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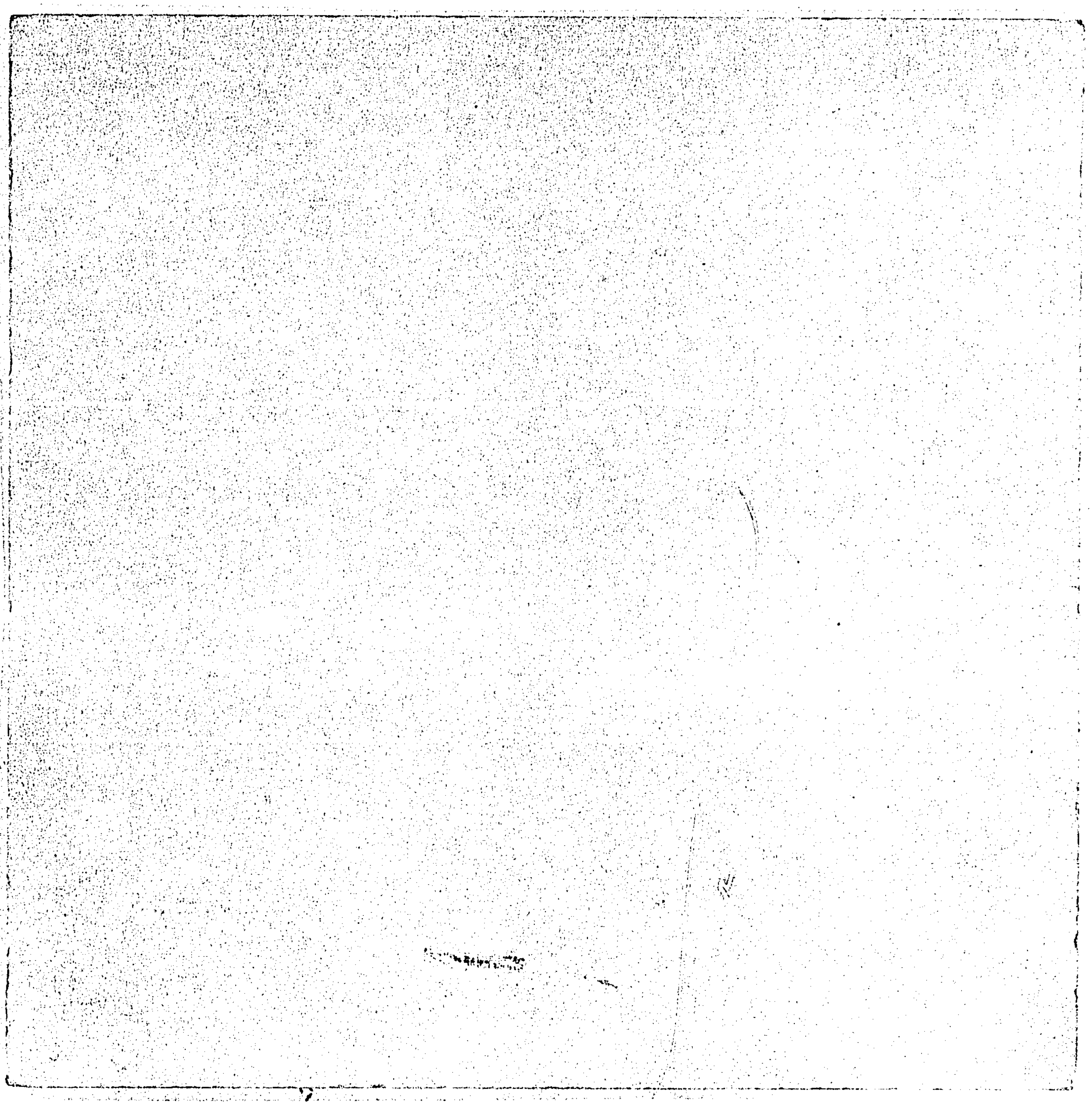

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Abstract

Illustrative experimental data are given on the performance of improved porous cellulose acetate membranes for the low pressure reverse osmosis treatment of dilute aqueous solutions of nickel chloride, nickel sulfate, nickel ammonium sulfate, and nickel plating solution. The presence of organic brightners in the plating solution did not affect membrane performance under the experimental conditions tested. The processing capacities of six membranes whose $(D_{AM}/K\delta)_{NaCl}$ values were in the range 3 to 11.2 x 10^{-5} cm./sec., for 90% water recovery, during the batchwise treatment of dilute nickel plating solution containing 1000 p.p.m. of Ni, were found to be in the range 20.8 to 35 gal./day. $ft.^2$ under an operating pressure of 300 p.s.i.g. and a feed flow rate corresponding to k_{NaCl}= 45 x 10^{-4} cm./sec.; under these operating conditions, nickel recovery in the concentrated solution was 98.9% in all cases. Data on membrane performance during a 45-day continuous test-run with a dilute nickel plating feed solution are also given.

ACKNOWLEDGMENT

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NOMENCLATURE

A = Pure water permeability constant, $\frac{\text{gr-mole } H_2O}{\text{cm}^2\text{-sec-atm}}$

$\bar{A}$ = area of film surface, sq. ft.

C = molar density of solution, gr-mole per cc.

C_1, C_2, C_3 = molar density of feed solution, concentrated boundary solution, and the product solution, respectively, gr-mole per cc.

C_{A1}, C_{A2}, C_{A3} = Solute-solvent weight ratio in the bulk solution, concentrated boundary solution, and the product solution

C_M = molar density of solution in the membrane phase, gr-mole per cc.

C_{M2}, C_{M3} = molar density of solution in the membrane phase in equilibrium with C_2 and C_3 respectively, gr-mole per cc.

D_{AB} = diffusivity of solute in water, cm^2 per sec.

D_{AM} = diffusivity of solute in membrane phase, cm^2 per sec.

$\bar{F}$ = fraction solute recovery in the batchwise concentration process.

f = solute separation

K = proportionality constant

k = mass transfer coefficient on the high pressure side of membrane, cm per sec.

l = thickness of the concentrated boundary solution.

M_B = molecular weight of water

M = solute molality

m_1 = solute molality in feed solution on the high pressure side of membrane

m_3 = solute molality in product solution

N_A = solute flux through membrane, $\frac{\text{gr-mole}}{\text{cm}^2\text{-sec}}$

N_B = solvent water flux through membrane, $\frac{\text{gr-mole}}{\text{cm}^2\text{-sec}}$

P = Operating pressure, atm.

PR = Product rate, gr. per hr. per s cm^2 of film area

PWP = Pure water permeability, gr. per hr. per s cm^2 of film area

q = product rate, gr. per day per sq. foot of film area

s = Film area, cm^2

S = function defined by equation (52)

t = time, day

$(V)i,(V)f$ = Initial and final volume of solution on the high pressure side of membrane during the batch-wise concentration process, cc.

W = Weight of solution, gr.

Ws = weight of solute in solution, gr.

Ww = weight of water in solution, gr.

X_A = mole fraction of solute

X_{A1}, X_{A2}, X_{A3} = mole fraction of solute in the bulk solution and the concentrated boundary solution on the high pressure side of the membrane, and in the product solution on the atmospheric pressure side of the membrane, respectively, at any point on the membrane surface at any time

X_{AM} = mole fraction of solute in membrane phase

X_{AM2}, X_{AM3} = values of X_{AM} in equilibrium with X_{A2} and X_{A3} respectively

Z = weight fraction of solute in the solution on the high pressure side of the membrane during the batchwise concentration process.

GREEK LETTERS

Δ = fraction recovery

δ = effective thickness of membrane, cm

Π = osmotic pressure of solution, atm

$\Pi(X_{A2}), \Pi(X_{A3})$ = osmotic pressure of solution corresponding to X_{A2} & X_{A3} respectively, atm.

ρ = density of solution, gr per cc.

ξ = normal distance towards membrane measured from edge of the concentrated boundary solution, cm

SUFFIXES

i = initial state of feed solution

f = final state of feed solution

I - INTRODUCTION

The application of reverse osmosis for the treatment of various waste waters, including plating wastes, has been illustrated (6). In the plating industry, the major pollution problem arises from the toxicity of the chemicals involved and the large volumes of water used in the rinsing operations, which also results in a significant loss of economically valuable chemicals. Therefore the treatment of rinse waters from metal finishing plants is of practical interest from the points of view of both water pollution control and waste recovery. This is particularly so with respect to nickel plating waste waters. Even in low concentrations, nickel is toxic to fish and sewage micro-organisms (4,7,8,15); the dilution of nickel plating rinse waters into the municipal sewage waters results in water pollution, loss of valuable nickel, and a reduction in the efficiency of the biological treatment processes (2,16). Application of reverse osmosis for the treatment of nickel plating waste waters can help reduce water pollution and recover the valuable nickel. Since the nickel plating solutions and the rinse waters from nickel plating baths usually have a pH between 4 and 6, these solutions are particularly amenable to reverse osmosis treatment at the ordinary room temperature using the well developed porous cellulose acetate membranes (21).

Reverse osmosis operation at low pressures is of particular interest to waste water treatment. The recent work of Kunst and Sourirajan (11, 12, 13, 14) has given rise to a new class of porous cellulose acetate membranes whose productivities are significantly high at operating pressures of 300 p.s.i.g. or less. For example, the CA-NRC-316 (or simply, Batch 316) type membranes (14) are capable of giving 91.3 per cent solute separation with

36.1 gallons of membrane permeated product per day per sq. foot of film area at 300 p.s.i.g. using an aqueous feed solution containing 3000 p.p.m. of NaCl, at a feed flow rate corresponding to a mass transfer coefficient of 45 x 10^{-4} cm./sec. on the high pressure side of the membrane; this productivity is higher than that obtainable from the CA-NRC-18 type membranes reported earlier (6) at 1000 p.s.i.g. Hence the recent work of Kunst and Sourirajan (11, 12, 13, 14) on reverse osmosis membrane science is of tremendous significance to the application of reverse osmosis for waste water treatment in general, and water pollution control in particular. The work described in this thesis illustrates the performance of the Batch 316 type porous cellulose acetate membranes for the low pressure reverse osmosis treatment of diluted nickel plating solutions.

II - COMPARISON OF SYSTEM METHODS

Currently the trend toward the salvage of materials has been evident. The reasons for this are: increased knowledge of recovery processes, savings due to metals and metal salts salvaged, improved economy in water consumption, and, of very real and practical importance, abatement of pollution.

Three methods of recovery are in general use in the metal finishing industry - evaporation, dialysis and ion exchange. A short description of these methods, their applications and limitations will be discussed. Reverse osmosis as a new separation technique and its potentials for water rejuvenation and pollution abatement is outlined.

Evaporation: (27)

Evaporation techniques are being employed by a number of electroplating plants for the concentration and recovery of plating rinse waters. Multiple rinse tanks with countercurrent flow provide an overflow liquor of reasonable concentration for charging to an evaporator. The partially evaporated or concentrated product can be returned to the plating tanks, to furnish make-up of both water and plating chemicals. Recently evaporation has been used to concentrate rinses from chromium, nickel and cyanide plating of copper, zinc and cadmium.

Vacuum evaporators are commonly used and multiple-effect evaporation is often important economically, because it needs no softening or other treatment before re-use. Glass lined equipment to limit corrosion is an extravagant necessity. In general processes involving phase changes are known to be of poor efficiency thus making them costly and for this reason prohibitive.

Dialysis: (26)

Dialysis is a membrane separation process requiring the placing of a semi-permeable membrane between a liquor and water stream. Because the membrane has properties permitting molecules to pass through it, the lower-weight molecules, which are smaller and faster, move through the membrane into the water stream. Most of the higher-weight molecules remain behind in the liquor, and the water stream carries with it a relatively pure product. Activating this process is the concentration gradient between the two streams, which acts as the driving force. As an example, a solution with metals and acids is put on one side of the membrane, the acids will pass through the membrane, leaving the metals behind, thus affecting a separation. The efficiencies of acid recovery ranges from 60% to 90% of the concentration of the contaminated waste solution. At the later range of recovery metal contamination becomes a problem thus making the process of limited applicability. Furthermore, the process is slow, of poor efficiency and requiring further processing by other means if metal recovery is to be accomplished.

Ion Exchange (26,28)

Use of ion-exchange techniques for the recovery of plating chemicals from rinses is by far the most popular and most successful method available to date. Aside from its use as a metalic cation remover it is often used for water demineralization. Here the removal of ionized dissolved solids as present in water supplies is accomplished through the exchange of ions. The exchange is a function of synthetic resinous particles which take ions from a solution passing through it and replace them with different ions. This removal is accomplished through two basic ion exchange steps:

1) Conversion of all salts to the corresponding acid by a hydrogen cation exchange resin, which exchanges hydrogen ion (H^+) for the metalic cations. After passage through this acid-regenerated resin bed, the water contains a mixture of acids corresponding in concentrations to the anions originally present.

2) Removal of acids by an alkali-regenerated anion exchange resin of the weakly basic type, which takes out the strong mineral acids such as hydrochloric, sulfuric and nitric. Generally this is sufficient for plating room requirements.

Disadvantages of this process are: operating costs, as well as equipment costs, for ion exchange operating on high concentration solutions are extremely high and, in fact, become prohibitive after a certain point (26). The down time for resin regeneration is considerable.

Although the ion-exchange technique for the recovery of such metals as copper, nickel, and chromium is wide-spread, only the large companies could justify the rather forbidding capital investment.

Reverse Osmosis (1,6,9,11,13,17,18,19)

In the last few years, growing interest has been expressed in a relatively new chemical engineering separation technique for water purification, namely, reverse osmosis. Although originally developed for the demineralization of sea and brackish waters to obtain potable supplies from their sources it is finding application in the treatment of a variety of domestic and industrial effluents for the recovery of by-products and the reclamation of water for reuse.

The heart of the reverse osmosis process is the "semipermeable membrane" a film which permits the passage of water but acts essentially as a barrier to dissolved substances. The contaminated water is pumped at an operating pressure of 300 p.s.i.g. through a chamber containing a suitably supported membrane. The water which permeates through the membrane is of high quality and is suitable for reuse, while the retentate, now in a relatively concentrated form, may be returned to a reservoir from which it can be recycled through the system until an appropriate degree of concentration has been attained. There are today a number of different types of reverse osmosis modulor units available commercially, each with its own particular advantages and disadvantages (30). All of these units are suitable for the treatment of dilute streams. Cellulose acetate membranes of the Kunst-Sourirajan (11,13) type are the most widely used, being characterized by a remarkable combination of high flux rates for water and high rejection of dissolved substances.

The method finds great promise for the removal of contaminants from plating waste streams and reuse of the purified rinse waters. An economic study (29) recently carried out with regards to the application of reverse osmosis for the recovery of nickel from contaminated rinse waters, has shown this method to be economically a highly attractive one. Credit to this, should be given to several factors: process simplicity, no phase change required, endurance of membranes, high dissolved solids rejection, limited down-time as membranes can be replaced while system is in operation, low operating pressures, high flux rates and relatively low capital investment required to set up an operating system. It is for these reasons that it is generally believed that reverse osmosis will displace the most successful and most popular method to date of water rejuvenation, the ion exchange process.

III - THEORETICAL CONSIDERATIONS

THE KIMURA - SOURIRAJAN ANALYSIS

This analysis is based on a generalised capillary - diffusion model for the transport of solute through the membrane, and is applicable for the entire possible range of solute separations in the reverse osmosis process. In this analysis, PWP is directly proportional to the operating pressure, and the proportionality constant obtained is called the pure water permeability constant, A; the transport of solvent water, N_B, through the membrane is proportional to the effective pressure where the proportionality constant is A; the transport of solute, N_A, through the membrane is due to pore diffusion through the membrane capillaries and hence proportional to the concentration difference across the membrane; and the mass transfer coefficient, k, applicable to the high pressure side of the membrane is obtained from a straightforward application of the film theory. The basic transport equations can be derived as follows:

Pure Water permeability constant, A:

$$A = \frac{\text{P.W.P.}}{M_B \times s \times 3600 \times P} \qquad (1)$$

In this analysis, A is a fundamental quantity; it is a measure of the overall porosity of the film; it corresponds to conditions of no concentration polarization; and it is independent of any solute under consideration.

Transport of solvent water, N_B, through the porous membrane:

$$N_B = A\Delta P = A\left[P - \{\pi(X_{A_2}) - \pi(X_{A_3})\}\right] \qquad (2)$$

$$= A\left[P - \Pi(X_{A_2}) + \Pi(X_{A_3})\right] \qquad (3)$$

At a given operating pressure, P, from any set of P.W.P., PR and f data, the values of A, N_B, and X_{A3} are obtained, and X_{A2} can be calculated from Equation 3; further, while A is a constant at a given P, N_B and X_{A3} are functions of feed concentration and feed flow rate (or degree of turbulence on the membrane surface). Equation 3, by itself, does not suggest the existance of any unique relationship (independent of feed concentration and feed flow rate) between X_{A2} and X_{A3}.

Equation 3 is applicable for systems where the kinematic viscocity of the product solution is not too different from that of pure water. This condition is reasonably satisfied in most cases of practical interest.

There is no easy method to determine X_{A2} experimentally. Hence, one cannot be certain that X_{A2} calculated from Equation 3 is the true value of X_{A2}. This uncertainty, however, is no limitation in this analysis so long as the correlation between the solute transport parameter and X_{A2} can be specified on the basis of Equation 3.

Transport of solute (N_A) through membrane phase:

The solute flux through the membrane is proportional to the concentration difference on either side of the membrane. Thus:

$$N_A = \frac{D_{AM}}{\delta} \cdot (C_{M2} \cdot X_{AM2} - C_{M3} \cdot X_{AM3}) \qquad (4)$$

None of the quantities on the right side of Equation 4 are known, (Fig. 1) and the dividing line in the membrane phase between the regions corresponding to X_{AM2} and X_{AM3} is only conceptual. Equation 4 can be transformed into one containing measurable quantities and a group of unknown quantities by assuming a simple linear relationship between X_A (concentration in the solution phase) and X_{AM} (concentration in the membrane phase).

Thus let

$$CX_A = K.C_M.X_{AM} \qquad (5)$$

so that

$$C_2X_{A2} = K.C_{M2}.X_{AM2} \qquad (6)$$

and

$$C_3X_{A3} = K.C_{M3}.X_{AM3} \qquad (7)$$

Equation 4 may now be written as

$$N_A = \left[\frac{D_{AM}}{K.\delta}\right](C_2X_{A2} - C_3X_{A3}) \qquad (8)$$

Since

$$X_{A_3} = \frac{N_A}{N_A + N_B} \qquad (9)$$

and hence

$$N_A = N_A.X_{A3} + N_B.X_{A3} \qquad (10)$$

$$N_A \ (1 - X_{A3}) = N_B.X_{A3} \qquad (11)$$

therefore

$$N_A = \left[\frac{X_{A3}}{1 - X_{A3}}\right] N_B \qquad (12)$$

Substitution of Equation 12 into 8, yields

$$\left[\frac{X_{A3}}{1 - X_{A3}}\right] N_B = \frac{D_{AM}}{K.\delta} \cdot (C_2X_{A2} - C_3X_{A3}) \qquad (13)$$

Rearranging Equation 13

$$N_B = \frac{D_{AM}}{K.\delta} \cdot \left[\frac{1 - X_{A3}}{X_{A3}}\right] (C_2X_{A2} - C_3X_{A3}) \qquad (14)$$

Mass transfer on the high-pressure side of the membrane:

The solute transfer from the concentrated boundary solution may be represented by the relation of Bird et al., (22)

$$N_A = X_A(N_A + N_B) - D_{AB}.C_1.\frac{dX_A}{d\xi} \qquad (15)$$

or by use of Equation 9

$$\frac{dX_A}{d\xi} - \frac{(N_A + N_B)}{C_1.D_{AB}} . X_A = - \frac{(N_A + N_B)}{C_1.D_{AB}} X_{A3} \qquad (16)$$

Where ξ represents normal distance toward membrane measured from edge of the concentrated boundary solution. Equation 15, expressing N_A as the resultant of two vector quantities, is a form of Fick's first law of diffusion. Under steady-state conditions, $(N_A + N_B)$ is the total flux through the membrane, and Equation 15 is applicable at any point in the reverse osmosis system. The boundary conditions for Equation 16 are when $\xi = 0 \quad X_A = X_{A1}$ (17)

and when $\xi = \ell \quad X_A = X_{A2}$ (18)

Solving Equation 16

$$X_{A2} = X_{A3} + (X_{A_1} - X_{A_3}) \exp\left[\frac{(N_A + N_B)}{C_1}\right] \cdot \frac{\ell}{D_{AB}} \tag{19}$$

or

$$\ln\left[\frac{X_{A2} - X_{A3}}{X_{A1} - X_{A3}}\right] = \frac{(N_A + N_B)}{C_1} \cdot \frac{\ell}{D_{AB}} \tag{20}$$

Defining the mass transfer coefficient, k, on the high-pressure side of the membrane in the conventional manner of the film theory (23, 24, 25)

$$k = \frac{D_{AB}}{\ell} \tag{21}$$

and by use of Equations 9 and 12

$$\ln . \left[\frac{X_{A2} - X_{A3}}{X_{A1} - X_{A3}}\right] = \frac{N_B}{k.C_1(1 - X_{A3})} \tag{22}$$

or

$$N_B = k.C_1(1 - X_{A3}) \;.\; \ln\left[\frac{X_{A2} - X_{A3}}{X_{A1} - X_{A3}}\right] \tag{23}$$

From the foregoing analysis, Equations 1, 3, 14, and 23 emerge as a set of basic equations describing the solute and solvent transport in a reverse osmosis process involving binary aqueous solutions and membranes having a preferential sorption for water from such aqueous solutions. For many solutions of practical interest, the molar density (C) may be assumed constant - i.e.,

$$C = C_1 = C_2 = C_3$$

so that the basic transport equations in reverse osmosis may be summarized as follows:

$$A = \frac{P.W.P.}{M.S \times 3600.P} \tag{1}$$

$$N_B = A.\left[P - \pi(X_{A2}) + \pi(X_{A3})\right] \tag{3}$$

$$N_B = C\,\frac{D_{AM}}{K.\delta}\,.\,\frac{(1 - X_{A3})}{X_{A3}}\,.\,(X_{A2} - X_{A3}) \tag{24}$$

$$N_B = k.C(1 - X_{A3})\ \ln\left[\frac{X_{A2} - X_{A3}}{X_{A1} - X_{A3}}\right] \tag{25}$$

and

$$N_A = C\,\frac{D_{AM}}{K.\delta}\,(X_{A2} - X_{A3}) \tag{26}$$

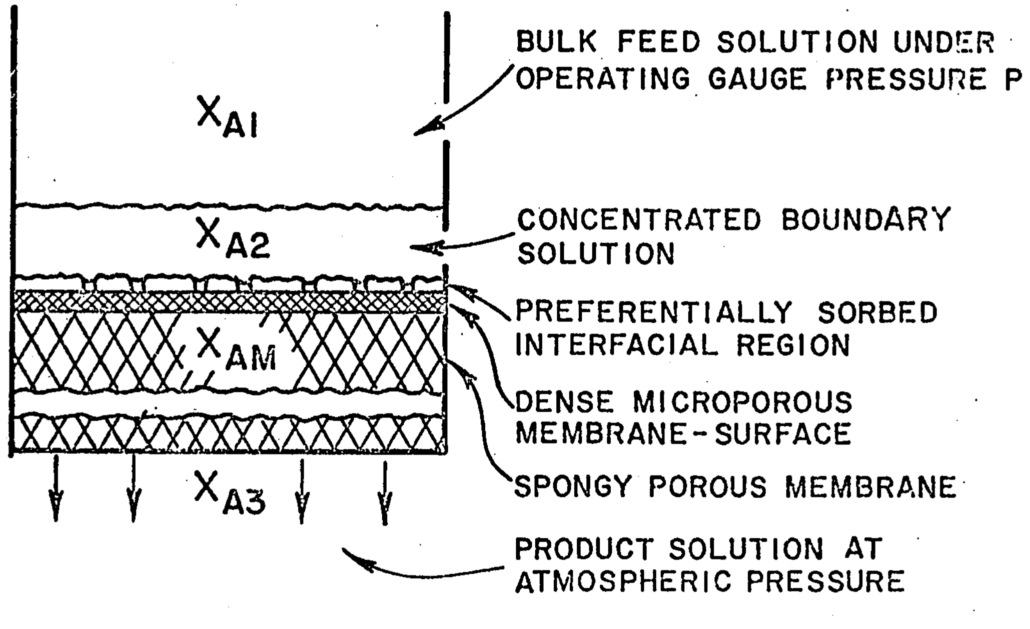


Figure 1

Reverse Osmosis Process Under Steady State Conditions.

Solute Recovery, Volume Change and Fraction Water Recovery During the Concentration Process (17)

In a batchwise reverse osmosis concentration process, it is of practical interest to know the increase in solute concentration and the weight fraction of solute recovery obtainable on the high pressure side of the membrane. This can be calculated as a function of volume fraction (Δ_w) recovery of membrane permeated water (17).

At any time, for the differential flow of water and solute transfer across the membrane,

$$C_{A1} = \frac{W_s}{W_w} \tag{27}$$

$$C_{A3} = \frac{dW_s}{dW_w} \tag{28}$$

$$f = 1 - \frac{C_{A3}}{C_{A1}} = 1 - \frac{dW_s}{dW_w} \cdot \frac{W_w}{W_s} \tag{29}$$

$$\therefore \quad (1 - f) \cdot \frac{dW_w}{W_w} = \frac{dW_s}{W_s} \tag{30}$$

When f is essentially constant at the given operating pressure for the entire concentration range involved, Equation 30 can be integrated to give

$$(1 - f) \log \frac{(W_w)_f}{(W_w)_i} = \log \frac{(W_s)_f}{(W_s)_i} \tag{31}$$

where the subscripts i and f represent respectively the initial and final state of the solution on the high pressure side of the membrane. Since

$$(C_A)_i = \frac{(W_s)_i}{(W_w)_i} \tag{32}$$

and

$$(C_A)_f = \frac{(W_s)_f}{(W_w)_f} \tag{33}$$

Dividing Equation 33 by 32, rearranging and taking logarithms of both sides, yields

$$\log \frac{(W_s)_f}{(W_s)_i} = \log \frac{(C_A)_f}{(C_A)_i} + \log \frac{(W_w)_f}{(W_w)_i} \tag{34}$$

Combining Equations 31 and 34,

$$\log \frac{(C_A)_f}{(C_A)_i} = f \log \frac{(W_w)_i}{(W_w)_f} \tag{35}$$

Let the fraction solute recovery in the concentrate ($\bar{F}$) be defined as

$$\bar{F} = (W_s)_f / (W_s)_i \tag{36}$$

Combining Equations 34 and 35

$$\log \bar{F} = (1 - \frac{1}{f}) \log \frac{(C_A)_f}{(C_A)_i} \tag{37}$$

Let V and ρ_1 represent respectively the volume and density of the solution on the high pressure side of the membrane.
From the relations

$$(V)_i = \frac{(W_w)_i + (W_s)_i}{(\rho_1)_i} \tag{38}$$

and

$$(V)_f = \frac{(W_w)_f + (W_s)_f}{(\rho_1)_f} \tag{39}$$

Dividing Equation 38 by 39

$$\frac{(V)_i}{(V)_f} = \frac{(\rho_1)_f}{(\rho_1)_i} \cdot \frac{(W_w)_i + (W_s)_i}{(W_w)_f + (W_s)_f} \tag{40}$$

The following can be written

$$1 + (C_A)_i = 1 + \frac{(W_s)_i}{(W_w)_i} = \frac{(W_w)_i + (W_s)_i}{(W_w)_i} \tag{41}$$

and

$$1 + (C_A)_f = 1 + \frac{(W_s)_f}{(W_w)_f} = \frac{(W_w)_f + (W_s)_f}{(W_w)_f} \tag{42}$$

Dividing Equation 41 by 42, rearranging and substituting into Equation 40 yields

$$\frac{(W_w)_i}{(W_w)_f} = \frac{(V)_i}{(V)_f} \cdot \frac{(\rho_1)_i}{(\rho_1)_f} \cdot \frac{1 + (C_A)_f}{1 + (C_A)_i} \tag{43}$$

Substituting Equation 43 into 35

$$\log \frac{(C_A)_f}{(C_A)_i} = f \quad \log\left[\frac{(V)_i}{(V)_f} \cdot \frac{(\rho_1)_i}{(\rho_1)_f} \cdot \frac{1 + (C_A)_f}{1 + (C_A)_i}\right] \tag{44}$$

Rearranging

$$\log \frac{(V)_i}{(V)_f} = \frac{1}{f} \quad \log \frac{(C_A)_f}{(C_A)_i} - \log\left[\frac{(\rho_1)_i}{(\rho_1)_f} \cdot \frac{1 + (C_A)_f}{1 + (C_A)_i}\right] \tag{45}$$

The fraction water recovery (Δ_w) from the initial charge can be calculated from the relation

$$\Delta_w = \frac{(V)_i - (V)_f}{(V)_i} = 1 - \frac{(V)_f}{(V)_i} \tag{46}$$

Equations 37, 45, and 46 can be used to obtain the correlations between Δ_w and $\bar{F}$ as a function of $(C_A)_f/(C_A)_i$

Processing Capacity of a Membrane (17)

The processing capacity of a membrane (= $V_1/\bar{A}t$) is defined as the volume of change (feed solution) that 1 square foot of the film can handle per day in a batch concentration process to increase the solute concentration from $(C_A)_i$ to $(C_A)_f$ on the high pressure side of the membrane at a given operating pressure. The calculation of the processing capacity for the case f = 1 can be illustrated as follows.

Let W and Z represent, respectively the total weight of solution and weight fraction of solute on the high pressure side of the membrane, and let q represent the product rate in weight units per unit area per unit time. Then

$$W.Z = \text{constant} = K = Wi.Zi \tag{47}$$

$$q = -\frac{1}{\bar{A}}\frac{dw}{dt} \tag{48}$$

$$\frac{d}{dt}(W.Z) = 0 \tag{49}$$

differentiating Equation 49 and substituting from Equations 47 and 48, we obtain

$$\int_{(Z)_i}^{(Z)_f} \frac{1}{q}\frac{dZ}{Z^2} = \frac{\bar{A}}{W_i Z_i}\int_0^t dt \tag{50}$$

or

$$\int_{(\bar{Z})_f}^{(\bar{Z})_i} \frac{1}{q}\, d\left(\frac{1}{\bar{Z}}\right) = \frac{\bar{A}.t}{W_i.Z_i} = \frac{\bar{A}.t}{K} \tag{51}$$

setting

$$\int_{(\bar{Z})_f}^{(\bar{Z})_i} \frac{1}{q}\, d\left(\frac{1}{\bar{Z}}\right) = S \tag{52}$$

$$\frac{W_i}{\bar{A}.t} = \frac{1}{(\bar{Z})_i S} \tag{53}$$

Since

$$Wi = Vi.\rho i \tag{54}$$

$$\text{Processing Capacity} = \frac{Vi}{A.t} = \frac{1}{(\rho_i).(\bar{Z})_i.(S)} \tag{55}$$

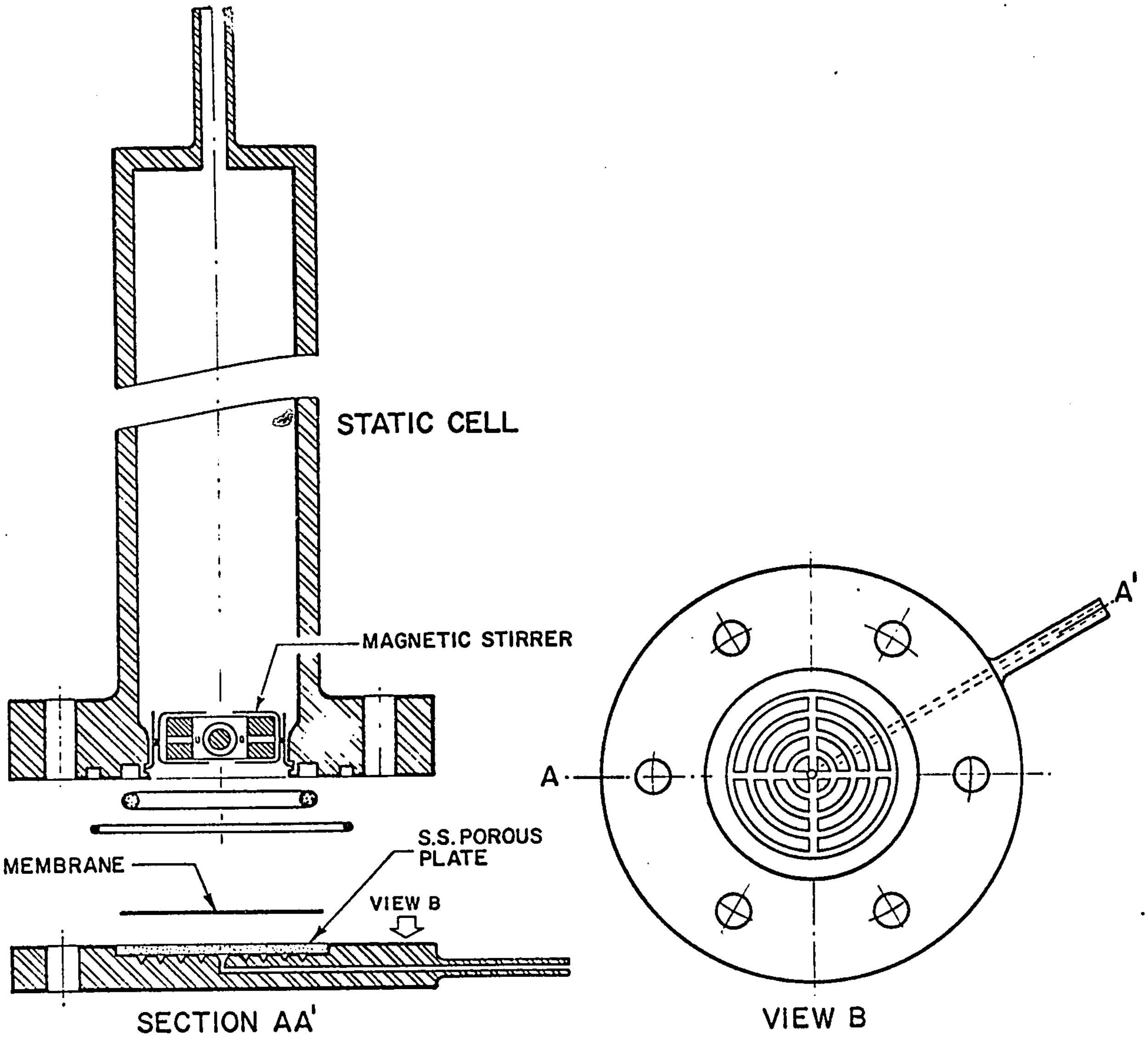
IV - EXPERIMENTAL WORK

1) The Static Reverse Osmosis Cell (18)

The static Reverse Osmosis Cell illustrated in Fig. 2 & 3 is a stainless steel pressure chamber consisting of two detachable parts. The film is mounted on a stainless steel porous plate embedded in the lower part of the cell through which the membrane permeated liquid is withdrawn at atmospheric pressure, Fig. 2 & 3. The upper part of the cell contains the feed solution under pressure in contact with the membrane. The two parts of the cell are clamped and sealed tight using rubber O-rings. Compressed nitrogen gas was used to pressurize the system. About 200 cc. of feed solution is used each time. The feed solution is kept well stirred during the experiment by means of a magnetic stirrer fitted in the cell about one quarter of an inch above the membrane surface. In most of the experiments 90% of the feed is recovered as products.

2) The Flow Type Reverse Osmosis Cell (18)

The flow type Reverse Osmosis cell used, shown in Fig. 4 & 5, was simple in design and construction and made of 310 Stainless Steel. It consisted of two detachable parts. The upper part was a high pressure chamber (Fig. 4) provided with inlet and outlet openings for the withdrawal of the membrane permeated product solution.
The outside diameter of the cell was 2 13/16 inches, and the inside diameter of the pressure chamber was 1 1/8 inches. The effective area of the membrane in the cell was 7.6 sq. cm. The wet preshrunk membrane was mounted on a stainless steel porous plate (2 inches in diameter x 1/16 inch thick) embedded in the lower part of the cell. A wet Whatman filter paper was placed between the membrane and the porous plate to protect the membrane from abrasion and also aid the flow of the product solution through the porous plate.



SECTION AA'

VIEW B

Figure 2

Static Cell

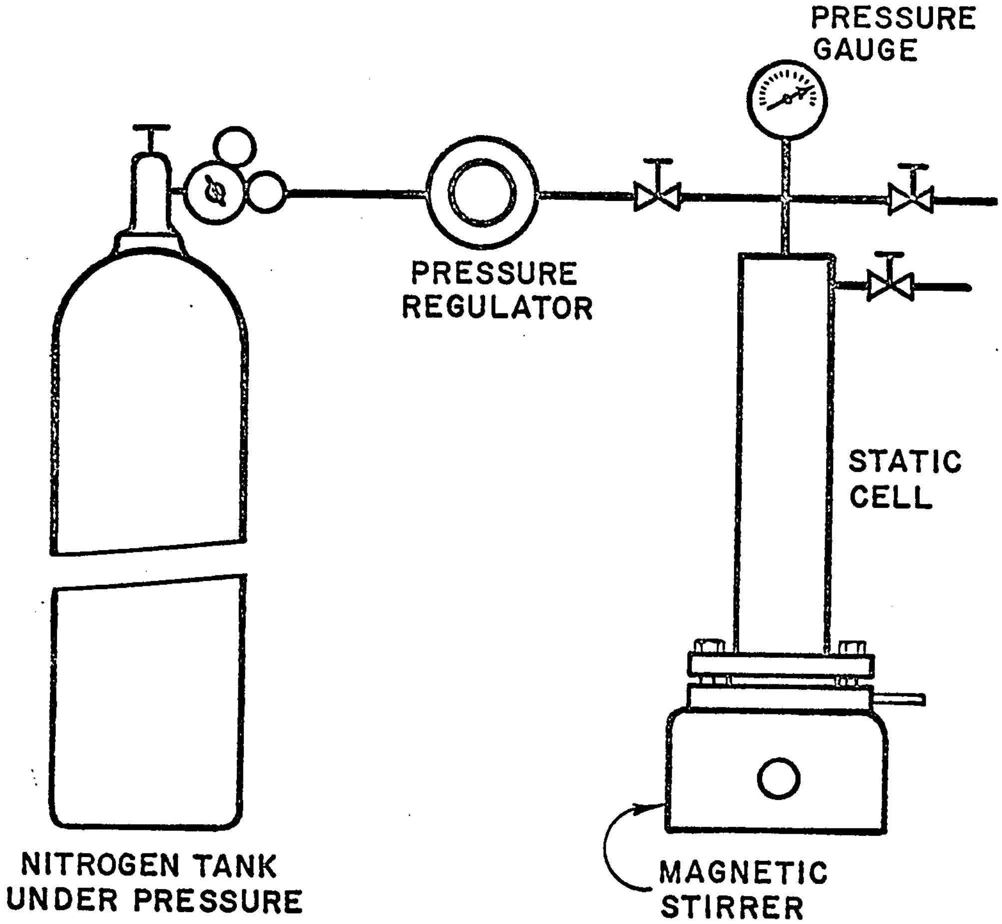


Figure 3

Static Cell Flow Diagram

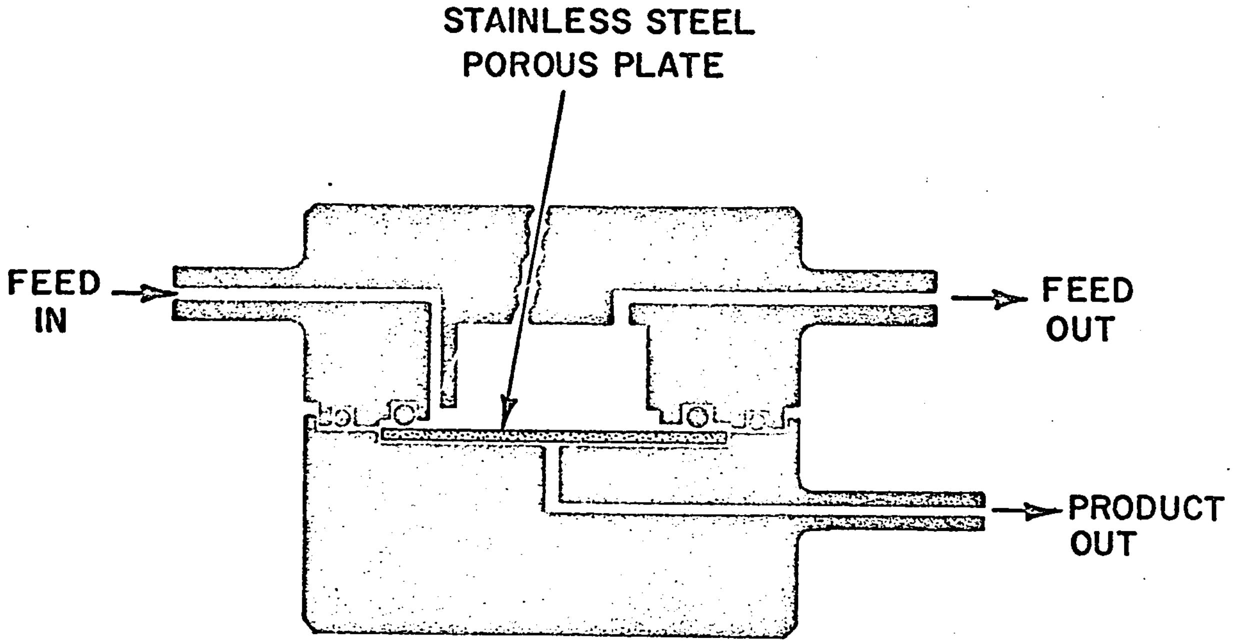


Figure 4

Flow Cell

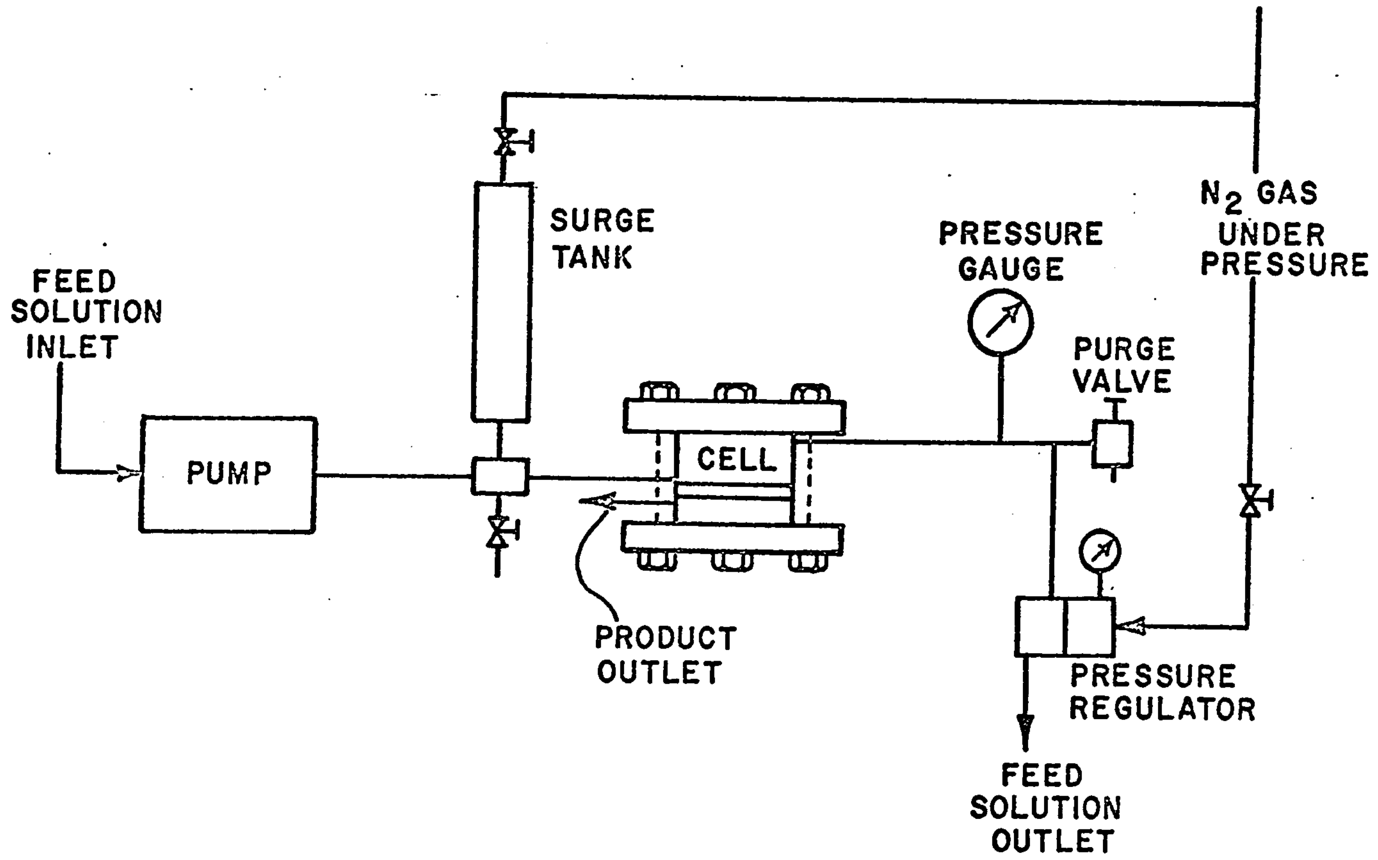


Figure 5

Flow Diagram of Apparatus for Reverse Osmosis Experiments

Under the operating conditions, the porous plate and the Whatman filter paper offered practically no resistance to fluid flow. The upper and lower parts of the cell were set in proper alignment with rubber O-ring contacts between the high pressure chamber and the wet membrane. A pressure tight joint was obtained by clamping the cell tightly between two thick end plates.

A flow diagram of the experimental arrangement is shown in Fig. 5. A motor-driven controlled-volume Milton Roy duplex pump was used to pump the feed solution under pressure through the cell. All parts of the pump coming into contact with the feed liquid were made of either Hastalloy or stainless steel. The surge tank, a stainless steel high pressure cylinder (2-inches o.d. x 1½-inches i.d. x 14 inches high), was used to minimize the pressure fluctuations in the cell. A porous stainless steel plate specified to have pores of average size equal to 5 microns was mounted between the pump and the cell to act as a filter for dust particles which might otherwise clog the pores on the membrane surface. Under the operating conditions the fluid pressure in the cell was indicated by a liquid sealed Ashcroft pressure gage. The purge valve was used to drain the system whenever necessary. A stainless steel Grove pressure regulator was used to maintain a constant operating pressure in the cell. Nitrogen gas under pressure from commercial gas cylinders was used to load the dome of the Grove regulator. Monel metal high pressure tubings and high pressure fittings made of 316 Stainless Steel were used throughout the system. For low pressure 400 P.S.I.G. ¼" copper tubing and brass Swagelock fittings were used.

Six cells were used in series (only one cell illustrated in Fig. 5) - so that six different membranes could be tested at the same time. Samples of the feed solution entering each cell could be drawn separately, if necessary. Membranes shrunk at different temperatures were used to give different level of solute separation at a given set of operating conditions. Unless otherwise stated, the experiments were of the short run type, each lasting for about 2 hours; they were carried out at the laboratory temperature; the effective area of the film used was 7.6 sq. cm., the reported product rates were those corrected to 25^{o}C. using the relative viscocity and density data for pure water.

3) Experimental Details

The nickel plating solution, and the organic additive solutions used in it, were obtained from the Canadian Hanson and Van Winkle Co., Toronto, Ontario, through the courtesy of Mr. Alan Olsen of the Udylite Corporation, Detroit, Michigan. The plating solution as received contained 101900 p.p.m. of Ni, and its pH was 4.0; when diluted 10, 100, and 1000 times with water, the pH of the solution became 5.9, 6.1 and 6.2 respectively. Most of the experiments were carried out at 300 p.s.i.g. with diluted nickel plating solutions containing 1000 p.p.m. of Ni; a few experiments were also carried out at lower pressures, at different concentrations of Ni, and also with similar dilute solutions of nickel chloride, nickel sulfate, and nickel ammonium sulfate (double nickel salt) which are commonly used in nickel plating solutions.

Two sets of experiments were carried out one using a nonflow-type cell, and the other using a flow-type cell. The apparatus employed in each case has been described in sections 1 & 2 of this chapter. All the experiments were carried out at the laboratory temperature. The film casting details are given in Table I. The membranes, preshrunk under water at different temperatures, were initially subjected to a pure water pressure of 350 p.s.i.g. for 1 to 2 hours before subsequent use in reverse osmosis experiments at operating pressures of 300 p.s.i.g. or less. In each experiment, the pure water permeation rate (PWP) and membrane permeated product rate (PR) per given area of film surface, and solute separation (f) were determined at preset operating conditions. Aqueous feed solutions containing 1500-3000 p.p.m. of sodium chloride were used to obtain data on membrane specifications and specify the mass transfer coefficient on the high pressure side of the membrane.

V - RESULTS & DISCUSSIONS

Specifications of Membranes and Feed Flow Conditions

Using the experimental (PWP), (PR), and f data at preset operating conditions for the feed system sodium chloride-water containing 1500 to 3000 p.p.m. of salt, the values of the pure water permeability constant, A, solute transport parameter, $(D_{AM}/K\delta)$, and the mass transfer coefficient, k, on the high pressure side of the membrane were calculated by the Kimura-Sourirajan analysis (9). The significance of A, $(D_{AM}/K\delta)$, and k on the performance of membranes in reverse osmosis has been extensively discussed, in the theoretical section III. The data on A and $(D_{AM}/K\delta)$ for NaCl at 300 p.s.i.g. given in Table II specify the films used in this work. Table II also gives the experimental solute separation and product rate data obtained with the films for the system 3000 p.p.m. $NaCl-H_2O$ at 300 p.s.i.g. at the feed flow rate corresponding to a mass transfer coefficient, k, of 45×10^{-4} cm./sec. These data illustrate the high productivity of the Batch 316 type membranes for reverse osmosis separation at low operating pressures compared to the performance of CA-NRC-18 type membranes reported earlier (6).

The feed flow conditions used in all the reverse osmosis experiments carried out in this work are specified in terms of the corresponding values of k obtained for the reference solution system sodium chloride-water containing 3000 p.p.m. of NaCl.

In order to compare the relative performance of different membranes for feed solution systems for which osmotic pressure and other physico-chemical data are not readily available, it is necessary to provide such specifications for membranes and feed flow conditions used, in terms of parameters easily obtainable for a convenient reference feed solution system.

Separation of Different Nickel Salts

Figure 6 illustrates the effect of pressure (100 to 300 p.s.i.g.) and concentration (100 to 1000 p.p.m. Ni) on solute separation and product rate obtained with a typical membrane preshrunk at 76°C using the feed solution systems $NiCl_2$-H_2O, $NiSO_4$-H_2O, $NiSO_4$ $(NH_4)_2SO_4$-H_2O and dilute nickel plating solutions. These experiments were carried out in the nonflow-type apparatus using 200 cc. of feed solution for each experiment in which the fraction of initial feed recovered as product was 50 to 80 per cent in the 100 p.s.i.g. runs, and 90 per cent in the other runs. The solute separation and product rate data reported are the overall average values obtained corresponding to the above levels of product recovery. With increase in product recovery, the concentration of the solution remaining on the high pressure side of the membrane, and the concentration polarization on the membrane surface increased with a consequent decrease in the instantaneous product rate, which set the limit to the product recoveries obtainable at the 100 p.s.i.g. runs. High levels of solute separations were obtained in all the experiments. The percent separation increased with operating pressure, and remained unchanged in the range of feed concentrations studied.

At a given operating pressure, the separation of nickel chloride was less than that of nickel sulfate. The solute separation in the dilute nickel plating solution was intermediate between the separations observed for the systems $NiCl_2$-H_2O and $NiSO_4$-H_2O; this is understandable since the nickel plating solution used contained both $NiCl_2$ and $NiSO_4$. The separation of the double nickel salt was essentially the same as that of $NiSO_4$.

The product rates increased with operating pressure almost linearly and they decreased with increase in feed concentration. In all experiments, it took more than six hours to collect the products corresponding to the high recoveries desired. Therefore the reported product rate data was at least partly influenced by membrane compaction effects.

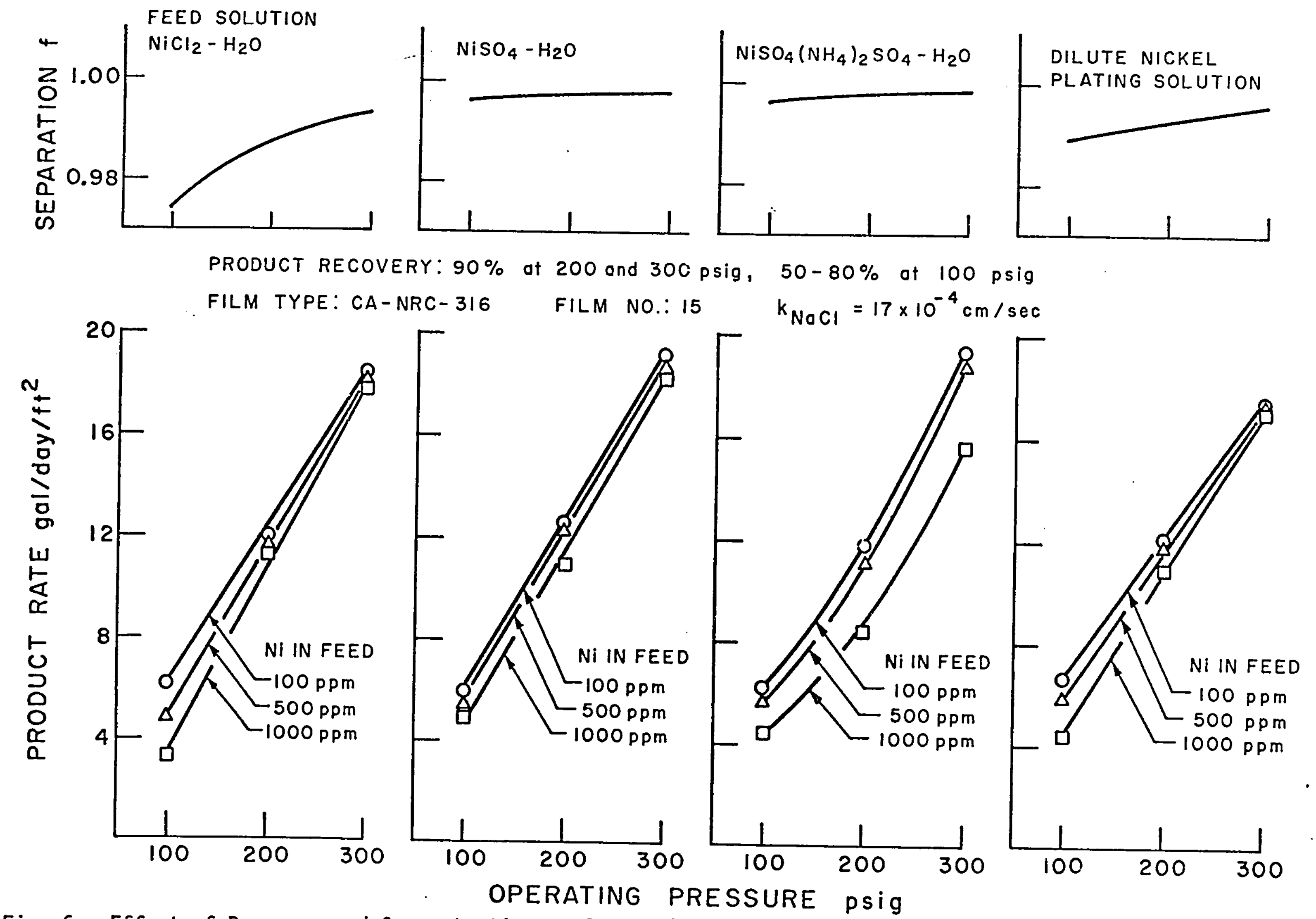


Fig. 6 - Effect of Pressure and Concentration on Separation and Product Rate for Dilute Aqueous Feed Solutions of Different Nickel Salts.

With respect to the dilute nickel plating solutions, the results showed that > 99 per cent solute separations could be obtained at pressures of 200 p.s.i.g. or above with the film used. In particular, at 300 p.s.i.g. using a feed solution containing 1000 p.p.m. Ni, a solute separation of 99.6 per cent and product rate of 17 gallons per day per sq. foot of film area were obtained corresponding to a product recovery of 90 per cent. These results are better than any reported earlier for the operating conditions employed, and they are considered significant enough for practical industrial use.

Effect of Organic Brightners on Membrane Performance

Industrial nickel plating solutions usually contain some organic brightner agents (3). These agents help to produce bright deposits over a wide range of current densities, to control stress in the deposit, and to permit wide tolerances of organic and inorganic impurities in the bath. Most nickel baths contain two classes of organic brightner solutions whose exact chemical compositions are usually kept proprietary by the companies concerned. One class includes such compounds as aryl sulfonic acids, aryl polysulfonic acids and their sodium salts, aryl sulfonamides and aryl sulfonimides, o-benzoic sulfinide or its salts, sulfonated aryl aldehydes and phenyl- or naphthyl-substituted alkyl sulfonic acids. The other class is represented by sodium formate, aldehydes and ketones, halogenated aldehydes, coumarin and derivatives, mercaptopyrimidines, amino-polyarylmethane, azo dyes, zinc and cadmium salts, thallium salts, and selenium salt (5). Brightners of the first class provide bright plate over a bright substrate and permit those of the second class to be present over an acceptably wide range of concentrations partly by reducing stress in the deposit. Brightners of the second class are used to build mirror-like luster on plating, but suffer from the fact that in the absence of brightners of the first class, they lead to excessive brittleness and stress in the deposit. In most modern baths, the two classes of brightners are used to complement each other. Hence the effect of brightners on the performance of membranes is of practical interest from the point of view of the reverse osmosis treatment of industrial nickel plating waste waters.

The effect of the Canadian Hanson nickel brightners, Numbers 61 and 63, present in their nickel plating solution, on the performance of the reverse osmosis membranes was investigated as follows. Their nickel plating solution had the following composition:

$NiSO_4.6\ 1/2\ H_2O$	:	48.2 oz/gal
$NiCl_2.6H_2O$		12.2 oz/gal
Boric Acid	:	6.0 oz/gal
Ni Brightner No. 61	:	1.1 % by volume
Ni Brightner No. 63	:	2.1 % by volume

The total amount of brightner solutions (3.2% by volume) as given in the above composition was designated as unit relative volume of brightner solution in feed. A stock nickel solution of composition given above minus the two brightner additives was prepared.

To 50 cc. aliquot of this stock solution, appropriate amounts of the two brightner solutions, No. 61 and 63, were added so that the relative volume of the brightner was 0.25, 0.5, 1, 2, and 4 times respectively of the above unit volume. Each aliquot was then diluted to obtain a feed solution containing 1000 p.p.m. Ni. This feed solution was then used in the reverse osmosis experiments at 300 p.s.i.g. in the non-flow type cell, and the solute separation and product rate obtained corresponding to 90% recovery were then determined. The results (Figure 7) showed that the presence of the brightner in the feed solution in the entire concentration range studied, had no effect on the performance of the membrane.

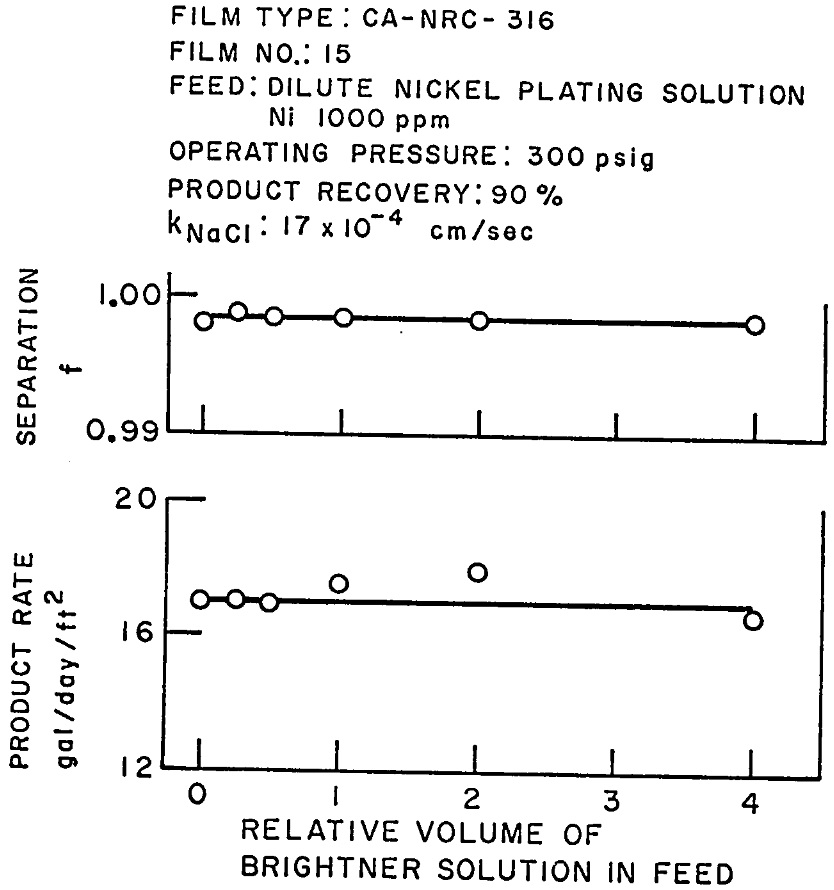


Figure 7

Effect of Organic Brightners in the Dilute Nickel Plating Feed Solution on Membrane Performance

Effect of Nickel Concentration in Feed on Membrane Performance

The experiments described below were carried out with dilute nickel plating solutions in the flow-type cells with which higher mass transfer coefficients were obtained on the high pressure side of the membrane. The values of K_{NaCl} obtained with the non-flow type cell used in the experiments described above were 17×10^{-4} cm./sec. compared to 45×10^{-4} cm./sec. obtained with the flow-type cells under the experimental conditions used.

One of the important characteristics of the Batch 316 type membranes is that they have initially a far larger number of smaller size pores on their surface layer compared to the CA-NRC-18 type membranes tested earlier (11, 14). Therefore it is not necessary to use high temperatures for film shrinkage in order to obtain a given average pore size. Further, since bivalent salts, such as those of nickel, are better separated than the monovalent salts, such as sodium chloride, (18) the average pore size needed to yield a given level of solute separation is higher for nickel salts than that needed for sodium chloride. In order to set the limits on membrane pore size needed for the particular application under study, the performance of several Batch 316 type membranes shrunk at temperatures below 76°C. were investigated for the separation of nickel from different concentrations of dilute nickel plating solutions.
The specifications of the membranes used, and a set of data on their performance with respect to the reference system $NaCl-H_2O$ are given in Table II. The films 12, 7, 11, 9, 6 and 5 were shrunk respectively at 65, 68, 72, 74, 75 and 75°C and subjected to a pure water pressure of 350 p.s.i.g. for 1 to 2 hours before their subsequent use in reverse osmosis experiments at 300 p.s.i.g.; the data on their $(D_{AM}/K\delta)_{NaCl}$ values and productivities (Table II) show that their average pore sizes decreased progressively in the given order.

The nickel concentration in the feed was varied from 100 to 12000 p.p.m. and the experiments were of the short-run type each lasting for about 2 hours or less. The solute separations and product rates obtained with the different films used are plotted in Figure 8. The results showed that the level of solute separation obtained was in the range 99.5 to > 99.9 with the films used, and the per cent solute separation remained essentially constant in the entire range of nickel concentrations studied. The high level (> 99.5%) of solute separation obtained with each one of the films tested shows that membrane pore sizes represented by $(D_{AM}/K\delta)_{NaCl}$ values up to 11.2×10^{-5} cm./sec. are appropriate for use in the treatment of dilute nickel plating waste waters under the experimental conditions used in this work. The product rates obtained are naturally highest with Film 12 and progressively less with membranes shrunk at higher temperatures.

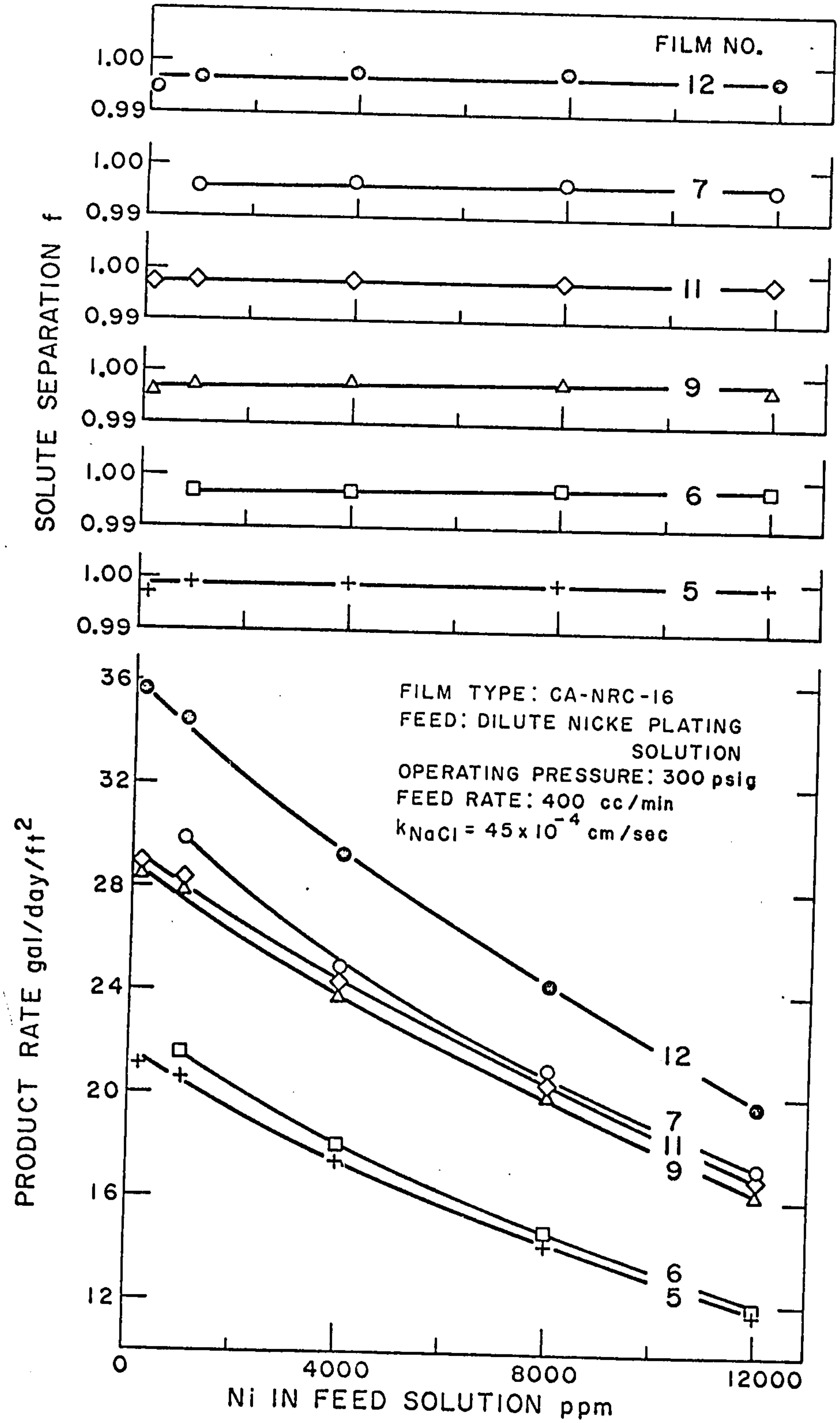


Fig. 8 - Effect of Nickel Concentration in the Dilute Nickel Plating Feed Solution on Membrane Performance.

Nickel Recovery and Processing Capacity of Membrane (17)

In a batchwise reverse osmosis treatment of nickel plating rinse waters, the increase in solute concentration and the weight fraction nickel recovery obtainable on the high pressure side of the membrane can be calculated as a function of volume fraction (Δ_w) recovery of membrane permeated water as was shown in the theoretical section. Equations 37, 45 and 46 can be used to obtain the correlations between Δ_w and $\bar{F}_s$ as a function of $(C_A)_f/(C_A)_i$ for given values of f (assumed constant) without reference to any particular membrane. This is illustrated in Figure 9 for the treatment of dilute nickel plating solutions containing 1000 p.p.m. Ni. The values of f = 0.995 and 1.000 were chosen for illustration since the performance of the actual membranes used with respect to f was in the range of 0.995 and 1.000 (Figure 9). The densities (ρ_1) of the dilute nickel plating solutions used containing 1000, 2000, 4000, 6000, 8000, 10000, and 12000 p.p.m. Ni were found to be 1.000, 1.003, 1.009, 1.015, 1.021, 1.027 and 1.033 g./cc. respectively. These values were used in the above calculations. For f = 1.000 (complete solute separation), $\bar{F}_s$ = 1.000 for all values of $(C_A)_f/(C_A)_i$; for f = 0.995, $\bar{F}_s$ decreases with increase in $(C_A)_f/(C_A)_i$. The values of Δ_w changed very little for the two values of f used. For example, when $(C_A)_f/(C_A)_i = 2.002$, Δ_w equals 0.5045 for f = 0.995, and 0.5026 for f = 1.000; when $(C_A)_f/(C_A)_i = 12.134$, the corresponding values of Δ_w are 0.9203 and 0.9193 respectively.

The processing capacity of a membrane has been defined (17) as the volume of charge (feed solution) that 1 square foot of film surface can handle per day in a batch concentration process to increase the solute concentration from $(C_A)_i$ to $(C_A)_f$ on the high pressure side of the membrane at a given operating pressure. The data on processing capacities of useful membranes are of utmost practical importance in reverse osmosis process design for the treatment of waste waters and solute recovery. For the case f = 1.000, the above data can be obtained from the relations (52) and (55). Since the values of f obtained with the membranes used in this work (Figure 8) were close to unity, equations 52 and 55 can be used in conjunction with the product rate data given in Figure 8. This was done again with respect to the treatment of dilute nickel plating solutions containing 1000 p.p.m. Ni. The experimental product rate data given in Figure 8 as a function of feed concentration were replotted in the form $\frac{1}{q}$ vs. $\frac{1}{Z}$, and the values of S (Equation 52) were obtained by graphical integration corresponding to the different values of $(C_A)_f/(C_A)_i$; Equation 55 was then used to calculate the processing capacities for each one of the films tested. These results are also plotted in Figure 9. The results show, for example, that in a batchwise operation, for 50% product water recovery, the processing capacities of the films 12, 7, 11, 9, 6, and 5 are 67.3, 58.0, 55.0, 53.9, 42.3, and 40.2 gal./day ft.^{2} respectively at the operating conditions of pressure and feed flow rate specified in Figure 8; under those conditions the solute recovery obtainable is > 99.7% in all cases. For 90% product water recovery, the corresponding processing capacities are 35.0, 30.1, 28.8, 28.3, 21.7 and 20.8 gal./day ft.^{2} respectively, under which conditions, the solute recovery obtainable is > 98.9%. In both cases, the concentrated nickel plating solution and the water recovered can be reused in the plating process.

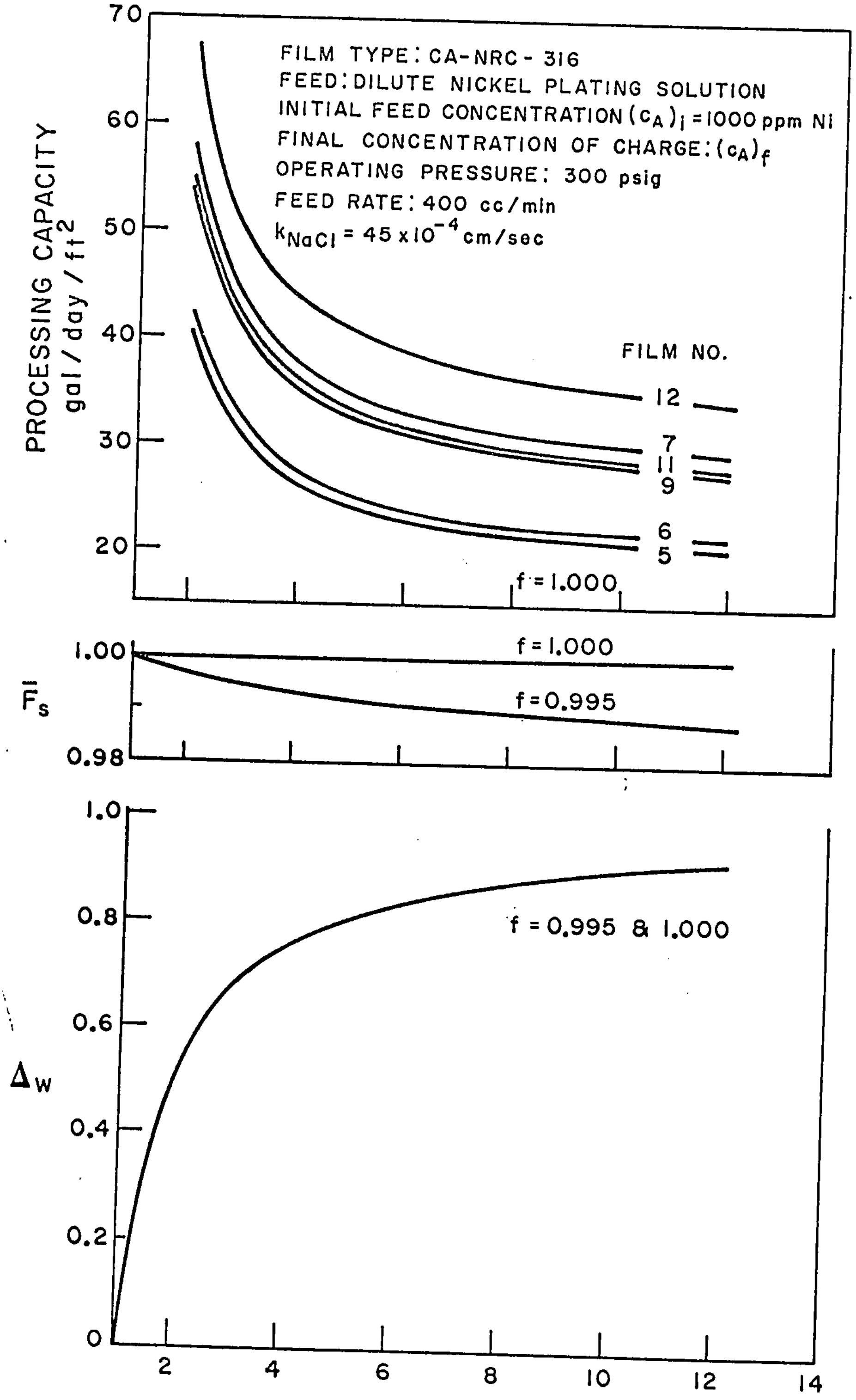


Fig. 9 - Water Recovery, Solute Recovery and Processing Capacities of Membranes for the Reverse Osmosis Treatment of Dilute Nickel Plating Feed Solutions.

Long Term Continuous Test Run Experiments

The data on processing capacities given in Figure 9 are based on product rates obtained in short-run experiments. Under conditions of continuous operation for longer periods of time, the membranes tend to compact resulting in lower product rates. The latter yields are the ones of practical interest from the point of view of process design. Hence a typical 45-day continuous test run was conducted to assess the performance of the films (Figure 8) under such conditions using dilute nickel plating solution as the feed. In this test run, the operating pressure used was 300 p.s.i.g., and the concentration of nickel in the feed solution was 1000 to 1100 p.p.m., the feed flow conditions were similar to those used in the short-run experiments. The solute separation and product rate characteristics of the films were periodically determined during the test period, and the results obtained are given in Figure 10.

There was practically no change in solute separation during the entire test period but the product rates dropped under conditions of continuous operation. Referring to the product rate data obtained with the films 12, 7, 11, 9, 6, and 5, at the end of 2 days of continuous operation the product rates were 94.3, 93.1, 93.1, 93.5, and 93.1% respectively of those obtained at the start of the run; at the end of 45 days of continuous operation, the product rates were 81.7, 87.5, 84.8, 88.5, 90.0, 91.0% respectively of those obtained at the start of the run. The effect of compaction on permeation rate is relatively more with membranes shrunk at lower temperatures. At the end of the test run it was found that there was some fine solid deposit on the membrane surfaces. This solid deposit was found to contain 56% ash whose spectrographic analysis showed the presence of Ni (30%), Fe (10%), Cu (5%), Zn (1%), Al (0.5%), Si (0.5%), Sn (0.2%), Pb (0.3%), Ti (0.1%),

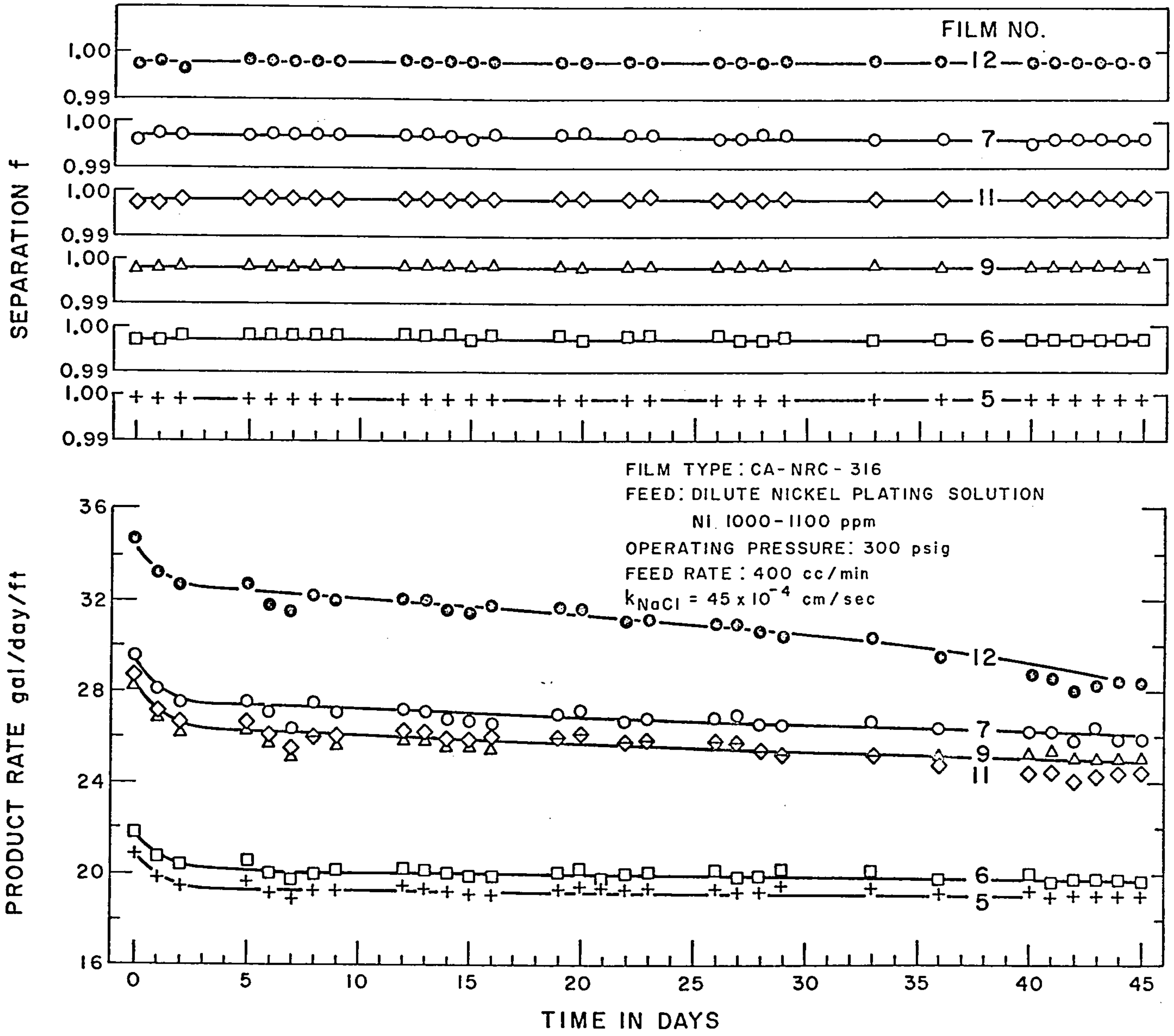


Fig. 10 - Performance of Membranes in Long Time Continuous Test Runs Using Dilute Nickel Plating Feed Solution

B (0.3%), Mg (0.02%), and Mn (0.02%). The origin of some of these metals could be the materials of construction used in the pump parts and fittings. It is possible that at least a part of the drop in product rates in the long-time run could be attributed to the above solid deposit on the membrane surfaces. In any case, on the basis of the results reported in Figure 10, it is reasonable to conclude that the practically realizable processing capacities of the membranes during continuous operation for extended periods of time would be 80 to 90% of those given in Figure 9. Even under these conditions, the performance of the membranes tested seems significant enough for consideration for practical industrial use.

VI - CONCLUSIONS

Nickel plating rinse waters are particularly well suited for the reverse osmosis treatment as the pH of such solutions is in a range where the hydrolysis effect on the cellulose acetate membrane is at a minimum. No pretreatment of the plating rinse waters is thus necessary and, barring physical defects, the membrane is assured longevity.

It has been amply demonstrated that rinse waters from nickel plating operations can be subjected to the reverse osmosis treatment, and 98.9 per cent of the nickel contents successfully recovered. The excellent separation and concentration abilities of the reverse osmosis process make it a highly attractive proposition from the point of view of water pollution control, metal reclamation and water conservation. Using the improved porous cellulose acetate membranes recently developed by Kunst and Sourirajan, this waste treatment process can be carried out successfully at low operating pressures.

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VIII - APPENDIX I

Table I. - Film Casting Details

Film Type: CA-NRC-316 (or simply, Batch 316)

Casting solution composition, weight %

Cellulose acetate (Eastman 398-3)	17.00
Acetone	69.20
Magnesium Perchlorate	1.45
Water	12.35

Temperature of casting solution: 0°C

Temperature of casting atmosphere: 23-24°C

Casting atmosphere: ambient air in contact with aqueous solution of 30 wt. % acetone

Solvent evaporation time: 6 minutes

Duration of film-setting in ice cold water: > 1 hr.

Nominal film thickness: 0.005 inch.

Table II. - Membrane Specifications and Performance Data

Film Type: CA-NRC-316
Operating Pressure: 300 p.s.i.g.
Feed: Sodium chloride-water

Film No.	Film Shrinkage Temp. °C.	A x 10^6 g. mole H_2O / $cm.^2$ sec.atm.	$(D_{AM}/k\delta)$ for NaCl x 10^5 cm./sec.	Feed: 3000 p.p.m. NaCl-water k=45x10^{-4} cm./sec. Solute sepn.%	Product rate gal/day $ft.^2$
5	75	2.84	3.00	96.5	22.2
6	75	3.02	3.50	96.1	23.6
7	68	3.73	6.30	94.2	29.1
9	74	3.50	5.40	94.7	27.3
11	72	3.60	5.80	94.5	28.1
12	65	4.62	11.20	91.3	36.1
15	76	2.39	1.90	96.5	____

Table III. - Physical Data for the System (NaCl-H_2O) at 25°C.

Molality	Mole Fraction x 10^3	Weight % Solute	Osmotic Pressure p.s.i.g.	Density of solution g./cc	Molar Density mole/cc. x 10^2	Kinematic Viscosity cm.^{2}/sec. x 10^2	Solute Diffusivity cm.^{2}/sec. x 10^5
0	0	0	0	0.9971	5.535	0.8963	1.610
0.1	1.798	0.5811	67	1.0011	5.535	0.9909	1.483
0.2	3.590	1.1555	133	1.0052	5.535	0.9054	1.475
0.3	5.375	1.7233	199	1.0091	5.535	0.9100	1.475
0.4	7.154	2.2846	264	1.0130	5.534	0.9147	1.475
0.5	8.927	2.8395	331	1.0169	5.534	0.9193	1.475
0.6	10.693	3.3882	398	1.0208	5.534	0.9242	1.475
0.7	12.453	3.9307	466	1.0248	5.534	0.9290	1.475
0.8	14.207	4.4671	534	1.0286	5.533	0.9338	1.477
0.9	15.955	4.9976	603	1.0322	5.532	0.9389	1.480
1.0	17.696	5.5222	673	1.0357	5.530	0.9440	1.483
1.2	21.160	6.5543	814	1.0427	5.526	0.9567	1.488
1.4	24.600	7.5640	959	1.0505	5.526	0.9685	1.492
1.6	28.016	8.5522	1109	1.0581	5.526	0.9802	1.497
1.8	31.408	9.5194	1262	1.0653	5.524	0.9923	1.505
2.0	34.777	10.4665	1419	1.0722	5.521	1.0044	1.513
2.2	38.122	11.3939	1580	1.0790	5.517	1.0206	1.521
2.4	41.444	12.3022	1745	1.0859	5.515	1.0365	1.530
2.6	44.743	13.1922	1915	1.0927	5.512	1.0530	1.539
2.8	48.019	14.0642	2089	1.0091	5.507	1.0683	1.548
3.0	51.273	14.9190	2270	1.1056	5.504	1.0840	1.556

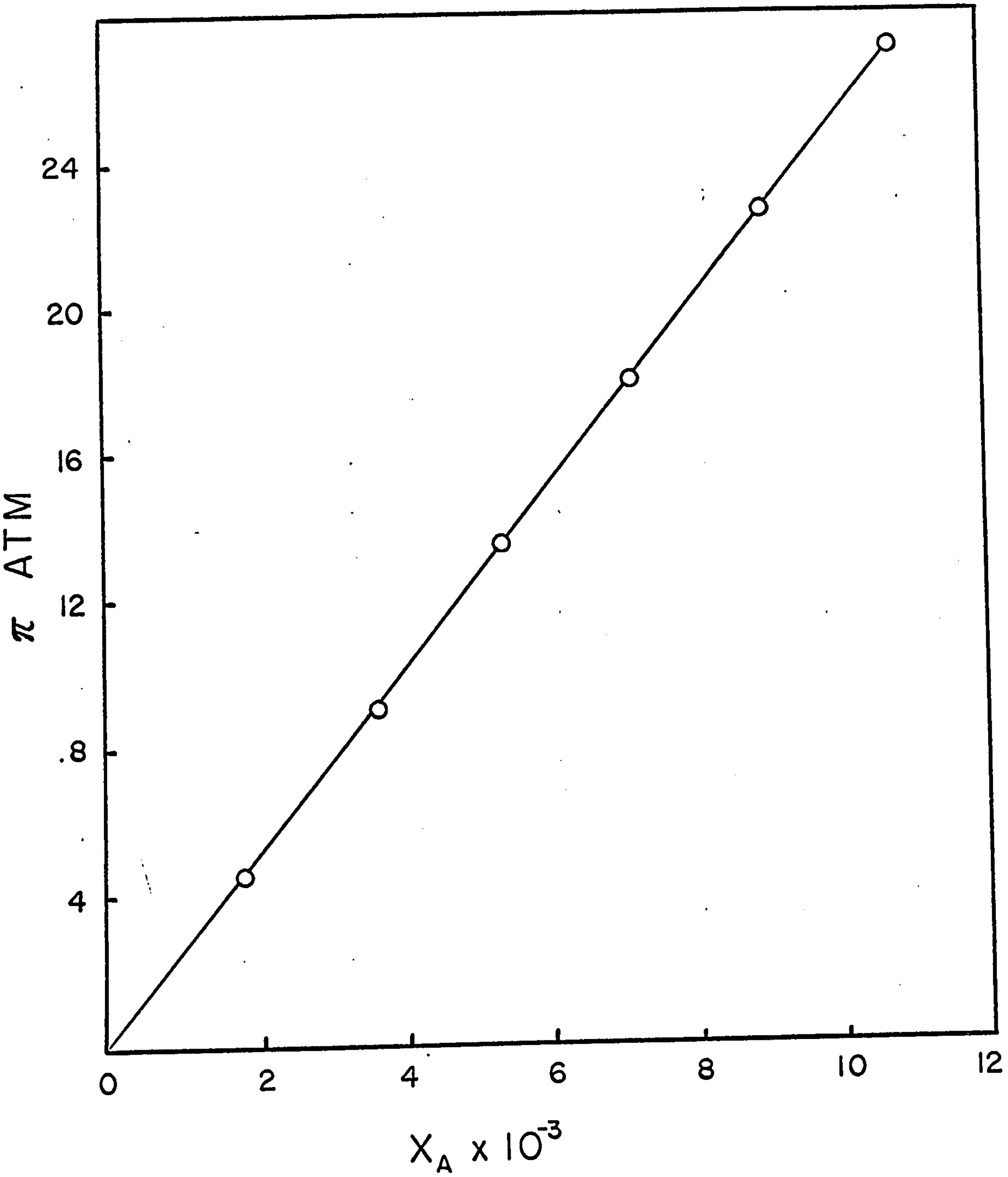

Fig. 11 - Plot of Osmotic Pressure vs Mole Fraction NaCl concentration

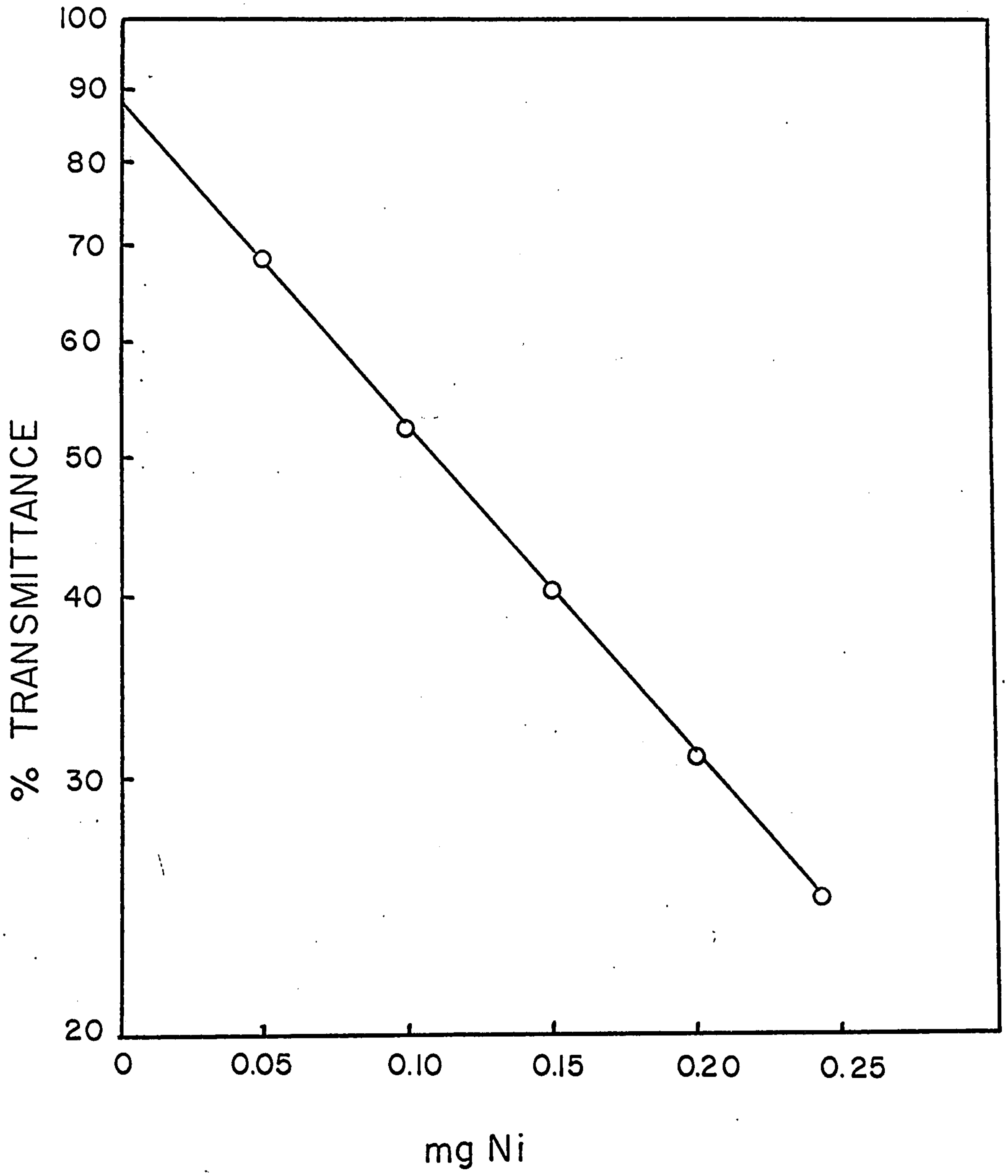

Fig. 12.- Calibration Curve for the Spectrophotometric Determination of Nickel by the Di-Methyl-Glyoxime Method.

IX - APPENDIX - II

SAMPLE CALCULATIONS

Membrane Specification Parameters

At a given operating pressure, P, from any set of (P.W.P.), (P.R.) and f data, the values of A, $\frac{D_{AM}}{k.\delta}$, and k can be calculated.

Calculations for membrane No. 7:

given: $\text{P.W.P.} = 41.29 \ \frac{\text{gr.}}{\text{Hr.-7.6 sq. cm.}}$

$\text{P.R.} = 34.90 \ \frac{\text{gr.}}{\text{Hr.-7.6 sq. cm.}}$

$f = 0.934$

$P = 22.6$ ATM.

$s = 7.6 \text{ cm}^2$

$m_1 = 0.0596$

$X_{A1} = 0.1073 \times 10^{-2}$

$M_B = 18$

1) Pure Water permeability constant, A

$$A = \frac{\text{P.W.P.}}{M_B.s.3600.P} \qquad (1)$$

$$A = \frac{41.29}{18\text{x}7.6\text{x}3600\text{x}22.6} = 0.3706 \times 10^{-5} \ \frac{\text{gr.-mde } H_2O}{\text{sq.cm.sec.atm.}}$$

2) Transport of solvent water, N_B, through the porous membrane

$$N_B = A\Delta P = A\left[P - \pi(X_{A2}) - \pi(X_{A3})\right]$$

$$N_B = A\left[P - \pi(X_{A2}) + \pi(X_{A3})\right] \qquad (2)$$

$$f = \frac{m_1 - m_3}{m_1} = 1 - \frac{m_3}{m_1} = 0.934$$

$$\frac{m_3}{m_1} = 1 - 0.934 = 0.066$$

$$\therefore \quad m_3 = 0.066 \times m_1 = 0.066 \times 0.0596 = 0.003938$$

$$m_3 = \frac{X_{A_3}}{1 - X_{A_3}} \cdot \frac{1000}{M_B}$$

$$0.03938 = \frac{X_{A_3}}{1 - X_{A_3}} \cdot \frac{1000}{18}$$

$$X_{A_3} = 0.0708 \times 10^{-3}$$

From the definition of molality

$$W_A + W_B = 0.003938 \times 58.45 + 1000 = 1000.23 \text{ gr}$$

By proportions, the weight fraction of solvent in the P.R. can be calculated

$$R = \frac{34.90 \times 1000}{1000.23} = 34.89$$

By definition of N_B

$$N_B = \frac{R}{M_B \cdot S \times 3600} = \frac{34.89}{18 \times 7.6 \times 3600} = 0.7077 \times 10^{-4} \frac{\text{gr-mole}}{\text{sec - cm}^2}$$

Substitution of N_B, A, P & $\Pi(X_{A3})$ into equation 2, yields $\Pi(X_{A2})$

$$0.7077 \times 10^{-4} = 0.3706 \times 10^{-5} \left[22.6 - \Pi(X_{A2}) + 0.16 \right]$$

$$\Pi(X_{A2}) = 3.69 \text{ ATM}$$

from Fig. 11

$$(X_{A2}) = 1.45 \times 10^{-3}$$

3) The solute transport parameter $\left(\frac{D_{AM}}{K.\delta}\right)$

$$N_B = C \left[\frac{D_{AM}}{K.\delta}\right] \quad \frac{(1 - X_{A3})}{X_{A3}} \quad . \quad (X_{A2} - X_{A3})$$

$$0.7077 \times 10^{-4} = 0.05535 \left[\frac{D_{AM}}{K.\delta}\right] . \frac{(1-0.0708\text{x}10^{-3})}{0.0708\text{x}10^{-3}} (1.45\text{x}10^{-3}-0.0708\text{x}10^{-3})$$

$$\left[\frac{D_{AM}}{K.\delta}\right] = 0.6562 \times 10^{-4} \quad \frac{\text{cm}}{\text{sec}}$$

4) Mass Transfer k, on the High-Pressure side of the Membrane

$$N_B = k.C\ (1 - X_{A3})\ .\ \ln \frac{X_{A2} - X_{A3}}{X_{A1} - X_{A3}}$$

$$0.7077 \times 10^{-4} = k.0.05535\ (1-0.0708 \times 10^{-3})\quad \ln\left[\frac{1.45\text{x}10^{-3}-0.0708\text{x}10^{-3}}{1.073\text{x}10^{-3}-0.0708\text{x}10^{-3}}\right]$$

$$\therefore\ k = 38.30 \times 10^{-4} \quad \frac{\text{cm}}{\text{sec}}$$

5) Determination of Fraction Solute Recovery, $\bar{F}_S$

$$\log \bar{F}_S = \left(1 - \frac{1}{f}\right) \log \frac{(C_A)f}{(C_A)i}$$

Assume $f = 0.995$

$$\frac{1}{f} = 1.005025$$

$$1 - \frac{1}{f} = 0.005025$$

P.P.M.	$\frac{(C_A)f}{(C_A)i}$	$\log \frac{(C_A)f}{(C_A)i}$	$(1-\frac{1}{f}) \log \frac{(C_A)f}{(C_A)i}$	$\log \bar{F}_S$	$\bar{F}_S$
1000	1.000	0.0000	0.000000	0.000000	1.000
2000	2.002	0.3025	0.001515	$\bar{1}.998485$	99.65
4000	4.012	0.6034	0.003032	$\bar{1}.996968$	99.31
6000	6.030	0.7803	0.003921	$\bar{1}.996079$	99.10
8000	8.056	0.9061	0.004554	$\bar{1}.995447$	98.96
10,000	10.091	1.0039	0.005045	$\bar{1}.994955$	98.84
12.000	12.134	1.0839	0.005447	$\bar{1}.994553$	98.75

6) Calculation of Variation of Volume Change, $\frac{Vi}{Vf}$

$$\log \frac{Vi}{Vf} = \frac{1}{f} \cdot \log \frac{(C_A)f}{(C_A)i} - \log \left[\frac{(\rho_1)i}{(\rho)f} \cdot \frac{1 + (C_A)f}{1 + (C_A)i} \right]$$

for f = 0.995

P.P.M.	$\log \frac{(C_A)f}{(C_A)i}$	$\frac{1}{f} \log \frac{(C_A)f}{(C_A)i}$	$\frac{\rho i}{\rho f}$	$\frac{1 + (C_A)f}{1 + (C_A)i}$
1000	0.0000	0.00000	1.00000	1.0000
2000	0.3025	0.30402	0.99701	1.0010
4000	0.6034	0.60643	0.99108	1.0030
6000	0.7803	0.78422	0.98522	1.0050
8000	0.9061	0.91065	0.97943	1.0071
10,000	1.0039	1.00894	0.97371	1.0091
12,000	1.0839	1.08934	0.96805	1.0111

for f = 1.000

1000	0.0000	0.0000	1.00000	1.0000
2000	0.3025	0.3025	0.99701	1.0010
4000	0.6034	0.6034	0.99108	1.0030
6000	0.7803	0.7803	0.98522	1.0050
8000	0.9061	0.9061	0.97943	1.0071
10,000	1.0039	1.0039	0.97371	1.0091
12,000	1.0839	1.0839	0.96805	1.0111

For f = 0.995

P.P.M.	$\left[\frac{\rho i}{\rho f}\cdot\frac{1+(C_A)f}{1+(C_A)i}\right]$	$\log\left[\frac{\rho i}{\rho f}\cdot\frac{1+(C_A)f}{1+(C_A)i}\right]$	$\log\frac{Vi}{V_f}$	$\frac{V_i}{V_f}$
1000	1.00000	0.00000 = 0.00000	0.00000	1.000
2000	0.99801	$\bar{1}.99913$ =-0.00087	0.30489	2.018
4000	0.99405	$\bar{1}.99741$ =-0.00259	0.60902	4.065
6000	0.99015	$\bar{1}.99570$ =-0.00430	0.78852	6.145
8000	0.98638	$\bar{1}.99404$ =-0.00596	0.91661	8.253
10,000	0.98257	$\bar{1}.99237$ =-0.00763	1.01657	10.390
12,000	0.97880	$\bar{1}.99069$ =-0.00931	1.09865	12.550

For f = 1.000

1000	1.00000	0.00000 = 0.00000	0.00000	1.000
2000	0.99801	$\bar{1}.99913$ =-0.00087	0.30237	2.010
4000	0.99405	$\bar{1}.99741$ = -0.00259	0.60599	4.036
6000	0.99015	$\bar{1}.99570$ = -0.00430	0.78460	6.090
8000	0.98638	$\bar{1}.99404$ = -0.00596	0.91206	8.167
10,000	0.98257	$\bar{1}.99237$ = -0.00763	1.01153	10.270
12,000	0.97880	$\bar{1}.99069$ = -0.00931	1.09321	12.395

7) Calculation of Fraction Water Recovery (Δ_W)

$$\Delta_W = 1 - \frac{V_f}{V_i}$$

For f = 0.995

P.P.M.	$\frac{V_i}{V_f}$	$\frac{V_f}{V_i}$	Δ_W
1000	1.000	1.0000	0.0000
2000	2.018	0.4955	0.5045
4000	4.065	0.2460	0.7540
6000	6.145	0.1627	0.8373
8,000	8.253	0.1212	0.8788
10,000	10.390	0.0962	0.9038
12,000	12.550	0.0797	0.9203

For f = 1.000

P.P.M.	$\frac{V_i}{V_f}$	$\frac{V_f}{V_i}$	Δ_W
1000	1.0000	1.0000	0.0000
2000	2.0105	0.4970	0.5026
4000	4.036	0.2476	0.7522
6000	6.090	0.1640	0.8358
8000	8.167	0.1223	0.8776
10,000	10.270	0.0974	0.9026
12,000	12.395	0.0807	0.9193

8) Density Data

Initial concentration 1000 P.P.M. or $(C_A)i = \frac{1}{999} = 1.001 \times 10^{-3}$

				ρ			
P.P.M.	$(C_A)_f$	$(C_A)_i$	$\frac{(C_A)f}{(C_A)_i}$	$NiCl_2$	$NiSO_4$	Ni-Plating	Double-Ni
1000	$1/999 = 1.001x10^{-3}$	$1.001x10^{-3}$	1.000	0.999	1.000	1.000	1.001
2000	$2/998 = 2.004x10^{-3}$		2.002	1.001	1.003	1.003	1.006
3000	$3/997 = 3.009x10^{-3}$		3.006	1.004	1.006	1.006	1.010
4000	$4/996 = 4.016x10^{-3}$		4.012	1.006	1.009	1.009	1.015
5000	$5/995 = 5.025x10^{-3}$		5.020	1.008	1.012	1.012	1.019
6000	$6/994 = 6.036x10^{-3}$		6.030	1.010	1.015	1.015	1.024
7000	$7/993 = 7.049x10^{-3}$		7.042	1.012	1.018	1.018	1.028
8000	$8/992 = 8.065x10^{-3}$		8.056	1.015	1.021	1.021	1.033
9000	$9/991 = 9.083x10^{-3}$		9.073	1.017	1.024	1.024	1.038
10,000	$10/990 = 10.101x10^{-3}$		10.091	1.019	1.027	1.027	1.042
11,000	$11/989 = 11.122x10^{-3}$		11.111	1.021	1.030	1.030	1.047
12,000	$12/988 = 12.146x10^{-3}$		12.134	1.024	1.033	1.033	1.051

9) Calculation of the "S" value and the processing capacity of a membrane

$$S = \int_{(\text{Ƶ})_f}^{(\text{Ƶ})_i} \frac{1}{q}\, d\,\frac{1}{\text{Ƶ}}$$

Calculations for membrane No. 12

Range $1/\text{Ƶ} = 1\text{x}10^{3}$ to $1/\text{Ƶ} = 0.5\text{x}10^{3}$

$$S = \frac{28270}{720\text{x}10^{4}} = 39.264\text{x}10^{-4}\ \frac{\text{DAY-Ft}^{2}}{\text{GR.}}$$

$$\text{Processing Capacity} = \frac{1}{(\rho 1)_i(\text{Ƶ})_i(S)}$$

$$\text{P.C.} = \frac{\left(2.642\ \frac{\text{GAL}}{\text{cc}}\right)}{\left(\frac{1\ \text{gr}}{1\ \text{cc.}}\right)\ (1\text{x}10^{-3})\ \left(39.264\text{x}10^{-4}\ \frac{\text{DAY-Ft}^{2}}{\text{gr.}}\right)^{2}} = 67.29\ \frac{\text{GAL.}}{\text{DAY-Ft}^{2}}$$

10) Processing Capacity Data for Membranes No.'s 12, 7, 11, 9, 6 and 5

Calculations for Membrane No. 12

P.P.M.	$\frac{1}{Z}$	P.R. $\frac{\text{GAL.}}{\text{DAY-Ft}^2}$	q $\frac{\text{GR}}{\text{DAY-Ft}^2}$	1/q $\frac{\text{DAY-Ft}^2}{\text{GR}}$	S $\frac{\text{DAY-Ft}^2}{\text{GR}}$	P.C. $\frac{\text{GAL.}}{\text{DAY-Ft}^2}$
1000	$1/1 \times 10^3$	34.5	130,203	7.6803×10^{-6}	------	-------
2000	$1/2 \times 10^3$	32.5	122,655	8.1529×10^{-6}	39.264×10^{-4}	67.29
4000	$1/4 \times 10^3$	29.5	111,333	8.9820×10^{-6}	60.447×10^{-4}	43.71
6000	$1/6 \times 10^3$	26.8	101,143	9.8870×10^{-6}	68.260×10^{-4}	38.70
8000	$1/8 \times 10^3$	24.3	91,708	10.904×10^{-6}	72.562×10^{-4}	36.41
10,000	$1/10 \times 10^3$	22.0	83,028	12.044×10^{-6}	75.415×10^{-4}	35.03
12,000	$1/12 \times 10^3$	19.8	74,725	13.382×10^{-6}	77.498×10^{-4}	34.09

Calculations for Membrane No. 7

P.P.M.	$\frac{1}{z}$	P.R. $\frac{GAL}{DAY\text{-}Ft^2}$	q $\frac{GR}{DAY\text{-}Ft^2}$	1/q $\frac{DAY\text{-}Ft^2}{GR}$	S $\frac{DAY\text{-}Ft^2}{GR}$	P.C. $\frac{GAL}{DAY\text{-}Ft^2}$
1000	$1/1\times10^3$	30.0	113,220	8.8324×10^{-6}	-------	-------
2000	$1/2\times10^3$	28.1	106,049	9.4296×10^{-6}	45.5611×10^{-4}	57.99
4000	$1/4\times10^3$	25.0	94,350	10.5988×10^{-6}	70.225×10^{-4}	37.62
6000	$1/6\times10^3$	22.8	86,047	11.6216×10^{-6}	79.415×10^{-4}	33.27
8000	$1/8\times10^3$	21.0	79,254	12.6177×10^{-6}	84.426×10^{-4}	31.29
10,000	$1/10\times10^3$	19.0	71,706	13.9458×10^{-6}	87.732×10^{-4}	30.11
12,000	$1/12\times10^3$	17.2	64,913	15.4052×10^{-6}	90.156×10^{-4}	29.31

Calculations for Membrane No. 11

P.P.M.	$\frac{1}{z}$	P.R. $\frac{GAL}{DAY\text{-}Ft^2}$	q $\frac{GR.}{DAY\text{-}Ft^2}$	1/q $\frac{DAY\text{-}Ft^2}{GR}$	S $\frac{DAY\text{-}Ft^2}{GR}$	P.C. $\frac{GAL}{DAY\text{-}Ft^2}$
1000	$1/1x10^3$	28.4	107,182	$9.3299x10^{-6}$	-------	-------
2000	$1/2x10^3$	26.8	101,143	$9.8870x10^{-6}$	$48.054x10^{-4}$	54.98
4000	$1/4x10^3$	24.4	92,086	$10.8594x10^{-6}$	$73.808x10^{-4}$	35.80
6000	$1/6x10^3$	22.4	84,538	$11.8290x10^{-6}$	$83.194x10^{-4}$	31.76
8000	$1/8x10^3$	20.4	76,990	$12.9887x10^{-6}$	$88.317x10^{-4}$	29.91
10,000	$1/10x10^3$	18.6	70,196	$14.2458x10^{-6}$	$91.694x10^{-4}$	28.81
12,000	$1/12x10^3$	16.8	63,401	$15.7721x10^{-6}$	$94.182x10^{-4}$	28.05

Calculations for Membrane No. 9

P.P.M.	$\frac{1}{z}$	P.R. $\frac{GAL}{DAY\text{-}Ft^2}$	q $\frac{GR}{DAY\text{-}Ft^2}$	1/q $\frac{DAY\text{-}Ft^2}{GR}$	S $\frac{DAY\text{-}Ft^2}{GR}$	P.C. $\frac{GAL}{DAY\text{-}Ft^2}$
1000	$1/1x10^3$	27.8	104,917	$9.5313x10^{-6}$	-------	--------
2000	$1/2x10^3$	26.2	98,879	$10.1134x10^{-6}$	$48.989x10^{-4}$	53.93
4000	$1/4x10^3$	24.0	90,576	$11.0404x10^{-6}$	$75.168x10^{-4}$	35.15
6000	$1/6x10^3$	21.8	82,273	$12.1546x10^{-6}$	$84.710x10^{-4}$	31.19
8000	$1/8x10^3$	20.0	75,480	$13.2485x10^{-6}$	$89.963x10^{-4}$	29.37
10,000	$1/10x10^3$	18.0	67,932	$14.7206x10^{-6}$	$93.407x10^{-4}$	28.29
12,000	$1/12x10^3$	16.3	61,516	$16.2559x10^{-6}$	$95.929x10^{-4}$	27.54

Calculations for Membrane No. 6

P.P.M.	$\frac{1}{z}$	P.R. $\frac{\text{GAL}}{\text{DAY-Ft}^2}$	q $\frac{\text{GR}}{\text{DAY-Ft}^2}$	1/q $\frac{\text{DAY-Ft}^2}{\text{GR}}$	S $\frac{\text{DAY-Ft}^2}{\text{GR}}$	P.C. $\frac{\text{GAL}}{\text{DAY-Ft}^2}$
1000	$1/1\text{x}10^3$	21.6	81,518	$12.2672\text{x}10^{-6}$	------	------
2000	$1/2\text{x}10^3$	20.3	76,612	$13.0527\text{x}10^{-6}$	$62,518\text{x}10^{-4}$	42.26
4000	$1/4\text{x}10^3$	18.0	67,932	$14.7206\text{x}10^{-6}$	$96.664\text{x}10^{-4}$	27.33
6000	$1/6\text{x}10^3$	16.4	61,894	$16.1567\text{x}10^{-6}$	$109.401\text{x}10^{-4}$	24.15
8000	$1/8\text{x}10^3$	14.8	55,855	$17.9035\text{x}10^{-6}$	$117.400\text{x}10^{-4}$	22.50
10,000	$1/10\text{x}10^3$	13.4	50,572	$19.7737\text{x}10^{-6}$	$122.044\text{x}10^{-4}$	21.65
12,000	$1/12\text{x}10^3$	12.0	45,288	$22,0810\text{x}10^{-6}$	$125.446\text{x}10^{-4}$	21.06

Calculations for Membrane No. 5

P.P.M	$\frac{1}{z}$	P.R. $\frac{GAL}{DAY\text{-}Ft^2}$	q $\frac{GR}{DAY\text{-}Ft^2}$	1/q $\frac{DAY\text{-}Ft^2}{GR}$	S $\frac{DAY\text{-}Ft^2}{GR}$	P.C. $\frac{GAL}{DAY\text{-}Ft^2}$
1000	$1/1x10^{3}$	20.6	77,744	$12.8673x10^{-6}$	------	------
2000	$1/2x10^{3}$	19.4	73,216	$13.6580x10^{-6}$	$65.656x10^{-4}$	40.24
4000	$1/4x10^{3}$	17.4	65,668	$15.2280x10^{-6}$	$101.161x10^{-4}$	26.12
6000	$1/6x10^{3}$	15.8	59,629	$16.7700x10^{-6}$	$114.347x10^{-4}$	23.11
8000	$1/8x10^{3}$	14.4	54,346	$18.4006x10^{-6}$	$122.564x10^{-4}$	21.56
10,000	$1/10x10^{3}$	13.0	49,062	$20.3824x10^{-6}$	$127.307x10^{-4}$	20.75
12,000	$1/12x10^{3}$	11.8	44,533	$22,4553x10^{-6}$	$130,771x10^{-4}$	20.20

11. Summary of Data for Plots in Fig. 9

P.P.M.	$\frac{(C_A)f}{(C_A)i}$	$(\bar{F})$ f=0.995	(Δ_W) f=0995	(Δ_W) f=1.00	Processing Capacity, GAL/DAY/Ft^2					
					(12)	(7)	(11)	(9)	(6)	(5)
1000	1.000	1.0000	0.0000	0.0000	-----	-----	-----	-----	-----	-----
2000	2.002	0.9965	0.5045	0.5026	67.29	57.99	54.98	53.93	42.26	40.24
4000	4.012	0.9931	0.7540	0.7522	43.71	37.62	35.80	35.15	27.33	26.12
6000	6.030	0.9910	0.8373	0.8358	38.70	33.27	31.76	31.19	24.15	23.11
8000	8.056	0.9896	0.8788	0.8776	36.41	31.29	29.92	29.37	22.50	21.56
10,000	10.090	0.9884	0.9038	0.9026	35.03	30.11	28.81	28.29	21.65	20.75
12,000	12.134	0.9875	0.9203	0.9193	34.09	29.31	28.05	27.54	27.06	20.20

END OF REEL

ARC-30