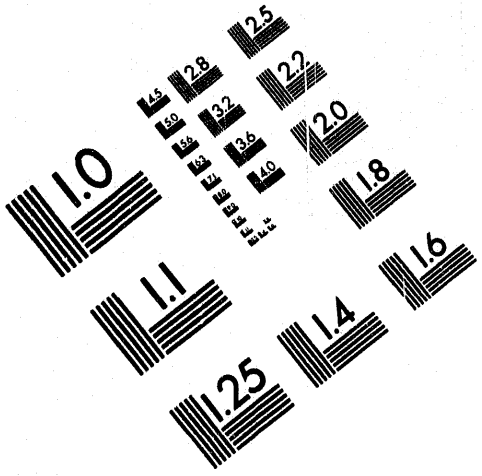

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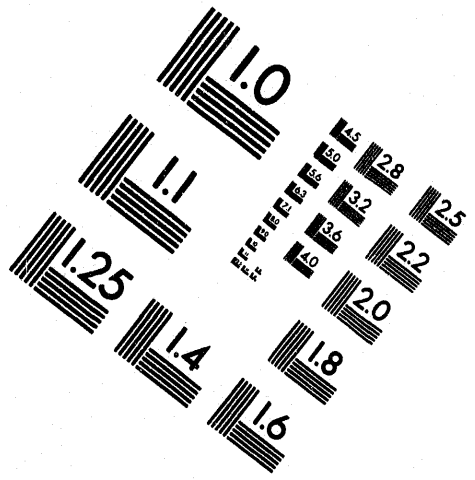

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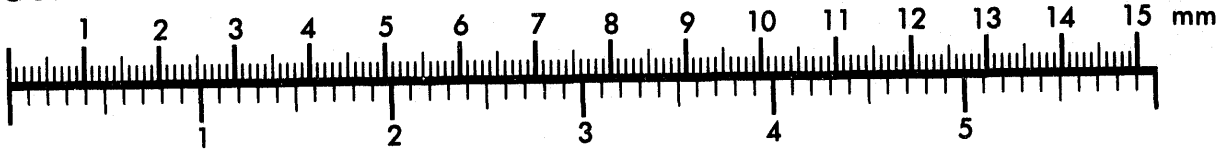

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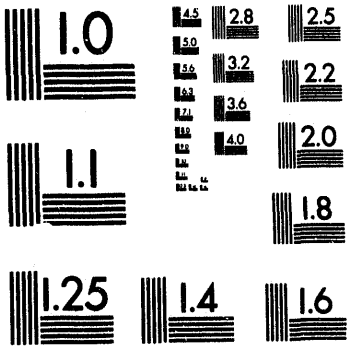

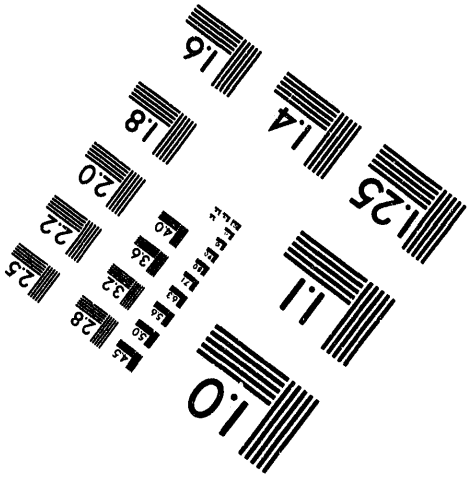

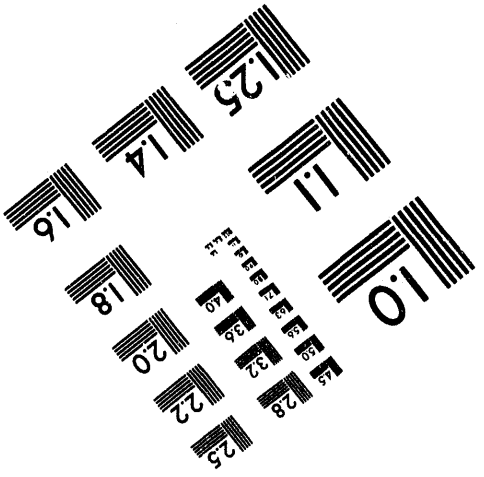

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DEVELOPMENT OF A MEMBRANE-BASED PROCESS FOR THE TREATMENT OF OILY WASTE WATERS

Contract No. DE-AC22-92MT92005

March 11, 1994

Contract Date: March 4, 1992
Anticipated Completion Date: May 4, 1994
Government Award for FY1992: [redacted]
Program Manager: Scott B. McCray
Principal Investigator: Scott B. McCray
Technical Project Officer (COR): Gene Pauling
Reporting Period: September 5 to December 4, 1993

from
Bend Research, Inc.
64550 Research Road
Bend, Oregon 97701-8599

NOTE: US/DOE Patent Clearance is **not** required prior to the publication of this document.

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None.

EXECUTIVE SUMMARY

This is a quarterly report from Bend Research, Inc., (BRI) to the U.S. Department of Energy (DOE) for work performed under Contract No. DE-AC22-92MT92005, titled "Development of a Membrane-Based Process for the Treatment of Oily Waste Waters." This report covers the period from September 5 to December 4, 1993.

The overall goal of this program is to develop a system based on reverse-osmosis (RO) membranes that can treat oily water economically. This system will be based on the use of thin-film-composite (TFC) membranes that consist of a selective coating placed on a solvent-resistant hollow-fiber support. For this program, we plan to develop solvent-resistant hollow-fiber supports and coat them with a "loose RO" coating. We developed the coating, which is designated TTM, in previous work for the treatment of oily waste waters.

During this reporting period, work was focused on operating the demonstration unit at a test site near Houston, Texas. During the next reporting period, we plan to prepare a draft of the final report for this program.

I. INTRODUCTION

This is a quarterly report from Bend Research, Inc., (BRI) to the U.S. Department of Energy (DOE) for work performed under Contract No. DE-AC22-92MT92005, titled "Development of a Membrane-Based Process for the Treatment of Oily Waste Waters." The purpose of this program is to develop a system based on reverse-osmosis (RO) membranes that can treat oily water economically. This system will be based on the use of thin-film-composite (TFC) membranes that consist of a selective coating placed on a solvent-resistant hollow-fiber support. For this program, we plan to develop solvent-resistant hollow-fiber supports and coat them with a "loose RO" coating. We developed the coating, designated TTM, in previous work for the treatment of oily waste waters. This report covers the period from September 5 to December 4, 1993.

During this reporting period, work was focused on performing a demonstration test of this technology. The demonstration unit was operated for more than 2000 hours. Over this time, the permeate produced by the modules contained less than 10 ppm oil and grease, meeting all current discharge regulations. From this standpoint, the demonstration test was a success.

However, several operational problems were encountered that affected the performance of the unit. First, scale formation, caused by a surge of scale-forming contaminants, led to a decrease in membrane performance. After cleaning, membrane performance returned to original values. Problems were also encountered with the prefiltration system; the filters plugged, which caused the feed-flow rate to decline. This also led to a decrease in membrane performance. We believe that by improving the design of the prefiltration system and improving control of operating parameters, the membranes would have maintained high performance throughout the demonstration test. At the end of this reporting period, the demonstration test had been halted.

II. PROJECT DESCRIPTION

This program is divided into seven tasks. A summary of the work to be performed in this program follows.

Task 1: Develop Hollow Fibers

Hollow-fiber supports will be made from high-strength, solvent-resistant polymers. These fibers will be physically robust and have 1) high permeability to assure high fluxes, and 2) smooth lumen (interior) surfaces to assure uniform coating of the hollow-fiber support with the permselective coating.

Task 2: Evaluate Hollow Fibers

The fibers made in Task 1 will be evaluated in a series of tests to ensure they are suitable for this application. These tests include 1) examining the texture of the lumen surface, 2) measuring the permeability of the fibers, 3) measuring the strength of the fibers, and 4) measuring the solvent resistance of the fibers.

Task 3: Make Small-Scale Modules

Small-scale hollow-fiber modules will be made using the fibers, and the lumens of the fibers will be coated with our loose-RO TTM coatings. The modules will be evaluated in standard tests to ensure the integrity of the coating.

Task 4: Test Small-Scale Modules

The small-scale modules made in Task 3 will be tested in the laboratory under controlled conditions. The modules will be evaluated on feed streams of oily waste waters obtained from various sources. Parametric and long-term tests will be conducted.

Task 5: Construct Large-Scale Modules

Large-scale modules will be constructed using standard module-preparation procedures developed at Bend Research. These modules will be constructed for use in a demonstration unit to be provided by the contractor.

Task 6: Operate Demonstration Unit

The effectiveness of this technology will be demonstrated by operating the large-scale modules on actual oily waste waters. Field-test sites will be identified and prepared

for the demonstration of this technology. A test plan will be developed before the demonstration is performed.

Task 7: Technology Transfer

Journal articles and conference and symposia presentations will be prepared. This technology will be transferred to the oil and gas industry and the scientific community.

III. PROJECT STATUS

III.A. Demonstration Unit

Figure 1 shows a simplified schematic of the demonstration unit. The feed to this unit is the effluent from a small three-phase separator.

III.B. Demonstration Tests

During this reporting period, we continued long-term tests of the demonstration unit using three hollow-fiber modules operated in parallel. The demonstration unit was operated for more than 2000 hours.

During the long-term tests, the modules were operated continuously without an operator at the test site. Data were collected by a remote sensing package and transmitted to a computer located off-site. Periodically, the data were examined by a technician and at least once a week an operator traveled to the test site to ensure all systems were operating properly. This type of operation is inexpensive, but allows only limited control over the system. Unfortunately, by the time difficulties in operation are encountered, it is often too late to take corrective measures.

Figure 2 shows the water flux through the hollow-fiber modules as a function of time during the demonstration test. As these data show, the flux varied significantly during the test. The reasons for this variation in flux are described below.

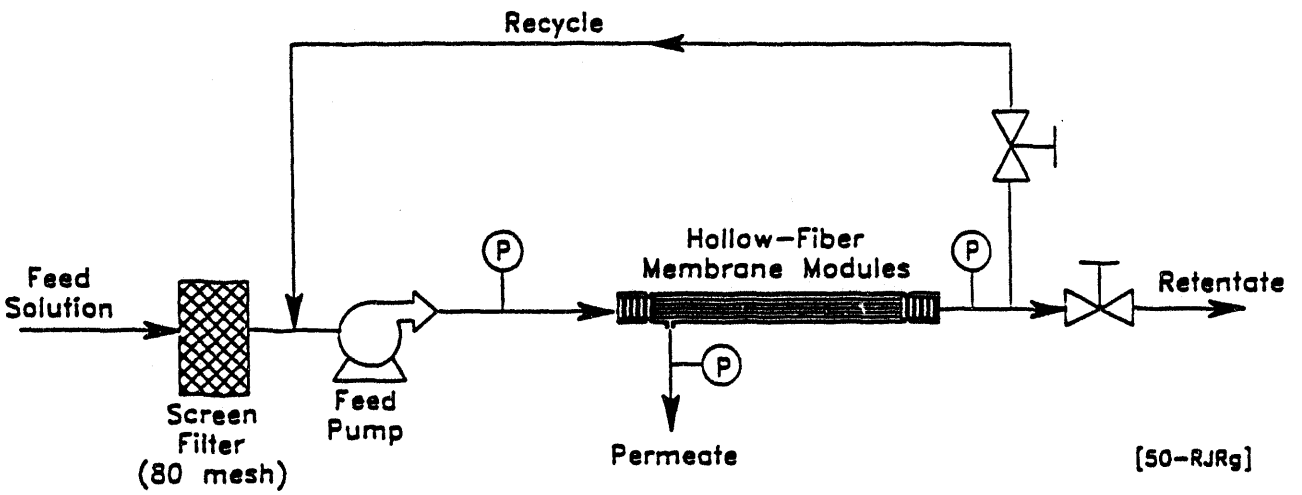

Figure 1. Simplified Schematic of the Demonstration Unit

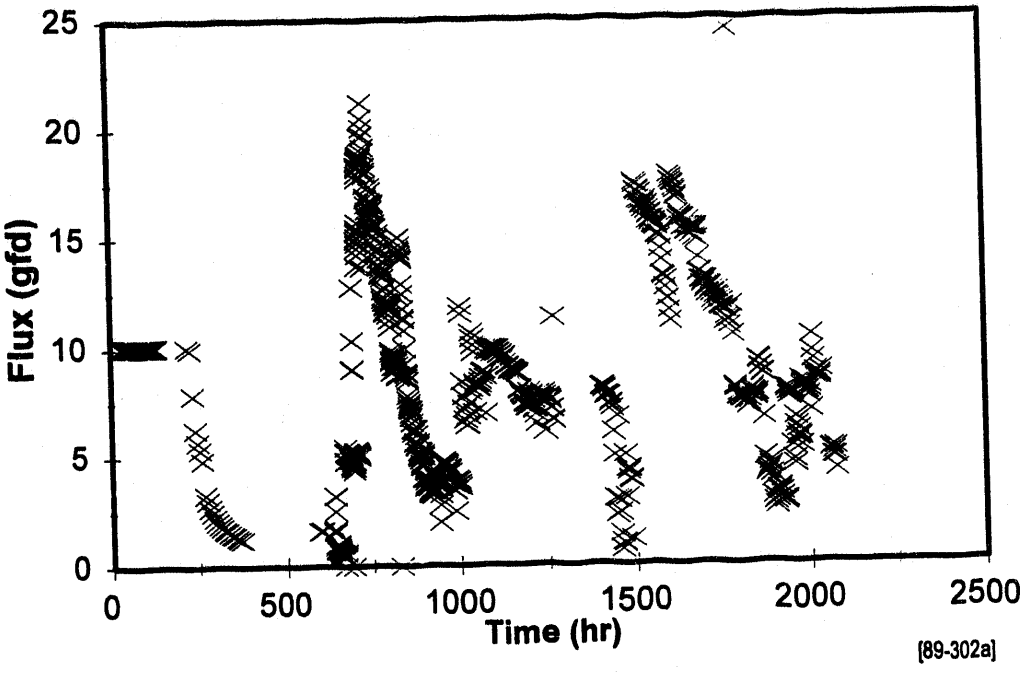

Figure 2. Water Flux Versus Time During Long-Term Test

Conditions: Feed: ~10,000 ppm salts (mostly Ca^{+2})
~1,000 ppm oil and grease
120 - 150 psig
Permeate: <7 ppm oil and grease

As the data in Figure 2 show, initially (during the first 200 hours) the flux through the modules remained constant at 10 gal/ft^2-day (gfd). This was because 1) the feed solution did not change appreciably over this time period, and 2) the operating conditions were adjusted to produce constant flux.

However, at about 200 hours, a surge of scale-forming contaminants entered the system. This led to scale formation on the membrane surface and a loss in flux. The reason for the loss in flux was not identified until about 600 hours; at that time the modules were cleaned with an acid solution to remove the scale. This cleaning restored the flux through the membrane.

The data in Figure 2 show that after cleaning (at about 700 hours), the flux through the membrane again decreased with time. This was due to variations in feed-flow rate, as shown in Figure 3. Indeed, at about 750 hours, the feed-flow rate to the modules accidentally dropped to 0, leading to 100% recovery in the system (Figure 4). When this occurred, a concentrated layer of oil formed on the membrane surface and a subsequent loss in flux occurred. However, when the feed-flow rate was restored to the original value (about 10 gallons per minute [gpm]), the flux stabilized.

At about 1400 hours of operation, a high concentration of silt entered the system, plugging the prefilters. This led to a decrease in feed-flow rate and a subsequent loss in flux. Silt remained a problem for the remainder of the test, leading to erratic feed-flow rates and fluxes.

Periodically during the long-term test, samples of the permeate stream were analyzed for oil and grease. In all cases, the permeate contained less than 10 ppm oil and grease. This concentration meets all existing discharge regulations.

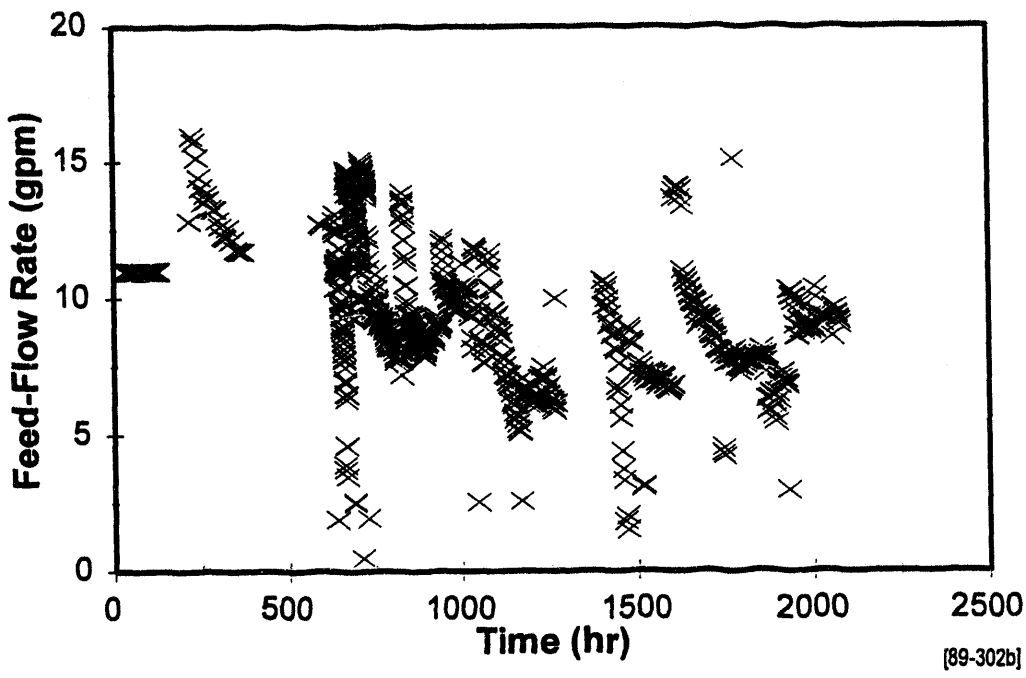

Figure 3. Feed-Flow Rate Versus Time During Long-Term Test

Conditions:	Feed:	~10,000 ppm salts (mostly Ca^{+2})
		~1,000 ppm oil and grease
		120 - 150 psig
	Permeate:	<7 ppm oil and grease

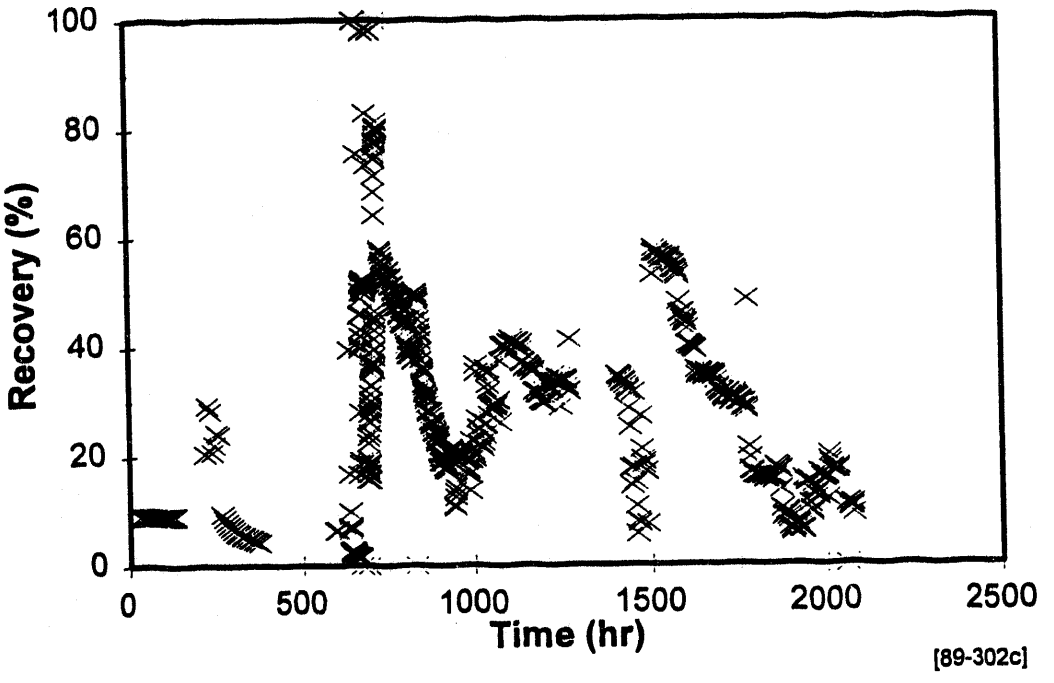

Figure 4. Recovery Versus Time During Long-Term Test

Conditions:	Feed:	~10,000 ppm salts (mostly Ca^{+2})
		~1,000 ppm oil and grease
		120 - 150 psig
	Permeate:	<7 ppm oil and grease

III.C. Conclusions Regarding Long-Term Test

The long-term tests of the hollow-fiber membrane modules were successful in that the modules produced permeate water of sufficient quality to meet all discharge regulations. However, the fluxes through the modules varied considerably during the test due to changes in feed composition and changes in operating conditions. The data indicate that a more-extensive pretreatment system (other than coarse filtration) will be needed to make this technology commercially viable.

Specific recommendations that would improve the performance of the system include the following.

1. A settling system should be located upstream of the unit to prevent larger particulates from entering the system. Such a settling system would also minimize the frequency at which a prefilter would need to be changed and/or cleaned.

2. Many oily-water treatment systems use induced gas flotation (IGF) to remove much of the free and emulsified oil from oily wastewaters. The hollow-fiber membrane technology studied here could treat the effluent from an IGF unit (which often does not meet discharge regulations), producing a permeate water that could be discharged. In some cases, the retentate from the membranes could be recycled back to the IGF unit, leading to high water recoveries.
3. The hollow-fiber membranes should be tested on a unit equipped with process control. This would prevent the membrane system from operating when conditions are undesirable (e.g., low feed flow).

We believe that these changes will improve the performance of the membranes, particularly regarding the flux through the system.

IV. PLANNED ACTIVITIES

During the next reporting period, we plan to prepare a draft of the final report. This is the only task remaining in this program.

V. SUMMARY

During this reporting period, we completed long-term tests of the demonstration unit at a test site near Houston, Texas. The test has now been completed and the demonstration unit has been returned to our commercialization partner, AquaAir Environmental.

Report Distribution List

Three copies to: Document Control Center, U.S. Department of Energy
Pittsburgh Energy Technology Center, P.O. Box
10940, MS 921-118, Pittsburgh, PA 15236-0940

References

None.

Publications

No new publications or presentations of this technology were prepared during this reporting period.

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