DEVELOPMENT OF AN ADVANCED TRANSVERSE FLOW NANOFILTRATION MEMBRANE PROCESS FOR HIGH PERFORMANCE DESALINATION PHASE II

By:
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Technical Service Center
Water Treatment Engineering and Research Group

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Water Treatment Engineering and Research
Denver, Colorado

EXECUTIVE SUMMARY

The U.S. Bureau of Reclamation has identified desalination of brackish waters using nano-filtration as a priority area of research under the Water Treatment Technology Program. Development of the most cost-effective and efficient membrane-based processes would address many water quality and supply problems encountered in the U.S., particularly in the more arid regions.

ZENON Environmental, Inc., was contracted to continue the development of its novel Moustica transverse flow hollow fiber nanofiltration module for desalination applications. The two-phase project involved development of a high tensile strength fiber suitable for brackish water application, production of suitable membranes, and re-design of the existing transverse flow concept for high-pressure applications.

In Phase I, high tensile strength base fibers were successfully developed, along with a chlorine-resistant nanofiltration membrane with suitable solute rejection characteristics. In addition, a module prototype was constructed of materials which could withstand the necessary operating pressures. Flux performance, when tested on synthetic brackish water, was lower than desired, and further module development was needed to improve the design.

In Phase II, the focus was to continue the development of an improved module by maxi-mizing surface area/volume ratio to improve productivity per module, optimizing flow distribution to minimize channeling, and modifying module design to reduce manufacturing costs.

The following improvements were made to the membrane and module during the course of Phase II:

- Significantly improved fibers with a flux of 20 USgfd and a rejection of >90% on 2,000 ppm $MgSO_4$ solution at 150 psi have been developed.

- Long-term testing of this membrane showed good performance under various test conditions, and the membrane also showed rejection of NaCl and other low molecular weight organic compounds.

- An entirely reconfigured transverse flow module was designed. The principle is based on a stack of "fiber positioning cards," which are positioned within an element housing to form a module containing 16 "cards."

- The module was designed to fit inside a standard S-inch RO pressure vessel, which would facilitate market acceptance and potential for the retrofit market.

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- A prototype module was constructed which was able to withstand a pressure up to 150 psi. Testing revealed that some redesign of the element housing and the fiber positioning card configuration is needed to extend operating pressures beyond this point.

- The packing density was increased to 63 ft^2/ft^3. It is anticipated that further refinements could result in a maximum additional 25% improvement.

An analysis of commercialization potential indicates that due to the level of automation required, and the cost of commercially available nanofiltration membranes, commercial production of this product is not yet viable because of the capital investment requirement to manufacture such a module.

The major advantages of this type of design are lower operating costs associated with higher mass transfer, lower pressure drop, and reduced pretreatment requirements, which could significantly impact overall process costs. Actual throughput using the improved fibers on brackish water was not measured as of yet+ The actual throughput or productivity of this module needs to be tested.

Once performance data is generated, the final commercial viability can be determined.

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Chapter 1
INTRODUCTION

1.1 Background and Objectives

Desalination of brackish waters using nanofiltration is one priority area of research identified by the U.S. Bureau of Reclamation Water Treatment Technology Program. In order for membrane-based desalination systems to be widely employed, the most cost-effective modules and systems need to be designed. ZENON Environmental, Inc., was contracted to continue the development of Zenon's novel Moustic™ transverse flow hollow fiber nanofiltration module for desalination applications and assess it's potential to meet BUREC objectives.

Phase I of this program, completed in 1995, resulted in the following improvements to the base Moustic™ design:

- A transverse flow module prototype was constructed which could be operated at the pressures necessary for brackish water desalination.

- Suitable high tensile base fibers were developed.

- Appropriate membrane chemistries were evaluated, and a chlorine-resistant nanofiltration membrane with suitable solute rejection characteristics was produced.

- Methods for application of the membrane were investigated.

The first phase of this work was published as Water Treatment Technology Report No. 9 and presents a description of the transverse flow concept as well as the initial development work completed. Testing of the module developed in Phase I on a synthetic brackish feed identified key areas where further improvements were necessary to make the concept commercially viable:

- Module design needs to be modified to increase flux, minimize channeling, and increase packing density (surface area/volume ratio).

- Fiber length needs to be maximized in order to minimize manufacturing costs associated with cutting and potting fiber ends.

This report presents the results of Phase II of the program, awarded under BUREC contract No. 1425-5-FC-81-20590. The primary objective of Phase II was to continue the development of an improved module design, building upon the improvements made in Phase I. The following specific module modifications needed to improve performance and reduce costs were the focus of this phase of development:

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1) Maximize surface area/volume ratio to improve productivity per module.

2) Improve flow distribution to minimize channeling and reduce pretreatment requirements.

3) Modify design to reduce manufacturing costs.

Chapter 2
FIBRE AND MEMBRANE PRODUCTION

Standard thin film composite reverse osmosis and nanofiltration membranes have two separate parts:

- A thin barrier layer (membrane) which serves as the separating layer.

- A microporous sublayer (base fiber) supporting the barrier layer.

It has been shown by Cadotte et al. (198 1) that polysulfone provides an excellent support for very thin, highly selective desalination membranes. In flat sheet membranes, the mechanical strength is provided by a fibrous web and, therefore, a thin support layer can be made. However, fibers must be self-supporting and, in this case, need to withstand compression pressures of 300-400 psi. Because of this, the fiber usually has a thick wall, and this results in low permeability. As such, the formulation used in flat sheets cannot be used in fiber spinning, and a new formulation with optimal characteristics needed to be developed.

Previous work has shown that a hollow fiber reinforced with fiberglass has a higher compression and collapse pressure. The dope composition, membrane thickness, and spinning conditions all affect performance, as measured by factors such as flux, compression pressure, and molecular weight cutoff (MWCO). The resulting base fiber must have the appropriate MWCO, pure water flux, and compression pressure for nanofiltration applications.

To meet this objective in Phase I of this program, base fibers were developed from three different polymers: polysulfone (PS), sulfonatedpolysulfone (SPS), and polyimide (PI). The fibers were reinforced using a hollow fiberglass braid as support. In addition to strengthening the fiber, this allowed the use of dopes with lower viscosity. High viscosity dopes yield fibers with lower flux and higher compression pressure. Methodology used in fiber spinning does not allow one to use low viscosity dopes. One way to overcome this difficulty is to make a thin membrane on a support so that most of the strength of the fiber will come from the support and the membrane only behaves as a separation layer. This would allow the coating of thinner base membranes, which in turn would maximize the flux while maintaining compression resistance.

2.1 Experimental Methods

2.1.1 Coating

A simplified schematic of a fiber coating device is shown in figure 2-l. A reservoir containing the polymer solution is pressurized with nitrogen and the polymer is forced into the spinning jet (a tube-in-tube jet). In unreinforced fibers, the solution extruded through the
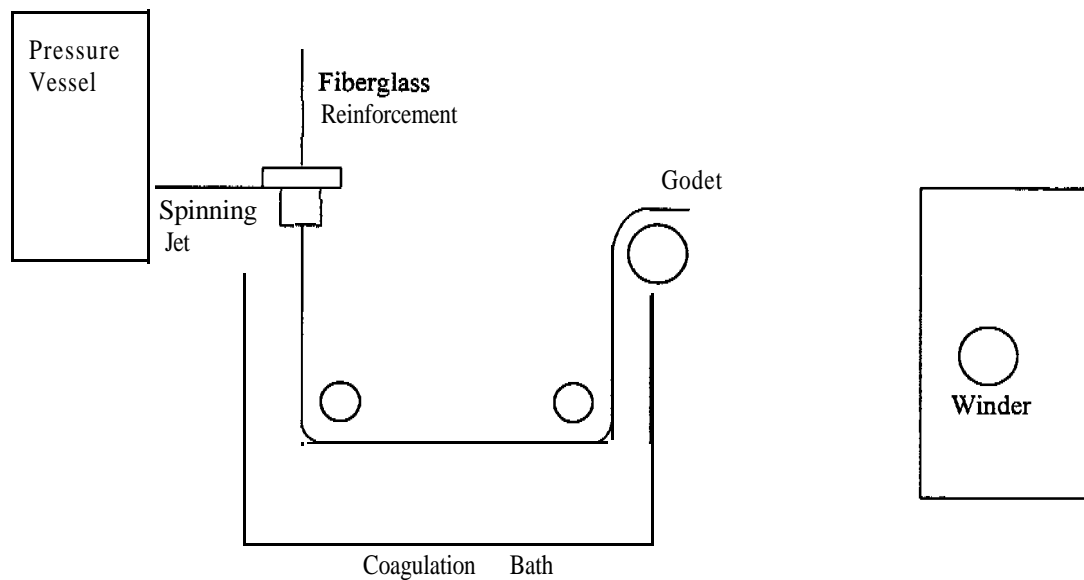
Figure 2-1 .-Hollow fiber spinning.

spinning jet is drawn into a coagulation bath. The center bore of the extruded fiber is maintained open by the use of a non-coagulant fluid under pressure. The size of the extruded fiber is controlled by the pressure in the dope vessel, the extruding speed, and the pressure of fluid in the center bore.

For reinforced fibers, the procedure is identical, except that a hollow fiberglass braid is used instead of a non-coagulant bore fluid. The fiberglass reinforcement passes through the first jet and is coated with polymer. The second jet removes the excess polymer. The resulting fiber proceeds through the coagulation bath.

The replacement of polymer solvent with water from the coagulation bath causes the viscosity of the polymer solution to increase. The speed of the replacement as well as the polymer formulation control the pore size of the resulting fiber. As more solvent is replaced by water, the polymer begins to solidify in the coagulation bath and can be further handled.

The fiber is then directed to a winder by use of a self advancing godet. The fiber is wound into bobbins. The wound fiber is then washed with water to remove the remainder of the solvent and additives and then impregnated with preservative before drying.

2.2 Compression Test

A simplified chart of the test installation is shown in figure 2-2. The compression pressure of the fiber was determined using water flux measurements. A looped fiber bundle containing 6 fibers was potted into a half-inch steel fitting. The fiber bundle was connected to a water reservoir. The outside of the fiber bundle was surrounded with water. The water reservoir was then pressurized with nitrogen to approximately 20 psi and the water flux recorded over
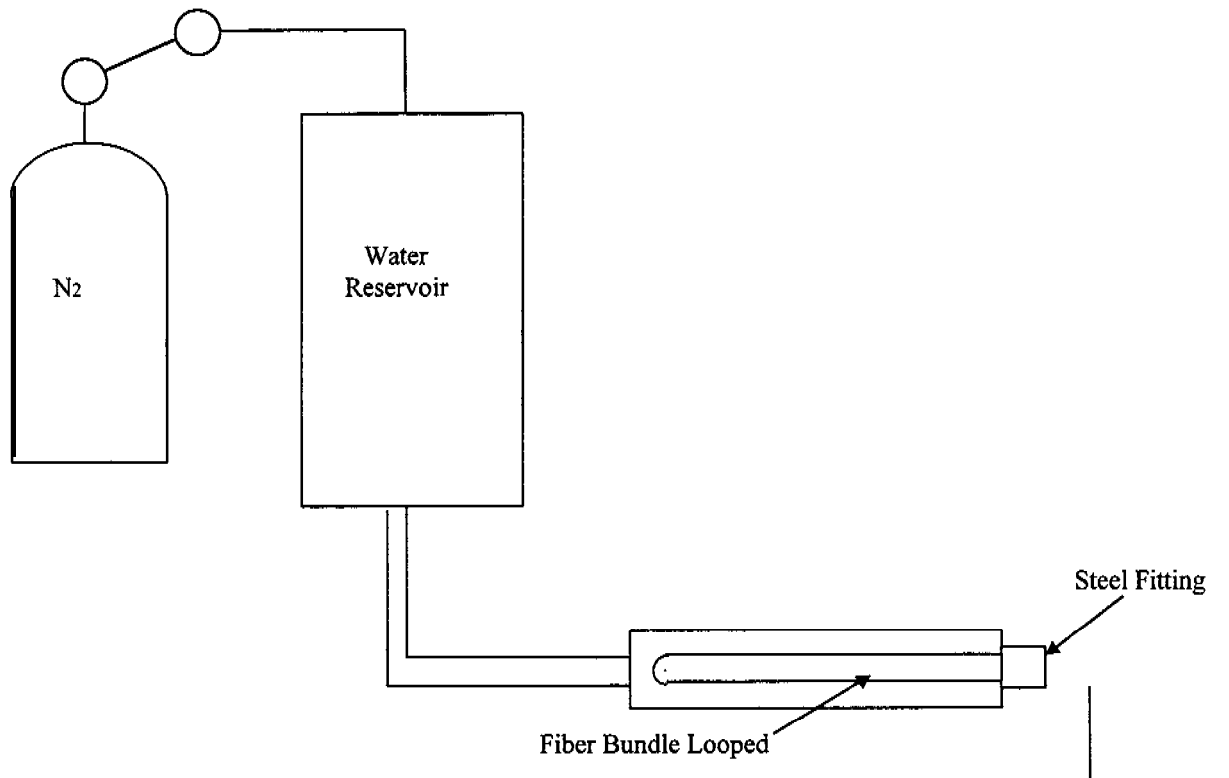
2-2

Figure 2-2.—Compression.

a 20-minute period (measured at 5-minute intervals). The nitrogen pressure was increased by 20 - 50 psi every 20 minutes up to 400 psi, or when the flux started to level off and stopped increasing with pressure. The compression resistance is determined by plotting the water flux as a function of test pressure. The point at which the curve begins to flatten is the compression pressure of the fiber.

In Phase I of this project, polysulfone base fiber coated with sulfonated polysulfone gave a nanofiltration membrane with the flux of 13 USgfd and a rejection of 70% on 2,000 ppm $MgSO_4$ solution at 200 psi test pressure. The membrane developed in Phase I had rejection of magnesium sulfate within the target criteria; however, the flux was lower than the target. It is our objective in Phase II to develop a membrane with high rejection >90% on magnesium sulfate and a flux of 20-25 USgfd at an operating pressure of 150 psi.

In Phase I, the polymer used in the fabrication of the base fiber was polysulfone. Although it was suitable for coating a nanofiltration thin film membrane, we had some difficulty with storage. It was observed that flux of the base fiber declined with time. Consequently, in Phase II, we decided to look at other polymers which have better hydrophilicity and higher glass transition temperature (T,).

2.3 Base Fiber Improvements in Phase II

In Phase I, the polysulfone base fiber, when coated with sulphonated polysulfone, gave a nanofiltration membrane with a flux of 13 USgfd and a rejection of 70% on 2,000 ppm $MgSO_4$ solution at 200 psi test pressure.

Although these membranes were suitable for treating brackish water, the flux and rejection were too low to be commercially viable. Another difficulty encountered during fabrication of the base fiber is that the flux of the base fiber changed with time (flux dropped).

As such in Phase II, the objective was to develop a more stable base fiber, which could not only be used at room temperature but could also be used at higher temperatures (>55 °C). The final coated nanofiltration membrane should have a 95% rejection on 2,000 ppm $MgSO_4$ and a flux $>$ 15 USgfd at 150 psi. In this phase, we chose the polymer polyethersulfone (PES) as base fiber material. The reason for this choice is that PES is much stronger, more hydrophilic, and has a higher T_g than polysulfone.

2.3.1 Development of Polyethersulfone Base Membrane

The target characteristics of the base membrane are:

- Pure water flux $>$ 100 USgfd at 150psi.

- Rejection (molecular weight cut off) $<$ 50,000 Daltons.

A. Optimization of Dope Composition

The dope consists of PES, polyvinylpyrrolidone (PVP), and the solvent dimethyl formamide (DMF). Dopes with varying amounts of PES and PVP were made and their properties (viscosity etc) were measured. The acceptable dopes were coated on a glass fiber braid (OD/ID 1.0/0.3mm). Initially, all fibers had a low bubblepoint and pin holes and, therefore, were not acceptable for coating the thin film composite membrane.

The dope making procedure and drying times were changed, and the resulting fibers obtained did not have any pin holes and had a good bubble point. Further, the spinning conditions were changed to optimize the flux and the rejection. Parameters considered were air gap, dope temperature, and the gelation temperature (temperature of bath 1 and bath 2).

Fibers having the following characteristics were chosen for the thin film membrane coating trials:

Pure water flux	< 100 USgfd at 50 psi
Rejection (MWCO)	> 60% dextran 10 K
Bubble point	> 15 psi

2.4 Membrane and Coating Development

2.4.1 Fiber Pretreatment

It is well known that in a solvent evaporative thin film coating method, the base fibers should be dry. However, in polymeric base fibers, pores collapse as soon as they are dried. Thus, the final coated membrane will have a very low flux. Several methods were tried to prevent the pore collapse when the fibers were dried.

a) Solvent Exchange

The base fiber was treated with methanol and isopropyl alcohol. After the treatment, fibers were soaked in water or air dried. Air dried membranes showed severe flux decline.

b) Hot Water Treatment

The base fiber was treated with boiling water and coated with standard SPS solution. The results obtained were not satisfactory.

c) Glycerine Treatment

The base fiber was treated with a glycerine solution and then coated with the standard coating solution. The results obtained were not satisfactory.

d) Chlorine Treatment

The base fiber was treated with chlorine and coated with the standard SPS coating solution. The fibers thus obtained did not have the required flux and rejection.

Another series of trials was conducted to develop and optimize a suitable pretreatment method.

The base membrane was treated with boiling water and glycerine and then coated with SPS coating solution under various conditions.

The coating methods used in the experiments are summarized below:

a) Base membranes were treated with boiling water and glycerol solutions. The membranes were then coated with SPS solutions of different concentrations and heated.

b) After treatment with boiling water and glycerol solution, the base membranes were coated with polyvinylmethylether (PVME)-methanol solutions of different concentrations. Then, PVME coated base membranes were coated with SPS solution and heated.

c) The boiling water and glycerol treated base membranes were coated with PVME and SPS at variation conditions. The main condition changes were: (1) PVME coated fibers were heated at 60 °C for 5 min., (2) there was no glycerol step after SPS coating, and (3) the base membranes were washed with methanol after boiling water and glycerol solution treatments.

d) The boiling water and glycerol treated base membranes were coated with PVME and SPS in different order.

e) The boiling water and glycerol treated base membranes were coated with PVME, PVA, and SPS. The coated membranes were heated at 100 °C for 10 min.

f) The untreated base membranes were coated with PVA and SPS. The reproducibility was determined. Change in the rejection and flux were measured with time for several membranes.

g) A polyacrylic acid was used as a precoating solution.

h) The SPS coated membranes were tested on 2,000 ppm $MgSO_4$. The rejection and flux were determined at 150 psi with a flow rate of 10 L/min.

Results of the trials indicated that PVA used as a precoating agent for the SPS coating on the PES base membrane provided the optimal membrane.

2.4.2 Optimization of Thin Film Coating

PVA-SPS coating trials were carried out at various conditions, using several base fibers to optimize the membrane. Base fibers used in this study were JB-5 to JB-11-3.

a) With JB-5 fibers, the effect of PVA of different molecular weight and concentrations on coating efficiency was investigated at different curing temperatures.

b) JB-6 fibers were washed with water for different time periods after spinning and were coated with PVA-SPS solutions. PVA solutions with two different concentrations were used, The reproducibility was determined at the same conditions.

c) The fibers (JB-7) were coated with PVA-SPS solutions. The PVA with different molecular weight and at different pH's were used. The effect of treatment times of PVA and SPS solutions on coating efficiency was investigated,

d) The fibers (JB-9) were coated with PVA-SPS solutions. The PVA solutions with different molecular weight and two concentrations were used. The reproducibility was determined at the same conditions.

e) The fibers (JB-11-3) were coated with PVA-SPS solutions. The PVA solutions with different molecular weight and two concentrations of SPS solutions were used. The coating period with PVA solution was changed from 10 min to 30 min. The number of times the coating solution could be used was determined.

2.4.3 Conclusions

While the coating efficiency strongly depends on the base fiber properties (molecular weight cutoff, bubble point, and flux), a defect free base fiber could be directly coated with SPS solution to produce fibers with a >90% rejection and 20 USgfd when tested on 2,000 ppm $MgSO_4$ at 150 psi.

2.4.4 Attempt to Increase the Rejection and Flux of SPS Coated NF Membrane

In an attempt to simplify the coating procedure, SPS coating trials were carried out under different conditions. Base fibers JB-11 and JB-13 were used in the experiments. Two different SPS solutions with different concentrations of dichloromethane were used for coating.

The following parameters were investigated:

- The effect of dichloromethane concentration in the SPS coating solution on rejection and flux was investigated (figure 2-3).

- The different water soluble polymer (P, PEIM, and P30) were used in the preparation of glycerol solutions.

- A P30 solution was coated on PES fiber directly.

Long-term testing of SPS coated fibers was then performed. A fiber with 94.6% rejection and 18 USgfd when tested on 2000 ppm $MgSO_4$ at 150 psi was obtained.

2.4.5 Long-Term Test

Fiber bundles with different membrane surface areas were tested at 150 psi. Figure 2-4 shows the rejection and flux change with time.
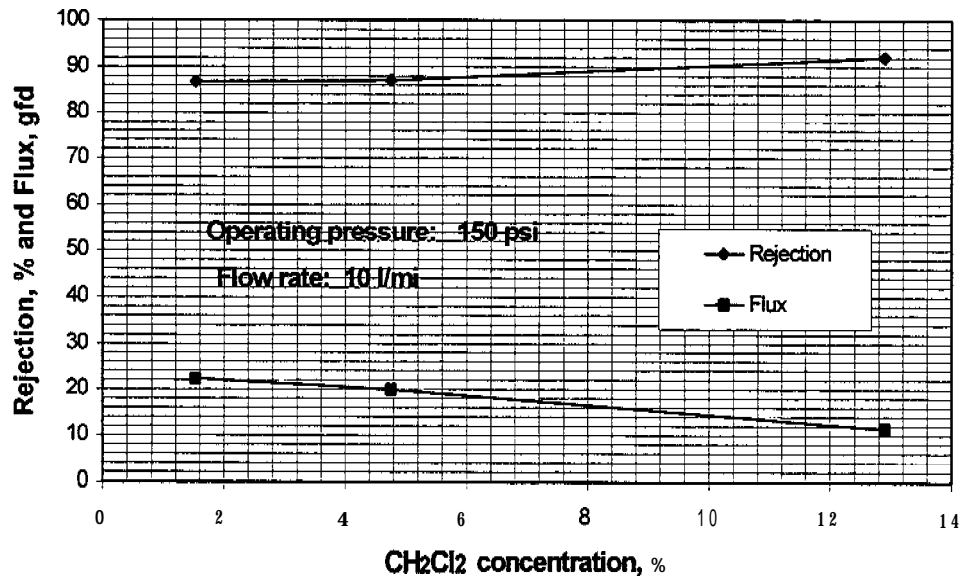
Figure 2-3.—The effect of concentration of dichloromethane in the SPS coating solution.

(JB-16-D-I)

Testing time (hour)
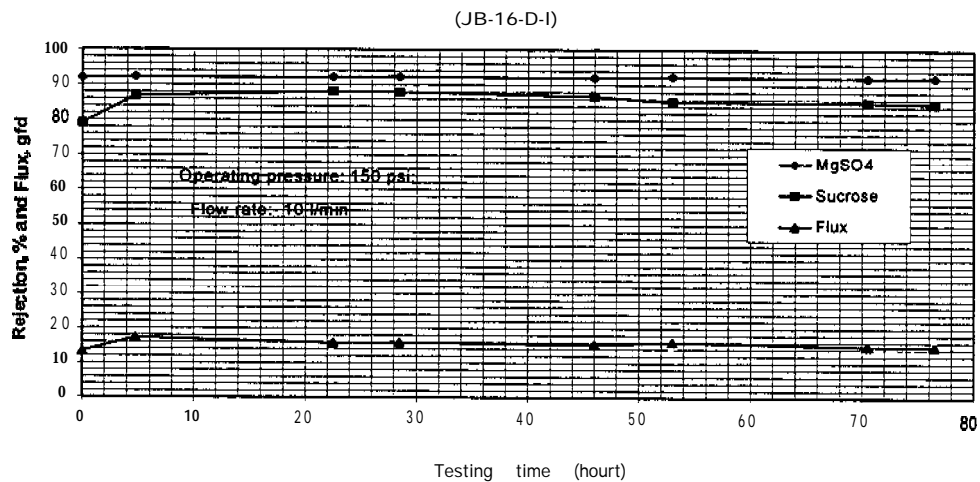
Figure 2-4.—Rejection and flux of a NF membrane using a feed containing 2,000 ppm $MgSO_4$ and 200 ppm sucrose.

These membranes were also tested on solutions which contained 2,000 ppm $MgSO_4$ along with organic compounds of different molecular weights. Results of this study are given in figure 2-5.

NF membrane JB-15-B1 showed the highest rejection of $MgSO_4$ and lowest rejection of Raffmose. The rejection of compounds from best to worst are:

- $MgSO_4$ > Sucrose > Polyethylene glycol (MW 1000) > Raffinose
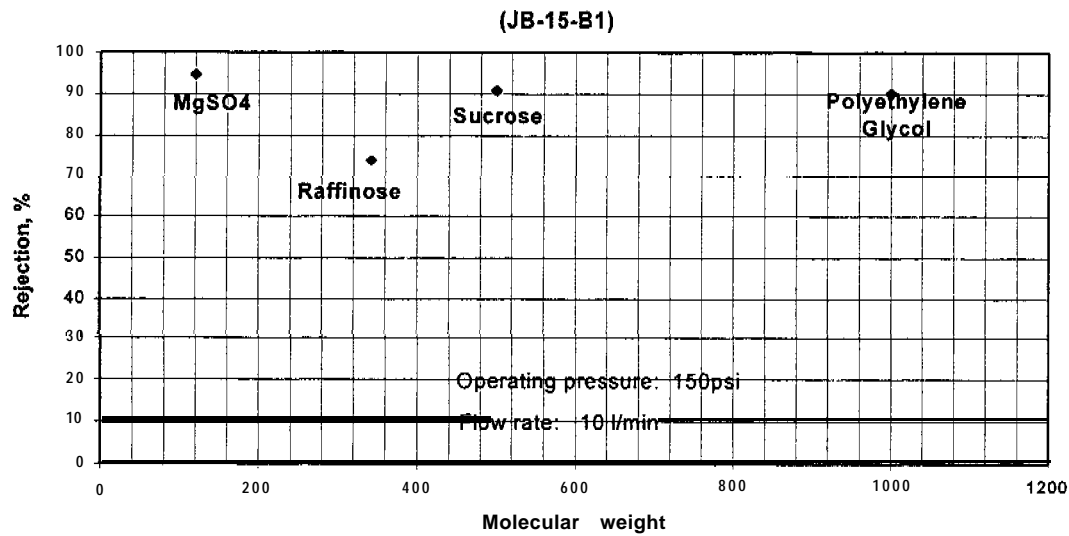
Figure 2-5.—The rejection of $MgSO_4$ and organic compounds.

Testing using a feed solution containing 2,000 ppm $MgSO_4$ and 200 ppm sucrose gave rejection of 92.1% of $MgSO_4$, 84.8% for sucrose, and 14.8 USgfd at the end of 78 hours run at 150 psi.

2.5 Conclusions

A SPS coated NF membrane has been developed. This membrane has a rejection >90% and flux 15 USgfd on 2,000 ppm $MgSO_4$ at 150 psi. A long-term test of this membrane shows that it performs well under various test conditions. This membrane also rejects NaCl and low molecular weight organic compounds.

Chapter 3
DEVELOPMENT OF A MODIFIED NANOFILTRATION MODULE

3.1 Module Design Improvements

A modified nanofiltration module was designed by Zenon which improves upon previous transverse flow concept for brackish water. The design forces feed to flow across, or transversely to, the axis of the fibres, which will take advantage of the principles of higher mass transfer and reduced fouling potential observed in previous generations of Zenon's Moustic™ modules. The newly developed prototype nanofiltration element can be seen in figure 3-1
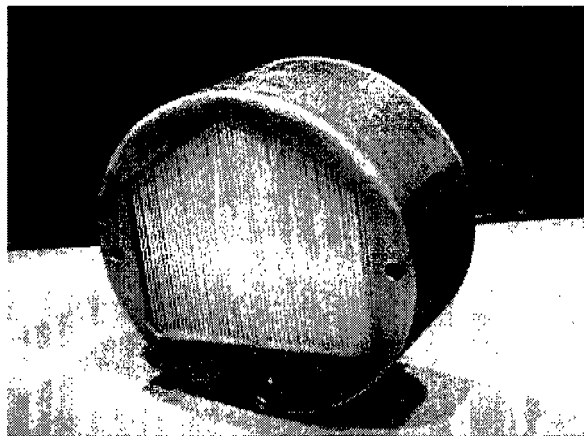
Figure 3-l .--Prototype nanofiltration element.

3.1.1 Prototype Description

The module embodies a true transverse hollow fibre membrane flow pattern. It is sized to fit inside a standard S-inch RO pressure vessel. The module is comprised of numerous elements stacked together within one pressure vessel. With the exception of the permeate collection chamber, the module elements are not pressure-loaded components. Therefore, the frame and shell of each element is solely for the purpose of positioning the fibre membranes and directing the feed flow water.

The periphery of the element is cylindrical in shape and will fit inside an 8-inch pressure vessel. Each element is 3.75 inches high. The feed flow region of the module has an irregular pentagonal shape. The fibres are oriented perpendicular to the flow and open at one end only to withdraw the permeate while the other end is embedded in a potting compound. An element is composed of 26 layers of fibre—each fibre layer containing 5 1 individual

fibres of varying length. The membrane surface area of each fibre layer fitted with 1.2 mm O.D. fibre is approximately 0.263 ft". The packing density of one nanofiltration element with 26 fibre layers is approximately 63 ft^2/ft^3.

As previously mentioned, the element itself is not a pressure vessel. Only the permeate collection chamber is subjected to working pressure loads. The key to the success of this design was to make a permeate collection chamber with sufficient strength to remain integral under working pressures. The new and novel approach to solving this problem is the fibre positioning card. The fibre positioning card, as shown in figure 3-2, serves two purposes: first, it is used in the manufacturing process to position and stabilize the fibre membrane segments during the assembly and potting process. This may be better understood by a description of the manufacturing process. Fibres are placed between the alignment pins on the card, the top row of pins direct the fibres to the permeate collection area, and the bottom row of pins ensure the fibres remain parallel and tensioned during the potting process. Fibres are lightly adhered to the card with hot melt adhesive. Second, the fibre positioning card serves as a reinforcing rib within the permeate chamber. The top edge of the card is designed to fit against the inside surface of the element housing. The top edge of the card has been reinforced since it will be subjected to working pressure loads within the permeate chamber.
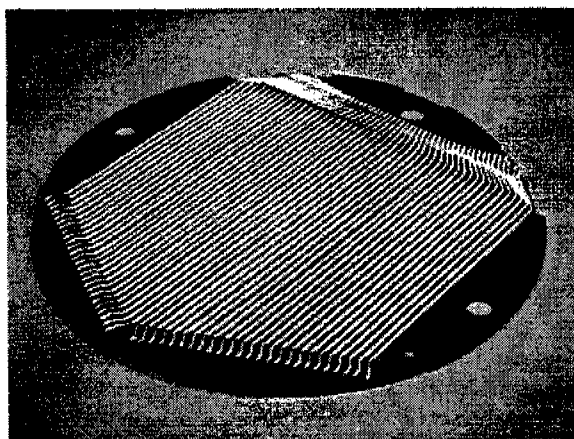
Figure 3-2.—Fibre positioning card
(with fibres in place).

The purpose of the element housing (see figure 3-3) is to contain a stack of fibre positioning cards. The element housing was machined from aluminum for prototype purposes; production versions would be injection molded in a similar material to the fibre positioning card. The element housing has O-ring grooves on the outside surface to seal multiple elements together to assemble a module. The inside of the housing has two grooves which travel the entire perimeter of the urethane sealing area. This feature was added to strengthen the urethane seal by increasing the surface area the urethane had to seal to. The ends of the element housing are stepped so they will be embedded in the potting compound to secure
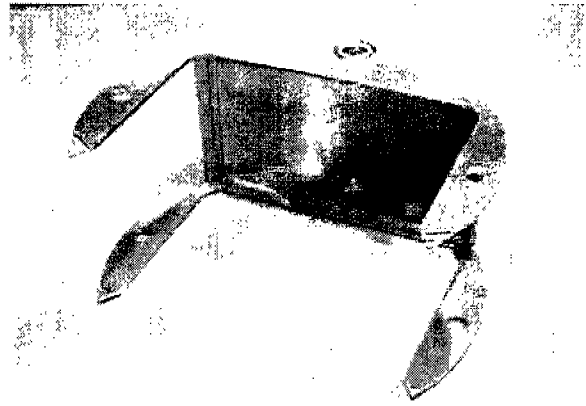
Figure 3-3.—Element housing.

the stack of fibre positioning cards. The holes at the sides are for alignment during potting and to attach multiple elements together. An element is assembled by placing a stack of 26 fibre positioning cards inside the element housing. The permeate collection area is then filled with hot liquid wax that will solidify at room temperature so that the ends of the fibres are just submerged. When the wax has solidified, the potting compound is poured on top so the fibres become sealed within the element housing. When the potting compound has cured, the wax is melted out of the permeate chamber. The other ends of the fibres are embedded in the potting compound to completely seal the fibre ends and secure the ends of the element housing to the stack of cards. A labeled completed nanofiltration element is illustrated in figure 3-4.

Polyurethane Sealed Fibre Ends

Nanofiltration Membrane Coated Fibre

Alignment / Module Assembly holes

Polyurethane Sealing Area

Permeate Chamber

Permeate Collection Hole

Figure 3-4.—Labeled nanofiltration element.

3-3

3.1.2 Process Advantages

It is estimated that the cost to manufacture these modules would be approximately the same or less than the cost of manufacturing our tubular membrane products on a $ per square foot basis. Packing density is higher than tubes but lower than spiral wound modules; however this may be more than offset by potential performance advantages. These include;

- A uniform flow velocity over all fibres, which assures uniform flow distribution and eliminates low velocity zones.
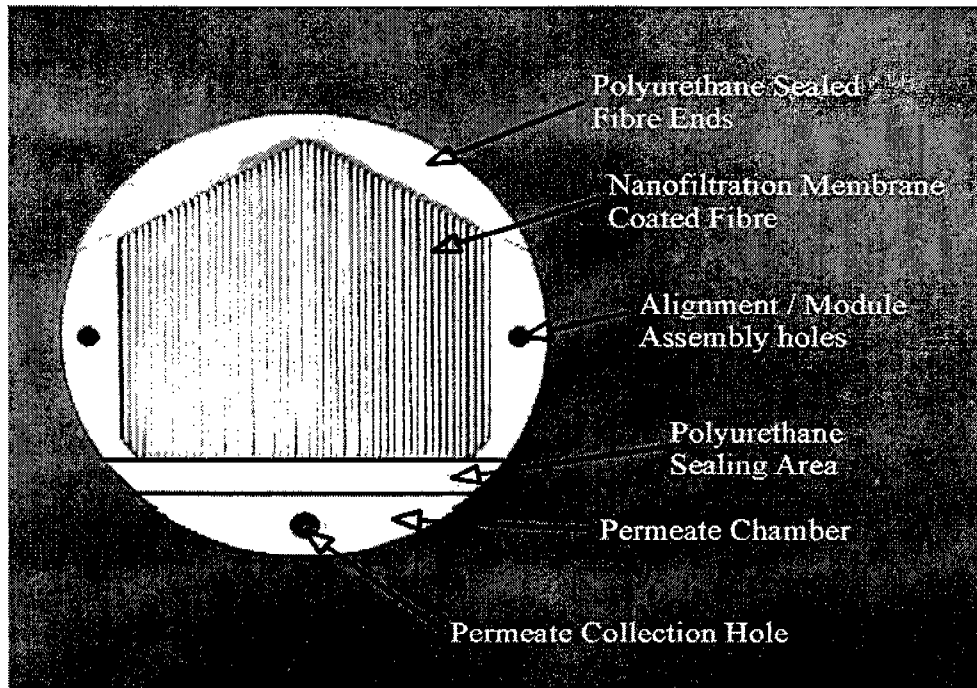
- True transverse flow design offers benefits of higher tolerance to solids and reduced fouling potential.

- Significantly higher Reynolds number than the previous transverse flow design, which serves to increase boundary layer mass transfer efficiency and results in higher solute rejections.

- Cylindrical design is well suited for higher pressures.

- Use of 8-inch pressure vessel design maximizes potential for retrofit market.

- Design maximizes the packing density achievable using true transverse flow principles, if modules are designed to fit into conventional pressure vessels.

- Significantly lower pressure drop than spiral modules when configured in series would allow higher overall recoveries.

3.2 Results of Pressure Testing

Initial air bubble testing at 20 psi of individual elements identified leaks at the urethane-element housing interface. Due to the smooth surface of the aluminum housing and the tight clearance between the fibre card stack, pinholes between the wall of the element housing and the urethane developed. Grooves on the inside surface of the element housing were added to increase the surface area the urethane had to adhere to. Subsequent bubble testing at 20 psi of an element with a modified housing was successful with no leaks in the urethane potting.

A single element was then assembled with inlet/outlet headers in an 8-inch pressure vessel. The vessel and the feed tank were filled with a crystal violet die so that any leaks in the module could be identified by coloured permeate. The vessel was pressurized to a line pressure of 40 psi to check for assembly leaks. The module showed no signs of leaks at 40 psi. With a static pressure tester, the pressure was increased to from 40 psi to 150 psi. The permeate became discoloured after the pressure reached 150 psi. The module was disassembled from the pressure vessel and inspected for major defects or delamination- No major defects were found. The module was bubble tested at 20 psi, which identified the leak point at an O-ring.

3.3 Conclusions

Although the module leaked after 150 psi, the urethane potting and permeate chamber were able to withstand the pressure. The headers are attached to the element housing using the two assembly alignment holes on the outer circumference of the housing. These two points are a significant distance form the O-ring and therefore do not provide optimum compression of the O-ring. The O-ring leak can be fixed by adding more compression between the headers and the element. This will require redesigning the element housing and the fibre positioning card to include more holes on the outer circumference of the housing.

Chapter 4
TECHNICAL AND ECONOMIC EVALUATION

4.1 Process Economics and Commercialization Potential

The major advantages of a module design of this type are the lower operating costs associated with higher mass transfer, lower pressure drop, and reduced pretreatment requirements and fouling potential.

The packing density of the prototype element is 63 ft^2/ft^3, which is significantly lower than commercially available spiral wound modules which have a packing densities in the range of 275 ft^2/ft^3. This is difficult to overcome in a transverse module design. The packing density could increase by reducing the spacing of the fibres, fitting more fibres on a positioning card, and reducing the thickness of the positioning cards to fit more cards in an element. However, it is important during the second dip coating that no fibres are touching each other so these changes would only increase the packing density approximately 25%. Longer fibre length would increase the packing density and decrease the manufacturing cost per unit area. The only way to significantly increase the length of fibre is to orient the fibres parallel to the axis of the module, but this eliminates the hydrodynamic advantages to the transverse design.

At this point in time, this module would not be commercially competitive with alternative available products on the market because of the capital investment required to manufacture such a module and the relatively low membrane packing density of this design. A more cost-effective solution to the problem of nanofiltration of brackish surface waters would be to use microfiltration as a pretreatment to conventional spiral wound nanofiltration. We believe Zenon's ZeeWeed® technology would be the most effective micro prefilter for this application,

4.2 Manufacturing Requirements

In order to manufacture a nanofiltration module of this type with repeatability and minimal defects that would be competitive with our existing technology requires the accuracy of automation. The fibre coating process would have to be fully automated to handle the volume of fibre required to produce large numbers of modules. The fibre coating line would most likely feed an automated machine which would lay-up the fibre on the fibre positioning card, glue it, and cut off the excess fibre. This step in the process requires repeatable accuracy and can be very tedious to do manually. At this point, the fibre positioning cards could be automatically stacked and assembled into an element housing and placed on another machine that would accurately dispense the wax into the permeate chamber. The element would then have to be cooled or left long enough for the wax to fully harden. Another automated machine would then accurately dispense urethane on top of the wax to seal off the permeate chamber. Once the urethane was cured, the other end of the fibres would have to be

sealed off in a two-step process once again by an automated machine. When the urethane was cured, the wax would need to be removed from the permeate chamber and the element could be quality checked by a fully automated test station. After testing, zero defect modules receive a second coating most likely by an automated dip process. The modules consisting of multiple elements would have to be assembled by hand and put on a system for final testing before being packaged as a finished product.

4.3 Development Needs

Before investment into a fully automated process, further module development is required to produce a more robust module design that can withstand higher operating pressures. Also, a reliable automated nanofiltration membrane coating process is required to determine whether the volume of fibre required can be produced for a full-scale production process.

Chapter 5
GOALS AND ACCOMPLISHMENTS

The ultimate goals of Phase I and II of this development effort were to:

- Develop an improved (higher tensile strength) nanofiltration hollow fiber membrane for brackish water applications (approximately 2,000 ppm total solute).

- Develop a transverse flow module for operation at higher pressures (150 - 225 psi).

- Assess the overall applicability and commercialization potential of the technology.

5.1 Nanofiltration Hollow Fiber Development

In Phase I, a polysulfone base fiber coated with sulfonatedpolysulfone was developed, which provided a flux of 13 USgfd and a rejection of 70% on 2,000 ppm $MgSO_4$ solution at 200 psi test pressure.

In Phase II, the following improvements were made to the base fiber and membrane formulation:

- Significantly improved fibers with a flux of 20 USgfd and a rejection of >90% on 2,000 ppm $MgSO_4$ solution at 150 psi have been developed.

- Long-term testing of this membrane showed good performance under various test conditions, and the membrane also showed rejection of $NaCl$ and other low molecular weight organic compounds.

5.2 Transverse Flow Module Development

In Phase I, a square transverse flow module prototype was constructed of materials which could potentially withstand operating pressures of up to 200. Testing of the fibers in module configuration indicated that fluxes of about 5.5 gfd were obtained at 200 psi, with rejection of about 75% at 200 psi.

The primary focus of Phase II was to improve the packing density and flux to improve productivity per module, improve flow distribution to minimize channeling, and modify design to reduce manufacturing costs.

In Phase II, the following significant accomplishments were achieved:

- An entirely reconfigured transverse flow module was designed using stereo lithography. The principle is based on a stack of "fibre positioning cards," which are positioned within an element housing to form a module containing 16 "cards."

- The module was designed to fit inside a standard 8-inch RO pressure vessel, which would facilitate market acceptance and potential for retrofit market. The novel design concept embodies an element which is not a pressure vessel in itself. Only the permeate collection chamber is subjected to working pressure loads.

- A prototype module was constructed using mold injection technology and subjected to pressure testing to assess its tolerance. The module was able to withstand a pressure up to 150 psi. A leak at that point was attributed to a leak point at an O-ring. Further investigation revealed that this could be resolved by adding more compression between the headers and the element, which will require re-designing the element housing and the fiber positioning card to include more holes on the outer circumference of the housing.

- The packing density was increased to 63 ft^2/ft^3. While this is still lower than commercially available spiral modules which have packing densities in the 250-275 ft^2/ft^3 range, this is a significant improvement from the last module generation. It is anticipated that further refinements could result in an additional 25% improvement; however, this is would be near the limit since the dip coating method envisaged for this module requires that no fibres are touching each other.

- It is estimated that the cost to manufacture these modules would be approximately the same or less than the cost of manufacturing our tubular membrane products on a $ per square foot basis. However, due to the level of automation required, and the cost of competing nanofiltration membranes, commercial production of this product is not yet viable because of the capital investment requirement to manufacture such a module.

- Actual throughput using the improved fibres on brackish water was not measured as of yet. The actual throughput or productivity of this module needs to be tested after pressure tolerance is achieved.

- The major advantages of this type of design are lower operating costs associated with higher mass transfer, lower pressure drop, and reduced pretreatment requirements, which could significantly impact overall process costs. Once performance data is generated, the final commercial viability can be determined.

Chapter 6
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Appendix A
DATA EXPERIMENTAL

TABLE A

The effect of rejection and flux on concentration of dichloromethane in the SPS coating solution

Membranes O.D. (mm)	Length (cm)	CH2Cl2 concentration (%)	Permeate rate (ml/min)	flux (gfd)	Permeability gfd/psi	Feed cond.	Permeate cond.	Rejection (%)
1.28	67	1.53	22/13	22.2	0.150	2.110	0.281	86.7
1.28	68	4.76	37/24	19.9	0.112	1.910	0.177	87.0
1.28	71	12.9	11/12	11.4	0.076	1.950	0.155	92.1

TABLE B

Rejection and flux of NF membrane using a feed containing 1000 ppm MgSO4 and 200 ppm sucrose

Fibers O.D.　0.120 mm
Fibers Length　69.0 cm

Testing time (hours)	Permeate rate (ml/min.)	Flux (gfd)	Permeability (gfd/psi)	MgSO4 Feed	MgSO4 Permeate	MgSO4 Rejection	Sucrose Feed	Sucrose Permeate
0	39/40	13.2	0.088	1.686	0.137	91.9	62.0	13.0
4.75	47/37	17.2	0.115	1.686	0.134	92.1	64.5	8.5
22.5	74/64	15.7	0.105	1.680	0.132	92.1	69.0	8.2
28.5	64/55	15.8	0.105	1.684	0.130	92.2	69.0	8.3
46.0	61/54	15.3	0.102	1.684	0.136	92.0	71.0	9.4
53.0	47/40	16.0	0.107	1.700	0.130	92.4	68.0	10.0
70.5	91/83	14.9	0.099	1.705	0.137	92.0	68.0	10.0
76.5	47/43	14.8	0.099	1.700	0.135	92.1	69.5	10.5

TABLE C

The rejection of MgSO4 and organic compounds

Organic Compounds	M.W.	Feed	Permeate	Rejection, %
MgSO4	120	1.84*	0.099*	94.6
Sucrose	342.3	58**	5.3**	90.8
Raffinose pentahydrate	594.5	71	18.4	74.1
Polyethylene glycol	1000	73	7.2	90.2

* Conductivity
** Total carbon analysis peak height

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This project was the second phase in the development of a novel Moustic™ transverse flow follow fiber nanofiltration module fo desalination applications. The two phases resulted in the development of a high tensile strength fiber suitable for brackish wate application, production of suitable membranes, and redesign of the existing transverse flow concept for high pressure applications. In phase I (WTTP #9), high tensile strength fibers were successfully developed, along with a chlorine resistant nauofiltration membrane with suitable solute rejection characteristics. In phase II, an improved module was successfully developed by maximizing surface area/volume ratio to improve productivity per module, flow distribution was optimized to minimize channeling, and the module desig was modified to reduce manufacturing costs.

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