Porous cylindrical media as supports for reverse osmosis desalination membranes
by Darrel Eugene Coverdell

A thesis submitted to the Graduate Faculty in partial fulfillment of the requirements for the degree of
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Abstract:
With the development of high flux, high salt rejection cellulose acetate membranes, reverse osmosis has become one of the important methods being considered for desalination. It combines flexibility and simplicity with thermodynamic efficiency. Good membrane performance at a high membrane density is important if reverse osmosis is to be successful. Using small diameter cylindrical porous media as supports for cellulose acetate membranes offers a solution to this problem.

Two membrane casting techniques and five different types of cylindrical porous media were investigated. The effects of support type, casting technique, casting solution, and various casting variables on membrane performance were studied. Membrane life investigations were also made.

Fluxes of 10-11 GSFD at 95% salt rejection were obtained when dip casting on ceramic supports using a casting solution containing l4.9% Eastman 400-25 cellulose acetate, 21.3% formamide, and 63.8% acetone. A 5-second solvent evaporating time and an 86°C heat treatment were used. For dip casting, acetone concentration ranges were established: for E398-3 cellulose acetate, 54-59% acetone; for e400-25, 60-65% acetone; and for E394-60, 63-68% acetone. A cellulose acetate-formamide ratio of 7:10 was used in all cases. Viscosities in the dip casting range vary from ~900 centipoise to ~3500 centipoise.

Fluxes of 16-18 GSFD at 93% salt rejection were obtained for extruded membranes on ceramic supports using a casting solution containing 14.9% Eastman 394-60 cellulose acetate, 21.3% formamide, and 63.8% acetone. A 5-second solvent evaporating time and an 86°C heat treatment were used. Extruded membranes always gave fluxes of 1.4 to 2.2 times those of similar dip cast membranes.

Porous ceramic, porous polyethylene, and porous polyvinylidene fluoride supports were the most promising. A membrane performance of 17 GSFD at 93% salt rejection was obtained on the ceramic; 11 GSFD at 92% salt rejection on the polyethylene, and 7.7 GSFD at 93% salt rejection on the polyvinylidene fluoride.

Support length and support diameter did not appear to affect membrane performance; however, support permeability does limit the length of porous rod that can be used. Use of tubular porous media is suggested.

Water flux declined about 15% during the first hours of operation, but remained constant thereafter provided the membrane surface remained clean. Salt rejection declined slightly during long runs.

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FOR

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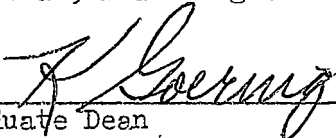
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ABSTRACT

With the development of high flux, high salt rejection cellulose acetate membranes, reverse osmosis has become one of the important methods being considered for desalination. It combines flexibility and simplicity with thermodynamic efficiency. Good membrane performance at a high membrane density is important if reverse osmosis is to be successful. Using small diameter cylindrical porous media as supports for cellulose acetate membranes offers a solution to this problem.

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I. INTRODUCTION

The world's need for fresh water is increasing at a very rapid rate -- a rate which will soon outstrip new supplies of fresh water in many areas. Finding new sources of good quality water at a reasonable cost is becoming a major problem.

Natural sources of water (wells, rivers, etc.) may currently be tapped at a cost of between 13 and 70¢ per thousand gallons (8). It is doubtful that new technology or equipment will be able to lower this cost or increase the natural supplies. On the basis of the high cost and unavailability of new natural fresh water supplies, it seems likely that soon it will be necessary to tap the world's vast quantities of brackish and sea water. This is especially true in the more arid areas such as the Middle East or the highly populated areas such as Southern California.

Until recently, thermal distillation was the only technique that had been developed for desalting water; however, because of the increased interest in desalted water, a number of new techniques are being developed. Commercial possibilities and research under the direction of the United States Office of Saline Water have enhanced the rapid development of these techniques. The more important processes for desalination now include: multistage flash distillation, falling film distillation, vapor compression distillation, electrodialysis (brackish water only), direct freezing, and reverse osmosis.

Distillation techniques are the most advanced. Using present day technology, costs of 80¢ to $1.10 per thousand gallons of fresh water for small multistage flash evaporation plants have been reported. For large plants (50 million gallons per day or more), this cost would be lowered to 20 to 40¢ per thousand gallons (8). Electrodialysis and reverse osmosis are also becoming important processes and costs are approaching those of distillation; especially in the area of brackish water desalting. Costs of 75.5¢ per thousand gallons have been esti-mated for a one million gallons per day reverse osmosis plant (2).

The principle of reverse osmosis is quite simple. Figure 1 illustrates osmosis processes. In ordinary osmosis, the pure water will pass through the membrane into the brine so as to dilute the brine solu-tion (Figure 1-a). No flow occurs at osmotic equilibrium (Figure 1-b); however, the flow of pure water may be reversed (Figure 1-c) by apply-ing a pressure greater than the osmotic pressure (osmotic pressure 1.15 psi for each 100 ppm NaCl) on the brine. This is called reverse osmosis.

Several inherent advantages exist for the reverse osmosis process. First, large energy requirements are not required as in the distillation techniques. Reid (17) reported that the minimum theoretical energy re-quired for desalting sea water is 2.63 Kw-Hr per thousand gallons. Dis-tillation techniques require up to 6 times this amount (23) while re-verse osmosis approaches this minimum energy requirement.

Figure 1. Osmosis Phenomena

Secondly, reverse osmosis operates at ambient temperatures which tends to lessen corrosion. Scaling problems are also virtually non-existant when compared to distillation techniques.

Another advantage is the simplicity of the reverse osmosis process and its equipment. The process is essentially an ultra-filtration of salt from the brine. The equipment consists of only two major items: a pump capable of pumping the brine to high pressures (600-800 psi for brackish water and 1200-1500 psi for sea water) and a membrane module in which the reverse osmosis process operates. The most important part is, of course, the membrane and the module in which it is mounted.

Membranes have improved considerably since Breton (3) first reported in 1957 that cellulose acetates act as semi-permeable membranes for sodium chloride solutions. Almost all other polymers have been tested as reverse osmosis membranes. Other reverse osmosis systems have also been fabricated from materials such as graphitic oxide (9), porous glass (11), and clay-like compounds. Many of these other polymers and systems do possess the ability to act as semipermeable membranes; but none possess the better desalination qualities of the cellulose acetate membranes.

The performance of cellulose acetate membranes has improved considerably. In 1958, Reid and Breton (18) obtained membranes 6 microns thick which produced a water flux of .945 GSFD (gallons per square foot

per day) and a salt rejection of 95%. Loeb and Sourirajan (14) obtained

fluxes of 1.265 GSFD and 99% salt rejection in 1961 by shrinking com-

mercially available cellulose acetate filters which were 100 microns

thick. As a result of their experience and an article by Mlle Dobry (7)

which suggested the use of magnesium perchlorate as a solvent in the

preparation of ultrafiltration membranes, Loeb and Sourirajan developed

a membrane with a flux of 5.4 GSFD and 99.8% salt rejection. A casting

solution containing acetone, magnesium perchlorate, water, and acetone

in the ratio 22.2-1.1-10.0-66.7 wt. percent was typical for preparing

these membranes. They also found that it was necessary to strictly

control the fabrication steps in order to obtain good results.

More recently, in 1964, Manjikian, Loeb, and McCutchan (15) pre-

pared casting solutions which contained non-electrolytes. A ternary

casting solution of cellulose acetate, formamide, and acetone was found

to produce good membranes. Membranes from this casting solution gave

fluxes of 20 GSFD and salt rejections of 95%. Today this type of cellu-

lose acetate membrane is the most widely used.

Electron microscopy studies of the high flux cellulose acetate

membranes have shown that they consist of two definite layers. The top

layer is a dense, very thin layer often referred to as the "active"

layer. It comprises between 1 and 10% of the total membrane thickness

and is on the order of .1 to .25 microns in thickness. This layer (on

the air-dried side of the membrane) is that portion of the membrane
which acts as the semipermeable barrier. The remainder of the membrane
is an amorphous porous substructure estimated to have a pore size of
between .1 and .4 microns (19).

Many theories have been proposed for the mechanism of water trans-
port through the membrane. Breton (3) proposed that water was trans-
ported through the membrane via alignment-type diffusion while some
salt and additional water was transported via hole-type diffusion with
no desalting. Sourirajan (22) proposed that a thin film of pure water
exists at the liquid-membrane interface. For pores with diameters of
greater than twice the film thickness, both pure water and saline water
will flow. Through the smaller pores, only pure water will pass. Using
these criteria, Blunk (1) postulated the following mechanism: "Water is
retained in the osmotic skin part of the membrane in such a way that it
still possesses the solubilizing properties attributable to its hydrogen
bonding capacity, but has largely lost the solubilizing properties
attributable to its high dielectric constant. Therefore, small species
whose solubilities in water are due partially (hydrogen-bonding univalent
ions) or wholly (nonelectrolytes) to their hydrogen-bonding capacities,
tend to pass through the membrane. On the other hand, small species
whose water solubilities are due primarily to the high dielectric con-
stant of water tend to be rejected. Those species include non-hydrogen-
bonding univalent ions, and all ions of valence greater than unity re-

gardless of hydrogen-bonding characteristics." (1)

The development of high flux membranes has presented an additional problem. For high flux membranes, a polarization effect due to a salt concentration buildup at the brine membrane interface becomes an important factor. This concentration buildup lowers the water flux (the effective driving pressure is lowered because of a higher osmotic pressure at the membrane surface) and increases the salt flux (the salt gradient increases). Brian (17), using a thin film model for turbulent flow, gave the following equation to describe the polarization effect:

$$\frac{C'}{C^b} = \frac{\exp\left[\left(\frac{v'}{j_D \bar{U}}\right) N_{Sc}^{\frac{2}{3}}\right]}{r + (1-r) \exp\left[\left(\frac{v'}{j_D \bar{U}}\right) N_{Sc}^{\frac{2}{3}}\right]}$$

where C' = salt concentration at the membrane interface, g/cm^3

C^b = salt concentration in bulk solution, g/cm^3

v' = product water flow velocity through the membrane, cm/sec

j_D = Chilton-Coburn mass transfer j-factor

$\bar{U}$ = average velovity over the cell, cm/sec

N_{Sc} = Schmidt number for salt diffusion; kinematic viscosity of solution, $(cm^2/sec)/$ molecular-diffusion coefficient in salt, (cm^2/sec)

r = salt rejection

This equation shows that the polarization effect is a strong function of desalinized water flux and feed velocity. Therefore, in the case of high flux, high salt rejection membranes, it is necessary to maintain a high brine flow rate past the membrane (turbulent flow) to prevent the polarization effect, thus keeping c'/c^b near unity.

Two other basic problems also exist with reverse osmosis systems. First, the pressures exerted on the membrane are quite high (600-800 psi for brackish water and even higher for sea water). The membranes described thus far are not capable of handling such pressures without additional support. Secondly, even though membrane performance has improved considerably, the desalinized water fluxes are still relatively low. This necessitates a reverse osmosis system to be designed so that a high membrane area per unit volume exists.

Several approaches have been used to solve these problems. An additional approach is being proposed by the author.

One of the first methods used was to mount the membranes in a porous tube (6). The porous tube would act as a support for the membrane. The brine would flow within the tube and the product would exit through the porous walls. Such membranes possess sufficient strength; but because larger diameter porous tubes are used, the membrane density is somewhat low. Also, the cost of mounting the membranes is relatively high. Recent advances in dynamically casting the membranes have given

more promise to this approach. Pilot plants of up to 5000 gallons per day have been successfully tested using the tubular design (6).

A second method is that of spirally winding the membranes to form a tubular module (5). Brine and a porous matrix alternate between membrane layers. The product water exits through the matrix material. Sealing problems, a telescoping effect, and finding a suitable matrix material are major problems. Despite these problems, pilot plants of up to 10,000 gallons per day have been successfully tested (5).

Another technique is that of mounting flat membranes in a "filter press type" arrangement (5). Again, spacers for brine and product flow alternate between membranes. Good membrane densities are obtained, but flow distributions, mounting of the membranes, and the large number of seals required are major disadvantages. Pilot plants of up to 50,000 gallons per day have been tested using this approach.

A fourth technique is that of using hollow fiber membranes (16). These are mounted in a shell and tube design with the brine flowing on the shell side and the product exiting through the hollow fibers. A system of this type produces a very high membrane density; however, because no membrane support exists, the fibers are thick-walled, resulting in low fluxes (< 1 GSFD and generally on the order of .1 to .2 GSFD). This system is one of the more promising as the equipment is simple and easily assembled and low brine flow rates may be used (low

fluxes, therefore no polarization problem); however, clogging is often a problem on the brine side because of the very small interstitial spaces between fibers. Up till now, fibers have not been successfully constructed of cellulose acetate. The polymers currently used for the fibers do act as barriers for many ions, but they are only imperfect barriers for Na^+ and Cl^- ions. This is the major disadvantage of this design. The development of membrane fibers which would act as Na^+ and Cl^- barriers would certainly make this approach more appealing.

The alternative approach being proposed by the author is one in which high flux cellulose acetate membranes are cast on the outer surface of small diameter porous rods or tubes. A shell and tube design would be used to mount the membranes. Such a system would be simple and would provide a high density of high flux cellulose acetate membranes. Also, the clogging problems encountered with the hollow fiber system would be substantially reduced due to a larger diameter tube and the resulting larger interstitial spaces.

Therefore, the object of the author's thesis is to determine the feasibility of casting reverse osmosis desalination membranes on small diameter porous tubular media as an alternative design for reverse osmosis systems. This includes the determination of casting conditions for casting membranes on porous tubular media, making performance tests on various porous media, studying important design factors, and briefly considering economic aspects.

Prior research considering the casting of membranes directly on porous substrates has been performed at Montana State University by Donald Wang (24). He cast cellulose acetate membranes upon a number of porous substrates, but found that an epoxy filled fiber glass material (Gelman Versapor filter material) proved most useful. Fluxes of 21 GSFD and 95% salt rejection were obtained for membranes cast on this material. These membranes were cast from a cellulose acetate - formamide - acetone (21.9, 31.2, and 46.9 weight percent, respectively) casting solution using a 5-second solvent evaporating time and a heat treatment at 85°C. Later work by Lai (10) showed that other porous materials also have promise as supports. Polyvinyl chloride was most promising. A flux of 21.3 GSFD and 97.3% salt rejection was obtained for membranes cast on this support. Casting conditions were the same as those used by Wang. The work of Wang and Lai has been used as a basis for this author's work.

II. EQUIPMENT AND PROCEDURE

A. Membrane Fabrication Techniques

Three different techniques were used for preparing membranes. The first of these was spreading the casting solution on flat porous media using a doctor blade. The other two techniques, used on porous cylindrical rods and tubes, were dip casting and extrusion. Figure 2, page 13, illustrates these techniques.

Membranes cast on the flat porous media were prepared in a constant temperature (70°F) and constant humidity (50% relative humidity) chamber. Casting was done on small glass plates using masking tape for clearance (about .005" clearance per layer of masking tape) and a smooth glass rod as a doctor blade. After spreading the cellulose acetate - formamide - acetone casting solution on the porous support, a measured solvent evaporating time was allowed. The membranes were then placed in cold (< 10°C) water for at least one hour for gelation. The membranes were annealed (heat treated) in hot water for four minutes at a desired temperature (usually between 75 and 90°C). Circular sections 2-1/2 inches in diameter were cut from the membrane for testing.

In dip and extrusion casting, 4-inch lengths of porous tubes or rods were used. Epoxy cement was used to seal one end of the tubes with the other end being left open for product flow. About one inch of the porous rods and tubes at the product end was sealed prior to casting,

1. SPREAD CASTING ON FLAT SUPPORT

1. GLASS PLATE
2. POROUS SUPPORT
3. MASKING TAPE
4. CASTING SOLUTION
5. GLASS ROD
6. MEMBRANE

TOP VIEW

END VIEW

SIDE VIEW

2. DIP CASTING

1. CASTING SOLUTION
2. POROUS ROD OR TUBE
3. MEMBRANE

3. EXTRUSION

1. CASTING SOLUTION
2. POROUS ROD OR TUBE
3. EXTRUSION DIE
4. MEMBRANE

Figure 2.   Casting Techniques

using a silastic sealant. This was done for mounting purposes and to
insure uniform flow past the tube length being tested. The porous rods
and tubes were saturated with water to prevent air bubbles from escaping
from the pores of the support during casting. Such bubbles often caused
leaks when complete saturation was not obtained.

Dip casting was performed by dipping the rods and tubes into the
casting solution, withdrawing the rod from the casting solution at a
nearly constant, relatively rapid rate ($\cong$ 2 inches per second), allow-
ing a measured solvent evaporating time, and then placing the membrane in
cold water for gelation. Casting was done at room conditions (approxi-
mately 75°F and variable humidity). All membranes with rods or tubes as
supports were annealed by immersing the membrane-coated rod in water at
60°C, heating the water at 5-6° per minute to a desired temperature, and
leaving it at that temperature for four minutes.

Extrusion casting on the porous rods and tubes differed from dip
casting only in that a die was dropped over the rod as the rod was with-
drawn from the casting solution. A clearance of .010 inches was allowed
between the rod and the die.

All membranes were stored in water when not being tested.

B. Membrane Test System

A flow diagram of the test equipment is shown on Figure 16, page 72. The system consisted of a 10-liter brine tank, a pump (Jaeco Model 753 S-8), and two parallel test lines, each containing one or more test cells and a back pressure regulator. The test lines could be operated buth at the same time or independently, as needed. All equipment was of plastic or stainless steel material to eliminate corrosion. A high pressure filter was used in some runs to maintain a clean system; however, it was found that frequent changing of the brine was more expedient.

High pressure nitrogen was used as back pressure against the circulating brine. The pump and brine circulation caused an increase in brine temperature. A cooling coil using cold tap water was used to maintain the brine temperature in the tank at $24 \pm .5°C$. Brine temperature at the membrane was $25 \pm .5°C$.

Two types of test cells were used; one for flat membranes, another for membranes cast on cylindrical porous media. The flat test cell (See Figure 17, page 73) consisted of two 304 stainless steel flanges, 4-1/2 inches in diameter, which were machined to desired dimensions. The upper flange contained a 2-inch diameter space for brine flow. This space tapered from 1/8-inch deep at the outer edge to 1/16-inch deep at the center to obtain an even flow distribution across the test cell.

The lower flange contained a 1/8-inch thick, 2-1/2-inch diameter porous (5-micron pore size) stainless steel plate upon which the membrane was supported. Product water passed through the porous support and exited through a 1/4-inch male Swagelok connector fitting. Eight 1/4-inch bolts and a neoprene gasket were used to seal the test cell.

The second type of test cell (Figure 18, page 74) consisted of a 1/2-inch diameter stainless steel tube with a Swagelok heat exchanger tee and a reducing union at the ends. In this case, the membranes were sealed into the heat exchanger fitting using General Electric RTV silastic sealant, a Swagelok front ferrule, and a fabricated reducing nut. See Figure 18 for details of the method used to seal membranes into the test cell. A 12-hour curing time was allowed for the silastic sealant. During this time it was necessary to keep the silastic area dry while maintaining the membrane section wet. Cell assembly consisted only of attaching the heat exchanger fitting containing the membrane to the test tube. The brine flowed on the outside of the membrane-coated porous rod and the product water exited by flowing lengthwise through the porous rod.

C. Test Procedure

After a membrane had been mounted in a test cell, the test cell was attached to a test loop. The brine was then circulated past the membrane at a gradually increasing pressure until the desired operating

pressure was reached. This was 800 psi for most runs. A 10,000 ppm (1%) NaCl brine solution prepared from deionized water and reagent grade NaCl was used in all runs.

Flow past the membrane was 8.7 cm/sec for the flat membrane and varied between 13.6 cm/sec and 43.8 cm/sec for the cylindrical membranes. The variation in the cylindrical membranes was due to the use of various diameter porous rods as supports. Performance was not affected by the variation in brine flow for the above velocity range. Also, membrane performance was not noticeably affected by pressure and flow surges from the reciprocating pump. Using a surge tank (50 feet of 1/4-inch diameter tube), the pressure surge could be lowered to 20 psi from 100 psi.

Product water rates and product water conductivity (resistance) were measured at 1/2, 2, 4, and 6 hours. During the first hours of operation, a flux decline occurs and the salt rejection increases slightly. By 6 hours, product flux and salt rejection are nearly constant. Except where noted, the data reported is that at the end of 6 hours.

Salt concentrations were measured using a conductivity bridge and two conductivity cells. These cells were calibrated and checked periodically using known brine samples. Conductivity cell #1 was used for determining product water NaCl concentration and conductivity cell #2 was used for determining brine concentrations. The calibration curve for

cell #1 showing a temperature dependence is shown in Figure 19, page 75.

Casting solution viscosities were determined using a falling ball viscometer, which was calibrated using glycerin at 0°C and 25°C. The viscometer could be used for only the more dilute casting solutions such as those used for dip casting. The accuracy of the viscometer was somewhat limited when using any of the casting solutions, but it did provide a good approximation for the viscosities of the various casting solutions.

III. CASTING MEMBRANES ON POROUS TUBES AND RODS

The use of porous tubes or rods as direct supports for reverse osmosis desalination membranes necessitates careful consideration of the membrane casting processes involved. Because the life of cellulose acetate membranes is relatively short (approximately 6 months to 1 year (17)), it is necessary that membranes be replaced periodically. Where direct membrane supports are used, two alternatives are open at the time of membrane replacement. Either the support may be discarded or the support may be reused. Expensive supports must be reused if they are to be justified economically. Cheap supports could possibly be discarded after use.

In consideration of the above alternatives, two casting techniques have been proposed by the author. Where supports must be reused, dip casting (at least for casting membranes on used supports) is suggested. A two-step process would be necessary for reusing supports. First, the old membrane must be removed by dissolution and then a new membrane would be dip cast upon the support. In the case where the porous media may be discarded or as a first membrane upon the porous media, extrusion is perhaps the best casting technique.

The more important casting variables for both dip casting and extrusion have been studied. These variables included casting solution composition, solvent evaporating time, and heat treatment temperature.

Other factors such as support type, support diameter, support perme-
ability, support length, and membrane life have also been considered.

A. Dilute Solutions on Flat Supports

The casting solutions used by Wang (24) and Lai (10) for casting
on flat supports are much too viscous for dip casting. Only a thick
"glob" occurs when a support is dipped into this solution. This neces-
sitated the development of a more dilute, less viscous casting solution.

For a number of membranes, Wang (24) used more dilute casting
solutions. He obtained less viscous casting solutions by reducing the
cellulose acetate content while maintaining the same formamide:acetone
ratio. Membranes cast on Gelman 0.9 micron Versapor porous media using
these dilute solutions gave poorer results. Lai (10) found similar
results when using other porous media. In both cases, the dilute, lower
viscosity casting solutions had a high formamide:cellulose acetate ratio.
This perhaps caused the poorer membrane performance. By maintaining a
constant cellulose acetate:formamide ratio and lowering the solution's
viscosity by increasing the acetone concentration, one may possibly ob-
tain membranes of good performance from dilute casting solutions. A
series of membranes cast on flat 0.9 micron Versapor porous material were
prepared from a dilute solution to check this approach.

First, the casting technique of Wang (24) was checked and his re-
sults using a viscous casting solution reproduced (see Runs V-5 to V-11,

Table XXIII, page 103). This casting solution (Casting Solution A)[*]
contained 21.9% Eastman 400-25 cellulose acetate, 31.2% formamide, and
46.9% acetone. Spread Casting (see Figure 2, page 13) using a 5-second
solvent evaporating time and an 85°C heat treatment temperature was used.
A less viscous (thin enough to be dip cast) casting solution (Casting
Solution B) was then prepared using 14.9% Eastman 400-25 cellulose ace-
tate, 21.3% formamide, and 63.8% acetone. This casting solution's vis-
cosity was approximately 3150 centipoise.

Preliminary membranes indicated that an increased clearance was
needed when using Casting Solution B. Increasing the casting clearance
to .010 inches from .005 inches eliminated imperfections in the mem-
branes and thus improved salt rejection. The imperfections were due to
flow of the casting solution into the pores of the support and to the
slight roughness of the support.

Because of the increased acetone (solvent) concentration, the
solvent evaporating time was thought to be a most important variable
when using dilute solutions. A series of membranes were prepared from
Casting Solution B on 0.9 micron Versapor supports using several differ-
ent solvent evaporating times: 5, 10, 20, 30, and 60 seconds. A heat
treatment temperature of 86°C was used in annealing the membranes.

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[*]All casting solutions used are designated by an alphabetic letter.
See Table VI, page 76, for a complete list of the compositions and
the viscosities of all casting solutions.

Figure 3 on the following page shows the performance[*] of these membranes as a function of the solvent evaporating time. Water flux decreased and salt rejection increased as the solvent evaporating time increased. A solvent evaporating time of 20 to 30 seconds produced membranes with fluxes of between 20 and 24 GSFD at salt rejections of 91 to 93%.

Using a 20-second solvent evaporating time, membranes were prepared at a higher heat treatment temperature of 88°C. This higher heat treatment temperature improved the salt rejection to 95.9% with the water flux declining slightly, but still remaining at 20.3 GSFD. This compares favorably with the flux of 19.5 GSFD and 95% salt rejection obtained for the best membranes cast on the Versapor support using the more viscous Casting Solution A.

Results indicate that using a more dilute casting solution as required for dip casting is practical; however, an increased casting thickness, a slightly longer solvent evaporating time, and a slightly higher heat treatment temperature were required to obtain good membrane performance on flat supports. Also, a less viscous casting solution which produces membranes of good quality can be obtained by increasing the acetone concentration while maintaining the cellulose acetate:formamide ratio constant at 7:10.

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[*] Each data point reported on the figures is an average for two or more membranes. Tables in the appendix provide details for each data point.

Figure 3. Performance of Membranes Cast on Versapor Using Dilute Casting Solutions. (See Table VIII, page   for data details.

B. Studies on Dip Casting

Porous ceramic rods (Norton RA-4021 alundum rods) 5/16" in diameter were used to study the more important casting variables for dip casting. The ceramic support was chosen because it is relatively strong, is dimensionally stable, is easy to work with, and it can be reused without adverse effect.

Effect of Casting Solution Composition

Of particular importance in dip casting are the casting solution viscosity and the solvent evaporating time. Several casting solutions were prepared using Eastman 400-25 cellulose acetate and three different acetone concentrations (thus different viscosities). These casting solutions, designated B, C, and D, had acetone concentrations ranging from 63.8% to 70.2% and viscosities ranging from 3150 to 995 centipoise.

Preliminary membranes on ceramic supports indicated that when dip casting on porous rods, solvent evaporating times of 5 to 15 seconds were sufficient to produce good quality membranes. Better air circulation and possible convection effects around the vertical cylindrical rod perhaps enhanced the solvent evaporation rate and made possible smaller solvent evaporating times when compared to flat supports.

A number of membranes were dip cast using casting solutions B, C, and D. A heat treatment temperature of 86°C and solvent evaporating

times of 5, 10, and 15 seconds were used. Figure 4 shows in detail the effect of the casting solutions composition (viscosity) on membrane performance for various solvent evaporating times. As the acetone concentration increases, the water flux increases while the salt rejection decreases. For longer solvent evaporating times, one may use slightly less viscous casting solutions to obtain the same or slightly better performance.

Those membranes prepared using the lower acetone concentrations (Casting Solution B, for example) were relatively thick; 1/64" to 1/32" as cast. Those of high acetone concentration were quite thin. When using dilute casting solutions, there is a point of maximum dilution at which the membrane surface begins flowing and cracking during the solvent evaporating time. New membrane of zero solvent evaporating time is thus always present. These areas have essentially a porous surface with no actual membrane present. The rapid deterioration of salt rejection (as in Figure 4) defines the beginning of the flow structure.

The point of maximum dilution is 65 ± .5% acetone when using E400-25 cellulose acetate, a cellulose acetate:formamide ratio of 7:10, and a solvent evaporating time of 5 seconds. This may be increased slightly to 67-68% acetone by increasing the solvent evaporating time to 10-15 seconds.

Figure 4. Effect of Casting Solution Composition on the Performance of Dip Cast Membranes. (See Table IX, page 79 for data details.)

Effect of Solvent Evaporating Time

The importance of the solvent evaporating time when using dilute solutions is shown by plotting membrane performance (water flux and salt rejection) as a function of the solvent evaporating time for two different casting solution compositions. This has been done in Figure 5. For Casting Solution B, the more viscous of the casting solutions, good performance (10.5 GSFD and 95.4% salt rejection) is obtained using a 5-second solvent evaporating time; however, for Casting Solution C, a 5-second solvent evaporating time produced membranes having poor salt rejection (< 65%). Increasing the solvent evaporating time to 10 seconds improved the salt rejection of these membranes to 90.5%.

For all solvent evaporating times, the membranes prepared from the more dilute solutions gave better water fluxes, but lower salt rejection. For example, at a 10-second solvent evaporating time and 86°C heat treatment temperature, Casting Solution C gave membranes having performance of 13.7 GSFD at 90.5% salt rejection while Casting Solution B produced membranes with 8.6 GSFD and 92.7% salt rejection. A similar comparison could be made at other solvent evaporating times. All membrane performances given (here and throughout the thesis) are an average performance for two or more membranes similarly prepared.

Figure 5. Effect of Solvent Evaporating Time on
the Performance of Dip Cast Membranes
When Using Different Casting Solutions.
(See Table X, page 81 for data details.)

Effect of Heat Treatment Temperature

When using membranes cast directly on porous supports, it was necessary to alter the heat treatment procedure. If the membrane-coated support was dipped directly into the hot heat treatment bath, bubbles caused by trapped air and vaporized acetone would form between the membrane and the support. These often caused the membrane to rupture. A gradual increase in heat treatment temperature was necessary to allow gradual release of the pressures within the support.

The effect of heat treatment temperature on dip-cast membranes is shown in Figure 6, page 30. Water flux declines and salt rejection increases as the heat treatment temperature increases. The slight rise in water flux at 86°C heat treatment (noted in much of the data) may be due to imperfections which arise at higher heat treatment temperatures. It is thought that the membrane shrinkage during annealing may cause cracks or other similar imperfections.

Comparison of Casting Solutions B and C over the range of heat treatment temperatures again shows that Casting Solution C, the more dilute of the two, produces membranes of better flux but slightly lower salt rejection. Best salt rejection for both casting solutions resulted when heat treatment temperatures of greater than 82°C were used.

Figure 6.   Effect of Heat Treatment Temperature on the Perform-
ance of Dip Cast Membranes. (See Table X, page 81
for data details.)

Effect of Cellulose Acetate Type

The viscosity of the casting solution can also be altered by using other cellulose acetate types. Several types of cellulose acetates are available. These differ only slightly in acetyl content, but they do vary widely in molecular weight, thus viscosity. Three types of cellulose acetate were chosen to check the effect of using other cellulose acetates. The three chosen were Eastman 398-3,[*] Eastman 400-25, and Eastman 394-60.

A number of casting solutions were prepared using these cellulose acetates. The lower the viscosity of the cellulose acetate, the less acetone it takes to achieve a desired casting solution viscosity. Table VI, page 76, shows the composition of the various casting solutions and their viscosities.

A number of membranes were prepared using three different casting solutions (viscosities) for each cellulose acetate type. Performance of these membranes is presented in Figure 7, page 32. For each type of cellulose acetate, there is a point of maximum dilution at which membrane performance begins to decline. The point of maximum dilution is 59 ± .5% acetone for 398-3 cellulose acetate, 65 ± 5% acetone for 400-25 cellulose acetate, and 68 ± 5% acetone for 394-60 cellulose acetate.

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[*]Acetyl content 39.8%, mean viscosity 3 sec -- range of 1.8 to 3.9 sec.

Figure 7. Effect of Cellulose Acetate on the Performance
of Dip Cast Membranes.

In all cases, the remainder of the casting solution consisted of cellulose acetate and formamide in the ratio of 7 to 10. The viscosity at the point of maximum dilution was approximately 900 centipoise. In all cases a flow structure in the membrane was noted for solutions with acetone concentrations higher than that at the point of maximum dilution. The flow structure was noticeable as cracks and lengthwise, wavy streaks in the membrane surface.

From the curves in Figure 7, an acetone concentration range has been established for dip casting for each of the cellulose acetate types tested. The lowest acetone concentration (most viscous casting solution) is determined by the point at which the membranes become thick and start to "glob" during casting. The maximum acetone concentration (least viscous casting solution) is determined by the point of maximum dilution discussed above. Acetone concentrations of between 54% and 59% for E398-3, between 60% and 65% for E400-25, and between 63% and 68% for E394-60 are required for dip casting. In all cases, a cellulose acetate: formamide ratio of 7 to 10 was used. Viscosities in the dip casting range vary from 900 to 3500 centipoise. The acetone concentration ranges are for a 5-second solvent evaporating time. The percent acetone could be increased slightly when using longer solvent evaporating times.

Figure 7 also compares the performance of membranes prepared from the various cellulose acetate types. E400-25 cellulose acetate produced

membranes of better performance than did the other cellulose acetate types. For example, at the point of maximum dilution, E400-25 membranes gave fluxes of 10 GSFD at 95% salt rejection compared to fluxes of 7.5 GSFD and 6.0 GSFD for E398-3 and E394-60, respectively, at salt rejections of 93.5%.

Attempts to use very dilute casting solutions were made by using multiple dipping. In this approach, the porous ceramic rods were dipped twice (or more) into a very dilute casting solution (Casting Solution E). A long solvent evaporating time ($>$ 1 minute) was allowed between dips with the final solvent evaporating time being 30 seconds. Membrane performance in all cases was poor with a flow structure being observed on the membrane surface.

Use of Warm Casting Solutions

Heating a casting solution also reduces its viscosity and may make possible the use of more concentrated casting solutions for dip casting. It was thought that thin membranes without the flow structure could possibly be obtained using this method. Several membranes were cast using warm (46°C) casting solutions. Performance of these membranes was poor (see Runs CR-27 to CR-30, page 109). Water fluxes were always relatively low ($<$ 10 GSFD) and most membranes contained pinholes. These pinholes, when dyed and observed under a microscope, appeared to be minute bubbles. Such bubbles may have originated from gases trapped in the

surface pores of the support or from the vaporization of the acetone in the casting solution. Because of the low fluxes and the defects, the use of warm casting solutions was discontinued.

C. Studies on Extrusion Casting

A modified extrusion technique (see Figure 2, page 13) was used to study the possibilities of extruding membranes upon the porous tubes and rods. An extrusion clearance of .010" was used to produce all extruded membranes. This technique produced relatively thin uniform membranes. Again ceramic rods were used in studies of the casting variables.

Effect of Casting Solution Composition

For extrusion casting, two different casting solutions were used to check the effect of casting solution composition; especially acetone content and viscosity. Highly viscous Casting Solution A (used by Wang (24) to produce his best membranes by spread casting) and Casting Solution B were used. Membranes were prepared using a 5-second solvent evaporating time and various heat treatment temperatures. Figure 8 shows the performance of these membranes.

Water fluxes were quite similar at lower heat treatment temperatures; however, at a heat treatment temperature of 86°C, Casting Solution B gave a water flux of 14.3 GSFD compared to 8.3 GSFD for Casting Solution A. Salt rejections were similar for both casting solutions over the heat treatment range.
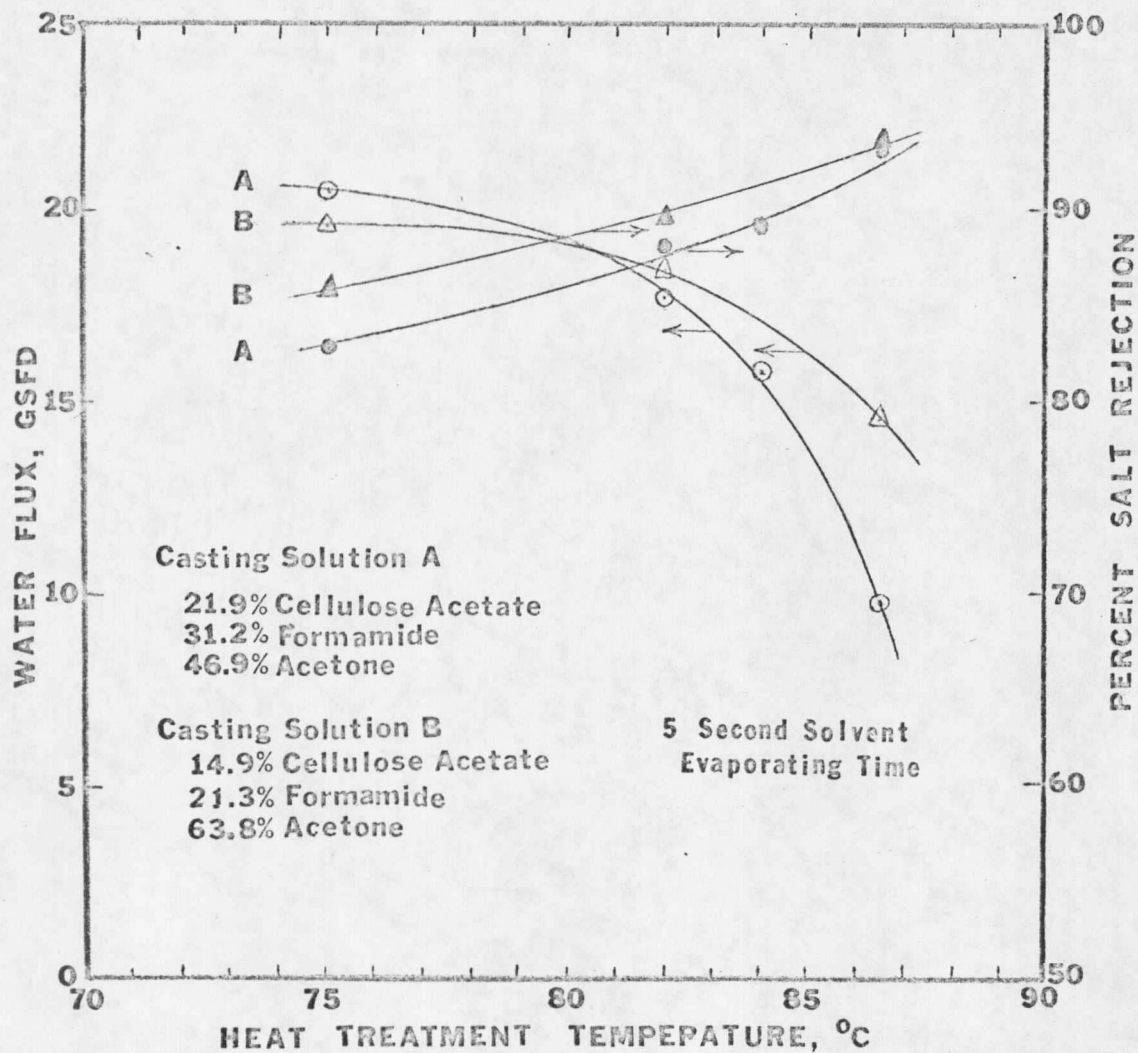
Figure 8. Effect of Casting Solution Composition on the Performance of Extruded Membranes.

Effect of Cellulose Acetate Type

The effect of cellulose acetate type on the performance of extruded membranes was studied using three different cellulose acetate types, each at three different levels of acetone concentration. A solvent evaporating time of five seconds and a heat treatment temperature of 86°C was used for all runs. Performance of these membranes is shown in Table I, page 38.

Best membrane performance (17.1 GSFD, 93.7% salt rejection) for extruded membranes was obtained when using Casting Solution K (cellulose acetate type 394-60). This casting solution was slightly below the acetone concentration range for dip casting. Use of high or low viscosity casting solutions generally resulted in poorer membrane performance. Salt rejection was similar for all cellulose acetate types.

D. Comparison of Dip and Extrusion Casting

The performances of dip cast and extruded membranes have been compared as a function of both heat treatment temperature and solvent operating time. Dip cast and extruded membranes were prepared on ceramic supports using Casting Solution B, heat treatment temperatures of 75, 82, 84, and 86°C, and solvent evaporating times of 5, 10, and 15 seconds. Results of these studies are shown in Figures 9 and 10, pages 40 and 41.

Table I: Effect of Cellulose Acetate Type on the Performance of Extruded Membranes.

Cellulose Acetate Type		High [a] Viscosity	Moderate [b] Viscosity	Low [c] Viscosity
E398-3	Casting Solution	F	G	H
	Water Flux (GSFD)	7.3	11.0	11.7
	Salt Rejection (%)	92.7	94.2	93.5
E400-25	Casting Solution	A	B	C
	Water Flux (GSFD)	8.3	14.3	9.1
	Salt Rejection (%)	91.1	93.9	91.1
E394-60	Casting Solution	J	K	M
	Water Flux (GSFD)	12.8	17.1	17.2
	Salt Rejection (%)	89.3	93.7	87.9

[a] Much above dip casting range -- ⋙ 3500 centipoise

[b] Near upper end of dip casting range -- ∼ 3500 centipoise

[c] Near lower end of dip casting range -- ∼ 1000 centipoise

Figure 9 indicates that extruded membranes have better performance than dip cast membranes for all heat treatment temperatures. At heat treatment temperatures of 82 to 86°C, fluxes of 15 to 18 GSFD and salt rejections of 90 to 94% were obtained for extruded membranes while fluxes of 10 to 11 GSFD and salt rejections of 95% were obtained for similar dip cast membranes. The lower flux of the dip cast membranes presumably results from the resistance to flow in the thick porous substructure of the membrane. Lack of imperfections results in a slightly higher salt rejection for the dip cast membranes. If a more dilute solution is used in preparing the dip cast membranes, their performance more closely approaches that of the extruded membranes. Water fluxes of 12 GSFD and salt rejections of 91.5% were obtained for dip cast membranes prepared from Casting Solution C.

Figure 10 compares the performance of dip cast and extruded membranes for various solvent evaporating times when using Casting Solution B. Salt rejections are nearly identical for membranes cast using either technique; however, water fluxes decrease rapidly when long solvent evaporating times are used for extrusion. It is thus quite important to use small (5 seconds or less) solvent evaporating times when casting membranes by extrusion. Longer solvent evaporating times (up to 15 seconds) may be used for dip casting without drastically affecting the water flux. The dependence on solvent evaporating time is primarily a result of the "as cast" membrane thickness. More sol-

Figure 9. Comparison of Dip Cast and Extruded Membranes for Various Heat Treatment Temperatures.

Figure 10. Comparison of Dip Cast and Extruded Membranes for Various Solvent Evaporating Times.

vent is available in the case of dip casting to prevent the membrane

skin from becoming thick.   Relatively thick surface films result when

extruded membranes are given longer solvent evaporating times due to

the small total amount of solvent present.

IV EVALUATION OF SUPPORT MATERIALS

A. Initial Screening of Supports

A number of materials are available in the form of porous media.
For use as a support for reverse osmosis desalination membranes, a
porous media must be of small pore size (on the order of microns or
less), must have good strength, and must be resistant to salt solutions.
For the author's work, the additional requirement of being available as
a small diameter rod or tube is necessary. Porous stainless steel,
porous ceramics, porous fiberglass, and certain porous plastics are
possibilities for membrane supports.

Five different porous support materials were obtained in the
form of small diameter porous rods or tubes. These included porous
tubular stainless steel (.5 microns pore size), two types of porous
ceramic material (unknown, but small, pore size), and two types of por-
ous plastic; polyethylene (10 micron pore size) and polyvinylidene
fluoride (25 micron pore size). Small diameter porous fiberglass
material, although of interest as supports, could not be obtained.
Table VII, page 77, provides a qualitative analysis of the various
support types used.

Ten membranes were prepared on stainless steel supports. The
performance of these membranes is shown in Table XXIV, page 106. In
all cases the product water flux was quite low. Microscopic inspection

of the support indicated that only a small portion of the support
(perhaps 10-20%) actually possessed a porous surface. The remainder
was shiny nonporous steel. This is the probable reason for the low
water flux of membranes cast on the porous stainless steel. Because of
the poor membrane performance and the high cost of the stainless steel,
the use of this support was discontinued.

Two types of ceramic materials were used; Norton RA-5021 alundum
tubes and Norton RA-4021 alundum rods. These materials were quite simi-
lar except that the RA-5021 material was fired to a higher temperature
during fabrication and thus was somewhat denser (less void space).
Both types of ceramic were brittle, necessitating care in handling.
Performance of membranes prepared on both types of ceramic supports
indicated that the RA-4021 material was at least as good as, or slightly
better than the RA-5021 material. RA-5021 ceramic, being of smaller
diameter and tubular in form, was more difficult to seal in the test
cell. Because of their similarity and the sealing problems, the use of
RA-5021 ceramic was discontinued. RA-4021 was evaluated further as a
membrane support.

Both of the porous plastic materials appeared to be promising as
supports for reverse osmosis membranes. The plastic materials are quite
strong and relatively cheap. A more complete evaluation of the porous
plastics as membrane supports was made.

B. Membrane Performance on Various Supports

Effect of Support Type on the Performance of
Dip Cast Membranes

After initial evaluation, the three most promising types of sup-
ports were compared. Using RA-5021 ceramic rods, polyethylene rods,
and polyvinylidene fluoride rods, a number of dip cast membranes were
prepared and performance evaluated to check the effect of support type.
Casting Solution B, a 5-second solvent evaporating time, and various
heat treatment temperatures were used. Results of this study are pre-
sented in Figure 11.

The ceramic rods produced membranes of better performance at all
heat treatment temperatures. For example, at a heat treatment tempera-
ture of 86°C, membranes dip cast on ceramic supports gave fluxes of
approximately 10.5 GSFD whereas similar membranes on polyethylene and
polyvinylidene fluoride gave fluxes of 7.4 GSFD and 4.5 GSFD, respec-
tively. Salt rejection was 94-95% for all support types at the above
fluxes.

The major differences between the ceramic and the plastic sup-
ports are the pore size and the wettability of the support. The ceramic
support is of unknown, but small pore size (on the order of 1 micron
as estimated from permeability data), while the pore size of the plas-
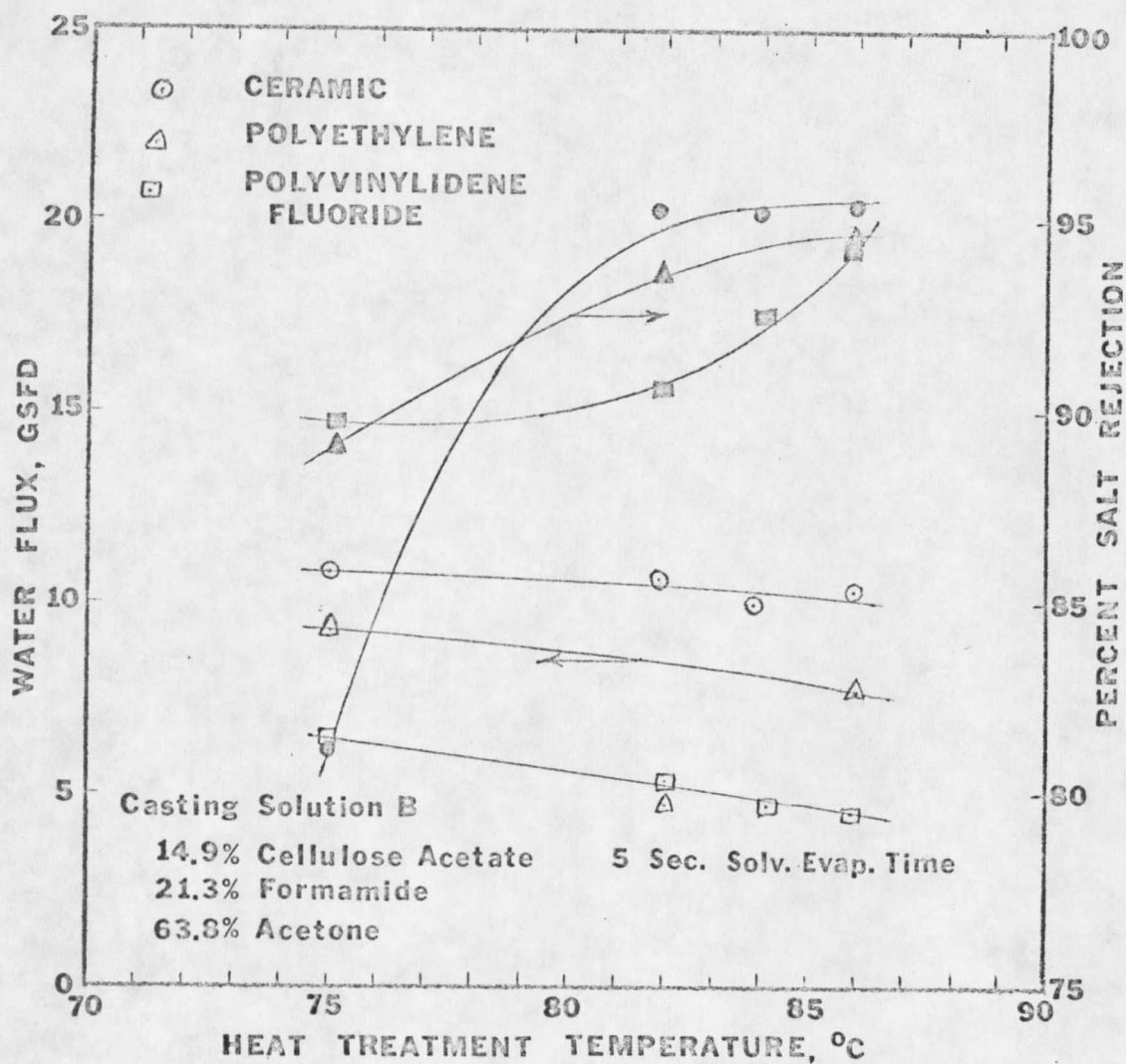tic supports is 10 and 25 microns for the polyethylene and polyvinyl-

Figure 11. Effect of Support Type on the Performance of Dip Cast Membranes.

idene fluoride, respectively. The larger pore size perhaps allowed the casting solution to flow into the pores. This would have increased the resistance to flow and reduced the water flux. The plastic supports were also hydrophobic while the ceramic supports were hydrophillic. This also may have slightly reduced the water flux. The effect of pore size and wettability are thought to reduce the water flux when using the plastic supports. It was not possible to obtain plastic supports of a smaller pore size.

Several attempts were made to increase the performance of dip cast membranes on the plastic supports by pretreating the support prior to casting. Membranes were prepared using Casting Solution G, a 5-second solvent evaporating time, and an 86°C heat treatment temperature. Table II shows the results of pretreating the support by various methods.

The effect of dip casting on wet and dry polyvinylidene fluoride supports was studied. Results (Table II) indicate that the use of water in the pores does improve water flux (by approximately 10-15%) presumably by preventing the casting solution from entering the pores of the support.

A further attempt to retard the flow of casting solution into the pores was made by coating the support with Non-Aqua stopcock grease prior to dip casting. In this case, the membrane (as cast) would not be in contact with the support. Upon operation, the water soluble stopcock

Table II: Effect of Polyvinylidene Fluoride Support Condition
on Membrane Performance.

Condition of Polyvinylidene Fluoride Support	Run No.	Water Flux (GSFD)	Percent Salt Rejection
Dry	PFR-29	4.60	94.9
	PFR-30	4.67	93.8
	PFR-37	4.82	95.5
	Avg.	4.70	94.7
Wet	PFR-31	5.08	94.9
	PFR-32	5.24	93.8
	PFR-33	5.94	97.6
	Avg.	5.42	95.4
Non-Aqua Coated	PFR-34	3.53	79.8
	PFR-35	3.14	78.9
	PFR-36	2.13	81.3
	Avg.	2.93	80.0
Detergent Washed - Wet	PFR-47	6.20	91.0
	PFR-48	5.35	97.5
	PFR-49	5.00	93.1
	Avg.	5.52	93.9

grease on the support would dissolve in the product water and be carried away. Performance of membranes prepared using this technique was poor both in respect to salt rejection (80%) and water flux (~ 3 GSFD).

A third technique was that of washing the support in detergent. This changed the support from hydrophobic to hydrophillic. This did not significantly improve results.

Effect of Cellulose Acetate Type When Dip Casting on Various Supports

The effect of using other cellulose acetate types when dip casting on each of the three support types was also studied. Casting solutions of approximately the same viscosity (upper end of the dip casting range) were used for all three cellulose acetate types. A 5-second solvent evaporating time and an 86°C heat treatment temperature were used in preparing the membranes. Table III shows the results of this study.

E400-25 cellulose acetate produced membranes of better flux on both the ceramic and the polyethylene supports. For example, on ceramic supports, E400-25 cellulose acetate gave an average flux of 10.5 GSFD compared to fluxes of 8.4 GSFD and 6.0 GSFD for E398-3 and E394-60 cellulose acetate, respectively. Similarly, on polyethylene, fluxes of 7.4, 5.4, and 6.5 GSFD were obtained for E400-25, E398-3, and E394-60 cellulose acetate, respectively. The cellulose acetate type had no

Table III: Performance of Membranes Dip Vast on Various
Supports Using Various Cellulose Acetate Types

Support Type	Cellulose Acetate Type	Casting Solution	Water Flux (GSFD)	Salt Rejection (%)
Ceramic	398-3	G	8.4	93.6
	400-25	B	10.5	95.4
	394-60	K	6.0	93.9
Polyethylene	398-3	G	5.4	96.1
	400-25	B	7.4	94.4
	394-60	K	6.5	92.3
Polyvinylidene Fluoride	398-3	G	5.4	95.4
	400-25	B	4.6	94.7
	394-60	K	5.5	92.0

large effect on the water flux of membranes dip cast on polyvinylidene fluoride supports. Also, no effect on the salt rejection could be determined.

Effect of Support Type on the Performance of Extruded Membranes

The effect of support type on the performance of extruded membranes was also studied. A series of membranes were prepared by extrusion using three types of cellulose on ceramic supports and one type of cellulose acetate (E400-25) on both the polyethylene and polyvinylidene fluoride supports. A 5-second solvent evaporating time and an 86°C heat treatment temperature were used. Table IV shows the performance of these membranes. A comparison to dip casting is also made in each case.

Membranes extruded on ceramic supports gave best performance. Water fluxes of 14.3 GSFD at 93.9% salt rejection was obtained when using Casting Solution B (E400-25 cellulose acetate). Casting Solution M (394-60 cellulose acetate) produced membranes having a water flux of 17.2 GSFD and 88.9% salt rejection. Water fluxes of 11.0 GSFD and 7.7 GSFD at ~92-3% salt rejection were obtained for the polyethylene and polyvinylidene fluoride supports, respectively, when using Casting Solution B and extruded membranes.