Investigations on Nylon 4 Membranes: Synthesis and Transport Properties. II. Transport Properties of Nylon 4 Polymer Membranes*

R. Y. M. HUANG, U. Y. KIM,** J. M. DICKSON,† D. R. LLOYD,† and C. Y. PENG, *Department of Chemical Engineering, University of Waterloo, Waterloo, Ontario, Canada N2L 3G1*

Synposis

The reverse osmosis, ultrafiltration, and dialysis properties of nylon 4 membranes to separations of sodium chloride, urea, a series of ethylene glycols and other compounds in the aqueous phase were investigated. The nylon 4 membranes were prepared from a formic acid solution with and without organic or inorganic additives. The effects of polymer concentration, amount of additives, casting time, and temperature on the membrane performance in terms of salt separation and product rate were investigated. The tensile properties of the nylon 4 membranes in both the dry and wet states were determined. It was found that the highest salt separation of a 0.1% sodium chloride solution did not exceed 53.3%. However, these membranes showed some interesting dialysis properties which were comparable to those of commercial cellophane and cellulose acetate membranes.

INTRODUCTION

Considerable interest has been shown in recent years in the synthesis and preparation of novel membranes for separation processes. The main efforts in the development of high-performance membranes has proceeded along three main lines. The first involves the crosslinking of water-soluble polymers by a variety of crosslinking agents.[1–4,5–7] A second approach has been the synthesis of water-swellable polymers,[8–10,11] while the third has been directed toward the development of polyamide polymers[12–16] for the preparation of the membranes.

Nylon 4 (polypyrrolidone) has been of particular interest among the aliphatic polyamides because of its hydrophilicity and has been regarded as a promising membrane material for separation purposes. The only previous work on nylon 4 membranes has been carried out by Lonsdale et al.[17] and by Orofino,[18] but these workers found that nylon 4 behaved erratically in terms of their transport fluxes. It was considered worthwhile, therefore, to reinvestigate the preparation of nylon 4 membranes through improvements in the synthesis of the polymer and in the casting methods for preparing the membranes. In part I of this series,[16] the synthesis of nylon 4 by the CO_2-activated anionic polymerization of pyrrolidone in the presence of potassium pyrrolidonate was described in detail. The purpose of this article is to report some improvements in the preparation of the mem-

CCC 0021-8995/81/061907-12$01.20

TABLE I
The Effect of Polymer Concentration on Nylon 4 Membrane Product Rate and Solute
Separation[a]

Membrane no.	Solvent system	C_p	Dry Temp., °C	Dry Time, min	PWP, (g/cm² sec) × 10⁵	PP, (g/cm² sec) × 10⁵	SEP, %
268	FA (60%)	12.0	80	30	3.83	1.31	28.30
270	FA (60%)	14.0	80	30	2.53	1.07	28.30
272	FA (60%)	16.0	80	30	2.95	1.24	24.30
274	FA (60%)	16.6	80	30	2.10	0.65	22.90
165	FA (60%)	10.0	80	30	1.42	1.13	37.30
166	FA (60%)	12.5	80	30	0.74	0.51	3.130
167	FA (60%)	15.0	80	30	0.53	0.48	29.80

[a] FA: Formic acid; C_p, polymer concentration; PWP, pure water permeability; PR, product rate; SEP, solute separation; polymer viscosity $\eta r = 2.69$, casting solution (salt-free), wet thickness 10 mils, feed solution 0.1% NaCl, and operating pressure 600 psi.

branes and to describe some of its transport properties in terms of reverse osmosis, ultrafiltration, and dialysis separations of various solutes in the aqueous phase.

EXPERIMENTAL

Polymer synthesis. The nylon 4 used in the present experiments was synthesized by the CO_2-initiated polymerization of 2-pyrrolidone using potassium 2-pyrrolidonate as the catalyst. A detailed description of the preparation has been reported in part I of this series.

Membrane preparation. The membrane was prepared from a formic acid casting solution of nylon 4 with or without added swelling agents. The membrane was formed by casting the solution on a glass plate and casting to predetermined thickness by means of a Gardner knife with an adjustable blade. The glass plate containing the casting solution was predried for 5 min at ambient temperatures and allowed to evaporate for a specified time at 80–90°C in the oven and cooled for 10 min. Then the glass plate with the film was immersed in a methanol nonsolvent bath, where it was maintained for 10 min at room temperature, enabling the residual solvent to be evaporated. The membrane was then peeled off and stored in deionized water for at least 12 hr before subsequent use.

TABLE II
The Effect of the Ratio of Formic Acid/Water in the Solvent System[a]

Membrane no.	Solvent system	Dry Temp., °C	Dry Time, min	PWP, (g/cm² sec) × 10⁵	PR, (g/cm² sec) × 10⁵	SEP, %
162	FA (50%)	80	20	1.05	0.80	31.25
163	FA (50%)	80	20	1.05	0.88	34.28
212	FA (90%)	80	10	1.39	1.37	30.67
213	FA (70%)	80	10	1.12	0.88	22.67
214	FA (60%)	80	10	1.26	0.93	22.67

[a] C_p 10%, thickness 10 mils, salt-free solution, 0.1% NaCl, and 600 psi.

TABLE III
The Effect of Polymer Viscosity[a]

Membrane no.	Solvent system	C_p	$[\eta]$	Dry Temp., °C	Time, min	PWP, (g/cm^2 sec) $\times 10^5$	PR, (g/cm^2 sec) $\times 10^5$	SEP, %
282	FA (60%)/nprOH (4/1, v/v)	10	4.4	90	60	1.89	2.21	19.44
283	FA (60%)/nprOH (4/1, v/v)	10	2.9	90	60	1.68	1.70	20.03
284	FA (60%)/nprOH (4/1, v/v)	10	2.4	90	60	1.47	1.16	20.83
285	FA (60%)/nprOH (4/1, v/v)	13	2.0	90	60	3.37	0.82	24.55
286	FA (60%)/nprOH (4/1, v/v)	13	1.7	90	60	2.53	1.24	23.87

[a] Thickness 10 mil, salt-free solution, 0.1% NaCl, and 600 psi.

Membrane testing. Tensile property measurements were carried out on an Instron table model TM instrument with a C-90-24 immersion testing attachment for the wet tests. The membranes were tested for their tensile strengths and elongations in both their dry and wet states.

Reverse osmosis properties. The reverse osmosis runs were carried out in a static stirred high-pressure cell (Amicon model 320). Details of the apparatus are provided elsewhere.[7] All the reverse osmosis runs were conducted at 600 psi at laboratory temperatures with sodium chloride aqueous solutions or those of other compounds. The pure water permeability (PWP), product rate (PR),

TABLE IV
The Effect of Nonsolvent Treatment

Membrane no.	Nonsolvent system	Immersion time, min	PWP, (g/cm^2 sec) $\times 10^5$	PR, (g/cm^2 sec) $\times 10^5$	SEP, %
290	A/W(1/3, v/v)	20	2.31	1.96	33.33
291	A/W(1/1)	20	2.74	1.39	36.23
292	A/W(3/1)	20	2.74	1.49	40.58
294	A/W(1/1)	20	1.68	1.33	34.26
295	A/W(3/1)	20	3.37	1.79	40.71
296	A/M(1/3)	20	1.77	1.58	36.99
297	A/M(1/1)	20	1.89	0.54	53.28
298	A/M(3/1)	20	2.53	2.21	44.55
309	acetone	20	6.84	2.53	30.00
310	methanol	20	2.21	1.81	52.86
302	A/M(1/1)	5	1.26	1.43	50.00
299	A/M(1/1)	10	2.74	1.20	53.33
300	A/M(1/1)	20	1.47	0.99	43.70
301	A/M(1/1)	40	1.89	1.14	44.44
304	A/M(1/1)	60	1.89	1.35	46.38
306	A/M(1/3)	10	1.89	1.87	43.84
307	A/M(1/3)	20	1.77	1.58	36.99
308	A/M(1/3)	40	2.21	1.94	37.67

[a] Polymer viscosity $\eta r = 2.25$, salt-free casting solution, C_p 10%, thickness 10 mil., drying time and temp. 20 min, and 90°C, 0.1% NaCl and 600 psi, A, acetone; M, methanol; W, water.

TABLE V
The Effect of Swelling Agents (Organic Nonsolvent)[a]

Membrane no.	Solvent system	Dry Temp., °C	Time, min	PWP, (g/cm² sec) × 10⁵	PR, (g/cm² sec) × 10⁵	SEP, %
191	FA (88%)/EtOH (2/1)	80	15	1.26	1.22	33.33
192	FA (88%)/EtOH (2/1)	80	15	1.05	1.14	33.33
193	FA (88%)/EtOH (2/1)	80	15	1.47	1.18	33.40
281	FA (60%)/nprOH (4/1)	90	20	2.53	1.52	33.57
	FA (60%)/nprOH (4/1)	90	20	2.15	1.52	33.10
	FA (60%)/nprOH (4/1)	90	20	2.53	1.58	34.51
197	FA (88%)/M/EG (4/1/1)	80	20	34.09	30.62	8.45
198	FA (88%)/Et/EG (4/1/1)	80	20	41.25	43.35	7.04
199	FA (88%)/prOH/EG (4/1/1)	80	20	22.73	23.57	7.70
232	FA (60%)/A/EG (8/1/1)	85	2	319.44	313.13	1.49
233	FA (60%)/A/EG (8/1/1)	85	2	512.63	487.37	0.75
234	FA (60%)/A/BA (8/1/1)	85	2	60.61	59.34	2.98

[a] C_p 10%, thickness 10 mil, 0.1% NaCl, and 600 psi.

Nonsolvent	bp, °C	Solubility Water	Alcohol
A: Acetone	56.5	∝	∝
M: Methanol	64.7	∝	∝
Et: Ethanol	78.4	∝	∝
nprOH: n-propanol	97.8	∝	∝
BA: Benzyl alcohol	204.7	4₁₇	∝
EG: Ethylene glycol	197.4	∝	∝
DMF: Dimethylformamide	153.0	∝	∝

and the solute separation (SEP) were determined. Solute separations were determined by the equation

$$\text{SEP} = \frac{C_{f1} - C_{f2}}{C_{f1}}$$

where C_{f1} and C_{f2} are the feed and product mole fractions, respectively. The sodium chloride concentrations were determined by using a Waters Associates differential refractometer model 403.

Dialysis properties. The dialysis system previously described in detail by Huang and Jarvis[5] was used for the experiments. The permeability coefficients were calculated from the slope of ln C_0/C_t-vs.-time plots. This system consists basically of a continuous-flow, flat type Plexiglass dialysis cell, where the solution to be dialyzed circulates in a loose loop past one side of the membrane. The

concentration is continuously monitored by a differential refractometer. On the other side of the membrane, deionized water circulated and transported the permeated solute out of the system. The effective area of the dialysis cell was 31.11 cm^2, and the initial NaCl solution concentration was 0.1% weight in water. The flowrates for both the solutions being dialyzed were 200 ml/min, and the operating temperature was 37°C.

Ultrafiltration measurements. The water flux was determined with the same static cells as used for the reverse osmosis runs, except that the pressure was at 50 psi at room temperature until steady state was achieved.

RESULTS AND DISCUSSION

Reverse Osmosis

In order to improve the nylon 4 membranes, variations were made to try and improve the solute separations and the fluxes. Two approaches were taken to improve the transport properties of the nylon 4 membranes: (1) improvement in the membrane casting method and formulation, and (2) the development of the nonsolvent treatment followed by a posttreatment. The casting solution used contained three components: (a) the polymer, nylon 4; (b) formic acid; and (c) water.

Among the solvents tested, formic acid was found to be the most favorable solvent. Tables I and II show the effect of the polymer concentration and the formic acid/water ratio in the solvent system on the nylon 4 membrane performance in terms of product rate (PR) and solute separation (SEP). As can be seen, both the PR and the SEP decreased slightly with increasing polymer concentration. Table III shows the effect of the polymer viscosity on the membrane performance, and polymers with adequate viscosities (molecular weights) must be used to ensure the formation of a tough and pliable membrane which can sustain the operating pressures. In this series of experiments, the nylon 4 membranes formed from samples of polymer with (η) = 2.0 and 1.7 at 10% polymer concentration proved to be too weak and soft to be a workable membrane, but by increasing the polymer concentration to 13%, the membranes began to show sufficient strength for the tests. Membranes which were prepared from higher-viscosity polymers showed slightly increased PR and SEP.

Table IV shows the effect of the nonsolvent treatment on membrane performance. It was expected that the nonsolvent would cause a modification of the membrane structure, similar to a shrinkage effect by drawing the polymer chains closer together. In the acetone/water system, decreasing the water ratio increased the SEP but decreased the PR slightly. Among the acetone/methanol systems, the acetone/methanol (50/50 ratio) showed the highest SEP with over 50%. Compared to acetone, methanol proved more effective in improving the solute separation but the resulting PR was lower. Long immersion times in the nonsolvent did not show better solute separation but decreasing the immersion time improved the results.

In order to improve the water fluxes of the membranes, it is appropriate to consider the formation of the porous structure in the membrane. One way of obtaining an assymetrical membrane is to prepare a solution of the polymer containing a mixture of good and poor solvents or inorganic salts. Swelling

TABLE VI
The Effect of Swelling Agents (Inorganic Salt)[a]

Membrane no.	Solvent system	Cs		Dry		PWP, (g/cm² sec) ×10⁵	PR, (g/cm² sec) ×10⁵	SEP, %
		Salt	%	Temp., °C	Time, min			
215-1	FA (70%)	LiCl	6	80	30	29.04	22.31	24.66
216	FA (60%)	LiCl	6	80	30	1.26	00.65	24.66
220	FA (60%)	LiCl	6	80	5	1.26	1.28	27.68
221	FA (60%)	LiCl	7	80	30	14.52	14.44	8.68
223	FA (60%)	LiCl	6	80	2	4.21	4.95	23.91
225	FA (60%)	LiCl	6	80	2	3.58	3.41	27.54
226	FA (60%)	CsCl	6	85	2	2.10	2.25	25.55
228	FA (60%)	CsCl	6	85	3	2.00	1.87	2.189
248	FA (60%)	CsCl	20	90	4	7.58	7.58	21.40
249	FA (60%)	CsCl	30	90	4	5.89	6.00	16.40
250	FA (60%)	CsCl	40	90	4	8.21	8.63	16.40
229	FA (60%)	RbCl	7	85	2	2.40	2.10	18.84
230	FA (60%)	RbCl	7	85	3	1.62	1.56	15.22
231	FA (60%)	RbCl	7	85	7	1.07	0.76	17.39
236	FA (60%)	RbCl	20	85	3	5.05	4.29	19.38
241	FA (60%)/nprOH (4/1)	Mg(ClO₄)₂	5	80	5	3.99	3.34	23.88
243	FA (60%)/nprOH (4/1)		10	80	5	2.53	1.01	31.34
245	FA (60%)/nprOH (4/1)		15	80	5	0.85	0.55	34.33
245-1	FA (60%)/nprOH (4/1)		15	90	5	1.26	1.39	30.15

[a] C_p 10%, thickness 10 mil.

TABLE VII
The Effect of Swelling Agent (NaCl)[a]

Membrane no.	Solute	PWP, (g/cm² sec) × 10⁵	PR, (g/cm² sec) × 10⁵	SEP, %
312	NaCl (12%	5.05	4.84	35.83
	MgSO$_4$	5.89	5.26	77.68
	Sucrose	5.68	5.47	68.87
	PEG 600	5.05	5.69	78.99
313	NaCl (14%)	4.21	4.73	32.04
	MgSO$_4$	5.47	4.84	80.80
	Sucrose	4.84	5.05	68.31
	PEG 600	4.42	5.26	83.10
314	NaCl (16%)	43.14	11.05	30.69
	MgSO$_4$	38.30	11.57	51.34
	Sucrose	41.88	10.31	60.23
	PEG 600	41.46	10.61	63.44

[a] Membranes 312, 313, and 314 contained swelling agent (NaCl) 12, 14, and 16%, respectively. C_p 10%; thickness 10 mil, drying temp. and time 90°C and 10 min; concentration of feed solution 0.1%; 600 psi.

agents are compounds which have the effect of producing a microgel in the membrane which increases the water content and the permeability to water. In general, this technique has been applied in the preparation of cellulose acetate

TABLE VIII
The Effect of Solute Selectivity[a]

Name	MW	Feed solution concentration, %	PSP, (g/cm² sec) × 10⁵	PR, (g/cm² sec) × 10⁵	SEP, %
Sodium chloride	58.45	0.1	2.03	1.78	33.50
Sodium chloride	58.45	0.5	1.96	1.89	16.44
Sodium chloride	58.45	1.0	1.96	1.90	16.18
Sodium chloride	58.45	1.5	1.95	1.89	16.14
Lithum chloride	42.40	0.1	1.79	1.94	18.80
Magnesium sulfate	120.38	0.1	1.79	1.85	92.15
Ethylene glycol	62.70	0.1	1.73	1.89	19.51
Di-EG	106.12	0.1	1.73	1.83	26.26
Tri-EG	150.18	0.1	1.73	1.83	43.92
Tetra-EG	194.23	0.1	1.79	1.96	68.95
Poly EG 200	190–210	0.1	1.96	1.96	67.71
PEG300	285–315	0.1	1.79	1.91	81.97
PEG400	380–420	0.1	2.06	1.96	86.50
Arabinose	150.08	0.15	1.85	2.00	59.01
Xylose	150.08	0.15	1.89	2.00	53.42
Glucose	180.10	0.15	1.96	2.04	69.97
Mannose	180.10	0.15	2.10	1.96	66.57
Galactose	180.10	0.15	2.10	1.96	70.33
Fractose	180.10	0.15	1.96	2.06	69.28
Cellobiose	342.18	0.15	2.03	2.03	89.41
Lactose	324.18	0.15	1.81	1.91	94.41
Maltose	342.18	0.15	1.87	1.96	91.57
Saccharose	342.18	0.15	1.89	2.08	90.53

[a] Membrane 281, FA (60%)/nprOH(4/1), C_p 10%; thickness 10 mil, drying temp. and time 90°C and 20 min; 600 psi.

TABLE IX*
Ultrafiltration Results[a]

| Membrane | Rate, g/hr | Water flux | |
		$(g/cm^2 sec) \times 10^5$	$g/hr\ m^2$
318	9.6	20.2	7272.7
319	4.7	9.9	3560.6
320	4.7	9.9	3560.6
Cellulose acetate KP 98	7.1		5658.4
Cellophane	3.0		2416.0

[a] The membranes were prepared from FA(60%)/nprOH(4/1) casting solution containing NaCl 16%(Mem 318), 14%(Mem 319, and 320). C_p 10%; thickness 10 mils; drying time and temperature 90°C and 20 min; reverse osmosis static cell, effective membrane area 13.2 cm², 50 psi, and room temperature (23 ± 1°C); cellulose acetate KP98 and cellophane are commercial membranes; Amicon model 52 ultrafiltration cell, an effective membrane area 12.5 cm²; 50 psi and room temperature (23 ± 1°C).

membranes with high water fluxes.[19,20] Several containing swelling agents both organic and inorganic were selected for the present experiments as shown in Tables V and VI. Water or alcohol is commonly used in preparing cellulose acetate acetone solutions for casting when preparing membranes. When combined with formic acid, however, they did not affect the transport characteristics of the membranes. On the other hand, the use of less volatile solvents such as ethylene glycol (EG), dimethylformamide (DMF), and benzyl alcohol (BA) dramatically increased the PR but decreased the SEP. These membranes appeared hazy or translucent depending on the amount of solvent used. Among the inorganic salts which were used, NiCl and NaCl were more effective in adjusting the water fluxes. The role of the inorganic salt is not clearly understood at the present, but the amounts used clearly affect the membrane performance. Other salts such as CsCl, RbCl, and Mg(ClO$_4$)$_2$ were used, but they did not produce any changes in the membrane performance.

Cellulose acetate membranes prepared from casting solution containing Mg(ClO$_4$)$_2$ gave higher water fluxes,[20] but in the case of the nylon 4 formic acid casting solution, the product rate decreased with increase in the amount of added magnesium perchlorate. It is possible that a large amount of the magnesium perchlorate remains trapped in the blind pores of the membranes. The membranes which were cast from solutions containing NaCl appeared translucent on visual inspection and showed a porous structure.

Table VII shows the effect of the amount of NaCl on the PR and SEP. They did not change much between 12 and 14% NaCl content, but the PR changed dramatically above a NaCl content of 16%.

TABLE X
Permeability Coefficients of Nylon 4 Membrane (Membrane 318)

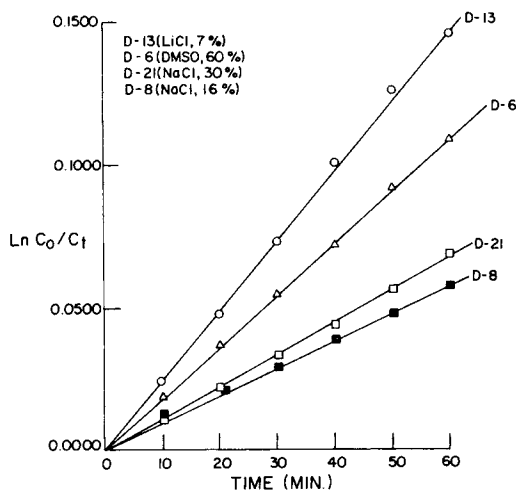| Permeant	Permeability coefficient $\times 10^4$, cm/min
Urea	71.63
Sodium	54.14
Ethylene glycol (EG)	44.05
Di-EG	42.44
Tri-EG	20.64
Tetra-EG	18.00

Fig. 1. Dialysis of sodium chloride through nylon 4 membranes containing LiCl, DMSO, and NaCl swelling agents.

TABLE XI
Tensile Properties of Various Nylon Membranes

Nylon	Tensile strength at break, lb/wt · in.^{2}		Elongation at break, %	
	Dry	Wet (under water)	Dry	Wet (under water)
Nylon 4	6330	1200	38.3	42.8
Nylon 6	6160	3120	75.9	64.2
Nylon 6,6	8017	4080	20.0	2.17

Table VIII illustrates the rejection characteristics of the nylon 4 membranes. It is interesting to note that MgSO$_4$ is rejected 92%, and carbohydrates such as lactose are rejected by as high as 94%. Ethylene glycol series and carbohydrates
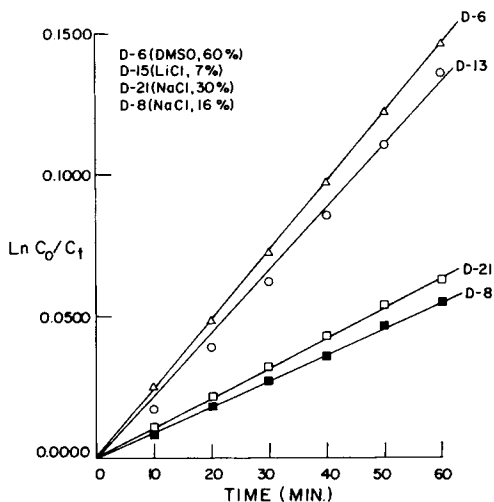
Fig. 2. Dialysis of urea through nylon 4 membranes containing LiCl, DMSO, and NaCl swelling agents.
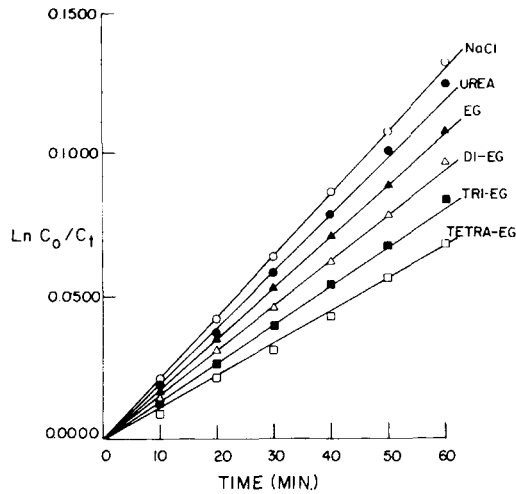
Fig. 3. Dialysis of NaCl, urea, and a series of polyethylene glycols through a nylon 4 membrane containing LiCl (7%) swelling agent.

showed an increasing rejection with increasing molecular weight. The solute rejection decreased with increasing solute concentration, but the product rate and separation did not change appreciably in the range of 0.5–1.5%. It is also interesting to note that the product rate remained constant through these series of experiments.

Ultrafiltration

Water flux is one of the most important characteristics of membranes. In general, commercial membranes are classified according to the criteria of water flux at 50 psi as follows: $<7 \times 10^{-5}$ g/cm^2 sec for reverse osmosis membranes, (7 to 14) $\times 10^{-5}$ g/cm^2 sec for dialysis membranes, and $>14 \times 10^{-5}$ g/cm^2 sec for
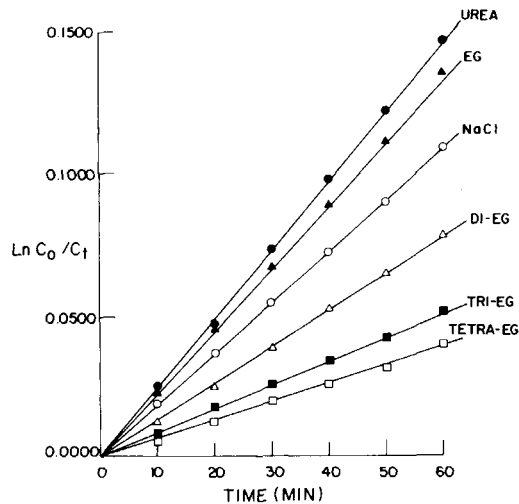

Fig. 4. Dialysis of NaCl, urea, and a series of polyethylene glycols through a nylon 4 membrane containing dimethyl sulfoxide (60%) swelling agent.
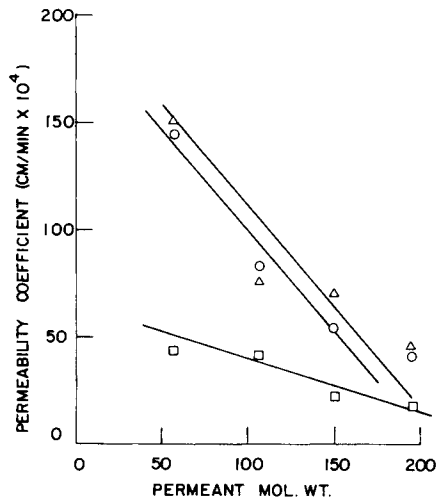
Fig. 5. Permeability coefficients of the ethylene glycol homolog depending on the permeant molecular weight. (O) D-6; (△) D-13; (□) MEM 318.

ultrafiltration membranes. Therefore, in order to find an appropriate application in each field, membranes with proper water fluxes were searched for. A wide range of water fluxes can be produced from nylon 4 by varying the amount of swelling agent used for adjustment. Controlled amounts of the swelling agent readily affect the water fluxes without loss of mechanical strength of the membranes. Slight changes on the concentration of LiCl resulted in dramatic changes in the water fluxes as can be seen in Table VI.

Table IX shows the ultrafiltration data for water permeation through nylon 4 membranes at an applied pressure of 50 psi and at $23 \pm 1°C$ room temperature. The membranes prepared from the casting solution containing 14% NaCl had values of water fluxes which were between those of commerical cellulose acetate membranes KP98 and cellophane. The water flux of membrane 318 was higher than that reported for the commercial membranes. This suggests that nylon 4 membranes containing NaCl with appropriate adjustment in the amount of salt added can be used not only for dialysis but also for ultrafiltration applications.

Dialysis

Dialysis runs were carried out for sodium chloride, urea, and a homogeneous series of ethythlene glycols for the nylon 4 membranes having 10 g/cm²·sec water fluxes. Table X and Figures 1–4 illustrate preliminary dialysis data obtained. The results are plotted in the form $\ln C_0/C_t$ versus time for the various permeants. It can be seen that all values of $\ln C_0/C_t$ change linearly with the time of dialysis and the values for urea and sodium chloride are generally higher than those for ethylene glycols. Table X shows the permeability constants for soidum chloride, urea, and the series of ethylene glycols. The effect of the permeant molecular weight of the ethylene glycol on the permeability through nylon 4 membranes is shown in Figure 5.

Tensile tests

The tensile tests for the nylon 4 were carried out in both the wet and dry states and are compared to those of nylon 6 and nylon 6,6.   The results are shown in Table XI.

CONCLUSIONS

The reverse osmosis properties of nylon 4 which were prepared under various conditions did not exceed a spearation (SEP) of 53.3%.   It has been shown however, that from casting solutions containing suitable amounts of organic or inorganic swelling agnets, there were considerable improvements in water fluxes and salt separations in the reverse osmosis experiments.   These membranes also exhibited dialysis properties which compare favorably with those of the commercial dialysis membranes, and transport properties of other types of nylons (nylon 6, nylon 6,6, nylon 6,10, nylon 12, and nylon 3) as well as blends of nylon 4 with these polymers and will be reported in a separate paper.

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