



Membranes for reverse osmosis desalination by direct casting on porous supports
by Donald Gong-Jong Wang

A thesis submitted to the Graduate Faculty in partial fulfillment of the requirements for the degree of DOCTOR OF PHILOSOPHY in Chemical Engineering
Montana State University
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Abstract:

Reverse osmosis stands out as the one method attracting the most world-wide attention for the desalination of saline water. It is a technically feasible process with high thermodynamic efficiency, flexibility and simplicity.

Cellulose acetate membranes have the most satisfactory desalinated water flux and most adequate desalination ability so far. Unfortunately, the hydrolysis of the acetate group in the cellulose acetate causes a short membrane life problem, This is the major problem at the current stage of development. It was believed that the membranes cast directly onto porous supports could reduce the high labor cost of membrane replacement as a shorter time and simpler procedure would be required to replace the membrane.

The purpose of this research was to investigate the membranes fabricated by direct casting onto porous supports. This is one of the most effective methods of attacking the membrane life problem indirectly by reducing the membrane replacement cost and to attack the membrane life problem directly by preventing the mechanical failures. The membranes were tested in test cells. Salt water under pressure was circulated through the cells on the upper side; product water was withdrawn on the other side. Enough circulation was maintained to reduce polarization effect.

Eighteen different kinds of porous materials were tested and two-hundred and eighty-four runs were made. Two simple practical fabrication techniques were developed. Also the process variables and the relationships of each variable as concerned with membrane fabrication by direct casting were determined. The optimum fabrication conditions for 1% NaCl feed concentration were found.

Membranes cast from dilute cellulose acetate-acetone binary solution need the support structure similar to the cellulose acetate. Under the standard test conditions of this research, membranes cast on cellulose and cellulose triacetate porous materials gave a range of water flux from 3.1 to 8.15 gal/ft² day and salt rejection from 91.8 to 82.5%. The difficulties of improving the membrane performance by this technique are discussed.

Membranes cast from acetone-formamide-cellulose acetate ternary solution on rigid porous epoxy supports showed definite promise. By using the standard test conditions of this study, the membranes gave an average water flux of 21 gal/ft² day with 95% salt rejection while certain commercial membranes under the same test conditions can only give an average water flux of 11 gal/ft² day with 95% salt rejection. Also, membranes showed an excellent reproducibility. The reasons for its high performance are discussed.

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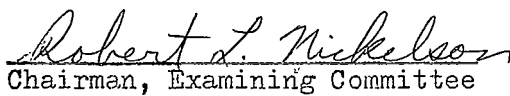
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ABSTRACT

Reverse osmosis stands out as the one method attracting the most world-wide attention for the desalination of saline water. It is a technically feasible process with high thermodynamic efficiency, flexibility and simplicity.

Cellulose acetate membranes have the most satisfactory desalinated water flux and most adequate desalination ability so far. Unfortunately, the hydrolysis of the acetate group in the cellulose acetate causes a short membrane life problem. This is the major problem at the current stage of development. It was believed that the membranes cast directly onto porous supports could reduce the high labor cost of membrane replacement as a shorter time and simpler procedure would be required to replace the membrane.

The purpose of this research was to investigate the membranes fabricated by direct casting onto porous supports. This is one of the most effective methods of attacking the membrane life problem indirectly by reducing the membrane replacement cost and to attack the membrane life problem directly by preventing the mechanical failures. The membranes were tested in test cells. Salt water under pressure was circulated through the cells on the upper side; product water was withdrawn on the other side. Enough circulation was maintained to reduce polarization effect.

Eighteen different kinds of porous materials were tested and two-hundred and eighty-four runs were made. Two simple practical fabrication techniques were developed. Also the process variables and the relationships of each variable as concerned with membrane fabrication by direct casting were determined. The optimum fabrication conditions for 1% NaCl feed concentration were found.

Membranes cast from dilute cellulose acetate-acetone binary solution need the support structure similar to the cellulose acetate. Under the standard test conditions of this research, membranes cast on cellulose and cellulose triacetate porous materials gave a range of water flux from 3.1 to 8.15 gal/ft² day and salt rejection from 91.8 to 82.5%. The difficulties of improving the membrane performance by this technique are discussed.

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INTRODUCTION

In 1952, when the United States began its formal desalting program, thermal distillation was the only one of nature's approaches which had been transformed into a well-established desalting technique. Recently, a variety of processes, such as: multistage flash distillation, long-tube vertical distillation, electrodialysis, reverse osmosis, etc., are being developed and perfected for the conversion of saline water.

Reverse osmosis is one of these processes and stands out as the one attracting the most world-wide attention among desalting enthusiasts.

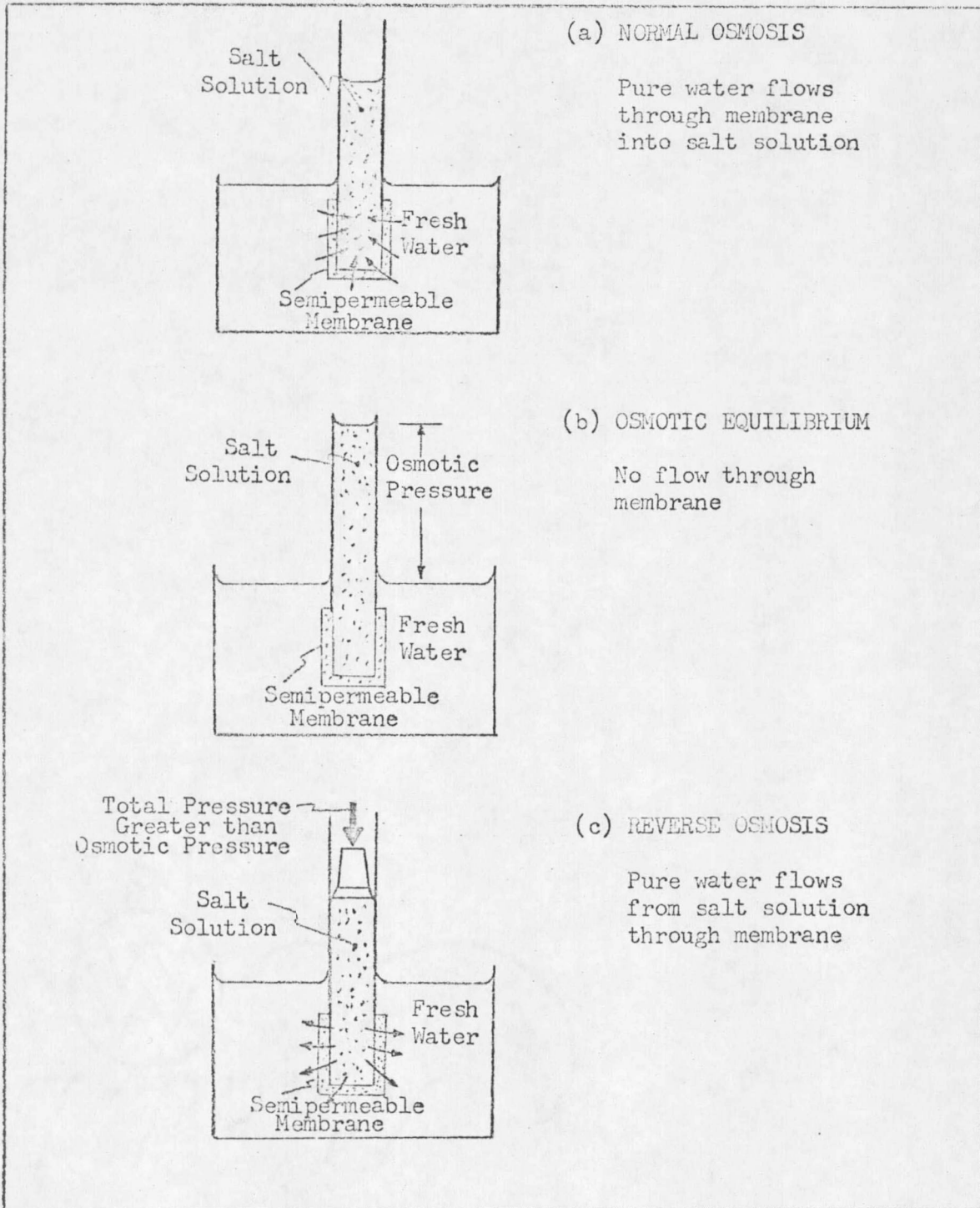
The basic principles underlying reverse osmosis have been understood for decades, and considerable work was done in the early part of this century with membranes that showed some ability to differentiate between water and dissolved salts. No effort was directed at developing reverse osmosis for the desalination of saline water until shortly after the federal desalination program was established. In early 1957 Breton⁽³⁾ reported that cellulose acetate film can be applied as a semipermeable membrane for sodium chloride solution and showed that reverse osmosis is a technically feasible process.

The principle of reverse osmosis is relatively simple. For example, when a sodium chloride solution is separated from water by a semipermeable membrane as in osmosis, water will flow through the membrane into the solution, as in Figure 1(a), until it reaches the osmotic equilibrium of that solution. At equilibrium, there is no net flow of water through the membrane, as in Figure 1(b). However if we apply an external pressure

which is greater than osmotic pressure (for example, a solution containing 1% NaCl has approximately 115 psi. osmotic pressure) the flow will be reversed, as in Figure 1(c). Since the membrane is impermeable to the salt, pure water is obtained from the saline solution. This is the principle of the reverse osmosis desalination process.

There are three factors which make this process appealing. First, in the view point of thermodynamics study, this process can be operated near the minimum work of separation. It was reported by Reid⁽¹⁶⁾ that the minimum energy requirement for producing 1000 gallons of water from sea water is only 2.63 Kw-Hr. Second, the process is not highly dependent on large scale plants to make it economical and small installations could be expected to produce water at a cost only slightly higher than large ones. Therefore, plant size is more flexible. Third, the process can be operated at ambient temperature, corrosion problems are less critical than distillation processes and insulation cost may be eliminated.

In 1957 Breton⁽³⁾ reported that cellulose acetate acts as semi-permeable membrane for sodium chloride solutions. Reid, Trautmann^(19,23) and other workers have tried a wide variety of other materials, including almost all types of existing high polymers (polystyrene, polyethylene, nylon, cellophane, cellulose acetate-butyrate,⁽⁹⁾ cellulose propionate,⁽⁸⁾ ethyl cellulose, etc.). Also many other new membrane materials are being and have been tested. Graphitic oxide membrane,⁽⁶⁾ porous glass membrane,⁽⁷⁾ and copolymer systems based on galactose methacrylate⁽²¹⁾ are examples of new membrane types. However, none of the materials tried



(a) NORMAL OSMOSIS

Pure water flows through membrane into salt solution

(b) OSMOTIC EQUILIBRIUM

No flow through membrane

(c) REVERSE OSMOSIS

Pure water flows from salt solution through membrane

Figure 1. Osmosis Phenomena

have shown as much promise as cellulose acetate.

Reid and Breton,⁽¹⁸⁾ Lonsdale, Merten and Riley,⁽¹²⁾ have shown that salt rejection increases and water flux decreases as the degree of acetylation of cellulose acetate is increased. By the proper selection of acetyl content, casting solution composition, fabrication technique, heat treatment and casting conditions, cellulose acetate membranes can be applied for the conversion of saline water.

An early hypothesis proposed by Breton⁽³⁾ stated that membrane desalination could be explained on the basis of flow through the membrane by two parallel mechanisms. Both water and salt are transported by "hole-type diffusion" with no desalination occurring. In addition, water alone is transported by "alignment-type diffusion". Sourirajan⁽²²⁾ proposed another hypothesis and explained that desalination occurs because of a thin film of pure water at the liquid-membrane interface and depends on the properties of interface. For pores with a diameter less than twice the thickness of the film, only water will flow. For larger pores both pure water and saline water will be transferred. On the basis of these experimentally based criteria, Blunk⁽²⁾ postulated the following mechanism for the passage or rejection of aqueous solutes by the membrane: "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 constant of water tend to be rejected. These species include nonhydrogen-bonding univalent ions, and all ions of valence greater than unity regardless of hydrogen-bonding characteristics."⁽²⁾

One of the serious problems with cellulose acetate membranes is the low rate of water transmission through them. Reid and Breton⁽¹⁸⁾ obtained a maximum membrane constant of 8.2×10^{-7} g/cm²·sec·atm (.945 GSFD* at 800 psi) and a salt reduction factor** of 25 (96% salt rejection***) for a membrane six microns thick, cast from acetone solution. Mahon⁽¹³⁾ attacked the low flux problem indirectly by making very fine tubes of cellulose triacetate to increase surface area per unit volume which can give a membrane constant, 5×10^{-8} g/cm²·sec·atm (.0575 GSFD at 800 psi) and a salt reduction factor between 100 and 25 (99%-95% salt rejection) for 10 microns wall thickness of the fine tubes. Based on his reported flux of 7×10^{-7} g/cm²·sec, each cubic foot would produce 200 gallons per day. Loeb and Sourirajan⁽¹¹⁾ attempted to increase the flux through the shrinking of commercially available cellulose acetate membranes in hot water. They obtained a membrane constant of 1.1×10^{-6} g/cm²·sec·atm,

* GSFD: gal/ft² day.

** Salt reduction factor: $100/(100 - \text{percent salt rejection})$.

*** Percent salt rejection: $100 \times [(\text{feed conc.} - \text{product conc.}) / \text{feed conc.}]$

(1.265 GSFD at 800 psi) and a salt reduction factor of 100 (99% salt rejection) for a membrane 100 microns thick.

As a result of experience gained with this porous cellulose acetate membrane shrinking technique, Loeb and Sourirajan initiated the development of high-flux semipermeable membranes. They encountered an article by Mlle. Dobry⁽⁵⁾ in which she suggested the use of saturated aqueous magnesium perchlorate as a solvent for cellulose acetate in the preparation of ultrafiltration membranes. They developed the first casting solutions containing electrolytes. The typical casting solution is a quaternary mixture of cellulose acetate - magnesium perchlorate - water - acetone in the proportion 22.2 - 1.1 - 10.0 - 66.7 wt %. They got the best results with a membrane constant of 4.7×10^{-6} g/cm²·sec·atm (5.4 GSFD at 800 psi and 10.2 GSFD at 1500 psi) and a salt reduction factor of 105 (99.8% salt rejection) for a membrane 250 microns thick. To obtain this performance, they found it is necessary to control rigidly the composition of the casting solution, the sequence and timing of the various steps of the membrane preparation and the temperature of the casting solution between -5°C to -10°C. "The sensitivity of the membrane to seemingly insignificant factors in its preparation is one of the most striking aspects of this problem."⁽¹⁾ Using casting solutions which contained nonelectrolytes, Manjikian, Loeb and McCutchan⁽¹⁴⁾ described a number of useful membrane casting solutions. In these, the number of components may be four, three or even two. Of the composition tested, the ternary mixture cellulose acetate-formamide-acetone, was

found to be the most useful. Membranes made from this mixture are equal to or better than those fabricated from casting solution containing electrolytes, and are simpler to produce. Today this type of cellulose acetate membrane has been increasingly developed and perfected for the conversion of both sea water and brackish water.

Unfortunately the hydrolysis of acetate group of this high polymer causes a most serious problem -- short membrane life as reported by Reid, Breton⁽¹⁸⁾ and Vos.⁽²⁴⁾ The decrease of both water flux and salt rejection as a function of operation time forces one to replace the membrane after a certain period of time and increases the over-all cost of this process. It has been reported that the labor cost of membrane replacement would be much higher than the cost of membrane itself. It is thought that by directly casting the membranes on porous supports, one can eliminate many of these problems.

The purpose of the author's research is to investigate a membrane which can cut down the high labor cost of membrane replacement and can increase the dependability, reproducibility and durability of the membrane itself. The over-all objectives of the research work are: (1) to develop the fabrication methods for membranes by direct casting; (2) to determine the process variables and the relationship of each variable as concerned with membrane fabrication by direct casting onto porous supports; and (3) to optimize the conditions of fabrication that will produce the highest quality reverse osmosis desalination membranes which can be easily handled.

The structure of high flux membranes containing electrolytes or non-electrolytes has been examined by Riley, Gardner, and Merten⁽²⁵⁾ using electron microscope techniques. They found that the membrane consists of a fine-pored matrix with a very thin dense layer (active layer) of cellulose acetate on the surface identifiable as the air-dried surface. For an original formulation of Loeb and Sourirajan⁽¹¹⁾ membrane, the dense surface layer (active layer) was estimated from the electron micrographs to be about .2 - .25 microns thick when the total membrane thickness was about 100 microns; the porous sub-structure was estimated to have a pore size on the order of .1 - .4 microns. The dense surface layer thickness would be a strong function of membrane fabrication conditions and a function of total membrane thickness. Furthermore, they indicated that the resistance to both flow of water and salt is in the dense surface layer.

The polarization effect due to concentration build-up on the membrane-brine interface for a high flux membrane has a significant effect on membrane performance. The concentration build-up in the boundary layer increases the salt flux due to the high concentration gradient and decreases the water flux because of effective applied pressure is reduced. In the case of turbulent flow, if we assume that the boundary layer is idealized as a thin film and eddy motion is negligible, a simplest model for the concentration boundary layer -- film-theory model may be applied. The film-theory model contains a number of simplifying assumptions known to be incorrect, but the effect of these assumptions upon the film-theory

predictions on the significance of the variables are rather small. For this thin film model, Brian⁽¹⁶⁾ gave the following equation to describe the salt concentration build-up at the membrane surface in terms of the permeation flux, the fluid mechanical parameter, and the Schmidt number N_{sc} for salt diffusion:

$$\frac{C_2'}{C_2^b} = \frac{\exp\left[\left(\frac{v'}{j_D \bar{U}}\right) N_{sc}^{2/3}\right]}{r + (1-r) \cdot \exp\left[\left(\frac{v'}{j_D \bar{U}}\right) N_{sc}^{2/3}\right]}$$

- where C_2' = salt concentration at membrane interface, g/cm^3
 C_2^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
 N_{sc} = Schmidt number for salt diffusion; kinematic viscosity of solution, cm^2/sec /molecular-diffusion coefficient in salt, cm^2/sec
 \bar{U} = average velocity over the cell, cm/sec
 r = salt rejection.

For high salt rejection membrane $r \approx$ unity, the above equation can be simplified to:

$$\frac{C_2'}{C_2^b} = \exp\left[\left(\frac{v'}{j_D \bar{U}}\right) N_{sc}^{2/3}\right]$$

The above equation shows that the concentration build-up in the boundary layer is a strong function of desalinated water flux and feed flow velocity, even if the bulk solution concentration is nearly unchanged. The high flux membrane with a low feed flow velocity will usually cause a serious polarization effect.

EQUIPMENT AND PROCEDURE

Test Cell

The test cell shown on Figure A1 was used for testing all reverse osmosis desalination membranes. Four identical cells were used. The test cells were made of stainless steel 304 blank flanges with 4.5" outside diameter and a 2" diameter test area. The membrane was mounted between the two halves of the cell, and salt water under pressure was circulated through the upper half. With the cone shape of the upper half surface, as shown on Figure A1, an even flow distribution across the test cell was obtained. This was studied by placing a glass plate over the test cell and injecting potassium permanganate solution into the incoming water stream. The concentration of potassium permanganate (indicated by color intensity) as it flowed across the cell showed that the flow distribution was quite good with no short-circuiting or stagnation areas.

The membrane was supported by a 1/8-inch porous stainless steel plate (Grade-H, pore size 5 microns, Pall Corp.). A 4.5" OD x 2" ID x 1/16" Neoprene rubber gasket was used between the membrane and the high pressure side of the cell. The two halves were held together by eight 5/16-inch stainless steel bolts, which were tightened stepwise to a perfect seal.

Membrane Test System and Flow Diagram.

The test system consisted of the four test cells, a pre-filter and a filter (5 microns), two parallel test lines which could be operated at the same time or independently and a plastic feed tank with a heater, stirrer, cooler and a thermo-probe connected to a temperature controller. The whole system was constructed of stainless steel and plastic to eliminate the corrosion problem. Circulation of salt water through the upper half of the test cells was provided by a stainless steel pump (Jaeco Model 753 S-8).

The flow diagram as used in typical runs with a 5 microns filter at high pressure side is shown on Figure A2. The pressure of one back pressure regulator was kept at 1200 psi and the other was kept at 800 psi. In this way, one test line merely served as a safety device. In many runs to avoid the leakage of the filter, the back pressure regulators were both kept at same pressure and the filter was connected at low pressure side of one of the two lines. The feed tank could be maintained as clean as when the filter was connected at high pressure side.

The pressure on the system was controlled by a regulator on the nitrogen cylinder and measured by a pressure gauge near the inlet of test cell. The product was withdrawn from the cell under its own pressure through a 1/4-inch Swagelok male connector and returned to the feed tank except when samples were taken. The product was collected in a graduated cylinder when sampling. After taking salt concentration measurements the product water was returned to the tank to maintain the feed concentration

constant at 10,000 ppm. sodium chloride.

In order to get meaningful data on desalination membranes, it is necessary to take account of the possible effects of liquid boundary layers on the membrane-salt water interface and to control the temperature in the test cell. A maximum feed flow rate 11.4 ml/sec was used. The average volume of the test cells was 8.3 ml., so that the feed in the cell was replaced every .73 seconds and the average feed flow velocity across the cell was 7.0 cm/sec. The temperature in the feed tank containing 10,000 ml. 1% NaCl, was controlled at $24.5 \pm .2^{\circ}\text{C}$ by an electronic temperature controller and the salt water in test cell was about $25 \pm .5^{\circ}\text{C}$. The temperature increase between tank and test cell was due to pumping and flow conditions. The operating pressure was 800 ± 35 psi in most runs unless otherwise specified.

Membrane Fabrication Equipment

A level glass surface table with the dimension of 12" x 11" was used for membrane casting. This was used to produce even membrane thicknesses. A constant temperature and humidity chamber was used for membrane casting after run No. TS-44. The chamber was constructed with a fiber glass body, a safety glass window ($10\frac{1}{2}$ " x 32") in front of the chamber, and two 6" diameter rubber plate covered working holes on the front chamber door (40" x 10"). The chamber contains lights, a heater, cooler, fan, two salt solution containers and a thermoprobe connected to an electronic temperature controller. In the most cases, the temperature

was kept at $24.5 \pm .2^{\circ}\text{C}$ by temperature controller and humidity was kept at about 50% humidity by using saturated $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ salt solution. Several 7" long and $3/8$ " diameter glass rods were used for spreading the casting solution. Vacuum insulated containers and stainless steel beakers were used for membrane heat treatments and gelations.

Membrane Test Procedure

After fabrication the membranes were immersed in distilled water overnight and allowed to equilibrate in distilled water. A membrane with a $3-1/16$ " diameter was cut from a water absorbed membrane. This was larger than the stainless porous support ($2-3/4$ " diameter) to eliminate possible leakage. The membrane was then firmly mounted in the test cell.

After the test cell was assembled, the system was filled with about 10,000 ml. of salt water. The salt water feed was made of reagent grade sodium chloride dissolved in distilled water. For most runs a 10,000 ppm sodium chloride solution was used. After the temperature of the feed reached 24.5°C in the tank, the pump was started and the pressure on the system was raised stepwise (usually 50 psi steps) at intervals of one-minute until the selected operating pressure was reached. This was usually 800 psi. At intervals thereafter the feed flow velocity was checked and adjusted to 11.4 ml/sec, the temperature and pressure were recorded, and the collected product water was analyzed. The feed concentration was also checked and maintained constant. The change in

