquid increases with the total pressure to which it is subjected at constant temperature. Considering the schematic arrangement of Fig. 1, the vapor pressures of the liquid on both membrane sides are related according to

$$\ln = \frac{p_I}{p_{II}} = \frac{\overline{V}(P_I - P_{II})}{RT} \tag{1}$$

where p_I = vapor pressure at Side I
p_{II} = vapor pressure at Side II
P_I = total pressure at Side I
P_{II} = total pressure at Side II
$\overline{V}$ = molar volume of the liquid

If $P_I > P_{II}$, $p_I > p_{II}$. In this case, the liquid will be isothermally evaporated at I and condensed at II. In a cell made of metal or any heat-conducting material, heat released at II (by condensation) is conducted back to I, where it is used to evaporate more liquid. Net energy consumption is only necessary to keep $P_I > P_{II}$.

This kind of experiment seems analogous to reverse osmosis. This point will be further examined in the Discussion section.

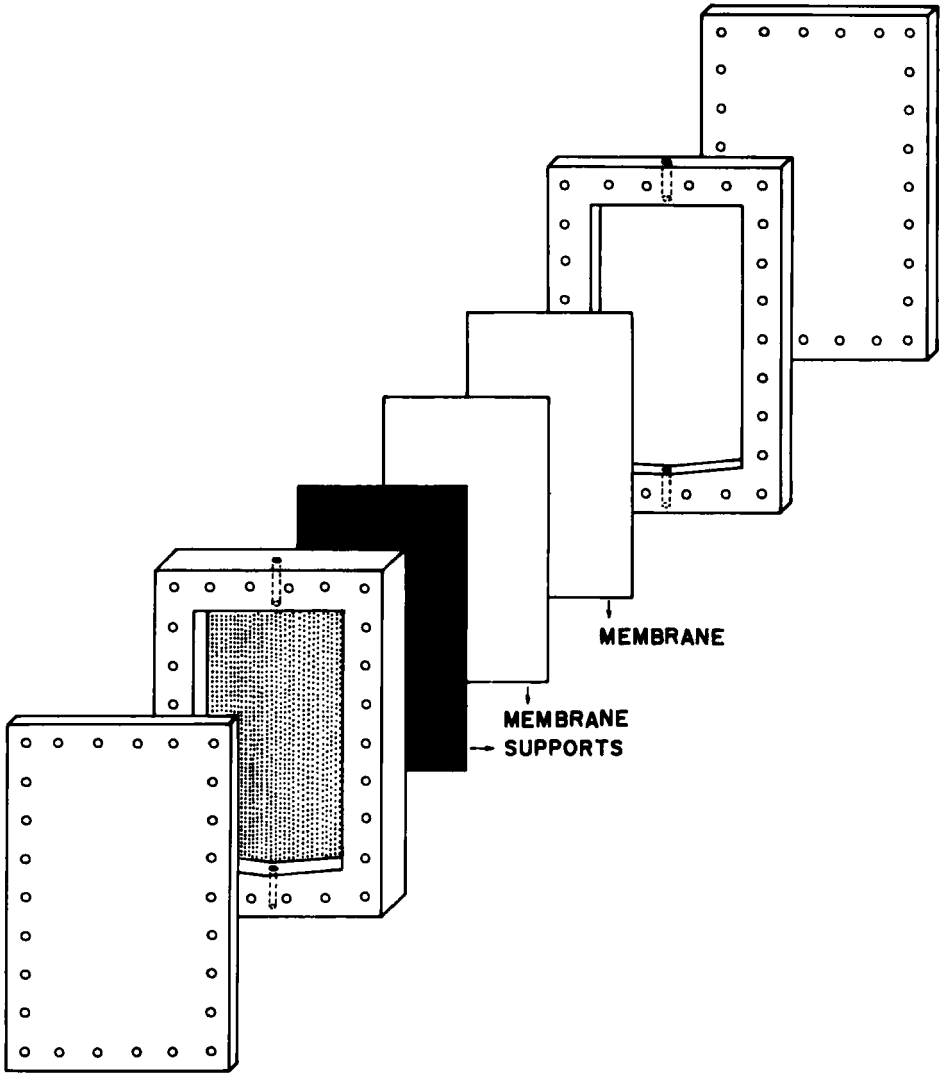

FIG. 2. Schematic view of the pressurized pervaporation cell.

EXPERIMENTAL

The pervaporation cell was built of 1020 SAE steel by Stäubli (Diadema, SP). It was surface-treated to prevent rusting. A schematic drawing is given as Fig. 2, and a photograph of the experimental set up is presented as Fig. 3.

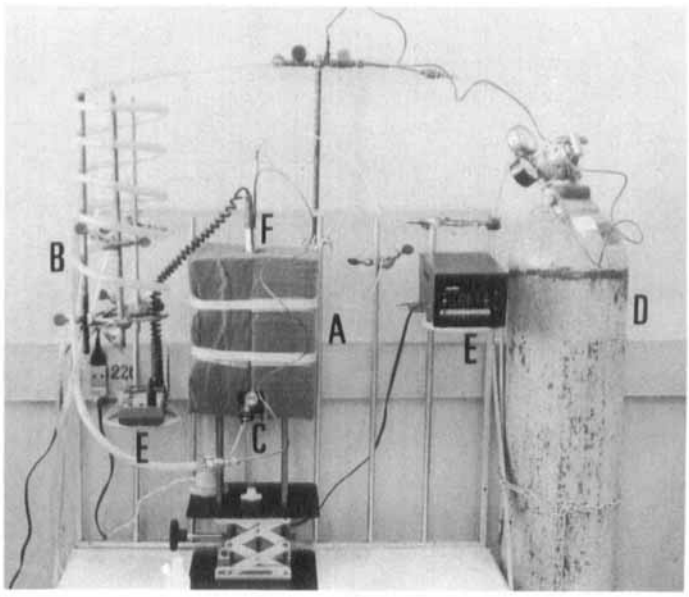

FIG. 3. A photograph of the assembled pervaporation apparatus. A: Pervaporation cell with insulating jacket. B: Feed solution reservoir coil. C: Permeate outlet. D: Gas reservoir. E: Thermocouple meters. F: Thermocouples.

Reagent grade chemicals were used throughout.

Membranes were cast by spreading Dow Corning RTV silicone adhesive over polyethylene films fixed over pieces of window glass with a double-face adhesive tape.

Infrared spectra showed that these films are made of poly(dimethyl siloxane).

RESULTS

Many different liquids were used in the pervaporation runs. The liquids used, other experimental conditions, and the resulting flow rates are given in Table 1.

We can observe a marked effect of both temperature and pressure in the flow rates. Beyond that, flow rates are higher for the less-polar liquids, following the order cyclohexane > butanol > ethanol > water. This is the same order as silicone swelling in these liquids.

To make sure that the flow rates are not due to leakage through minute holes in the membrane or in cell gaskets, the assembled cells were tested

TABLE 1
Pressure and Temperature Effects on Pervaporation Rates

Liquid	T (°C)	ΔP (atm)	Permeation rate (mL/min)	Membrane thickness (μm)
Cyclohexane	28	3	0.15	102
		6	0.26	102
		12	0.41	102
Ethanol	25	6	0.0035	115
		12	0.011	115
	75	6	0.1	115
Water	27	9	$<10^{-4}$	90
Butanol	25	9	0.013	115
	70	2.5	0.025	115
		5	0.05	115
		7.5	0.06	115
		10	0.07	115
		12.5	0.085	115
		15	0.12	115

with solutions containing the dyes methylene blue and Blue Dextran. The absence of dye in the permeate was taken as evidence of absence of leakage.

Other hydrocarbons and chlorinated liquids showed very high flow rates. However, silicone swelling was also very strong, and the existence of swollen membranes was too short to allow repeated measurements to be made.

Many binary solutions were also examined. The corresponding results are in Table 2. Flow rates are higher in solutions richer in less polar component (e.g., see the CCl_4/ethanol and ethanol/water solutions).

In the case of ethanol/water, the separation factors are very low; much lower than those found for the same membranes in normal pervaporation experiments (*7*).

DISCUSSION

The basic idea put forward in this paper could, in principle, be used for isothermal distillation experiments. This requires large pressure gradients which are difficult to handle in normal distillation but which can be easily obtained by using a membrane.

One obvious question is: Is this different from reverse osmosis? Perhaps not much, in operational terms. To answer this question, we are now making a detailed examination of pressure and temperature effects on flow rates to determine permeabilities, respective activation energies, and actual energy inputs.

TABLE 2
Permeation Rates of Solutions in Pressure-Driven Pervaporation

Feed solution	T (°C)	P (atm)	Flow rate (mL/min)
n-Butanol/ethanol:			
50% (v/v)	22	9	0.03
Toluene/ethanol:			
25% (v/v)	30	9	0.049
33% (v/v)	29	9	0.053
40% (v/v)	23	9	0.064
Toluene/butanol:			
10% (v/v)	26	9	0.092
Methanol/chloroform:			
20% (m/m)	26	9	0.0042
CCl_4/ethanol:			
20% (m/m)	23	12	0.011
40% (m/m)	24	12	0.12
Ethanol/water:			
20% (m/m)	73	10	0.0036
40% (m/m)	70	10	0.0071
60% (m/m)	70	10	0.013
100%	70	10	0.029

Irrespective of the nomenclature used, it seems that the association of pressure and above-room temperatures is a convenient way to purify liquids by using a membrane.

Regarding the low separation factors observed using water–ethanol solutions, we assign this to the superimposition of an osmotic pressure-driven liquid flow across the membrane. Indeed, even if pure ethanol flows across the membrane and condenses on the permeate side, its retention over the membrane should drive water across it due to the resulting high osmotic pressures (which in the present experiments would be in the 10^2 bar range).

For this reason we think that this technique can only be successful for the separation of volatile liquids from substances to which the membrane is really impermeable (such as the dyes used in this work). Nevertheless, the intrinsic advantages due to high thermodynamic efficiency should make this a useful alternative in volatile liquid purification and recovery.

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