Effects of Manufacturing Variables and the Development of Replaceable Membrane Elements for Brackish Water Tubular Reverse Osmosis Systems

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Effects of Manufacturing Variables and the Development of Replaceable Membrane Elements for Brackish Water Tubular Reverse Osmosis Systems

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Contract No. 14-30-2665
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FOREWORD

This is one of a continuing series of reports designed to present accounts of progress in saline water conversion and the economics of its application. Such data are expected to contribute to the long-range development of economical processes applicable to low-cost demineralization of sea and other saline water.

Except for minor editing, the data herein are as contained in a report submitted by the contractor. The data and conclusions given in the report are essentially those of the contractor and are not necessarily endorsed by the Department of the Interior.
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I. OBJECTIVES AND SUMMARY

In accordance with the stated objective of subject contract, emphasis during the investigation of the effects of manufacturing variables on the quality and reliability of tubular reverse osmosis modules described in this report was placed on: (a) development of reliable and reproducible methods for casting of high-flux, brackish water desalination membranes, and (b) quality control of the tubular membrane-support composite elements. Amendment #3 to the contract modified the program objectives to include the development of replaceable membrane elements and of equipment suitable for their production and easy insertion into tubular supports. The results of the work carried out is summarized in the paragraphs below.

- A method for extruding in a reproducible manner good quality, high-flux brackish water desalination membranes formulated from a blend of cellulose di- and triacetates was developed. Mean values at 800 psi pressure of product water flux and sodium chloride rejection ratios of 9/16-in. diameter reverse osmosis tubes, equipped with separable Dacron sleeves lined with a blend membrane and heat treated at 93°C, were determined to be 29.6 gfd with a standard deviation of ± 3.85 gfd, and 90.9% with a standard deviation of ± 2.56%, respectively. Reverse osmosis tubes annealed at 90°C exhibit average flux and rejection ratio values of 35.1 gfd and 87.9%, respectively. Consequently, the objective set forth in the Scope of Work, namely, to establish methods for the production of high-flux tubular reverse osmosis membrane elements with a minimum flux of 30 gfd and 85% NaCl rejection, has been obtained.

- Variations in product water flux and sodium chloride rejection ratio attributable to batch-to-batch variations in the blend membrane casting solution were determined to be ± 7.65% and ± 4.6% of the respective mean values.
The storability of membrane casting solution was verified up to two months at temperatures of 40°F and 75°F; storage at 120°F for durations in excess of two weeks was found to affect the casting solution’s characteristics.

The effects of heat-induced agglomeration of the casting solution, of the surface finish and width of the extrusion nozzle’s gap, of particulate matter either on the extrusion nozzle’s inner plug, or on the substrate filter paper or in the gel bath, of the dimensional stability of the reverse osmosis tubes, and of the vibration induced by the sinusoidally varying dynamic loads imparted by the braiders were investigated as potential causes of the previously experienced erratic variations in the integrity of membrane lining. It was determined that the vibrations resulting from the kinematics of the braiders can cause defects in the extruded membrane.

An end fitting configuration for reverse osmosis tubes equipped with separable, membrane lined Dacron sleeves was developed. The number of ends and twist of the continuous-strand E-glass rovings was optimized and hydrostatic pressurization to destruction of membrane support tubes with this structure was found to result in true isostatic breaks of the tubes' wall. Average burst strength of the 9/16-in. I.D., 0.075-in. thick wall tube intended for working pressures up to 800 psi, is 3850 psi. Tube end fittings were found to withstand successfully tube bursts at pressures as high as 4400 psi.

Impregnation of the braided fiberglass membrane support tube structure with a crosslinked resin formulated from a 90%/10% blend of Union Carbide PKHS-1 phenoxy and Ciba Products Company XU-165 epoxy results in a substantially improved distribution of the resin within the matrix. On the basis of the "accelerated aging" tests carried out under Contract -2644,
impregnation with this resin appears to reduce substantially the susceptibility to hydrolysis of the fiberglass/resin composite structure.

- A technique was developed for low-cost production of membrane substrate tubes by ultrasonic seaming of strips of Dacron. The desalination properties of the membrane extruded onto Dacron are superior to those obtainable on filter paper. The most significant advantage arising from the use of the Dacron substrate tube however is that, because of its wet strength, it lends itself for employment as a separable, membraned sleeve which is easily replaceable without the necessity of discarding the entire reverse osmosis tube assembly.

- Major equipment which was developed to facilitate production of reverse osmosis tubes with replaceable membrane elements included: (a) the Dacron seamer and sizing assembly shown in the photograph and drawings, Figures 9, 10 and 11; (b) the membrane element insertion fixtures illustrated in the photographs and drawings, Figures 13 through 16, and (c) the elastomeric seal and cupro-nickel eyelet insertion and expansion fixture, Figures 17 and 18.

- Replaceable membraned elements, end fittings and seals were successfully tested both at the factory on a 5000 ppm sodium chloride solution and at the OSW Brackish Water Test Center, Roswell, New Mexico. The data acquired verified three salient features of the membrane employed, namely, high-flux, flux stability, and good salt retention.

- Simulated shutdowns and startups, i.e. pressure cycling of the reverse osmosis tubes between 0 and 800 psig up to 300 times at the rate of 20 times per hour, did not have any
measurable effect on desalination performance. 30,000 pressure cycles between a low of 600 psig and a high of 1000 psig at the rate of 3 cycles per minute did not have any effect on burst strength. Pressure cycling between the same limits but at the rate of 15 cycles per minute were found to cause about 40% degradation of burst strength after 10,000 cycles. Cycling at the same rate but between a low of 400 psig and a high of 1200 psig was found to cause tube failure after some 6,000 cycles.

II. TECHNICAL DISCUSSION

The work performed is presented below by the tasks set forth in the Scope of Work of subject contract and Amendment #3 thereto.

A. INVESTIGATION OF THE EFFECTS OF MANUFACTURING VARIABLES

1. Development of Reliable and Reproducible Methods of Casting High-Flux Brackish Water Desalination Membranes

The salt rejection ratio of the 9/16-inch I. D. reverse osmosis tubes manufactured using the pilot production equipment developed under Contracts 14-01-0001-2146 and -2163 was found to vary erratically. Dye check and microscopic examination of tubes with low salt retention revealed that the high salt-flux observed was caused by leakage through discrete defects and not by excessive inherent salt permeability of the membrane lining.

In compliance with the established quality control procedure, the integrity of the reverse osmosis tubes is verified by "100% inspection" which is carried out in two steps. At the initiation of the heat treatment cycle each tube is leak-checked at a pressure of 50 psig; subsequently every tube is subjected to desalination test at a pressure of 800 psig using a saline solution containing sodium chloride at a concentration of 10,000 ppm. The defects, with the relatively low-flux* membrane which was formulated using cellulose

*10 gfd at 650 psig in 9/16-inch I. D. reverse osmosis tubes
acetate E400-25 and propionamide, consisted of pinholes. Introduction of
the high-flux blend membrane resulted in doubling of the footage of reverse
osmosis tubes which had to be scrapped because of membrane defects.
Examination of tubes manifesting gross leakage during the leak-check re-
vealed longitudinal streaks along which there was insufficient membrane
lining or the membrane's active layer was damaged; as a rule, these
streaks were found to terminate with an agglomerate. Minor leakage,
usually not resulting in visible sprays and manifesting themselves only in
high salt permeation, were found to be caused by localized discontinuities
in the membrane, such as bubbles and "short streaks." The high rate of
incidence of such membrane defects were peculiar to the operations on the
pilot production line. Such defects did not occur with laboratory setup used
for membrane extrusion experiments in the course of membrane development
work. This observation led to a systematic investigation of the effects of the
attributes of the pilot production process which are different from the ones
practiced with the laboratory setup. The all-to-frequent, but erratic
occurrence of the membrane defects, indicated that some process parameters
were either not-at-all or, at best, only inadequately controlled. Accordingly,
the objective of the investigation was to identify the uncontrolled process
parameters which caused the great variability in membrane integrity.

The laboratory setup is designed to perform a single
operation, namely, the extrusion of the membrane casting solution onto the
interior of the substrate tube; with the pilot plant equipment, the braided glass
fiber filament/resin composite structural tube is formed at first, and then is
subsequently lined with the extruded membrane material. The pilot pro-
duction equipment is illustrated by the photograph and schematic in Figures 1
and 2. The sequence of processing operations starts at the top of the setup
with the sizing, scarfing and wrapping around the hollow mandrel of a filter
paper tape as required for forming the membrane substrate tube. For
achieving the desired structural strength, the substrate tube is encased by
three layers of fiberglass braid; accordingly, there are three braider head
assemblies, each comprising carriers and bobbins. The interior and outer
braids, applied by the top and bottom braiders, respectively, are formed by both longitudinal and diagonally-crossing rovings which are interwoven; the middle braid consists solely of a herringbone pattern without any longitudinal strands. Below each of the braiders there is a resin applicator to which the resin is fed by individual, positive displacement-metering pumps from a storage container, a vacuum jacket for stripping-off the resin solvent and an infrared heater for curing the resin. The reverse osmosis tube, as it is being formed, is conveyed downward into the gellation tank by several sets of hard rubber-covered external drive wheels. In order to facilitate hardening of the tube as it emerges from the infrared heaters, the tubes are air-cooled just ahead of the drive wheels. The hollow center mandrel is terminated just above the level of the water in the gellation bath by means of a nozzle through which the membrane casting solution, which is conveyed by means of a positive displacement metering pump, is extruded onto the interior of the substrate liner of the reverse osmosis tube. Upon passing over the extrusion nozzle, the membrane lined tube enters the low-temperature gellation bath.

The operating parameters of the pilot production line are controlled and, therefore, they were not believed to contribute to the product variability observed. The following uncontrolled process parameters were suspected as the potential cause of the erratic variations in membrane integrity:

1. Variations in the characteristics (viscosity, composition, etc.) of the membrane casting solution
2. Conditions at which the casting solution is stored
3. Temperature-history of the casting solution
4. Membrane extrusion nozzle setting and casting bubble
5. Variations in the diametric clearance between the extrusion nozzle and the substrate tube's interior.
The investigation of the effects aforementioned variables are described in the paragraphs below.

Inasmuch as it was the practice to commit the membrane casting solution to use on the basis of measurement of its viscosity, a laboratory evaluation of the validity of this attribute was undertaken. Twelve casting solution batches, 1 gallon each, were prepared according to the standard procedure. Since batch-to-batch variations in the characteristics of the commercially available lacquer-type cellulose acetates are known to affect membrane quality, the entire process information acquisition program was carried out using a single batch of both cellulose di- and triacetates; all solvents used were of certified reagent grade and packaged in "batch-size" small containers to prevent contamination by repeated opening and closing. Viscosity of each of the casting solution batches was measured at 25°C with a Model RVT Brookfield viscometer using a #6 spindle at 5 rpm. Membrane was sheet-cast from each of the batches on aluminumized Mylar and dried for 16 seconds prior to gelling in 1°C water; subsequently it was annealed at 70°C for 3 minutes, and the osmotic properties determined using six, 3-inch diameter circular specimens of each membrane, at 800 psi applied pressure with an aqueous 1% sodium chloride solution. Viscosity of the twelve batches of casting solution, average product water flux and sodium chloride rejection ratios of the respective membrane specimens are listed in Table 1. Mean values of the characteristics enumerated were: viscosity - 50400 cps, flux - 44.4 gfd, and salt rejection ratio - 87.89%. The respective standard deviations amount to ± 2200 cps ± 3.4 gfd, and ± 4.06%, corresponding to 4.4%, 7.65% and 4.6% of the respective mean values, thus indicating a tight distribution of the data. The data reveal no apparent correlation between the casting solution viscosity and the osmotic properties of the membrane. The
precision of viscosity measurements was determined to be ± 1400 cps; the variations in the values of the characteristics listed in Table 1 are also affected by the precision in casting solution preparation, membrane sample casting and testing operations; these factors, however, are not readily quantifiable. Consequently, for practical purposes the values listed in Table 1 have to be regarded as a measure of batch-to-batch variations. The data acquired indicate that the viscosity of the casting solution can be used as a check for proper formulation but not as a prediction of the membrane's osmotic properties.

Batches of casting solution are prepared and stored at a controlled temperature of about 70°F. When needed, they are transferred into the cylindrical storage tank located above the top platform of the pilot production line shown in Figure 1. Since neither duration nor the temperature of the storage in that tank are controlled, the potential effects of the variability of these characteristics upon the membrane casting solution were evaluated. For that purpose, the three 1-gallon batches of casting solution were stored, one each at the following temperatures: 40°F, 75°F, and 120°F. At approximately one week intervals the viscosity of the casting solution batches were measured and membrane sample was cast from each of them. To facilitate the task, each batch was separated into two bottles - one for viscosity measurements and one for membrane casting. The data thus acquired are listed in Table 2. As can be seen, storage at either 40°F or 75°F for durations up to two months has no significant effect on the salient characteristics of the membrane casting solution. On the other hand, storage at 120°F for durations exceeding two weeks results in a significant change in the casting solution. Since the daily average temperature of the casting
solution in the pilot production line's storage tank does not exceed 75°F, its "aging" cannot be considered to have caused the variability in membrane quality.

Mean values of the flux and sodium chloride rejection ratio of the membranes cast from the batch of casting solution stored at 75°F amounted to 44.4 gfd and 87.01%. These values are virtually the same as the mean values shown in Table 1 which pertain to the membranes cast from the 12 batches of solution which were employed for the determination of batch-to-batch variations. On the other hand, the standard deviation for a single batch, namely, +2.6 gfd and +2.68%, amount to only 85% and 65%, respectively, of the corresponding batch-to-batch standard deviations. This confirms that the variations in osmotic properties of membrane specimens cast from the same batch of casting solution are less than those which are attributable to batch-to-batch variations.

The shape of the most frequently recurring defect, namely, a longitudinal streak along which the membrane's active layer was either missing or damaged, suggested that it may have been caused by temporary, partial plugging of the gap in the extrusion nozzle. Consequently, several manufacturing test runs were conducted with the objective of reducing, if not eliminating, the effect of variables which could cause plugging of the nozzle gap by particulate matter. Figure 3 is a schematic illustration of the membrane extrusion.

The casting solution as it flows downward within the hollow center mandrel, passes through the infrared heating zones and its temperature could be raised sufficiently to result in the formation of agglomerates which could cause temporary obstruction or plugging of the extrusion nozzle gap. Prevention of the casting solution from "overheating" was attempted by water-cooling the inside of the mandrel. Manufacturing runs were performed
using two different cooled mandrel configurations; calori-
metric measurements established their rates of cooling as
150 and 1100 Btu/hr. Some 3400 ft of reverse osmosis
tubing was produced using these cooled mandrels; results of
these processing tests are shown in Tables 3 and 4. The
percentage of tubes which passed the high pressure desali-
nation test amounted to 39.2% and 54.3% employing the
mandrels cooled at the rates of 150 and 1100 Btu/hr,
respectively.

Casting solution sticking to the wall of the tubing through
which it is conveyed to the extrusion nozzle could start to
agglomerate as a consequence of prolonged residence in the
zones affected by the infrared heaters. The occurrence of
such a situation could be prevented by periodic removal of
the casting solution hanging on the tube wall. The results of
the processing tests performed after thorough cleaning of
the entire membrane casting solution supply system are
shown in Table 5. Some 1800 ft of reverse osmosis tubing
was processed and 33.6% of the tubes passed the high
pressure desalination test.

During experimentation with the laboratory extruder setup,
blebs were noticed in the casting solution as it was being dis-
charged by the extrusion nozzle. To insure that such blebs
will quickly pass through the nozzle and not obstruct ex-
trusion of the casting solution, the internal nozzle passages
were highly polished in the direction of flow. Some 750 ft
of tubing was processed with the polished nozzle and 37.8%
of the tubes passed desalination test. The results are pre-
sented in Table 6.
To prevent obstruction of the nozzle by gelled blebs, the nozzle extrusion gap was increased from 7 mils to 15 mils. Some 700 ft of reverse osmosis tubing was produced with the opened up gap; the results are shown in Table 7. 55.5% of the reverse osmosis tubes passed desalination test.

Dust and chips from the substrate filter paper scarfing was observed to settle onto the filter paper strip. Since this phenomenon could result in plugging of the extrusion nozzle by particular matter, 1200 ft of reverse osmosis tubes were produced with the substrate paper vacuum-cleaned on both sides just before passing into the paper former around the mandrel. The data are shown in Table 8; 50% of the tubes passed desalination test.

Actuation of the flying cut-off saw used for rough sizing of the length of the reverse osmosis tubes causes stirring of the water in the gel bath. Consequently, sawdust and/or impinging water may impede the extrusion of the casting solution. Accordingly, tubes were hand-cut at a point just below the extrusion nozzle. Some 1300 ft of tubing were processed in this manner; data are shown in Table 9. 41.5% of the tubes passed desalination test.

Evidence of gelled casting solution under the surface of the extrusion nozzle inner plug indicates the occurrence of back-wetting. This phenomenon can be prevented by solvent saturation of the air-stream which is supplied immediately below the nozzle to maintain adequate pressure. The air supply for the casting bubble was passed through a mixture of solvents in the casting solution to prevent back-wetting and thus the formation of particulate matter which could damage the freshly deposited membrane lining. Some
1600 ft of reverse osmosis tubes were processed in this manner and 50% of the tubes passed desalination test. The data are listed in Table 10.

One of the prerequisites to the deposition of a faultless membrane liner coat is that the inner surface of the substrate tube be at all times perfectly concentric with the lip of the extrusion nozzle. Any eccentricity would result in uneven diametric distribution of the clearance between nozzle and tube interior and, therefore, in irregularities in the extruded membrane lining.

To investigate the effects of dimensional stability of the reverse osmosis tubes, two additional infrared heaters were installed following the top two resin applicators. The extra heat was to increase the tube hardness and thus its dimensional stability at the extrusion nozzle. Some 1300 ft of tubing was processed in this manner; data are shown in Table 11. 45.8% of the tubes passed desalination test.

Owing to their kinematics, the carriers installed in the braiders impart to the pilot production setup sinusoidally varying dynamic loads and the resulting vibrations can affect extrusion of the membrane casting solution. Consequently, a membrane extruder, which is separate from the pilot plant equipment, was constructed by Aerojet/Envirogenics as an adjunct to its other capital asset facilities for the production of braided glass fiber filament/resin composite reverse osmosis tubes. This setup was activated with the change-over from the filter paper membrane substrate liner which is adhesively bonded to the structural tube to the separable sleeve made of Dacron (see Paragraph 4 - Substrate) and its effectiveness was readily apparent. Of the first 500 reverse osmosis tubes membraned using this new equipment, more than 360, i.e. 72.5%, passed the desalination test. The
quality of the membrane this extruder is producing repro-
ducibly demonstrates that the erratic performance of the
pilot plant's extrusion nozzle was caused by the vibrations
imparted by the braidiers. With this newly installed setup,
ocurrence of the longitudinal streaks along which the
membrane's active layer is defective has been eliminated.

The reproducibility in the quality of high-flux blend membrane lining
produced by means of the membrane extruder, which is separate from the
line containing the braidiers, is illustrated by the product water flux and salt
rejection ratio histograms shown in Figures 4 and 5. 9/16-in. diameter
reverse osmosis tubes of a nominal 14 ft length, equipped with membrane
lined separable Dacron sleeves and heat treated at 93°C, exhibit at 800 psi
an initial mean flux of 29.6 gfd; standard deviation is ± 3.85 gfd, i.e. 13% of
the mean value. The mean value of the corresponding sodium chloride
rejection ratio is 90.9% with a standard deviation of ± 2.56%. The heat treat-
ment temperature of 93°C was selected for insuring that the mean value of the
sodium chloride rejection ratio will exceed 90% for the production lot ratio
under consideration. Tubes annealed at 90°C exhibit average flux and re-
jection ratio values of 35.1 gfd and 87.9%, respectively. Consequently, the
objective set forth in the Scope of Work, namely, production of high-flux
tubular membrane elements with a minimum of 30 gfd and 85% NaCl rejection,
has been attained.

2. End Fitting Evaluation

Tubular reverse osmosis pilot plants containing 128, 9/16-in.
I.D. reverse osmosis tubes of a nominal length of 14 ft were employed in the
course of the field tests the Office of Saline Water conducted to determine the
technical feasibility of utilizing the reverse osmosis process to reduce the
salinity of waters flowing into the Colorado River. The two such units, one
of which was tested on the Virgin River near LaVerkin Springs, Utah, and the
other on the surface stream flowing in Las Vegas Wash near Henderson,
Nevada, exhibited wide disparity in the performance of tube end fittings: no
end fitting failures occurred to the unit at LaVerkin Springs during the over
4-1/2 months operation, while 23 tubes, amounting to 18% of the initial
complement, pulled loose from the end fittings during the about 3-1/2 months operation at Las Vegas Wash. The configuration of the seal assembly employed in these field tests is illustrated in Figure 6. Its marginality was attributed to the stress concentrations at the sharp termination of the stainless steel seal cage (drawing TD-0483 on Figure 6), which may have resulted in local stresses which were beyond the ultimate capability of a particular tube. As a corrective action, flaring of the seal cage termination, as illustrated in the drawing in Figure 7 was proposed. Prior to the start of evaluating the effectiveness of this modification, the change-over to the separable, membrane lined sleeve caused the configuration to become obsolete.

Figure 8 illustrates the tube end fitting configuration which was developed for use with the separable, membranated substrate liner and to fit the same receptacle socket as the ones shown in Figures 6 and 7. The separable membrane sock, Item 5 on the drawing shown in Figure 8, is held in place by the expanded cupro-nickel eyelet, Item -3, a Kraton elastomeric seal, Item -2 which is sandwiched between the eyelet and membrane sleeve, and is cuffed around and attached to the O-ring carrier fitting, 2F100084. This latter part, made of polycarbonate, 20% glass fiber reinforced, is injection-molded directly onto the structural tube's end. The Kraton end fitting cuff is secured to the O-ring carrier by means of a neoprene snap ring, Item -4. The plastic retaining nut is identical to that employed in the seal assembly shown in Figure 6.

3. Structural Support Tube Strength Improvement

In the course of the optimization of the quality of glass fiber, twist of yarn and spooling procedures performed under Contract -2163, it was found that the number of ends and the extent of twist of both the longitudinal and hoop glass strands affect the resin content of the composite material and thus the reverse osmosis tube's apparent stiffness. This latter attribute proved to be quite critical, inasmuch as the originally elected commercially available thermoplastic phenoxy resin (Union Carbide Corp., PKHS-1) was
found to be subject to plasticization by water which, in turn, made the structural tubes to become pliable when wet and thus easily damaged. Since the process developed under Contract 14-30-2568 for the conversion of the thermoplastic phenoxy resin into a thermoset through crosslinking it with American Cyanamid Co. Cymel-301 brand melamine resin, using a 20% solution of para-toluene sulfonic acid morpholine salt in dimethylformamide as the catalyst results in a substantial reduction in the extent of plasticization by water and thus in a smaller loss in the wet stiffness of the tubes, the structural support tube improvement program was carried out using the crosslinked resin.

The quasi "second generation" reverse osmosis tube manufacturing line, which was constructed and procured by Aerojet/Envirogenics as its own capital asset equipment, incorporates 4 braiding and 3 longitudinal strand application stations vs the 3 braiding and 2 longitudinal yarn applicators in the equipment illustrated schematically in Figure 2 which were acquired under Contract -2146. The "second generation" line, because of the greater freedom in the selection of glass fiber distribution, was judged to be better suited for the investigation of the braiding patterns required for the attainment, or at least close approximation, of the ideal isostatic tube structure.

Optimization of the glass distribution was carried out using continuous-strand E-glass rovings (ECG-75) with the intent of achieving the closest possible approximation of the isostatic structure without changing the tube's outside diameter and thus without necessitating change in the tube end fitting dimensions. On the basis of theoretical considerations, the number of ends and extent of twist which are required for the attainment of the isostatic structure are shown in Table 12. This pattern resulted in excessive outside diameter. The patterns subsequently tried are also indicated in Table 12; the last one shown is considered to be optimum inasmuch as with the crosslinked resin it yields a nominal wall thickness and outside diameter of 0.075 and 0.72 inches, respectively, and upon burst at an average pressure of 3200 psig it exhibited a true isostatic break.
As a consequence of wetness induced strength degradation of fiberglass, both the longevity and the strength of fiberglass/resin composite structures exposed to wet environments are greatly affected by the extent of resin impregnation. Certain, newly developed resins are exhibiting exceptionally good "wetting" characteristics, i.e. upon application to braided or woven fiberglass structures they tend to disperse within the matrix and thus coat the rovings. The epoxy resin, XU-165 of the Ciba Products Company, is one of such compounds. On the basis of destructive testing performed after soaking in 500 ppm aqueous hydrofluoric acid solution, structural tubes impregnated with a 90/10 blend of Union Carbide PKHS-1 phenoxy resin and the Ciba XU-165 epoxy appear to be much less susceptible to hydrolysis than tubes processed only with phenoxy resin. In addition, with the blended resin the burst strength of the structural tubes increased to 3850 psig.

The optimum braiding pattern with the 3 braiding and 2 longitudinal strand applicator equipment which was acquired under Contract -2146 was determined to be as follows:

<table>
<thead>
<tr>
<th>Station</th>
<th>Braider Ends</th>
<th>Twist/inch</th>
<th>Longitudinal Ends</th>
<th>Twist/inch</th>
</tr>
</thead>
<tbody>
<tr>
<td>#1</td>
<td>4/0</td>
<td>1</td>
<td>4/0</td>
<td>1</td>
</tr>
<tr>
<td>#2</td>
<td>6/0</td>
<td>1</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>#3</td>
<td>6/0</td>
<td>1</td>
<td>12/0</td>
<td>1</td>
</tr>
</tbody>
</table>

This pattern differs from the one which was found to be optimum for the line containing 4 braiders and 3 longitudinal strand applicators. While the difference was found to have no effect on the tube's burst strength, it results in markedly different elongation and flexural characteristics. At a pressure of 800 psig, the elongation of tubes produced with the 3 braiding/2 longitudinal strand applicator equipment was determined to be 0.32% which is almost 50% greater than the 0.22% value for tubes manufactured by means of the line containing 4 braiders and 3 longitudinal strand applicators. On the other hand, no
A measurable difference was detected in the expansion of the tubes' internal diameter; at a pressure of 800 psig, this attribute was found to be 6 mils for both types of tubes. As can be seen from the data tabulated below, both the resin content and the amount of solvent extractables are affected by the features of the tube manufacturing equipment used.

<table>
<thead>
<tr>
<th></th>
<th>Average Resin Content</th>
<th>Average Solvent Extractables</th>
</tr>
</thead>
<tbody>
<tr>
<td>3 braider/2 longitudinal</td>
<td>11%</td>
<td>26.4%</td>
</tr>
<tr>
<td>strand applicators</td>
<td></td>
<td></td>
</tr>
<tr>
<td>4 braider/3 longitudinal</td>
<td>14.6%</td>
<td>20.8%</td>
</tr>
<tr>
<td>strand applicators</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

The improvements in glass distribution and resination which are realized using the 4 braider/3 longitudinal applicator line manifest themselves by the increase in tube stiffness and reduction in the susceptibility to handling damage. Of the structural tubes made using the setup with 4 braiders and 3 longitudinal strand applicators, less than 2% were damaged as a consequence of handling; of the tubes produced using the 3 braiders/2 longitudinal applicator equipment, some 20% had to be scrapped because of handling damage.

4. **Substrate**

On the basis of the membrane substrate selection program, which was carried out under Contract 14-01-0001-1764, the C. H. Dexter, Grade X1611 long-fibered, manila hemp filter paper was chosen because of the following attributes:

- High tensile strength, to permit transport through the paper forming machine with minimum breakage.

- High compressive modulus in the dry condition to prevent excessive pressure on the mandrel, and consequent drag from the tension in the braid.

- At least one surface free of loose protruding fibers.
Good formability to insure a properly shaped tube.

High wet strength, both tensile and compressive.

Subsequently, however, it was found that the osmotic properties with certain membrane formulations are inferior when cast on the Dexter X1611 filter paper to those obtained by casting the membrane onto aluminized Mylar. In the course of the search for alternate substrate materials which do not limit the magnitude of the product water flux that can be realized, filter cloth made out of synthetic fibers such as Nylon or Dacron were found to be superior to filter paper. However, the high cost of circular-loom-woven seamless tubes made out of such fabrics made this approach too costly. Consequently, at that time, the use of the selected Dexter filter paper was judged to be the most cost effective.

Ultrasonic welding equipment of 1000 watts or larger capacity which can be operated continuously, became commercially available recently and enable low-cost production of substrate tubes by ultrasonic seaming of a strip of Dacron or Nylon filter cloth. 45-in. wide rolls of Travis Mills Corporation's Dacron and Nylon fabrics (#601 and #5050) of which suitability of membrane material has been established some time ago cost $0.96 and $0.51 per yard, respectively, and the rolls can be slit into some 20 strips for ultrasonic welding of nominal 9/16-in. diameter tubes. Accordingly, the material cost of the Dacron or Nylon substrate tube amounts to 1.4 cents and 0.75 cents per lineal foot, respectively, i.e., nearly the same as the one cent for the long-fibered, manila hemp filter paper, Dexter Grade X1611. Although desalination performance with both Nylon and Dacron substrate materials appears to be identical, Dacron, in spite of its higher cost, has been selected because its water absorption is negligible.

The most significant advantage arising from the use of the Dacron substrate tube is that, because of its wet strength, it lends itself for employment as a separable, membraned sleeve which is easily replaceable without the necessity of discarding the entire reverse osmosis tube assembly. Consequently, to enable the realization of the resulting reduction in membrane replacement costs, the filter paper membrane substrate lining which, because
of adhesive bonding is integral with the structural tube, has been replaced by the separable membraned sleeve made out of Dacron.

B. EQUIPMENT FOR PRODUCTION OF REVERSE OSMOSIS TUBES WITH REPLACEABLE MEMBRANE ELEMENTS

1. Dacron Substrate Liner Seaming and Sizing

The Dacron Seamer Assembly is illustrated in the photograph in Figure 9. The Dacron fabric, after rough cutting and sizing, is dispensed from a roll through an alignment fixture for final cutting. The fixture then preforms the trimmed material which passes around the mandrel and forming guides for final forming, moves between a shaped horn and the mandrel through which an ultrasonic power source is attached. This power source supplies current through the horn that accomplishes the actual seaming. The finished sleeve then "floats" along an air-driven guide until passing through an electric eye which triggers a cutting tool, cutting the finished substrate liner to the desired length. This electric eye can be so positioned to activate the cutters for any length of substrate liner desired. The drawings 4A100150, Sheets 1 and 2 (Figures 10 and 11), depict the seamer assembly configuration as described, and the drawing 4A100164 in Figure 12, the sock conveyor on which the seamed liner "floats" until cut to size.

Upon completion of the mechanical and performance check-out of the seamer assembly, the adequacy of seamed Dacron sleeves with respect to their diametric dimensions and seam strength was verified.

Conformance to the desired outside diameter was verified by inserting Dacron socks into precision drawn stainless steel tubing of which bore is identical to the nominal inside diameter of the fiberglass/resin composite membrane support tubes.

Acceptability of the inside diameter and of the seamed liner interior's roundness was verified through the performance of membrane extrusion runs. The fact that
the osmotic properties of the membrane thusly deposited confirmed to specified values, i.e. flux and sodium chloride retention 25 gfd and 90%, respectively, at 800 psi indicated that the liner's interior was satisfactory for the deposition of good quality membrane coating.

Membraned Dacron sleeves encased in fiberglass/resin composite structural tubes were subjected to hydraulic "pounding" by cycling the internal pressure between 0 and 800 psig, about 20 times per hour. The fact that sleeves subjected to 300 such pressure cycles did not suffer any deterioration in salt retention indicates the adequacy of the strength of the seam.

2. Membrane Element Insertion Fixtures

Two different substrate sleeve insertion fixtures have been developed.

The one shown in the photograph in Figure 13 and drawing 2T101030, Figure 14, consist of a V-block support, tube drive mechanism, a mandrel with substrate end securing attachments and a linear mandrel actuator.

For insertion of the Dacron sleeve, either a precision drawn stainless steel processing tube or the fiberglass/resin composite structural tube is positioned onto the V-block support and inserted over the mandrel. The tube drive mechanism is then engaged, driving the tube over the mandrel. The linear mandrel is then extended through the tube. A substrate sleeve is then placed on the V-block and secured at the ends to the mandrel. The linear mandrel is then retracted
partially into the tube which is then driven over the entire substrate. Finally, the substrate securing attachments are removed and the substrate sleeve encased by either the stainless steel processing or fiberglass/resin composite structural tube and is ready for further processing.

The other insertion fixture shown in Figure 15 and drawing 2T100220, Figure 16, consists of an inflatable mandrel with double membrane substrate attachment, process tube holding clamps, and a mandrel drive mechanism with a grooved membranated substrate support belt. It is intended for two operations:

(a) removal of membranated substrate from process tube, and

(b) for insertion of the membranated sleeve into the structural tube.

The process tube with membranated substrate is stored for membraning in the water filled process tank. To remove the membranated substrate support, engage the tube holding clamps and drive the inflatable mandrel in the deflated condition into membranated process tube. The membranated substrate is then attached to the mandrel and the mandrel inflated. The mandrel is then driven with the membranated substrate from the process tube onto a grooved belt. The holding clamps are then disengaged and the process tubes are removed from the tank. The braided structural tube is then placed into the holding clamps and the clamps engaged. The membranated substrate and mandrel are
then guided into the structural tube. The mandrel is then deflated, the substrate attachments removed from the mandrel, and the mandrel is then retracted from the structural tube. The lift mechanism is then actuated to drain the structural tube with the membrane and removed from the tank.

As a consequence of eliminating the necessity of encasing the Dacron sleeve into a process tube and of the happenstance that with the "dry" setup shown in Figures 13 and 14, which is more convenient to operate, the membraned sleeve can be inserted into the structural tube fast enough to prevent drying of the membrane, the wet setup is used less frequently.

3. End Fitting and Seal Development

Drawing 2F100096, Figure 8, depicts the end fitting and seal configuration for use with the replaceable membraned sleeve. The first operation in the manufacture of this configuration is the injection molding of the O-ring carrier fitting (2F100084) directly onto the structural tube end. Upon assembly, an extruded elastomeric seal and cupro-nickel eyelet, Items -2 and -3 of Figure 8, are inserted. Expansion of the eyelet is accomplished using the swaging fixture shown in drawing 2T101062, Figure 17, and photograph, Figure 18. This fixture pulls equally in both directions along the longitudinal axis so as not to impart any differential axial force which might wrinkle or more either the substrate support or the eyelet. After expansion of the eyelet, the elastomeric seal and substrate leader are wrapped around the leading edge contour of the O-ring carrier with the tool depicted in drawing 2T101078, Figure 19.

More than 110 fourteen-ft long reverse osmosis tubes fitted with replaceable membrane elements, end fittings and seals of the configuration as shown in Figure 8 (drawing 2F100096), were tested on a 5000 ppm sodium chloride solution at a pressure of 800 psi for a duration amounting to almost 300 hours during the two month period between December 15, 1970 and
February 15, 1971. The setup containing the test tubes was operated during the period for about 8 hours every working day and was subjected to more than 100 startup and shutdown cycles. The replaceable membrane sleeves, end fittings and seals performed satisfactorily without any malfunctioning, whatsoever. However, some of the structural tubes showed evidence of developing weak spots in the immediate vicinity of the in-board end of the molded-on O-ring carrier fittings.

As mentioned above, pressure cycling of the reverse osmosis tubes between 0 and 800 psig up to 300 times, at the rate of 20 times per hour, did not have any measurable effect on desalination performance. Pressure cycling between a low of 600 psig and a high of 1000 psig at the rate of 3 cycles per minute did not have any noticeable fatigue effect, inasmuch as the burst strength after 30,000 such cycles was found to be the same as that of virgin tubes. On the other hand, pressure cycling between the same limits but at the rate of 15 cycles per minute was found to cause definite degradation in burst strength; the burst pressure after 10,000 cycles was measured to be some 1500 psig lower than that of virgin tubes. When cycling between a low of 400 psig and a high of 1200 psig at a rate of 15 cycles per minute, the average number of cumulative cycles resulting in failure amounted to some 6000. Accordingly, it appears that the extent of fatigue damage depends on the magnitude of applied peak stress and frequency of pressure cycling but not on the amplitude of pressure variations. The mode of failure of the tubes, namely, partial or complete rupture in the vicinity of the in-board edge of the molded-on O-ring carrier fittings, was found to be the same regardless whether the failure was caused by hydrostatic burst or by pressure cycling-induced fatigue. This observation led to the conclusion that the structural tube could be strengthened by making the transition in the section modulus at that location more gradual and/or increasing modulus of elasticity of the tube wall.

9/16-in. diameter reverse osmosis tubes with separable Dacron sleeves lined with membrane formulated from a blend of cellulose di- and triacetates were shipped to and field tested at the OSW Brackish Water Test Center, Roswell, New Mexico. The data acquired verify three salient features of the membrane employed, namely, high-flux, flux stability and
good salt retention. Product water flux at the start of the testing was determined to be 25.7 gfd at a feed pressure of 600 psig. After more than a month of operation (about 800 hours), the flux at 600 psig was 24.8 gfd, i.e. only .9 gfd less than at the start of the test. On the basis of these data, the intrinsic flux decline exponent can be calculated to be less than 0.02, and a terminal flux after one year of operation in excess of 20 gfd forecast. Electrical conductivity measurements indicate that a total dissolved solids rejection ratio of 91-92% was realized.
## TABLE 1
MEMBRANE CASTING SOLUTION VISCOSITY
AND OSMOTIC PROPERTIES

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<th>Casting Solution Batch No.</th>
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<th>Flux gfd</th>
<th>NaCl Rejection %</th>
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Standard Deviation  
\[ \pm 2200 \]  \[ \pm 3.4 \]  \[ \pm 4.06 \]
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*Membranes cast from samples used for viscosity measurements.*
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<th>TUBE PROCESSING &amp; TESTING</th>
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\[
\frac{159}{174} = 91.4\% \\
\frac{153}{157} = 97.4\% \\
\frac{60}{153} = 39.2\%
\]

Overall Yield = 91.4 × 97.5 × 39.2 = 34.7%
### TABLE 4

**R. O. TUBE TEST REPORT**

**PROCESSING TEST NO. T-105**

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\[
\frac{35}{40} = 87.5\%
\]

\[
\frac{35}{35} = 100\%
\]

\[
\frac{19}{35} = 54.3\%
\]

Overall Yield = 87.5 x 100 x 54.3 = 40.5%
### TABLE 5

**R. O. TUBE TEST REPORT**  
**PROCESSING TEST NO. ** T-101  
**Line No.** OSW-1

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\[
\frac{114}{118} = 96.5\%
\]

\[
\frac{116}{116} = 100\%
\]

\[
\frac{39}{116} = 33.6\%
\]

**Overall Yield = 96.5 \times 100 \times 33.6 = 32.4\%**
### TABLE 6

**R. O. TUBE TEST REPORT**

**PROCESSING TEST NO. T-106**

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\[
\frac{46}{47} = 98\%
\]

\[
\frac{45}{46} = 97.8\%
\]

\[
\frac{17}{45} = 37.8\%
\]

Overall Yield = 98 \times 97.8 \times 37.8 = 36.2\%
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\[
\frac{13}{46} = 28.3\% \quad \frac{9}{13} = 69.2\% \quad \frac{5}{9} = 55.5\%
\]

Overall Yield = 28.3 x 69.2 x 55.5 = 10.9%
### TABLE 8
R. O. TUBE TEST REPORT
PROCESSING TEST NO. T-103

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<tr>
<td>3/17</td>
<td>42</td>
<td>41</td>
<td>0</td>
</tr>
<tr>
<td>Totals</td>
<td>75</td>
<td>63</td>
<td>0</td>
</tr>
</tbody>
</table>

\[
\frac{63}{75} = 84\% \\
\frac{62}{63} = 98.5\% \\
\frac{31}{62} = 50\%
\]

Overall Yield 84 x 98.5 x 50 = 41.4%
<table>
<thead>
<tr>
<th>PERIOD (MANUFACTURING)</th>
<th>TUBE PROCESSING &amp; TESTING</th>
<th>R.O. Test</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Heat Treat</td>
<td>End Fit</td>
</tr>
<tr>
<td>Day</td>
<td>No. Mfg</td>
<td>Shifts</td>
</tr>
<tr>
<td>3/12</td>
<td>47</td>
<td>24</td>
</tr>
<tr>
<td>3/13</td>
<td>39</td>
<td>21</td>
</tr>
<tr>
<td>Totals</td>
<td>86</td>
<td></td>
</tr>
</tbody>
</table>

\[
\frac{70}{86} = 81.4\%
\]
\[
\frac{69}{70} = 98.7\%
\]
\[
\frac{29}{70} = 41.5\%
\]

Overall Yield: \[81.4 \times 98.7 \times 41.5 = 33.4\%\]
### TABLE 10

**R. O. TUBE TEST REPORT**

**PROCESSING TEST NO. T-107**

<table>
<thead>
<tr>
<th>PERIOD MANUFACTURING</th>
<th>TUBE PROCESSING &amp; TESTING</th>
<th>HEAT TREAT</th>
<th>END FIT</th>
<th>R.O TEST</th>
<th>FAILED</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>No. Tubes</td>
<td>Passed</td>
<td>Shorts</td>
<td>Failed</td>
</tr>
<tr>
<td>Day</td>
<td>Mfg</td>
<td>Shifts</td>
<td>Day</td>
<td>Swing</td>
<td></td>
</tr>
<tr>
<td>3/31</td>
<td>23</td>
<td>2</td>
<td>21</td>
<td></td>
<td></td>
</tr>
<tr>
<td>4/1</td>
<td>43</td>
<td>25</td>
<td>18</td>
<td></td>
<td></td>
</tr>
<tr>
<td>4/2</td>
<td>36</td>
<td>15</td>
<td>21</td>
<td></td>
<td></td>
</tr>
<tr>
<td>4/3</td>
<td>3</td>
<td>3</td>
<td>0</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Totals</td>
<td>105</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

\[
\frac{94}{105} = 89.5 \%
\frac{94}{94} = 100\%
\frac{47}{94} = 50\%
\]

Overall Yield = 89.5 x 100 x 50 = 44.8%
### TABLE 11

**R. O. TUBE TEST REPORT**  
**PROCESSING TEST NO. T-108**  
Line No. OSW-1

<table>
<thead>
<tr>
<th>PERIOD</th>
<th>MANUFACTURING</th>
<th>TUBE PROCESSING &amp; TESTING</th>
<th>R O Test</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>No. Mfg</td>
<td>Shifts</td>
<td>No. Tubes</td>
</tr>
<tr>
<td>Day</td>
<td>Day</td>
<td>Swing</td>
<td>Passed</td>
</tr>
<tr>
<td>4/7</td>
<td>29</td>
<td>12</td>
<td>17</td>
</tr>
<tr>
<td>4/8</td>
<td>21</td>
<td>10</td>
<td>11</td>
</tr>
<tr>
<td>4/9</td>
<td>33</td>
<td>19</td>
<td>14</td>
</tr>
<tr>
<td>4/10</td>
<td>1</td>
<td>1</td>
<td>0</td>
</tr>
<tr>
<td>Totals</td>
<td>84</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

\[
\frac{59}{84} = 70.2\% \\
\frac{59}{59} = 100\% \\
\frac{27}{59} = 45.8\%
\]

Overall Field = 70.2 \times 100 \times 45.8 = 32.1%
## TABLE 12
GLASS DISTRIBUTION WITH 4 STATIONS

<table>
<thead>
<tr>
<th>Station</th>
<th>Braider Ends</th>
<th>Twist</th>
<th>Longitudinal Ends</th>
<th>Twist</th>
<th>Braider Ends</th>
<th>Twist</th>
<th>Longitudinal Ends</th>
<th>Twist</th>
<th>Braider Ends</th>
<th>Twist</th>
<th>Longitudinal Ends</th>
<th>Twist</th>
<th>Braider Ends</th>
<th>Twist</th>
<th>Longitudinal Ends</th>
<th>Twist</th>
</tr>
</thead>
<tbody>
<tr>
<td>#1</td>
<td>4/0</td>
<td>1</td>
<td>3/0</td>
<td>1</td>
<td>4/0</td>
<td>1</td>
<td>3/0</td>
<td>1</td>
<td>4/0</td>
<td>1</td>
<td>3/0</td>
<td>1</td>
<td>4/0</td>
<td>1</td>
<td>3/0</td>
<td>1</td>
</tr>
<tr>
<td>#2</td>
<td>3/0</td>
<td>3</td>
<td>4/0</td>
<td>1</td>
<td>3/0</td>
<td>3</td>
<td>4/0</td>
<td>1</td>
<td>3/0</td>
<td>3</td>
<td>4/0</td>
<td>1</td>
<td>3/0</td>
<td>3</td>
<td>4/0</td>
<td>1</td>
</tr>
<tr>
<td>#3</td>
<td>5/0</td>
<td>3</td>
<td>-</td>
<td>-</td>
<td>5/0</td>
<td>3</td>
<td>-</td>
<td>-</td>
<td>4/0</td>
<td>1</td>
<td>-</td>
<td>-</td>
<td>4/0</td>
<td>1</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>#4</td>
<td>6/0</td>
<td>3</td>
<td>6/0</td>
<td>1</td>
<td>5/0</td>
<td>3</td>
<td>6/0</td>
<td>1</td>
<td>4/0</td>
<td>1</td>
<td>6/0</td>
<td>1</td>
<td>5/0</td>
<td>3</td>
<td>6/0</td>
<td>1</td>
</tr>
</tbody>
</table>
Figure 1

Pilot Production Equipment
Figure 2
Figure 3. Extrusion of Tubular Membranes
9/16" I.D. REVERSE OSMOSIS TUBES; LENGTH 14' (NOMINAL)
BLEND MEMBRANE

PRODUCT WATER FLUX DISTRIBUTION
at 800 psi and heat treat temperature at 93°C
1% NaCl test solution

Mean 29.6 gfd

Standard Deviation
± 3.85 gfd

Figure 4
9/16" I.D. REVERSE OSMOSIS TUBES; LENGTH 14' (NOMINAL)
BLEND MEMBRANE

SODIUM CHLORIDE REJECTION RATIO DISTRIBUTION
at 800 psi and heat treat temperature at 93°C
1% NaCl test solution

Mean 90.9%

Standard Deviation
± 2.56%

63.3%

5.5% 9.2% 22.0%

<70 70-80 80-90 >90

Sodium Chloride Rejection Ratio, %

Figure 5
NOTES:

- R.O. TUBE
- NUT-RETAINING TD-0484 (LEXAN)
- SEAL - INNER (LEXAN)
- PARKER 2-19 O'RING
- CAGE SEAL TD-0483 (S.S.)
- SEAL RUBBER TD-0488 (NEOPRINE)

**Figure 1**

---

**Figure 6**
Figure 7
Membrane Element Insertion Fixture

Figure 15
Figure 18

Eyelet Swaging Fixture