Appendix 2.3 B

Task 2.3 B: Identification of Factors that Influence Fiber Breakage

Submitted by:

Amy E. Childress
University of Nevada, Reno
Department of Civil Engineering

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Legal Notice

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Preface

The Public Interest Energy Research (PIER) Program supports public interest energy research and development that will help improve the quality of life in California by bringing environmentally safe, affordable, and reliable energy services and products to the marketplace.

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- Buildings End-Use Energy Efficiency
- Industrial/Agricultural/Water End-Use Energy efficiency
- Renewable Energy
- Environmentally-Preferred Advanced Generation
- Energy-Related Environmental Research
- Strategic Energy Research

What follows is the final report for *Electrotechnology Applications for Potable Water Production and Protection of the Environment*, Contract No. 500-97-044**,** conducted by the Metropolitan Water District of Southern California. The report is entitled "Electrotechnology Applications for Potable Water Production and Protection of the Environment: Task 8 Identification of Factors that Influence Fiber Breakage." This project contributes to the Industrial/Agricultural/Water End-Use Energy Efficiency area.

For more information on the PIER Program, please visit the Commission's Web site at: http://www.energy.ca.gov/research/index/html or contact the Commission's Publications Unit at 916-654-5200.

Executive Summary

Introduction

The use of microfiltration (MF) or ultrafiltration (UF) followed by reverse osmosis (RO) has become the industry standard for demineralization of municipal secondary effluent in high quality industrial reuse applications and groundwater recharge projects.

Purpose

Microporous membranes provide exceptional pretreatment for RO provided the MF or UF membrane fibers maintain their integrity. This study was initiated to identify any correlation between membrane and module properties and membrane fiber failure (i.e., loss of integrity).

Project Objectives

Module potting technique, membrane symmetry, fiber modulus of elasticity, fiber thickness, module flow pattern (inside-out or outside-in), and membrane material were investigated. The approach combined mathematical modeling of structure-fluid interactions with analysis of membrane failure made at the Orange County Water District (OCWD) pilot- and demonstration-scale facility.

Project Outcomes

SEMs images were created for the five membrane fibers. Tensile testing results of the hollow fiber membranes was performed. A structural and a fluid model of a dynamically potted module with an elastomer overlay were developed using the symmetry, thickness, and strength data.

Conclusions

Correlations between membrane and module properties and membrane fiber failure (i.e., loss of integrity) were difficult to make because only two membrane fibers (the PM100s and PVDF fibers) underwent both materials testing and performance testing. Preliminary modeling results

found the existence of additional stresses at the fiber/potting juncture which might possibly lead to the formation of fractures.

Commercialization Potential

Information acquired from the study of microporous membrane fiber integrity will assist with the development and manufacture of longer lasting MF and UF membranes and modules for the reclamation market. Reductions in operation and maintenance costs associated with loss of membrane fiber integrity will allow this technology to be more competitive with other less effective existing technologies.

Recommendations

Future efforts should include the evaluation of immersed hollow fiber membranes as well as evaluation of the impact of backwashing (using both air and water) on hollow fiber membrane integrity. The current model for pressure-driven membranes could be modified to evaluate suction-driven membranes or to evaluate the effects of air and water backwashing. Similar to the current investigation, results from the modified structure-fluid model would be combined with analysis of membrane failure for OCWD membrane systems.

Benefits to California

Preventing microporous fiber breakage will have a significant effect on water treatment and wastewater reclamation in California and throughout the world. The performance of reverse osmosis membranes in indirect potable reuse and the efficacy of disinfection processes (chlorination and ultraviolet irradiation) in direct non-potable reuse are directly dependent on MF and UF fiber integrity.

Abstract

In order to identify any correlation between membrane and module properties and membrane fiber failure (i.e., loss of integrity), module potting technique, membrane symmetry, fiber modulus of elasticity, fiber thickness, module flow pattern (inside-out or outside-in), and membrane material were investigated. The approach combines mathematical modeling of structure-fluid interactions with analysis of membrane failure made at the Orange County Water District (OCWD) pilot- and demonstration-scale facility. The performance of five membrane modules were tested. Fibers from two of these modules were supplied for scanning electron microscope (SEM) analysis and tensile testing. Additionally, fibers from three membrane modules that were not tested for performance were supplied for SEM analysis and tensile testing. Therefore, a total of five fibers underwent SEM analysis and tensile testing. The strongest fibers were the polyacrylonitrile (PAN) and the polyvinylidene (PVDF) membrane fibers. The weakest fiber was the polyethylene (PE) membrane fiber. The PM fibers have intermediate strengths

The membrane module was modeled using ADINA, Automatic Dynamic Incremental Nonlinear Analysis, which models time-dependent structure-fluid interactions using finite element analysis. The structural model, ADINA, was used to determine structural deformations. The structural model is composed of a porous pipe, representing the hollow fiber, and a block, representing the potting material. It was found that additional stresses at the juncture of the potting material and the hollow fiber membranes exist. These stresses likely lead to the formation of fractures.

Introduction

Background and Overview

The use of microfiltration (MF) or ultrafiltration (UF) followed by reverse osmosis (RO) has become the industry standard for demineralization of municipal secondary effluent in high quality industrial reuse applications and groundwater recharge projects. Microporous membranes provide exceptional pretreatment for RO provided the MF or UF membrane fibers maintain their integrity. Generally, the incidence of fiber failure can be divided into four categories: damage by chemical attack (oxidation), damage during operation resulting from faulty installation (compression), damage due to the presence of foreign bodies (scoring and cleaving), and damage due to faulty membrane/module structure (stress/strain). Failure due to oxidation can be easily attributed to the incompatibility of chemicals in the feed water with the membrane material. Similarly, failure due to scoring and cleaving can be readily identified by examining a failed module. However, failure resulting from an external load, applied under normal operating conditions, necessitates identification of load points and shear together with consideration of the modulus of elasticity of the membrane and potting materials.

It is typically thought that mechanical properties are not very important in membrane processes because the membrane is held by a supporting material. However, hollow fibers are self supporting and therefore the mechanical properties become more important. For example, when a high pressure is applied to a fiber of a low tensile modulus material, the fiber will break. However, a material with a high tensile modulus can easily withstand higher pressures. With the proper choice of fiber diameter and wall thickness, the fiber can withstand substantially higher pressures (Mulder 1991).

The microporous membrane modules consist of 3,000 to 20,000 hollow fibers that are held in place with a resin. The resin, which is usually an epoxy or urethane, can be cured statically or dynamically (Figure 1). The statically potted resin tends to wick up the fibers by capillary forces, so that a sharp edge may form at the fiber surface (Figure 1a). This sharp edge may contribute to breakage of the fiber. An elastomer overlay may be applied to reduce this edge effect (Figure 1b). Additionally, a dynamic potting procedure can be used to minimize the sharp edges (Figure 1c). Membrane symmetry may also play a significant role in maintaining hollow fiber integrity.

Project Objectives

This study was initiated to identify any correlation between membrane and module properties and membrane fiber failure (i.e., loss of integrity). Specifically, module potting technique, membrane symmetry, fiber modulus of elasticity, fiber thickness, module flow pattern (inside-out or outside-in), and membrane material are being investigated. The approach combines mathematical modeling of structure-fluid interactions with analysis of membrane failure made at the Orange County Water District (OCWD) pilot- and demonstration-scale facility.

Project Approach

Experimental Approach

The performance of five membrane modules were tested (Table 1). Fibers from two of these modules were supplied for scanning electron microscope (SEM) analysis and tensile testing. Additionally, fibers from three membrane modules that were not tested for performance were supplied for SEM analysis and tensile testing. Therefore, a total of five fibers underwent SEM analysis and tensile testing.

Table 1. Microporous membrane and module properties

Membrane	A	B	C	D	E	F	G	H	I
	PM 882	PM 100s	PM 100d	PE	PAN 13	PAN 80	PVDF	PP pressure	PP submerse
Type	UF	UF	UF	MF	UF	UF	MF	MF	MF
Material	PS	PS	PS	PE	PAN	PAN	PVDF	PP	PP
Symmetry	A	A	A	S	A	A	S	S	S
Pore Size		100,000 MWCO	100,000 MWCO	0.1 µm	13,000 MWCO	80,000 MWCO	0.1 µm	0.2 µm	0.2 µm
Flow	inside-out	inside-out	inside-out	outside-in	outside-in	outside-in	outside-in	outside-in	outside-in
Potting Type	static	static	dynamic	static	dynamic	dynamic	dynamic	dynamic	dynamic
Elastomer		no	no	yes	yes	yes	no	no	no
Performance Testing	pilot scale	pilot scale	no data	no data	no data	no data	demo scale	pilot scale	pilot scale
Fiber Supplied?	no	yes		yes	yes	yes	yes	no	

Several SEMs images were created for the five membrane fibers. Surface and cross-sectional images of the fibers were evaluated. The hollow fiber symmetry and thickness were confirmed using the cross-sectional images.

Tensile testing of the hollow fiber membranes was performed using Instron testing equipment. The purpose of this testing was to determine how strong the fibers are and how much deformation can be expected given a certain load. In the test, the membrane fiber is held by grips on either end and the elongation is monitored as the fiber is pulled in tension at a constant rate. The result is a load-elongation curve. By normalizing this curve for the fiber geometry (i.e., dividing the load by the original cross-sectional area of the sample and dividing the elongation by the initial length), a stress-strain curve for each membrane fiber is developed. The modulus of elasticity is then determined by the slope of the stress-strain curve in the elastic region. The modulus of elasticity (or Young's modulus) represents the stiffness of the material, or its resistance to elastic strain. This manifests itself as the amount of deformation in normal use below the yield strength.

In the experimental portion of the investigation, clarified secondary effluent provides feed water for the pilot plants. Modules A-G are backwashed at 20-minute intervals for 2 to 3 minutes with MF/UF permeate. In addition, some of the membrane are scoured with air for two minutes at six-hour intervals. Modules H and I are backwashed with air followed by a feed flush every 18 to 20 minutes.

Modeling Approach

The membrane module is being modeled using ADINA, Automatic Dynamic Incremental Nonlinear Analysis, which models time-dependent structure-fluid interactions using finite element analysis. The structural model, ADINA, is used to determine structural deformations. The structural model is composed of a porous pipe, representing the hollow fiber, and a block, representing the potting material (Figure 2). Boundary conditions for the porous pipe include translational degrees of freedom in the transverse directions, while the longitudinal direction has been fixed. It is assumed that rotations do not occur. The potting material has been fixed in all directions. Pressure and distributed loading on the structures are being considered. A preliminary model depicting fracture (large displacement/large strain type of analysis) at the junction between the pipe and potting material is being undertaken in order to model breakthrough. The fluid model, ADINA-F, is used to determine fluid flow. The fluid model is composed of a block of fluid surrounding the pipe. The fluid is assumed to have constant properties, (i.e., constant viscosity, density, and surface tension coefficients). The boundary condition is a prescribed fluid velocity. Iterative solutions are obtained for both the structural and the fluid models. Within one time step, ADINA-F is run until convergence. The loads on the structure due to fluid flow are then passed onto the ADINA model, which is run until it converges. The new structural geometry is then passed back to the ADINA-F model. A user-specified convergence criteria (fracture criteria) within a specified tolerance determines the end of the simulation.

Project Outcomes

SEM Analysis

SEM images for the five fibers are shown in Figure 3. Although the thickness was difficult to measure exactly, the approximate thickness estimated from the difference between the outside radius and inside radius of the fiber was confirmed for each of the samples. Additionally, the PM-100, PAN 13000, and PAN 80000 were found to be asymmetric and the PE and PVDF fibers were found to be symmetric.

Tensile Tests

Stress-strain curves for each of the five fibers are shown in Figure 4. At least 3 specimens were evaluated for each fiber tested. The average and standard deviation of the modulus of elasticity for each of the samples is given in Table 2.

Table 2. Average modulus of elasticity and standard deviation for the five membrane fibers

Membrane	**A**	**B**	**C**	**D**	**E**	**F**	**G**	**H**	**I**
	PM 882	PM 100s	PM 100d	PE	PAN 13	PAN 80	PVDF	PP pressure	PP submerse
Modulus of Elasticity (psi)	no fiber	15783 ±2696		6535 ±244	21631 ±3945	31515 ±1681	20120 ±2150	no fiber	no fiber

The strongest fiber is the PAN 80000. The high strength of the PAN 80000 was anticipated prior to testing. In making this fiber, the phase inversion process was slowed down to allow for more partitioning of the solvent and consequently, more void space. The intention of the manufacturer was to create a stronger fiber. The weakest fiber is the PE. The PAN 13000, PVDF, and PM fibers have intermediate strengths

Performance Analysis

The PM-882 module had the lowest performance of the five membrane modules that were tested. This module experienced many sudden drops in pressure and had to be cleaned frequently. The PM-100 (static) module also performed poorly, although marginally better than the PM-882 module. Frequent cleaning, low transmembrane pressure, and coliform breakthrough were the main problems with this membrane. The PP pressure module performed better than the PM-882 and PM-100 modules, but not as good as the PP submersible module. Both the PP submersible module and the PVDF module had the highest performance. Both modules were operated for long periods (approximately three weeks) between cleanings. Over the lifetime of the PVDF module, no fiber breakages have been detected.

Figure 5 is an example of a dynamically potted membrane which includes an elastomer overlay, from both a top and a side perspective. Five fibers are included in the unit. The boundary conditions allow translational movement in the y and z directions, but no translational movement in the axial (x) direction. Rotational degrees of freedom have been fixed in all directions. The fluid structure interface boundary includes the outer and inner faces of the fibers and the top of the elastomer. Pressure loading occurs on the outer fiber faces and the top surface of the elastomer.

Figure 6 is the fluid model counterpart of the structural model just described. There is a block of fluid on top of the elastomer boundary and surrounding the outside of the fibers. There is a no slip boundary condition on the outside walls of the fluid block, corresponding to the no slip conditions on the outer walls of the entire unit. The fluid structure interface boundary corresponds to those mentioned before, including the inner and outer faces of the fibers and the top of the elastomer. A prescribed fluid velocity is applied to the top surface of the fluid block.

An example of preliminary results for the ADINA-F model is shown in Figure 7. This figure shows the y-displacement of water by showing the block of fluid outside of the fibers and the fluid flowing down the inside of the fibers. The green color shows no y-displacement, while the red and blue colors show fluid displacement. As water hits the surface of the elastomer, it is being displaced near the fiber-elastomer juncture. This indicates that there are additional stresses at the juncture which might possibly lead to the formation of fractures.

Conclusions and Recommendations

Conclusions

Correlations between membrane and module properties and membrane fiber failure (i.e., loss of integrity) were difficult to make because only two membrane fibers (the PM100s and PVDF fibers) underwent both materials testing and performance testing. Koch decided not to supply a PM882 fiber for material testing. Similarly, Memcor did not supply a fiber from the PP submersible or the PP pressure vessel. Additionally, no performance data was available for the PM 100d, PE, PAN 13000, or PAN 80000 membrane modules.

Preliminary modeling results found the existence of additional stresses at the fiber/potting juncture which might possibly lead to the formation of fractures. Further modeling was impeded by limitations of the ADINA software. Although several ADINA updates were received over the course of the investigation and enhanced capabilities were to be forthcoming, the software never reached the initially stated potential. For this reason, current and future modeling efforts are focusing on more advanced software, ANSYS.

Commercialization Potential

Information acquired from the study of microporous membrane fiber integrity will assist with the development and manufacture of longer lasting MF and UF membranes and modules for the reclamation market. Reductions in operation and maintenance costs associated with loss of membrane fiber integrity will allow this technology to be more competitive with other less effective existing technologies.

Recommendations

Future efforts should include the evaluation of immersed hollow fiber membranes as well as evaluation of the impact of backwashing (using both air and water) on hollow fiber membrane integrity. The immersed hollow fiber membranes have been found to delaminate or crack in the area where the hollow fiber meets the potting material. The current model for pressure-driven membranes could be modified for the suction-driven membranes. Similar to the current investigation, results from the structure-fluid model would be combined with analysis of membrane failure for OCWD demonstration-scale submersible membrane systems.

The process of backwashing hollow fiber membranes may be responsible for the widening of the pores or the weakening of the material properties of hollow fiber membranes. To investigate the effects of backwashing on hollow fiber membrane performance and integrity, the structure-fluid model would be further modified to be able to evaluate the effects of air and water backwashing. Results from this model would again be compared to observations and measurements taken at the OCWD pilot- and demonstration-scale facility.

Benefits to California

Preventing microporous fiber breakage will have a significant effect on water treatment and wastewater reclamation in California and throughout the world. The performance of reverse osmosis membranes in indirect potable reuse and the efficacy of disinfection processes (chlorination and ultraviolet irradiation) in direct non-potable reuse are directly dependent on MF and UF fiber integrity.

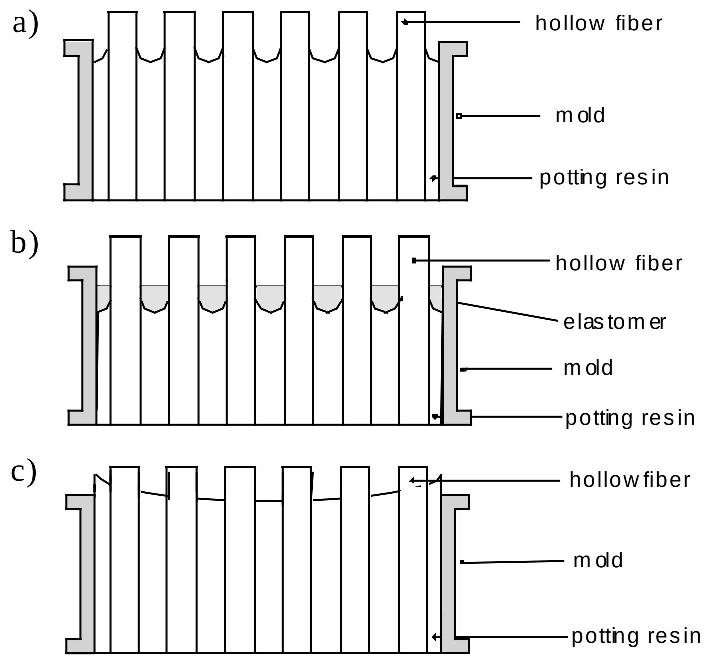

Figure 1. Fiber bundle potted under (a) static conditions, (b) static conditions with an elastomer overlay, and (c) dynamic conditions.

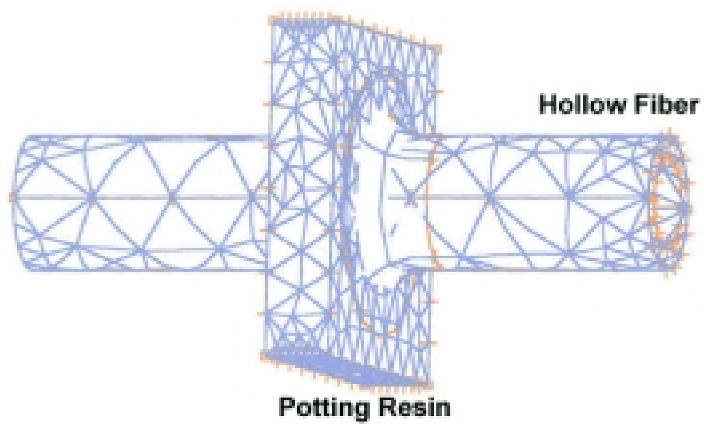

Figure 2. Structural model composed of porous pipe (representing a hollow fiber membrane) and a block (representing the potting material).

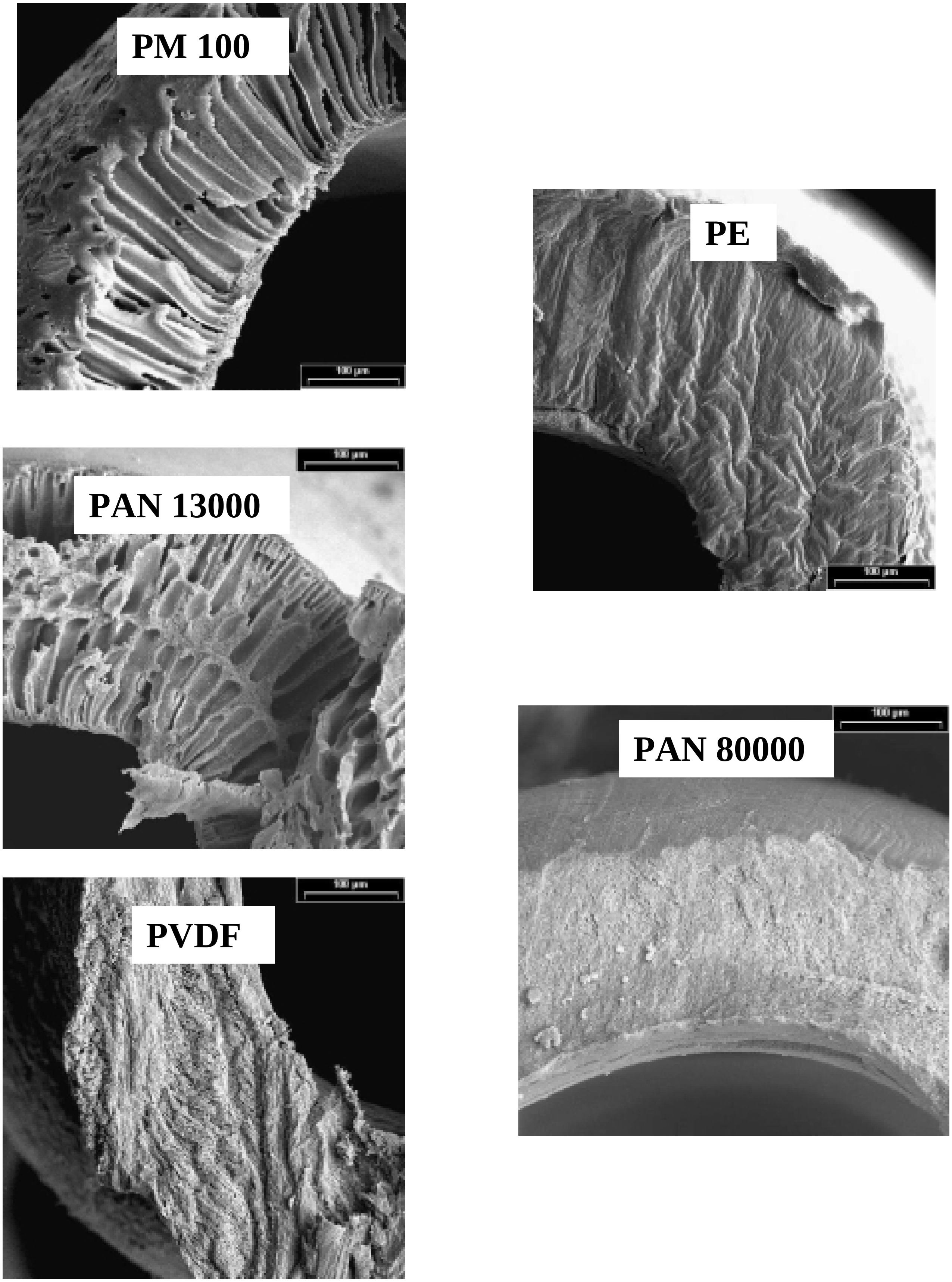

Figure 3. SEM images of the five hollow fiber membranes.

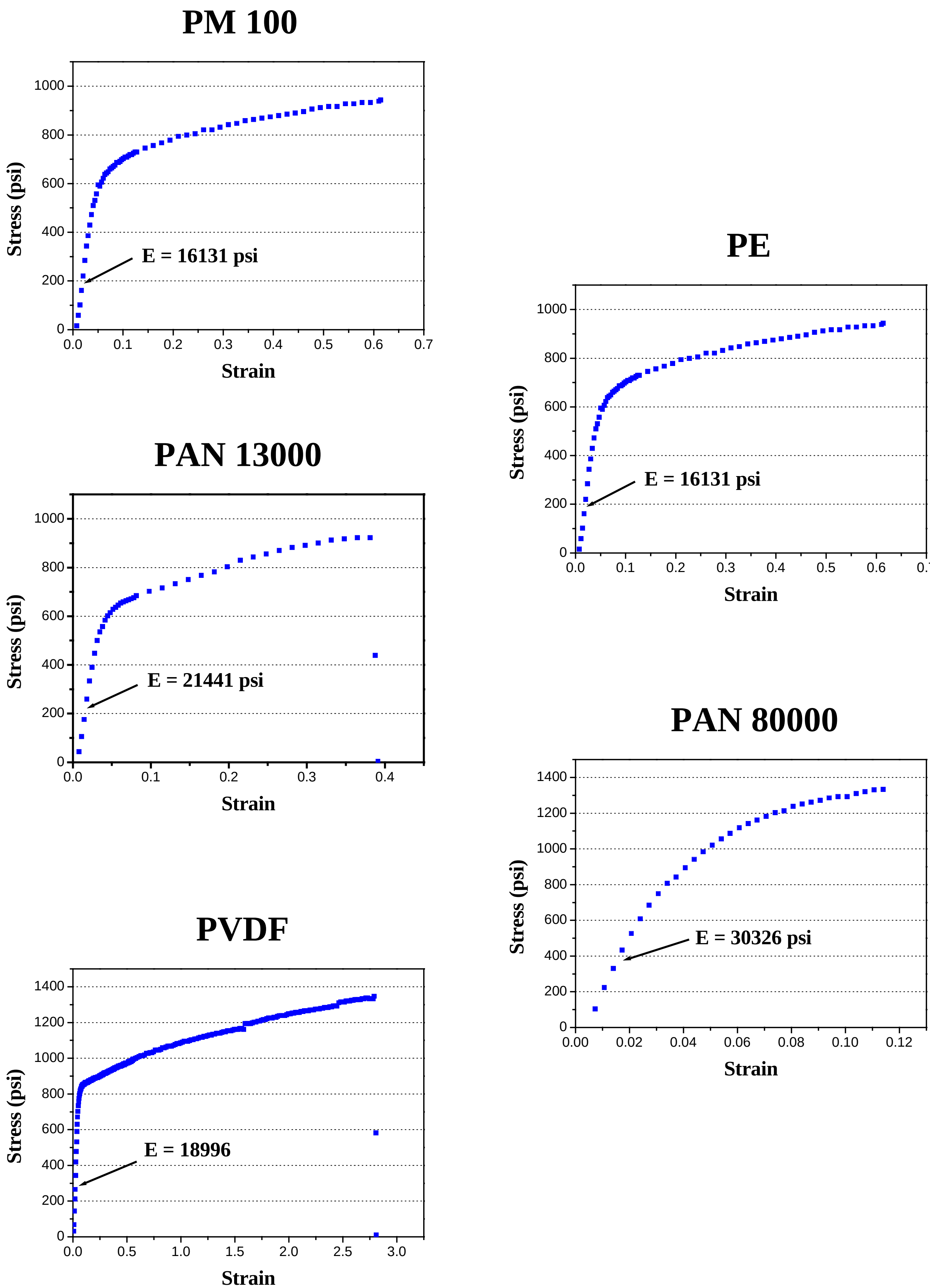

Figure 4. Stress-strain curves for the five hollow fiber membrane samples.

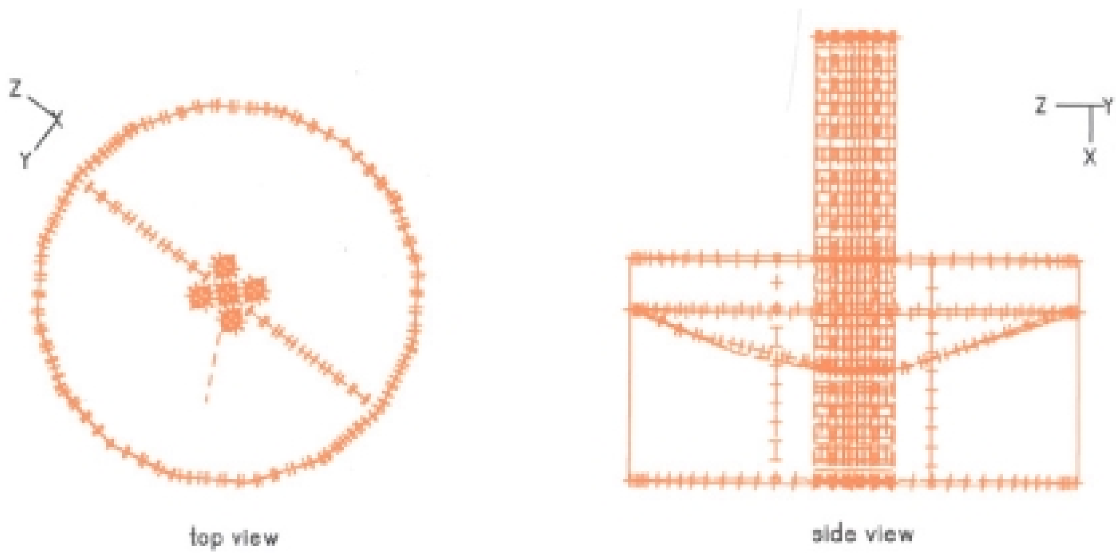

Figure 5. Structural model of a dynamically potted module with elastomer overlay.

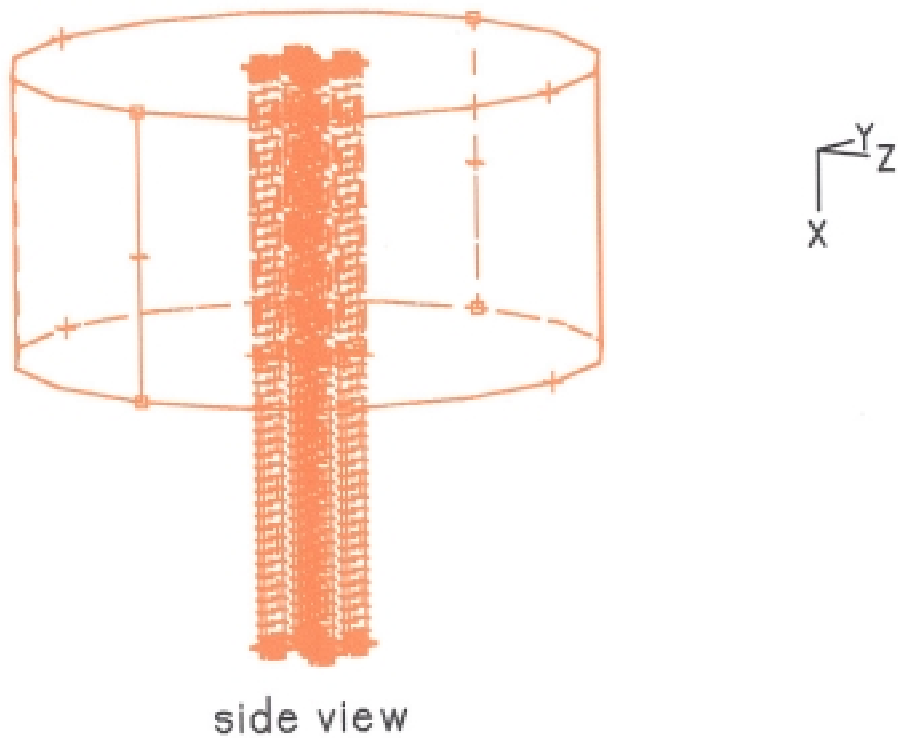

Figure 6. Fluid model of a dynamically potted module with elastomer overlay.

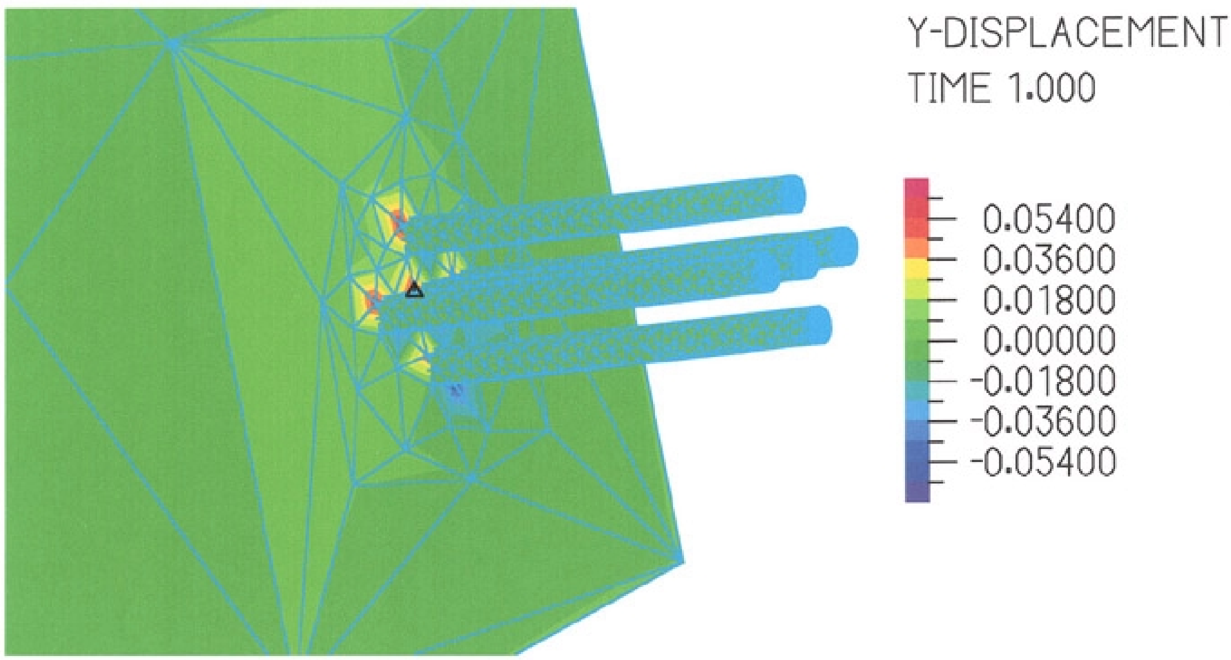

Figure 7. ADINA simulation showing the y-displacement of water for a dynamically potted module with elastomer overlay.