

Some Experimental Parameters Affecting Performance of Composite Reverse Osmosis Membranes Produced by Plasma Polymerization

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Synopsis

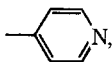
Reverse osmosis (RO) composite polymer membranes were prepared from the organic monomers 3-butenitrile, propyleneimine, 4-vinylpyridine, and allylamine by plasma polymerization in a radio-frequency discharge. RO performance data (water flux and salt rejection) were obtained as a function of discharge power and deposition time. Membranes were subjected to RO testing at 45°C for up to 150 hr to investigate possible membrane deterioration. No appreciable degradation was observed. Water flux increased significantly with time at 45°C, and improved salt rejection at 45°C was observed in most cases.

INTRODUCTION

This study was initiated to further investigate the relationship of the structure of nitrogen-containing groups in selected monomers to the performance of reverse osmosis (RO) membranes produced by plasma polymerization. Nitrogen-containing monomers have been demonstrated^{1,2} as a class of compounds effective in providing highly functional membranes for RO when polymerized in a plasma.

In this study, RO testing was conducted at 45°C to accelerate any possible degradation of membrane performance, i.e., water flux and salt rejection. Although monomers with specific nitrogen-bonded structures were selected, it should be noted that a proportion of nitrogen-containing groups in plasma-polymerized membranes are unlike those found in the original monomers.² For example, there is a strong tendency for nitrile groups to form in the polymer during plasma polymerization, independent of the nature of the nitrogen structure in the monomer.

A previous study by Bell and co-workers³ indicated that RO membranes formed from the plasma polymerization of allylamine (AAM) degraded significantly after elevated-temperature testing. In addition to further work with AAM, which contains —NH₂ groups, monomers containing



—C≡N, and —CH=NH were investigated in this study to provide additional

insight into structural factors relating to possible membrane degradation. According to the proposed mechanism for degradation, AAm forms nitrile groups when polymerized. This was confirmed by analysis of the infrared spectrum. It was proposed³ that the nitrile groups reacted with residual polymer free radicals to become converted to aldimine- and ketimine-type structures. These nitrogen structures, similar to their known organic reaction behavior, were degraded by hydrolysis to NH_4OH in the RO saline solution. There was a complete loss of nitrogen in the surface of the membrane, as evidenced by ESCA analysis in this study.

Therefore, it was believed that various monomers in the present study would reflect the relative stability of nitrogen structures in membranes toward possible degradation. Prior to elevated-temperature RO studies, deposition rates, times, and the power going into the discharge for the various monomers were studied for their effects on RO performance. This preliminary characterization of plasma polymerization behavior and membrane performance was undertaken to learn what plasma conditions were necessary to form optimum membranes for RO testing.

EXPERIMENTAL

Monomers and Substrate Material

The following monomers were employed:

Monomer	Formula	bp, °C
Allylamine (AAm)	$\text{CH}_2=\text{CHCH}_2\text{NH}_2$	52–53
3-Butenenitrile (3-BN)	$\text{CH}_2=\text{CHCH}_2\text{CN}$	117–119
Propyleneimine (PI)	$\begin{array}{c} \text{H} \\ \\ \text{CH}_3-\text{CH}-\text{CH}_2 \\ \\ \text{N} \end{array}$	66
4-Vinylpyridine (4-VP)	$\text{CH}_2=\text{CH}-\text{C}_5\text{H}_4\text{N}$	53–55

All reagents were obtained from Eastman Organic Chemicals, with the exception of PI, which was obtained from K & K Fine Chemicals. All compounds were distilled, except for 4-VP, and all were thoroughly degassed by several freeze-thaw cycles under vacuum prior to use.

The substrate material was acquired from the Roga Division of Universal Oil Products. The microporous substrate was an asymmetric cellulose acetate-cellulose nitrate (CA-CN), having an average pore size of 250 Å in the dense skin layer.

Apparatus and Membrane Preparation

The plasma apparatus employed in this study was essentially the same as that reported in the initial study² and as modified by Bell and co-workers³ to enable better pressure and flow rate measurement. Since several hundred membranes have been produced in this system, it was considered desirable to present here a more detailed schematic of the reactor portion of the deposition system. This is shown in Figure 1.

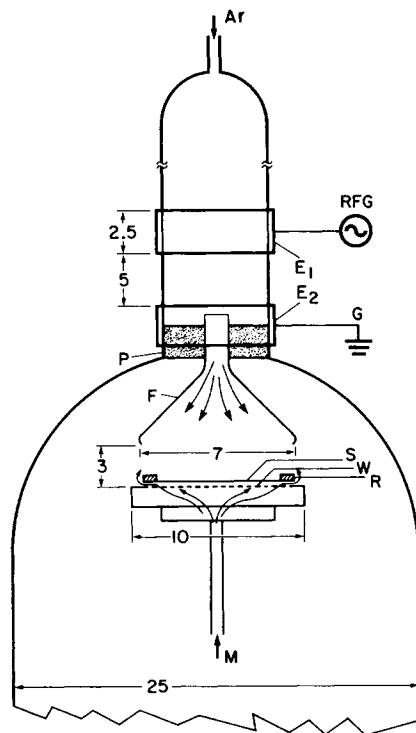


Fig. 1. Bell jar plasma polymerization system. All numbers are dimensions in centimeters. RFG, Radio-frequency generator and associated electronics; E_1 and E_2 , RF and ground electrodes, respectively; P, Buna plug; F, funnel; S, substrate; R, support ring; W, wire screen; M, monomer inlet.

The membranes were formed by the interaction of the argon plasma, produced in the electrode region (E_1 , E_2), with monomer vapor that entered through a pedestal holder and exited through small distribution orifices, and finally through a fine-mesh wire screen on which the substrates lay flat. A 7.6-cm-diameter piece of substrate material was held in a flat position by a metal ring of the same outer diameter as the cellulosic substrate. The center 5.25-cm diameter of the substrate was exposed during deposition.

The flow of argon was $16.0 \text{ cm}^3/\text{min}$ (S.T.P.) for all the membrane preparations. The argon plasma was expanded through a funnel inserted in a Buna A rubber plug and encountered the monomer vapor in a countercurrent flow pattern. Mixing of the monomer and argon plasma initiated polymerization on the substrate surface. Monomer flowed radially out and around the circumference of the substrate support ring, and any unreacted monomer, other gaseous products, and argon were subsequently pumped away at an exit in the bell jar base plate.

In all of the experiments described here, the monomer and argon flow rates were kept constant for membrane preparation, while other parameters, such as radio-frequency (RF) power and deposition times, were varied. Table I lists the pressure conditions used for preparing membranes from the various monomers.

A significant aspect of the reactor configuration here was that the substrate

TABLE I
Pressures at Nondischarge Conditions

Monomer	P_M , torr ^a no discharge	P_{M+Ar} , torr ^b no discharge
3-BN	0.026	0.270
PI	0.020	0.263
AAm	0.050	0.283
4-VP	0.005	0.261

^a Pressure of monomer.

^b Pressure of monomer + argon.

TABLE II
Effect of Backed Versus Unbacked Universal Oil Products Membrane Support
on Water Flux and Salt Rejection^a

Monomer run pair	Flux, GFD ^b		NaCl rejection, %	
	Backed	Unbacked	Backed	Unbacked
BN-7B, BN-25U ^c	0.6	4.29	32.0	83.5
BN-34B, BN-35U	371	4.18	5.4	89.1
BN-36B, BN-37U	14.8	2.96	60.4	84.0
PI-6B, PI-9U ^d	40.0	7.99	33.0	85.7
PI-2B, PI-1U	very high	22.1	11.3	74.7

^a RO test conditions: 10 g/l. NaCl; 600 psi applied pressure; test temperature, 20°C.

^b GFD = gallons/(ft² day).

^c All BN runs at 30 W power, 1500 sec deposition time.

^d All PI runs at 30 W power, 1000 sec deposition time.

and monomer, and consequently polymerization and the membrane during its formation, were essentially out of the high electric field region which was mainly between E_1 and E_2 . It was believed that minimal polymer degradation was achieved using this design. The purpose of the funnel was to provide a high forward velocity for the argon so that minimum back diffusion of the monomer into the high electric field region took place. Such was the case, since negligible polymer formation occurred in the electrode region. If the funnel was not present, polymer formation took place rapidly in the electrode region, and the polymer film was markedly degraded, even charred for example, at 60 W input power. Thus, with the substrate downstream from the electrode region, a better structured polymer film (i.e., minimal degradation) formed at temperatures slightly above ambient.

The pump-down time and the length of time that was required to adjust flows of monomer and argon for the desired deposition condition prior to initiating the plasma were held constant. Prior to the RO tests, the membrane area was reduced in size by cutting with a die to a 5.0-cm diameter. All membranes were tested at 600 psi applied pressure in an RO facility previously described.⁴ The NaCl feed solution concentration was 10 g/l. The membrane water permeability of the Universal Oil Products CA-CN substrate was 270×10^{-5} g/(cm² atm sec). Prior to raising the temperature of the RO system to 45°C, all membranes were run at 18°C for 24 hr.

In initial tests in this study, as well as in previous work on AAm,^{2,3} the substrate was backed during deposition with an equivalent size disk of glassine paper. This impermeable backing was employed to allow deposition only on the shiny or skin side of the substrate. It was believed that the back side of the substrate should be masked to prevent any deposit there from obscuring effects of deposition on the skin side. However, the effect of backing versus not backing the substrate was found to be dramatic in an adverse way (see Table II). In initial runs with some of the monomers, unexpected erratic water fluxes and NaCl rejections were observed. This behavior was traced to the backing of the substrate. Membranes from allylamine did not appear to be as radically dependent on the presence of a backing material as membranes prepared from other monomers. Therefore, all the data to be reported subsequently will relate to membrane preparation without a backing of glassine paper.

RESULTS

Membrane Performance and Deposition Time

Hudis and Wydeven⁵ found that when ethylene–nitrogen was copolymerized for a long time, the subsequent membranes exhibited a dramatic decrease in salt rejection and a sharp increase in water flux. For example, NaCl rejections and water fluxes were 99.5% and 2 GFD (gallons/ft²/day), respectively, for a membrane prepared at an 800-sec deposition time, while at 1200 sec, these values became 10% and 85 GFD. Their reactor was a parallel-plate internal electrode arrangement, and the substrate rested on the high-voltage electrode and was therefore directly in the field during polymerization. The bell jar apparatus used in the present and previous studies^{2,3} did not produce any significant decrease in membrane performance over deposition time periods longer than required for optimum performance. However, to verify this for the present apparatus, two monomers, BN and PI, were selected for study. Table III shows the case for BN deposited at 30 W for times up to 2000 sec. Decreased water flux and increased salt rejection were noted until 1200 sec, undoubtedly due to continuing pore closure of the substrate.³ At 1200 sec, the pores were apparently closed and the per cent salt rejection was not markedly affected by further increase in membrane thickness. Similar behavior has been noticed for AAm, and the pore closure has been followed by SEM for various deposition times.³

For PI membranes, Table III, there was some indication of a decreased rejection over the range of 700–1500 sec deposition time, while the water flux declined. PI membranes always exhibited rather poor adherence to the substrate, and it is possible that at longer deposition times the poor adhesion became a problem which contributed to reduced rejection. Alternately, the membrane polymer may have degraded with longer deposition times, as suggested by Hudis and Wydeven.⁵ In any case, the observed decrease of about 10% in salt rejection for PI was far less over the deposition time range than previously observed by Hudis and Wydeven for ethylene–N₂. As they pointed out, their films were deposited in the regions where the plasma was generated, i.e., directly in the electric field where the ultraviolet radiation could have been quite intense and could have led to fragmentation or scissioning of polymer chains. The results of the present study suggest that deposition times can be roughly twice as long

TABLE III
Flux and NaCl Rejection for Various Deposition Times^a

Deposition time, sec	Flux, GFD	Rejection, %
3-BN ^b		
750	7.9	53.3
1200	2.3	88.7
1500	6.42	85.0
1800	2.57	90.7
2000	0.16	86.3
PI ^c		
700	7.5	90.2
1000	3.3	93.0
1500	1.1	81.9

^a RO test conditions: 10 g/l. NaCl; 600 psi applied pressure; test temperature, 20°C.

^b 30 W deposition.

^c 50 W deposition.

as necessary to get initial complete substrate pore coverage yet achieve acceptable membrane performance without significant membrane degradation. This is attributed largely to the reactor configuration used for membrane synthesis, i.e., polymerization outside the region of the high electric field.

Deposition Rates

Deposition rate, $\mu\text{g}/(\text{cm}^2 \text{ min})$, was determined by weight gains on 4.84- cm^2 microscope cover slips over known time intervals. Figure 2 shows the rates of deposition for the various monomers as a function of discharge power. The rates for AAm and PI tended to increase with discharge power; 4-VP polymerization decreased, and 3-BN remained steady with increasing power. The rate behavior of 4-VP may imply that the heterocyclic ring was opening at higher discharge power, with more gas-phase products and less condensed polymer being formed. The rates were essentially linear with power.

RATES OF POLYMERIZATION, $\mu\text{gm}/\text{cm}^2/\text{sec}$, vs POWER

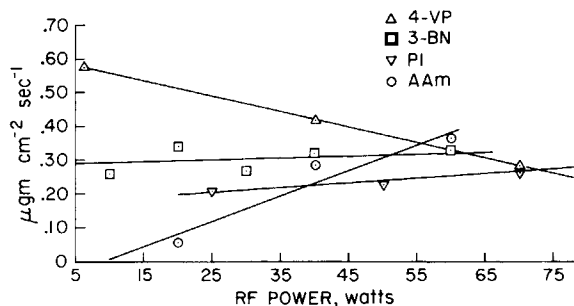


Fig. 2. Deposition rates in $\mu\text{g cm}^{-2} \text{ min}^{-1}$ vs. RF power, W, for the various monomers prepared under the same conditions as for membrane production. Total deposition time, 2000 sec.

TABLE IV
Flux and NaCl Rejection of Monomers 3-BN, PI, AAm, and 4-VP
for Various Power Levels^a

RF power, W	3-BN		PI		AAm		4-VP	
	Flux ^b	Rej. ^c	Flux	Rej.	Flux	Rej.	Flux	Rej.
10	2.74	57.5	10.6	19.0	70.2	45.8	—	—
15	—	—	—	—	—	—	0.13	96.6
20	1.96	76.9	—	—	12.07	52.1	—	—
30	2.30	88.7	22.1	74.7	20.25	56.0	3.5	88.0
40	4.90	90.2	5.96	70.0	14.29	87.0	—	—
50	2.10	89.9	4.3	93.0	0.14	70.0	1.2	93.8
60	1.34	83.3	0.3	88.0	0.07	13.0	1.75	98.6
70	0.4	40.6	1.17	90.1	—	—	—	—

^a Deposition times: 3-BN, 1200 sec; PI, 1000 sec; AAm, 480 sec; 4-VP, 900 sec.
RO test conditions: 10 g/l. NaCl; 600 psi applied pressure; test temperature, 20°C.

^b Flux values in gallons/(ft² day).

^c Rejection, per cent of NaCl concentration.

Membrane Performance and Discharge Power

The RF power coupled into a discharge produces desired reactions up to a certain power level, after which the additional power merely adds heat to the gas and surroundings. In the present experimental arrangement, the cellulosic substrates will begin to deform after a 600-sec exposure to argon plasma above 80 W discharge power.

Table IV shows the dependence of water flux and salt rejection on power for 3-BN, PI, AAm, and 4-VP membranes. For membranes formed from 3-BN, PI, and AAm, the rejection increased with increasing power and decreased significantly for 3-BN and AAm at the highest powers. In the case of membranes formed from 3-BN and AAm, the rejection clearly goes through a maximum in the range of 30–50 W; after 60 W, the rejection decreased significantly.

It appears that a poorer, more degraded membrane structure resulted at high powers for 3-BN and AAm. Bell and co-workers³ noticed considerably better membrane performance at a high-power level than in this study (82% salt rejection, at 80 W power input). However, their study took into consideration the fact that as power increased, the rate of polymerization increased for AAm. To compensate for this factor, they measured the polymerization rate for AAm and adjusted the deposition time to allow an equivalent mass (hence membrane thickness) to develop while varying the discharge power. At 80 W, the deposition time was 155 sec as opposed to 480 sec at 60 W in Table IV of this study. The rapid degradation of membrane performance at these latter conditions reflects the fact that longer deposition times coupled with higher powers can indeed be deleterious to membrane performance.

Concurrently, as the deposition rate increased with power, so did the membrane thickness at constant deposition time, except for 4-VP. This observation is reflected in the values obtained for flux. In the case of 4-VP, the lowest flux was seen at the lowest power. This is consistent with the fact that the most mass, hence membrane thickness, had been deposited at the minimum power (Fig. 2). As power increased, the rate of deposition decreased, producing a thinner

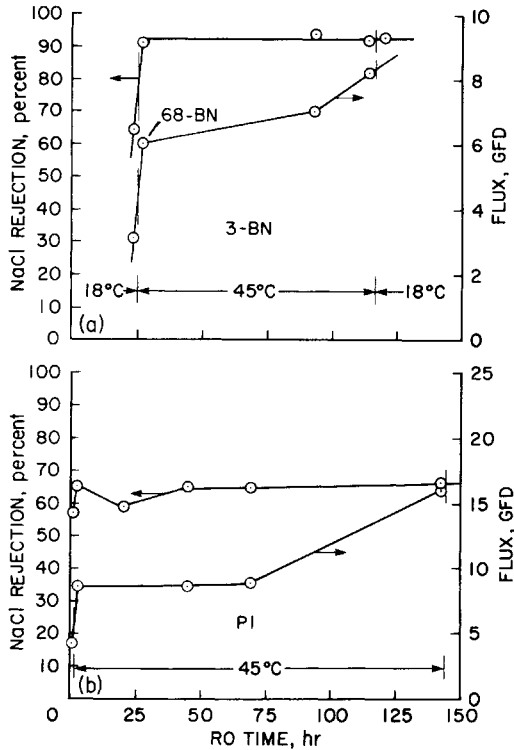


Fig. 3. Water flux and salt rejection during RO test time for (a) membrane prepared from 3-BN at 1200 sec and 60 W; the point 68-BN refers to data taken from a different run of an identically prepared membrane, and (b) membrane prepared from PI at 1000 sec and 45 W.

membrane, and the flux was seen to increase. At 60 W, the membrane was still structurally favoring high rejection.

Bell et al.³ produced membranes from AAm of equivalent thickness by adjusting the deposition times at various powers and consequently found no such dependence of flux on power increase. Therefore, in optimizing the performance of membranes derived from previously unexamined monomer candidates, the trade-off between deposition time and power going into the discharge must be carefully assessed toward their effects on water flux and salt rejection.

Membrane Performance at Elevated Temperature

The choice of monomers was made primarily to investigate the possible effect of the structure of nitrogen in the monomer on ultimate membrane performance during RO at elevated temperature (45°C) and for longer RO test times than previously studied (up to 140 hr), to accelerate any possible deterioration which might occur.

A previous ESCA study³ indicated for AAm that most surface nitrogen was lost after RO at elevated temperatures. A reaction mechanism was invoked which was based primarily on the rapid conversion of the amine group to a nitrile during polymerization and subsequently, under RO conditions, to aldimine- and ketimine-type structures, with subsequent hydrolysis of the latter. Figure 3a

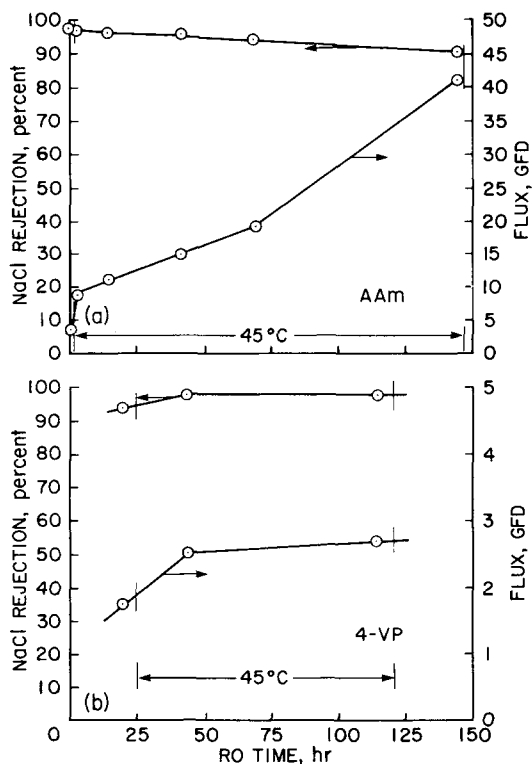


Fig. 4. Water flux and salt rejection during RO test time for (a) membrane prepared from AAm at 480 sec and 30 W, and (b) membrane prepared from 4-VP at 900 sec and 50 W.

shows the behavior of 3-BN during RO at 45°C for up to 115 hr. No significant degradation of RO performance was observed. The water flux at 45°C more than doubled its original value at 18°C.

The membrane from PI monomer, Figure 3b, had an initial rejection of 58% at 18°C. After 144 hr at 45°C, the rejection was actually somewhat higher, 66%, while water flux increased by a factor of 3 over its value at 18°C.

The membrane from AAm monomer, Figure 4a, exhibited a decrease in rejection from an initial 98% to about 90% at 45°C over 144 hr. The water flux increased to nearly four times the initial value at 45°C over this time period. While it appears that some decrease in membrane performance has occurred, based on the decrease in salt rejection, there is not the rather catastrophic decrease from 83% to 48% after 72 hr at 50°C, as observed by Bell and co-workers.³

ESCA Results

ESCA analysis (Table V) of the polymer membrane was performed before and after RO at elevated temperature. Consistent with the observation of Bell et al.,³ the nitrogen peak intensity decreased for AAm and for all membrane materials studied by a factor of 2 or more; AAm nitrogen intensity decreased by a factor of 4. Yet no significant correlation appears to exist between this reduction in nitrogen content and membrane stability over the test period and test con-

TABLE V
Corrected Elemental Core Electron Line Intensities, $I/1000$

Monomer	Before RO			After RO		
	N	C	O	N	C	O
3-BN	21.4	118	31.8	10.7	200	96.4
PI	45.9	315	50.0	18.4	219	91.0
AAm	50.0	250	67.5	12.5	259	80.6
4-VP	19.0	278	53.8	7.13	176	134.0

ditions used. We have found that IR spectra of many plasma-produced polymers derived from monomers containing nitrogen in different functional groups indicate that in almost all cases, a strong tendency exists for nitrile groups to form, as evidenced by a pronounced peak appearing at 2200 cm^{-1} . The densities of these nitrile groups are not known with certainty; but a significant fraction of the nitrogen-containing precursor groups must have converted to nitrile. The nitrogen loss, as evidenced by ESCA, is possibly due to the further conversion during RO of these nitrile groups to aldimine/ketimine structures which subsequently hydrolyze, as suggested by Bell et al.³

Some of the ESCA interpretation must be taken with caution. After long RO elevated-temperature testing, the entire hyperfiltration recirculation system can release trace constituents from various materials of construction into the RO feed solution. In fact, during RO at 60°C , the feed solution has been observed to turn green, suggesting corrosion of Monel metal parts. Therefore, ESCA, being a surface-limited analytical technique, analyzes to a depth of possibly a few tens of angstroms. If any foreign material accumulation is occurring on even a minute level, this could result in a false elemental analysis of apparent surface composition. Independent testing is presently underway to verify the ESCA results.

DISCUSSION

Figures 3 and 4 indicate that during RO test times of many hours, the water flux is increasing while salt rejection remains steady by comparison. Yasuda and co-workers⁶ also observed that a membrane derived from copolymerization of acetylene- $\text{H}_2\text{O}-\text{N}_2$ in a plasma exhibited a flux of 1.2 GFD at ambient temperature, while after 30 min of RO test time at 80°C , the flux rose to 2.3 GFD; however, no membrane degradation was indicated, since the salt rejection held constant at 96.2%.

In the present study, water flux was seen to increase rather markedly in the initial stages of heating from 18° to 45°C . Since water flux continued to increase over many hours during the test period, this suggested that a slow structural change was occurring in the membrane itself. Decreased viscosity of the salt solution at 45°C cannot account entirely for the magnitude of the water flux increase.

A more expanded membrane structure may provide for the inclusion of an increased number of water molecules hydrogen-bonded to hydrophilic groups in the polymer network. These additional sites would favor a higher diffusional transport of water through the membrane due to the increased number of dif-

TABLE VI
RO Performance of Membranes of Polymerized Ethylene-N₂ Mixtures^a

Test time, hr	Temperature, °C	NaCl, % rejection	Water flux, GFD
24	21.5	96.9	0.63
28	44	98.9	1.6
32	60	98.7	1.8
48	22	99.1	0.58
56	68	97.9	2.21
72	20	99.1	0.55

^a Data of C. C. Johnson and T. Wydeven, to be submitted for publication. RO test conditions: 10 g/l. NaCl; 600 psi applied pressure.

fusion pathways and greater density of sites available for the successive formation, breakage, and reformation of hydrogen bonds.

Rosenbaum et al.⁷ investigated the diffusion of an NaCl solution through conventional cellulose acetate membranes from 24° to 50°C. They measured a water flux which doubled over this temperature range and a salt flux that nearly doubled. One of their significant conclusions required the necessary creation of additional water sites in the membrane, hence water content, to serve to solvate salt ions.

Walch⁸ has noted that there is a requirement for hydrophilic groups to be well dispersed in RO membranes in order to have appreciable water flux without coupled salt flow. The density of these groups and their susceptibility to hydrolysis or other chemical alteration during RO could be related to the stability, improvement, or deterioration of membranes with time. All three cases were observed to some extent. A small amount of degradation, interpreted as a decrease in NaCl rejection, was observed for AAm. Rather steady salt rejections for 3-BN and PI, and a modest decrease in salt rejection for 4-VP, were observed.

In work to be submitted for publication by Johnson and Wydeven⁹ on the preparation and RO performance of membranes from copolymerization in a plasma of mixtures of ethylene-N₂, the data showed a similar trend to the data on the membranes produced from the monomers of this study. Table VI summarizes some of their results. The water fluxes are seen to be essentially reversible when the RO test temperature was reduced to 20–22°C after intermediate higher temperature excursions. This lends further validity to the assumption that the change in the membrane is not produced by a permanent chemical alteration of the polymer itself, but rather by a temperature-dependent, temporary modification of the membrane structure related largely to the degree of inclusion of hydrogen-bonded water sites and solvated ions at elevated temperatures. It is suspected that the reversibility of water flux, as a function of membrane expansion and contraction after heating and cooling, will be a fairly general observation of plasma-produced membranes.

On cooling, the membrane apparently contracts and excludes a certain amount of the hydrogen-bonded water molecules that assisted in water transport through the membrane. It is also evident from Table VI that water flux has increased by nearly 350% at 68°C over the values measured at 20°C. The salt rejection

has remained rather steady without membrane deterioration over the test period.

Another interesting feature of the membranes tested in this study and also exhibited by membranes from ethylene-N₂ polymerization (Table VI) was the rather significant improvement in membrane per cent salt rejection in the first hour or two of heating. This was particularly evident for membranes from 3-BN. Increased salt rejection could result from more complete pore closure of residual uncovered or partially covered micropores that allowed coupled water and salt flow. The pore filling could have been brought about by polymer or organic material from other parts of the membrane. The mechanism for pore filling could be a combination of the applied hydraulic pressure and the increased mobility of the material closing the pores at higher temperatures. This improvement in membrane structural integrity was apparently a permanent change since there was not a return to lower salt rejections at room temperature.

If membrane deterioration is occurring, this may be regarded as owing to both chemical and physical factors. Chemical degradation may proceed for AAm as suggested by Bell et al.³ under their test conditions, although the present study indicated that considerably more stable membranes can be produced from AAm. In addition, certain monomers in the present study, such as those containing nitrile or imine groups, should have led to membranes which would be even more prone to degradation than that which was observed for amine-containing polymers. Physical degradation in the polymer membrane at elevated temperature could be the ultimate production of such an expanded and swollen network that larger holes are being produced allowing more and better diffusional pathways for salt ions coupled with water. Perhaps lower molecular weight materials are being slowly leached out of the membrane at higher temperature, creating pores of larger sizes.

In summary, the following conclusions are made:

1. As pointed out by Yasuda et al.,⁶ the nature of the initial monomer may not be as critical in dictating ultimate membrane RO performance as the selection of the plasma conditions attending polymerization.
2. In assessing a new monomer candidate, the deposition rate behavior versus the discharge power must be determined. In addition, screening experiments must be performed to determine what deposition times and RF powers provide optimum water flux and ion rejection.
3. Erratic flux and rejection values for membranes may be attributable to the design of the experiment, backing versus unbacking the substrate, and other membrane handling or storage conditions prior to RO testing.
4. In elevated-temperature RO testing, several membranes prepared under identical plasma conditions to give optimum performance (as pointed out in item 2 above) should be evaluated to give true confidence in the trends of water flux and salt rejection with time.
5. After long-term RO testing, ESCA data should be interpreted with caution. Moreover, ESCA does not provide all the information about the internal structure of plasma-produced membranes.
6. Plasma-produced membranes may benefit from an initial heating period to anneal out residual micropores or other unfavorable structural factors prior to room temperature operation.
7. One method that may be used to achieve higher water fluxes, after it has

been determined that little or no membrane deterioration is taking place, is to run the RO system at elevated temperature. This is particularly dramatic for AAm membranes (see Fig. 4a) where flux increased from 7 to 42 GFD at 45°C.

This work was supported by NASA Contract NAS 2-8767 to the Tegal Corp. The authors wish to express their gratitude to James Orasco for preparing the many membranes involved in this study. Catherine Johnson (NASA) also assisted in various portions of this work, for which the authors are appreciative. Dr. E. V. Ballou provided insight and helpful discussions concerning the RO testing and the evaluation of membranes at elevated temperature. The ESCA analyses were performed by Dr. M. Millard of the U.S.D.A., Albany, California.

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Received January 13, 1976

Revised March 10, 1976