DOE/ID/12906--T1

DE92 016817

RESEARCH AND DEVELOPMENT TO OVERCOME FOULING OF MEMBRANES

Annual Report

June 1992

Work Performed Under Contract No. DE-FC07-88ID12906

Prepared for the
U.S. Department of Energy
Under DOE Idaho Field Office
Sponsored by the Office of the Assistant Secretary
for Conservation and Renewable Energy
Office of Industrial Technologies
Washington, D.C.

Prepared by
SRI International
333 Ravenswood Avenue
Menlo Park, CA 94025-3493

SRI International

First Annual Report • November 1990

RESEARCH AND DEVELOPMENT TO OVERCOME FOULING OF MEMBRANES

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SRI Project PYU-8330
Covering Period October 1, 1989 to October 31, 1990

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SUMMARY

During this first year of the program, we have successfully accomplished the main objective of demonstrating the feasibility of using piezoelectrically assisted ultrafiltration to reduce membrane fouling and enhance the flux through ultrafiltration membranes. A preliminary economic evaluation, accounting for the power consumption of the piezoelectric driver and the extent of permeate flow rate enhancement, has also shown that piezoelectrically assisted ultrafiltration is cost effective and economically competitive in comparison with traditional separation processes.

Piezoelectric transducers, such as a piezoelectric lead zirconate titanate (PZT) disc or a piezoelectric horn, driven by moderate power, significantly enhance the permeate flux on fouled membranes, presumably because they promote local turbulence. Several experiments were conducted on polysulfone and regenerated cellulose UF membranes fouled during filtration of model feed solutions. Solutions of poly(ethylene glycol) and of high-molecular weight dextran were used as models. We found that we could significantly increase the permeate flux by periodically driving the piezoelectric transducer, horn, or PZT disc, by application of moderate power over short periods of time, from 20 to 90 seconds. Enhancements as high as a factor of 8 were recorded within a few seconds, and enhanced permeate fluxes were maintained over a prolonged period (up to 3 hours). The prolonged flux enhancement makes it feasible to drive the piezoelectric transducer intermittently, thereby reducing the power consumption of the piezoelectric driver.

As piezoelectric drivers of sonically assisted ultrafiltration, PZT disc transducers are preferred over the piezoelectric horn because of their small size and ease of adaptability to ultrafiltration test cells. The horn transmits sonic energy to the UF membrane through a titanium element driven by a separate piezoelectric transducer, but a piezoelectric ceramic disc transmits energy directly to the UF membrane. Moreover, because piezoelectric ceramic elements can be fabricated in several configurations, they are potentially feasible for piezoelectrically driven ultrafiltration spiral-wound membrane modules.

Piezoelectrically assisted ultrafiltration by means of conventional UF membranes backed with piezoelectric polyvinylidene fluoride (PVDF) films showed the poor properties of PVDF films

as piezoelectric drivers. Even when high voltage pulses were applied to polymorph PVDF membranes, no significant permeate flux enhancement was recorded.

Detailed feasibility studies were conducted to measure the long-term effect of a piezoelectric horn on ultrafiltration. Flux was enhanced by a factor of 4 for the filtration of a 1.0% polyethylene glycol solution through a regenerated cellulose UF membrane when a nominal power of 10 watts was applied to the piezoelectric horn in contact with a 47-mm membrane. This specific experiment was run over 21 days, after having reached constant fouling of the membrane.

Even higher permeate flux enhancements were obtained with piezoelectric PZT disc drivers. Permeate flow rate was increased by a factor of 8 during the filtration of a 0.45% dextran solution through a UF polysulfone membrane (47 mm diameter) assisted by a 2.5-mm-thick lead zirconate titanate piezoelectric disc driven at 40 watts (2.3 W/cm^2). We have not yet attempted to optimize the power consumption and performance of the piezoelectric PZT disc driver. PZT disc experiments have been repeated many times over a period of one month with consistent enhancement of the flux. No cross flow velocity experiments have been carried out in detail. However, the feed flow rate was maintained between 1.4 and 1.6 L/min.

Our experiments show that the PZT disc produces higher flux enhancements (up to a factor of 8) than the piezoelectric horn (up to a factor of 4). We believe that the PZT disc is a more efficient driver since it is free floating on the permeate side of the ultrafiltration membrane, while the piezoelectric horn that is adapted to constitute the top portion of the test cell is immobilized and therefore does not efficiently transmit energy to the membrane.

Because of the high efficiency and ease of adaptability of ceramic element drivers, we plan a detailed study of their long-term performance on piezoelectric-driven ultrafiltration in Year 2. This study will provide a basis for applying piezoelectrically assisted ultrafiltration to industrial processes such as whey protein filtration and solvent recovery in deasphalting. We will also pursue the development of ceramic piezoelectric drivers with controlled porosity to overcome the major limitation of the PZT disc drivers and still provide a backing support to the ultrafiltration membrane. Further, we will configure one or more backing supports prototypical of the supports necessary for winding into spiral modules.

CONTENTS

INTRODUCTION

This study on "Research and Development to Overcome Fouling of Membranes" is the joint development of SRI's Department of Polymer Chemistry and Technology and its Membrane Separation Program. Our main objective is to develop innovative low-cost technological solutions to minimize fouling of membranes during ultrafiltration based on piezoelectric effects. Ultrafiltration (UF) is being increasingly applied as a separation process in liquid industrial waste treatment, in food processing, and in the pharmaceutical and medical industries. However, commercial acceptance of ultrafiltration has been severely limited because membranes do not perform consistently for extended periods. Fouling of UF membranes results in a serious flux decline of the permeate and increases the cost of using a membrane separation process as a unit operation.

Ultrafiltration (UF) is being increasingly applied as a separation process in liquid industrial waste treatment, in food processing, and in the pharmaceutical and medical industries. Ultrafiltration applied to industrial processes, such as whey protein filtration and solvent recovery in deasphalting would be energy-saving, effective, and economically competitive with traditional separation processes if the membrane performances could be improved. Indeed, commercial acceptance of ultrafiltration has been severely limited because membranes do not perform consistently for extended periods. Fouling of UF membranes results in a serious flux decline of the permeate and increases the cost of using a membrane separation process as a unit operation.

UF membranes are vulnerable to fouling by pore blockage because they are hydrophobic and because they have low surface porosity and uneven pore sizes. Fouling generally proceeds as follows:

- The flux declines rapidly due to the buildup of solutes near the membrane surface.
- Macromolecules such as proteins are adsorbed on the hydrophobic membrane material and plug the pores.
- Particles deposited by convection and cake solids are compressed.

The rate of decrease of the flux of the permeate through the membrane depends significantly on the particular composition of the liquid treated and on the interaction between solute and the membrane.

Several approaches have been tried to reduce fouling of membranes. Pretreatment of the membrane with hydrophilic surfactants and polymers to increase the initial flux and reduce the flux decline gives only a marginal and short-term improvement. Prefiltration of the feed solution with membranes of larger pore size adds the cost of another unit operation. Backwashing unplugs the blocked pores and dislodges the cake; however, it implies interruption of operation and can decrease membrane life.

To overcome fouling of membranes, SRI International is develcping unique piezoelectric backing for ultrafiltration membranes capable of producing local turbulence next to the membrane to minimize concentration polarization and the rate of buildup of solutes and particulate matter on the membrane surface.

RESULTS AND DISCUSSION

To demonstrate the feasibility of using piezoelectrically assisted ultrafiltration to reduce fouling, we undertook three approaches. We used thin ceramic piezoelectric discs, a piezoelectric ultrasonic horn, and PVDF membranes as drivers to transmit energy to commercially available UF membranes. The effectiveness of the driver in producing local turbulence next to a fouled UF membrane was evaluated by monitoring the permeate flux enhancement.

We used regenerated cellulose with a molecular weight cut off (MWCO) of 10,000 and polysulfone with a MWCO of 100,000 as ultrafiltration membranes. We tested aqueous solutions of polyethylene glycol with molecular weight of 10,000, a derivative of polyethylene glycol with molecular weight of 15,000-20,000, and dextran with molecular weight of 162,000. Before testing the effect of the piezoelectric driver on ultrafiltration, we allowed the UF membranes to foul to an approximately constant permeate flux. The piezoelectric driver was applied on the permeate side. The metallized PVDF films and ceramic transducer were carefully encapsulated by an insulating polymer coating to ensure proper electrical insulation. For this purpose we used room temperature vulcanized (RTV) rubber, polyvinylchloride (PVC), and epoxy polymers.

We found that PZT discs have significant advantages over PVDF membranes or the piezoelectric ultrasonic horn. Piezoelectric PVDF films perform poorly in reducing fouling, most likely because of their insufficient electromechanical conversion. Indeed, the piezoelectric strain constants (d_{ij}) of PVDF ($d_{31} = 0.5 \times 10^{-12}$ C/N and $d_{33} = -7.5 \times 10^{-12}$ C/N) are quite low in comparison with other piezoelectric materials (C/N means coulombs per newton). The piezoelectric strain constants are defined as follows:

$$S_i = d_{ji} E_j$$

where S_i is a strain component, and E_j is the applied electric field; the subscripts i and j refer to orthogonal directions, length and thickness, respectively, within the specimen. Since large d_{ij} coefficient are desirable in piezoelectric driver applications, ceramic transducers, such as PZT, appear best suited for ultrasonically assisted ultrafiltration (PZT has $d_{31} = -93.5 \times 10^{-12}$ C/N and $d_{33} = 223.0 \times 10^{-12}$ C/N).

A PZT disc transducer is potentially more practical than the piezoelectric ultrasonic horn. The ultrasonic horn is a bulky piezoelectric driver that must be fastened to the ultrafiltration test cell, but a PZT disc can easily fit in a conventional cross-flow ultrafiltration flow cell with minimal modifications. PZT discs as thin as 2.5 mm produced significant flux enhancements. More important, thin piezoelectric discs are potentially feasible in commercial spiral-wound membran modules.

A cross-flow ultrafiltration test apparatus was designed and assembled exclusively for this project, as described below.

CROSS-FLOW ULTRAFILTRATION TEST APPARATUS

A cross-flow ultrafiltration test apparatus was assembled for long-term evaluation of the membrane fouling. The apparatus is designed to circulate feed solution through two test cells that hold ultrafiltration membranes (Figure 1). This configuration allows the accurate and simultaneous evaluation of UF membranes driven and not-driven by a piezoelectric transducer, under the same operating conditions. The feed pump is a centrifugal five-stage pump with a maximum flow rate of 4 gallons per minute and a maximum pressure of 125 psig. The pressure in the system is adjusted through a back-pressure regulator. Our standard operating conditions are a pressure of 50 psig with a feed flow rate of about 1/2 gallon per minute. The feed stream is split so that the piezoelectric and non-piezoelectric cells can be tested side by side. The non piezoelectric-driven cell is identical to the piezoelectric-driven cell in all respects except that it does not have any power supplied to the piezoelectric driver. The residue streams from each of the cells is recombined before passing through a throttle valve. The throttle valve has the function to maintain the pressure in the system. Because equal lengths of tubing have been used to supply the feed to both cells, the feed streams to each cell are approximately similar. We measure the permeate flux through each membrane. Any difference in flux directly measures the effect of the piezoelectric vibration on the fouling of the UF membrane. Both the permeate stream and the combined residue stream can be sampled for analysis. To maintain constant feed conditions, the test apparatus is designed with total recycle. A copper coil with cooling water is inserted into the feed tank to maintain constant temperature in the system. The temperature of the feed is monitored with a thermocouple.

Commercial UF membranes with 10,000 and 100,000 molecular weight cut-off (MWCO) were used in this study. We studied polysulfone membranes mounted on a polypropylene support (Millipore type PTGC) and low-protein binding regenerated cellulose membranes bound to a polypropylene support (Millipore type PLGC). Stainless steel support filters with ten and twenty micron pores were used to provide structural support to the UF membranes, if required.

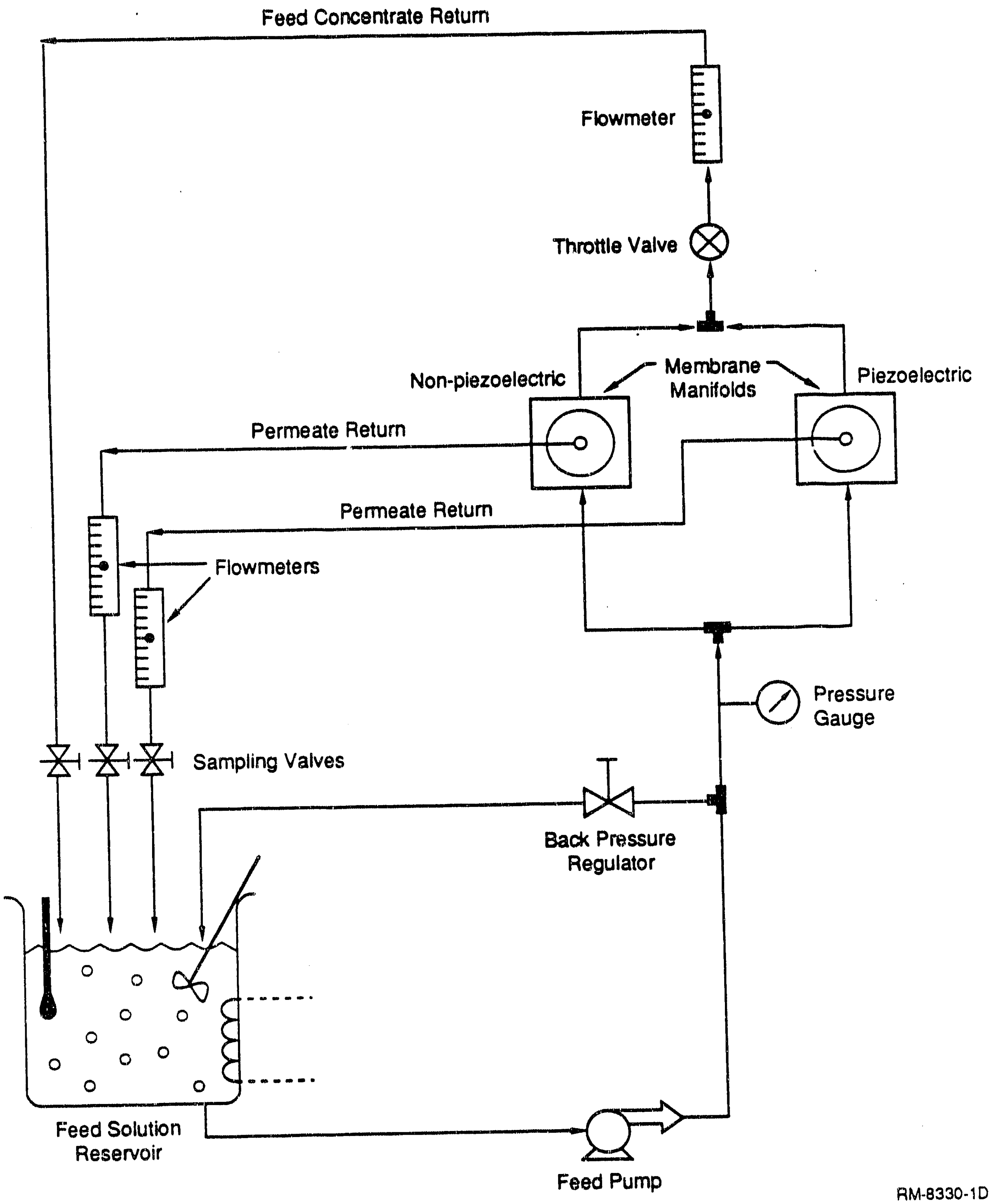


Figure 1. Cross-flow ultrafiltration test apparatus.

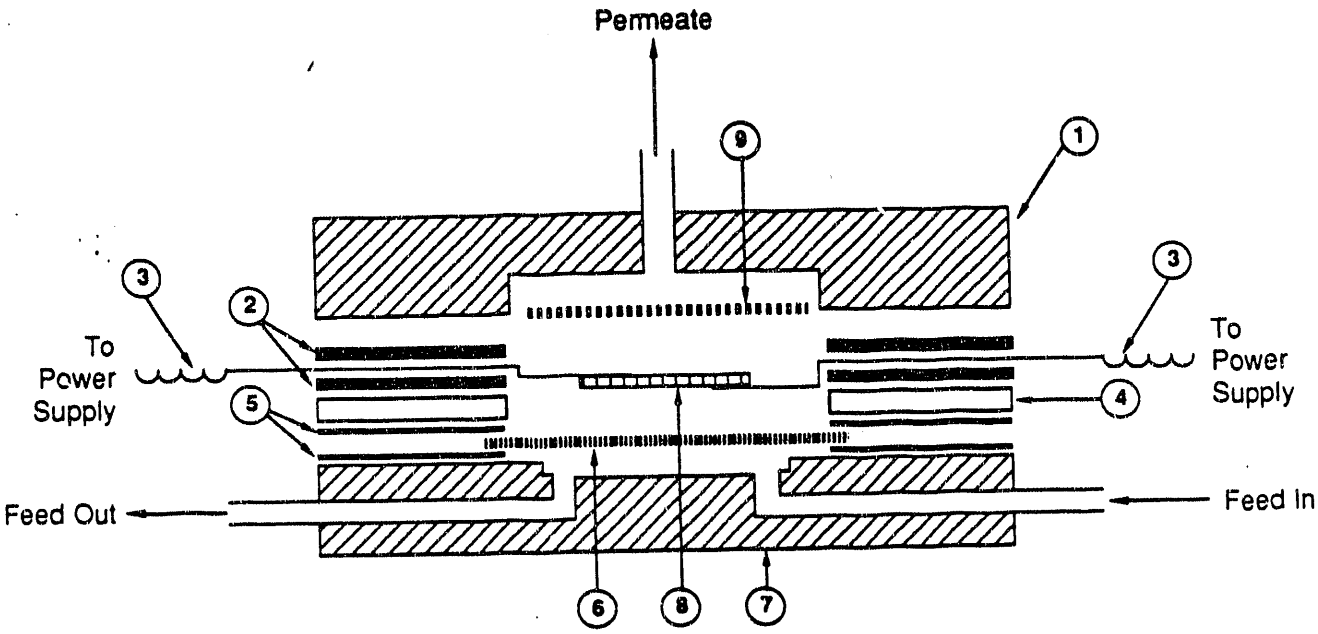
As a standard procedure, between different runs, the system is thoroughly cleaned to prevent contamination. A 2% solution of citric acid is run for one hour to remove traces of metal from the system. Deionized water is then run for fifteen minutes for rinsing the system. A 0.2% Micro detergent solution is run for one hour to remove proteins and amino acids. Several more deionized rinses follow. Care was taken to run the final two rinses with Milli-Q (18 megaohm) water. The entire cleaning procedure is always carried out with no membrane so as to ensure a thorough cleaning of both sides of the system.

LEAD ZIRCONATE TITANATE (PZT) PIEZOELECTRIC DISC ASSISTED ULTRAFILTRATION

Piezoelectrically assisted ultrafiltration by means of a piezoelectric lead zirconate titanate disc is an entirely new approach to reduce fouling. By application of moderate voltages to a PZT disc on the permeate side of the UF membrane, a significant enhancement of permeate flow rate is monitored. We believe that the PZT driver produces local turbulence next to the UF membrane to minimize concentration polarization and the rate of build up of solutes and particulate matter on the membrane surface.

Ring and disc-shaped PZT transducers were tested to transmit sonic power to UF membranes. The transducers were placed on the permeate side of the UF membrane, where they are not subjected to high pressure. Disc-shaped PZT transducers provided the best performances. Electrical wires were connected to a metallized PZT disc of 38 mm. diameter and of 2.5 mm thickness. The disc and the electrical leads were electrically insulated by encapsulation with a fluoroepoxy coating. The schematic representation of the PZT disc driven ultrafiltration test cell is illustrated in Figure 2. The PZT disc is not in direct contact with the UF membrane, but "floats" on the permeate side. By application of a moderate power, such as 40 watts, mechanical strain is produced along orthogonal directions. Dimensional changes of thickness, length or width, depending on the resonance frequency, are responsible for the generation of local turbulence on the permeate side, which is transmitted to the UF membrane.

It is highly desirable to operate the piezoelectric element at one of the resonance frequencies, since under these conditions the resistance of the element is minimal, thus allowing a high degree of power conversion into vibrational energy. For the disc shaped PZT transducer, fundamental resonance frequencies (ν_r) of the radial and thickness mode can be calculated as follows:



1. Top half
2. Thick rubber gaskets
3. Electrical leads
4. LEXAN ring
5. Thin rubber gaskets
6. Ultrafiltration membrane
7. Bottom half
8. PZT disc transducer
9. Microfilter

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Figure 2. PZT disc-driven ultrafiltration test cell.

$$\nu_r = \frac{V}{2t}$$

where ν_r is the resonance frequency; V is the velocity of sound in the PZT disc; and t is the measurement of the disc dimension through which the strain deformation takes place. In our calculations, we have used a value of 4000 ms^{-1} for the velocity of the sound in PZT. This value represents an average of the values reported for different PZT formulations. It should be also notes that the physical description of the transducer by this equation is a simplification that does not take in account that the PZT disc is held by two metal strips and is coated by a polymer film.

We have calculated a 52 KHz symmetric radial resonance frequency for a PZT disc of 38 mm. of diameter while the symmetric thickness frequency is at about 800 KHz for a disc thickness of 2.5 mm.. Both these values are in reasonable agreement with the resonance frequencies of 47 KHz and 750 KHz observed from experimental measurement of the impedance as a function of frequency.

PZT disc assisted ultrafiltration tests were run by operating the piezoelectric transducer at 47 KHz , that represents the radial resonance mode of vibration of the disc. A simplified representation of the symmetric vibration mode is shown as follows. This radial resonance leads to buckling of the disc resulting in a displacement perpendicular to the disc.

We believe that under the operating conditions this radial deformation is responsible for the local turbulence next to the membrane, expected to reduce fouling and enhance the permeate flux through the UF membrane.

The feasibility of PZT disc-driven ultrafiltration was tested in the following experiments. A 0.45 % solution of dextran (M_w 162,000 daltons) in water was filtered through a Millipore polysulfone UF membrane with a nominal MWCO of 100,000. The insulated PZT disc was inserted in the ultrafiltration test apparatus. The feed solution was filtered at 50 psig under cross-flow conditions for a few days to reach steady fouling conditions. The permeate flux was 0.34 mL/min. Forty watts of power was applied to the PZT transducer for 90 seconds by means of the power supply shown in Figure 3. The permeate flow rate increased to 2.78 mL/min. We turned off the power, and after more than one hour and half the permeate flux was still higher than the permeate flux before power was applied. The permeate flux changes are shown in Figure 4. Under these conditions, piezoelectric vibration increased the permeate flux as much as by a factor of 8.

In a similar test, we filtered a 0.58% dextran solution with a Millipore UF membrane with nominal MWCO of 100,000. At 50 psig a steady fouling condition was reached with a permeate flux of 0.30 mL/min. Forty watts of power was applied to the PZT transducer for 90 seconds, and the permeate flux increased to 2.1 mL/min (a factor of 7). Permeate volumes were collected at constant intervals of time, and the cumulative volumes were plotted versus time (Figure 5). The effect of only 90 seconds of piezoelectric vibration is quite evident on the trend of the permeate volume collected. It took 3 hours for the system to return to the original permeate flux of 0.30 mL/min. It should be noted that no attempt has yet been made to optimize power consumption and performance of the piezoelectric ceramic disc driver.

The configuration of the PZT transducer is an important parameter, as shown by some experiments we did with a PZT ring-shaped transducer instead of the PZT disc. The ring had an internal diameter of 3/4 inch and an external diameter of 1 inch, and a thickness of 1/2 inch. The ring was metallized on the inner and outer surfaces and poled radially. We attached electrical leads to the two metallized surfaces and the ring was completely coated with a thin layer, of epoxy for electrical insulation. In air the PZT ring showed a minimum impedance at 40.394 KHz, corresponding to the radial resonance mode of vibration of the ring. At this frequency, in-phase dimensional changes of the inner and outer ring diameter take place. However, when immersed in water, this radial mode of displacement was severely attenuated, as shown by experimental measurements of impedance as a function of frequency. Nevertheless, a PZT ring-assisted ultrafiltration test was carried out by resonating the PZT ring in the frequency range of approximately 30 ± 10 KHz . The voltage applied to the ring was amplified by means of a amplifier. Under these conditions, even at maximum amplification, the PZT ring transducer assisted ultrafiltration showed an insignificant permeate flux increase of about 5% .

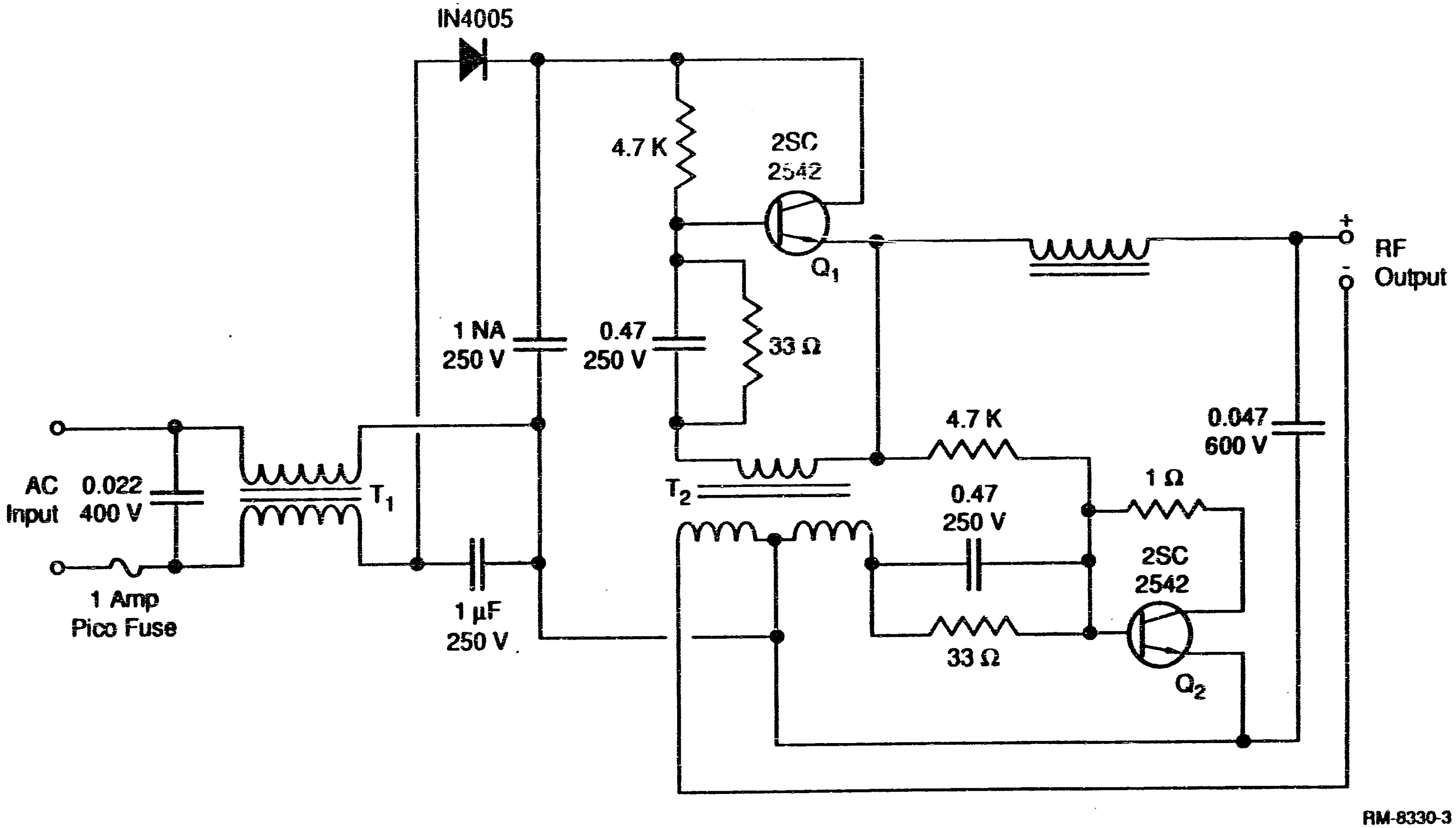


Figure 3. 40-watt power supply for the PZT transducer.

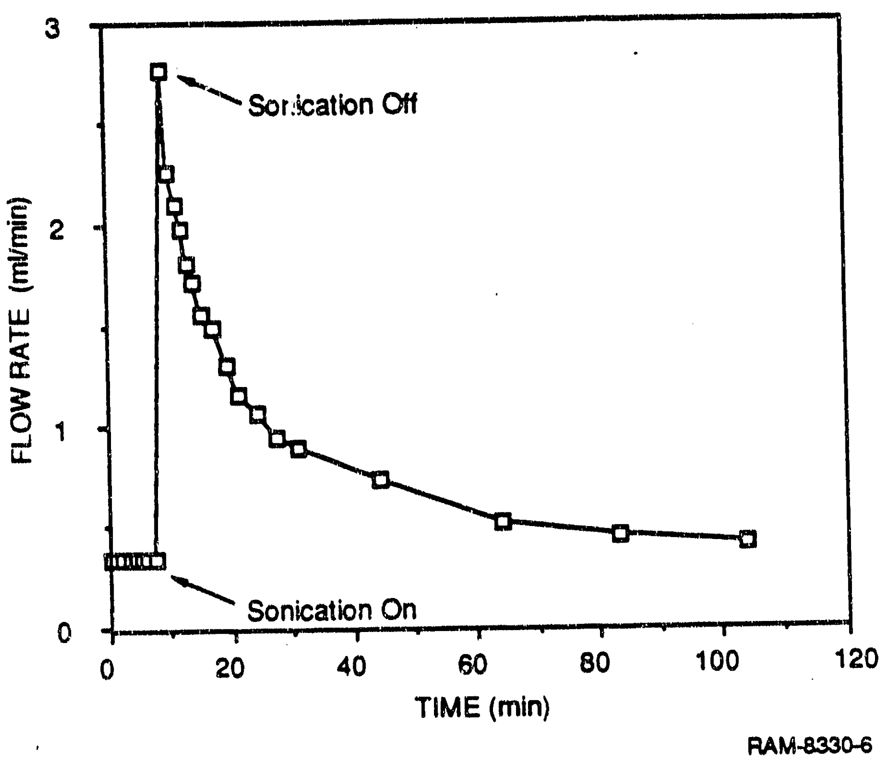


Figure 4. Effect of sonication by a PZT disc transducer on the ultrafiltration flux of a 0.45% dextran (MW 162,000) solution with a 100,000 MWCO polysulfone membrane.

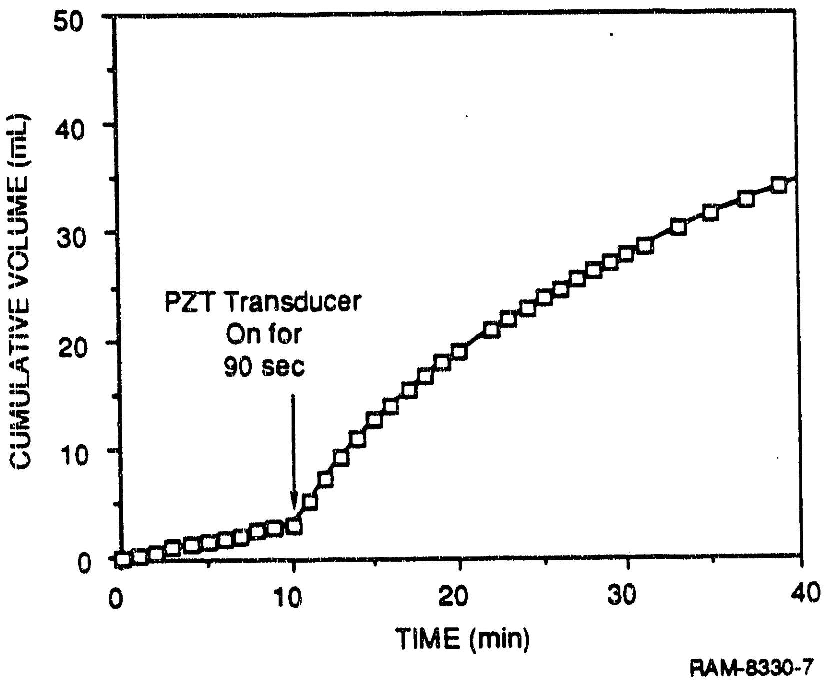


Figure 5. Cumulative permeate volumes of PZT disc-assisted ultrafiltration of a 0.58% solution of dextran (MW 162,000) with a 100,000 MWCO polysulfone membrane.

The reproducibility of each of the above illustrated experiments was confirmed by repeated tests. Determinations of the enhancement of permeate flux by piezoelectric disc were conducted several times over a period of one month. The permeate flux was consistently enhanced by a factor of 8. The piezoelectric disc diameter was 38 mm and the membranes diameter was 2 x 76 mm, thus representing area coverage of 12%. We have experimented with area coverage of as low as 5%, although these experiments need to be further examined with appropriate power supplies. While similar experiments are consistent, it is also evident that the absolute permeate flux enhancement is a function of parameters, such as composition and flow rate of the feed solution (as expected). These observations will need to be quantified and correlated during the next phase of this study.

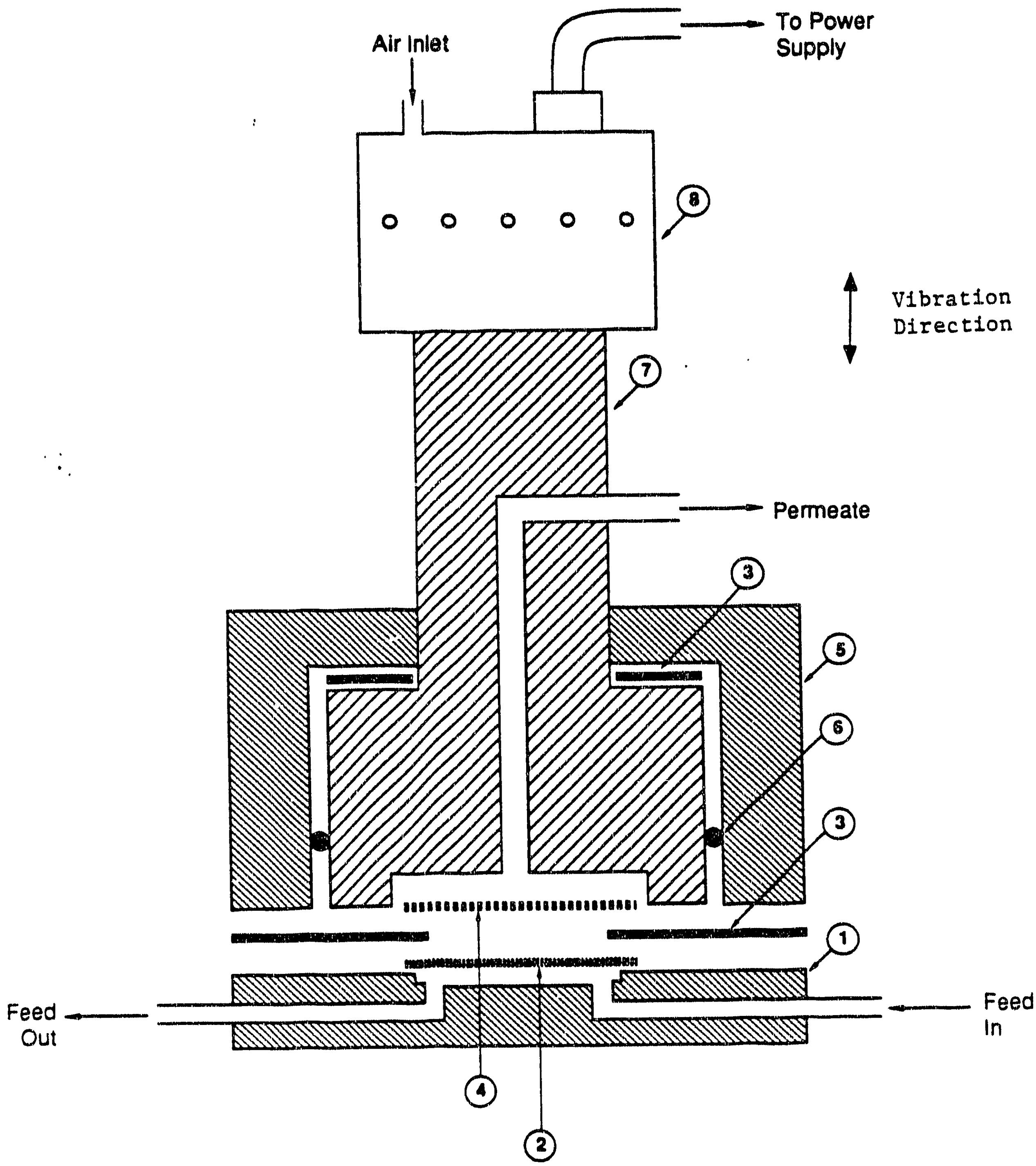
PIEZOELECTRIC HORN-ASSISTED ULTRAFILTRATION

We used a piezoelectric horn capable of transmitting up to 200 watts of power to a titanium tip at the frequency of 20 KHz. The horn was maintained in close contact to the UF membrane, on the permeate side, through a microporous stainless steel filter. The horn was adapted to constitute the top portion of the test cell, as shown in Figure 6. The permeate was therefore collected through an output channel drilled through the horn.

Feasibility tests of the piezoelectric horn assisted ultrafiltration were carried out on a simplified ultrafiltration apparatus where the whole feed solution was forced through the UF membrane without recirculation of the feed and of the permeate stream. Two experiments, with and without piezoelectric assistance were conducted and compared. A 1% solution of poly(ethylene glycol) with average molecular weight of 10,000 daltons was prepared and used as feed through a 47 mm. 10,000 MWCO poly(sulfone) UF membrane, mounted on a polypropylene support. A microporous stainless steel support filter was used to give structural support to the UF membrane. The feed solution was pressurized at 80 psig.

The ultrafiltration test experiments, with and without power applied to the piezoelectric horn, were run independently on the same membrane.

Data were first collected for a filtration experiment of 1% poly(ethylene glycol) solution without driving the horn. The permeate cumulative volume was plotted at different times (Figure 7, Curve 1). Once the experiment was concluded, the same ultrafiltration membrane was backwashed overnight by circulation of deionized water without opening the flow cell. The flow rate of Milli-Q water was used as a criterion to monitor the proper cleaning of the membrane.



1. Bottom half
2. Ultrafiltration membrane
3. Rubber gasket
4. Microfilter
5. Top half
6. O-ring
7. Titanium horn
8. Piezoelectric element housing

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Figure 6. Piezoelectric horn-driven ultrafiltration test cell.

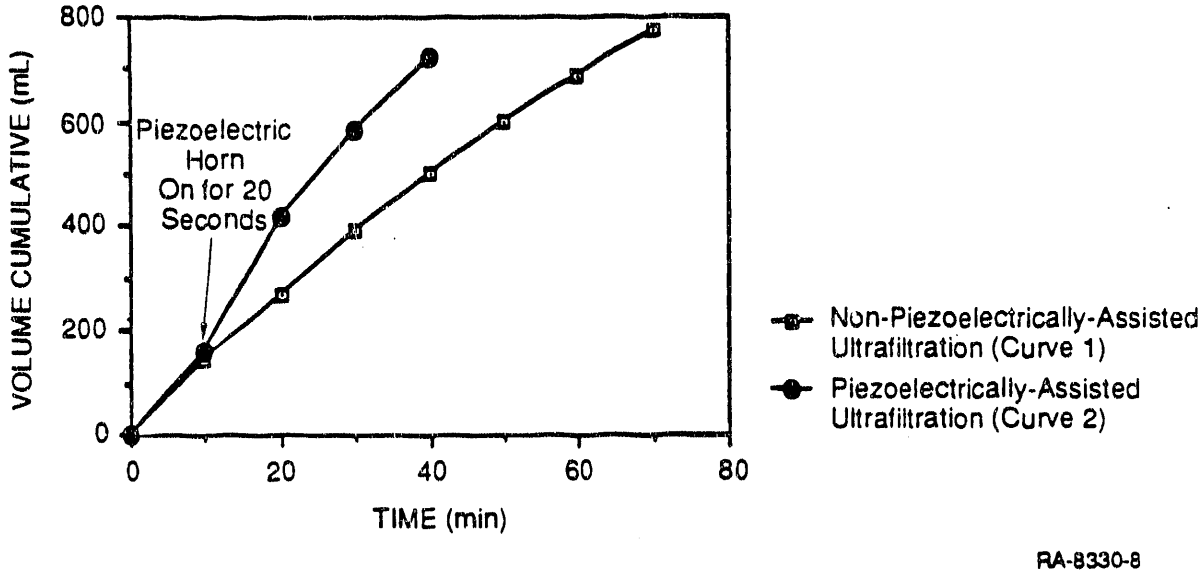

Figure 7. Cumulative permeate volumes of the ultrafiltration of a 1% poly (ethylene glycol) solution.

The piezoelectrically-assisted ultrafiltration was carried out with a piezoelectric horn.

Piezoelectric horn-assisted ultrafiltration of 1% poly(ethylene glycol) solution was then carried out again on the same membrane. The permeate cumulative volume during the first 10 minutes of the experiment was found to be reproducible and comparable with the previous experiment (Figure 7, Curve 2). The horn was then driven at a nomimal power of 20 watts for twenty seconds. The permeate cumulative volume significantly increased, and was constantly higher for the whole duration of the experiment.

Because of this encouraging result, we went on with the test of piezoelectric horn assisted ultrafiltration in a cross-flow test apparatus. Testing procedures and protocol were established, the most suitable testing conditions were identified, and experimental data on ultrafiltration flux enhancement due to sonication produced by an ultrasonic horn were generated. A summary of the experiments conducted, in chronological order, is shown in Table 1.

To obtain baseline flow rates of the permeate through a fouled membrane, long term tests on the cross-flow filtration apparatus were carried out. Thus, we ran a 0.1% polyethylene glycol solution with an average molecular weight of 10,000 for 19 days (Experiment #1). Millipore's low binding regenerated cellulose membranes with a 10,000 MWCO were used for the above experiment. The fouling data show that the cells are subjected to the same inlet conditions and that fouling is qualitatively in accord with the expected behavior (Figure 8).

As described in our original proposal, we had planned on studying a synthetic whey solution. The main component of synthetic whey, besides water, is casein. Therefore a 0.8% casein solution was mixed in the feed tank (Experiment #2). However, we could not completely dissolve the casein. We therefore opted for the study of high molecular weight dextran (162, 000 daltons). Dextran is available in a wide range of molecular weights and is relatively inexpensive.

For the Experiment #3 we prepared a 1% dextran solution and we used Millipore's PTHK membranes with molecular weight cut-off of 100,000. Within thirty-five minutes, the permeate flow rate was no longer measurable. We drove the piezoelectric horn at different nominal powers for one minute each time, and we observed significant increments of the permeate flux that lasted between ten and fifteen seconds. In all cases, we observed a delay of the permeate flux increase from the moment of the sonication start of approximately five seconds. This experiment confirmed previous data registered in a flow-through filtration system.

In the Experiment #4 we tested a 1% polyethylene glycol compound solution (MW 15,000-20,000) using a 10,000 MWCO low binding regenerated cellulose membranes. Fouling was observed very quickly. We did not even try to piezoelectrically drive the horn to enhance flow

Table 1

SUMMARY OF THE PIEZOELECTRIC HORN-ASSISTED ULTRAFILTRATION EXPERIMENTS

No.	Membrane	Solute/Mol Wt.	Feed Composition (wt%)	Results/Comments
1	Regenerated cellulose, 10,000 MWCO	Polyethylene glycol/10,000	0.1	Experiment continued for 19 days. Steady decline of flux over time.
2	Regenerated cellulose, 10,000 MWCO	Casein/unknown mol wt	0.8	Problems with the dissolution of solute; system fouling.
3	Polysulfone, 100,000 MWCO	Dextran/162,000	1.0	Permeate flux became negligible within 35 minutes from the start of the experiment; however, the vibration of the piezoelectric horn did improve the flux somewhat
4	Regenerated cellulose, 10,000 MWCO	Polyethylene glycol compound/ 15,000-20,000	1.0	Permeate flux became negligible almost from the beginning
5	Regenerated cellulose, 10,000 MWCO	Polyethylene glycol compound/ 15,000-20,000	2.6*	Reasonably good results. The vibration of the piezoelectric horn enhanced the permeate flux approximately by a factor of 2
6	Regenerated cellulose, 10,000 MWCO	Polyethylene glycol compound/ 15,000-20,000	1.0*	Good results. The vibration of the piezoelectric horn enhanced the permeate flux approximately by a factor of 4. Duration of experiments: 21 days.
7	Polysulfone, 100,000 MWCO	Dextran/162,000	0.1*	Good results. The vibration of the piezoelectric horn enhanced the permeate flux approximately by a factor of 5. Duration of experiments: 15 days.
8	Polysulfone, 10,000 MWCO	Polyethylene-imine/50,000-60,000	2.5	Good results. The vibration of the piezoelectric horn enhanced the permeate flux by a factor of 4.

*added gradually to feed water

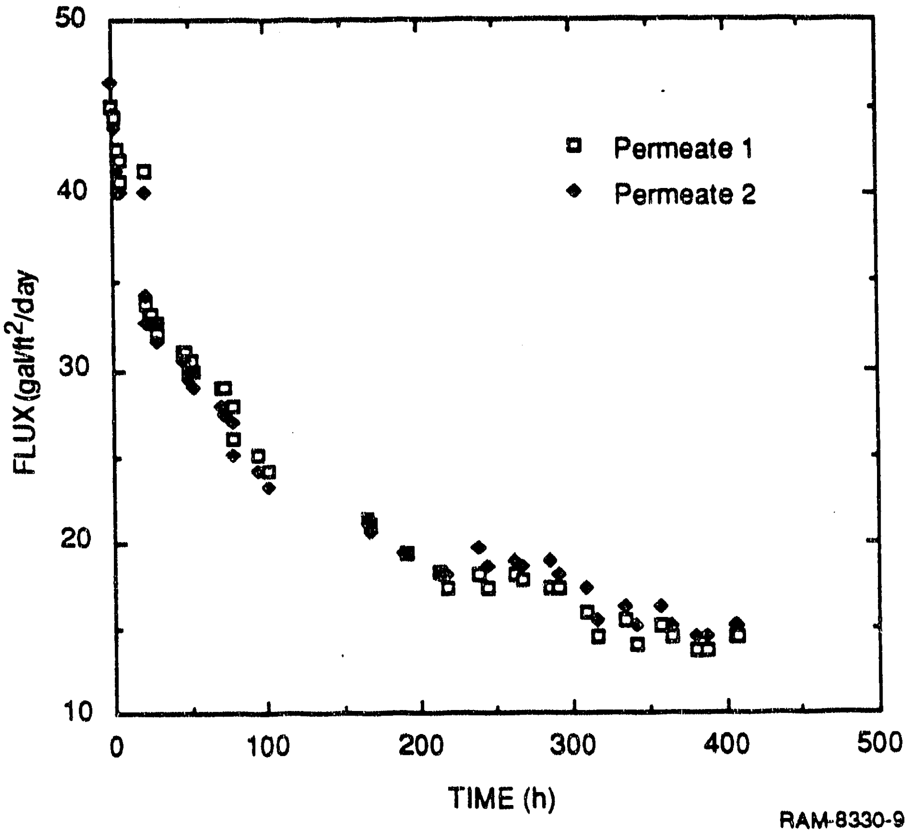

Figure 8. Fouling data for a 0.1% solution of 10,000 MW polyethylene glycol with 10,000 MWCO regenerated cellulose membranes.

rates, and instead we cleaned thoroughly the apparatus for another test with a more convenient concentration of poly(ethylene glycol).

By gradually adding polyethylene glycol to the feed solution we had better control of the feed composition, and we could test the ultrafiltration of a 2.6% poly(ethylene glycol) solution (Experiment #5). We applied six power pulses to the UF membrane, sixty seconds long, over a period of one hour. The piezoelectrically horn-assisted ultrafiltration enhanced the permeate flux approximately by a factor of 2.

A more systematic study on the effect of sonication produced by the horn was conducted in the Experiment # 6. A 1% polyethyleneglycol solution was studied with a regenerated cellulose UF membrane (MWCO 10,000) and a 0.1% dextran solution with a polysulfone UF membrane (100,000 MWCO) were studied, respectively. Several sonication pulses were applied during each run. Figure 9 shows the short-term variation of the permeate flux with time for the polyethylene glycol experiments. The upper dotted line represents the flux of pure water through the membrane. Before the piezoelectric horn was driven to enhance ultrafiltration, the membrane was fouled over a period of a few days. The flux through the fouled membrane is indicated by the lower dotted line. The piezoelectric horn was then periodically driven for about two minutes by applying a formal power of 10 watts. The permeate was substantially enhanced by more than a factor of 4 when the horn was on. Figure 10 shows the long-term effect of sonication on long-term performance of the membrane. Initially the feed solution was progressively concentrated to quickly reach steady state fouling conditions. Then the flux was periodically enhanced by driving the piezoelectric horn with 10 watts power (2 minutes every 50 hours, on average). The figure compares the flux of the piezoelectrically-driven cell and non-piezoelectrically-driven cells. The permeate flux of the piezoelectrically-driven cell is higher than that of the non-piezoelectrically driven cell at the end of 21 days. To examine in detail this effect, we should verify whether any pore size variation of the UF membrane takes place because of vibration. A possible approach would be to inspect the membrane by scanning electron microscopy (SEM) at the end of the sonication test. Such experiments will be carried out during the second year. Our preliminary economic evaluation was calculated on the basis of this particular experiment, because of the availability of long term data. The evaluation (Appendix A) illustrates that PZ-assisted ultrafiltration will make energy saving membrane technology substantially more attractive to industry.

The ultrafiltration of dextran (0.1 %) with molecular weight of 162,000 through polysulfone 100,000 MWCO (Experiment # 7) showed similar results. The permeate flux was enhanced by approximately a factor of 5 when the horn was piezoelectrically driven. The

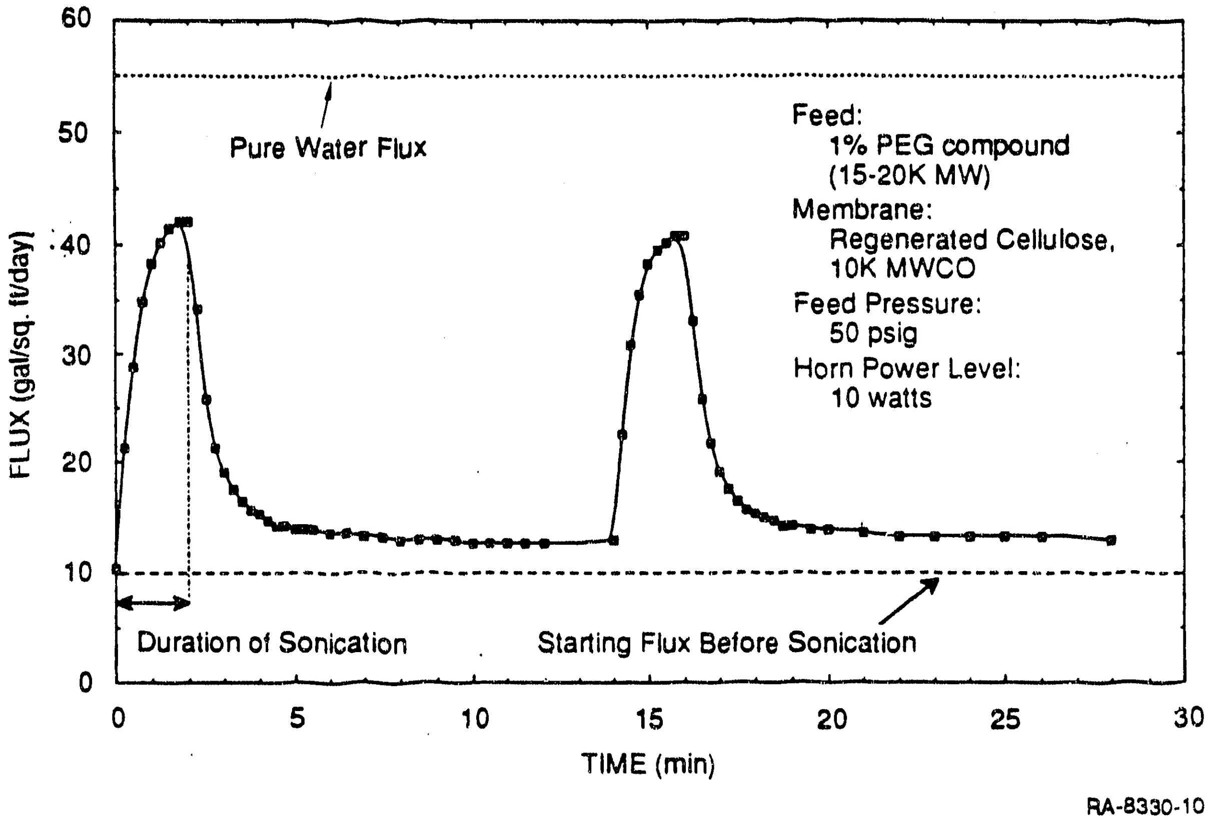

Figure 9. Effect of the piezoelectric horn on the ultrafiltration flux of 1.0% polyethylene glycol solution.

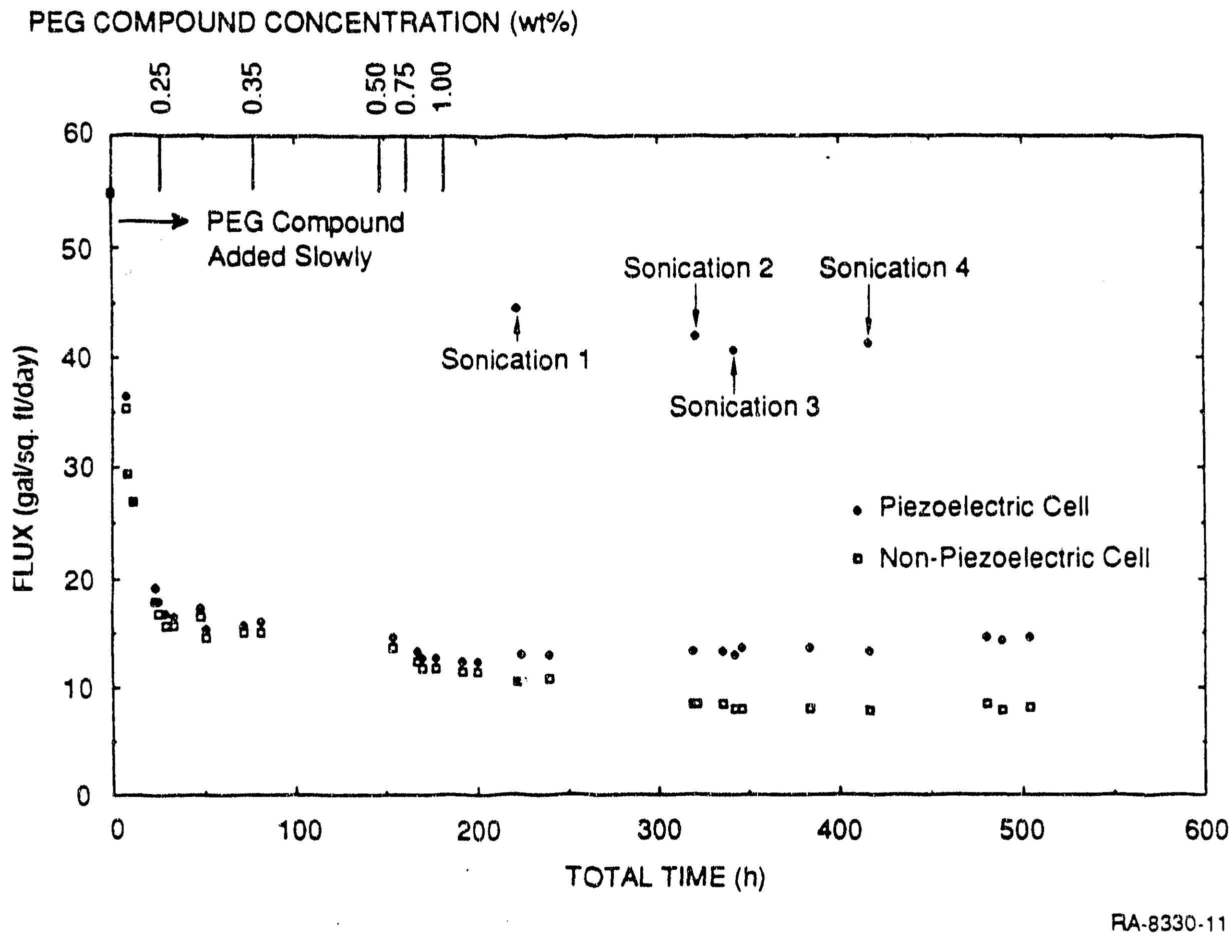

Figure 10. Long-term ultrafiltration performance on 1.0% polyethylene glycol solution. Piezoelectrically-assisted ultrafiltration was carried out by a piezoelectric horn.

ultrafiltration of poly(ethylene imine) (2.5 %), molecular weight 50,000-60,000, through polysulfone 10,000 MWCO also showed good results with a flux enhancement by a factor of 4.

PVDF FILM ASSISTED ULTRAFILTRATION

Polyvinylidene fluoride is a well known polymer with piezoelectric properties. As other piezoelectrically active materials, PVDF films can be deformed by selective application of charge to electrically conductive coatings deposited on the opposite sides of the PVDF films. Our objective was to laminate metallized PVDF films, electrically insulated by room temperature vulcanized (RTV) rubber or polyvinylchloride with UF membranes to test their capability to produce local turbulence next to the membrane pores therefore reducing the fouling and enhancing the permeate flux.

A summary of the most representative experiments carried out on PVDF membranes as piezoelectric transducers is illustrated in Table 2.

Since ultrafiltration usually takes place at pressures in the range of 50 to 100 psi, we first sought to verify whether, under these conditions the PVDF films vibrate when an external electrical bias is applied. Two metallized PVDF films, separated by a rubber gasket, were compressed at 50 psi. Either one or both the films were coated by a thin film of RTV rubber (Experiments #1 and 2). One of the films was driven using a function generator, while the other film was connected to an oscilloscope to monitor the vibration of the first membrane. Even if we could not quantify the overall effect, the result of this experiment showed that the piezoelectric effect responsible for the sonication of the PVDF film under external bias takes place also when pressures of about 50 psig are applied. Moreover, we also found that the piezoelectric properties of the piezoelectric film are not significantly affected by the use of the insulating polymer coating.

To use PVDF film as piezoelectric transducer for ultrafiltration, metallized PVDF films bearing holes of about 5 mm diameters, electrically insulated by a PVC coating were laminated to UF membranes, and preliminarily tested in a flow-through ultrafiltration apparatus. Filtration experiments were conducted with piezoelectric activation (application of a bias to the metallized PVDF film) and without, with a feed solution of polyethyleneglycol in deionized water. As shown in experiments #3, 4 and 5, we did not observe any permeate flux enhancement by using PVDF films either driven by a function generator, or by a pulse generator (higher voltages). The membrane vibration frequency was also varied from 20 KHz to 500 kHz without any positive

Table 2

SUMMARY OF THE EXPERIMENTS CONDUCTED BY USING PVDF MEMBRANES AS PIEZOELECTRIC TRANSDUCERS

No.	Description of Experiment	Results/Conclusion
1	Test of a RTV coated PVDF piezoelectric transducer driven by a function generator. An uncoated PVDF film was used to detect the signals generated by the above transducer under a pressure of 50 psig. The sensor and transducer membranes were separated by a rubber gasket.	The RTV coated PVDF piezoelectric transducer is not dampened as the vibration signals are detected by the sensor PVDF film.
2	Same as in Expt. #1 except that the sensor PVDF film is also coated by RTV to assess the effect of dampening	Vibration signals are detected by the RTV coated PVDF sensor indicating no major reduction of piezoelectric properties due to the RTV coating.
3	Filtration of a 0.5% solution of poly (vinyl-pyrrolidone), 40,000 Mw, through polysulfone UF membrane, 10,000 MWCO. The PVDF piezoelectric transducer was driven by a function generator.	No enhancement of permeate flux was observed.
4	Filtration of a 0.1% solution of poly (vinyl-pyrrolidone), 40,000 Mw through polysulfone UF membrane, 10,000 MWCO. The PVDF piezoelectric transducer was driven by a function generator.	No enhancement of permeate flux was observed.
5	Filtration of a 0.1% solution of poly (vinyl-pyrrolidone), 40,000 Mw, through polysulfone UF membrane, 10,000 MWCO using a glass micro fiber filter as backing. A pulse generator was used to drive the PVDF piezoelectric transducer.	No enhancement of permeate flux was observed.
6	Filtration of 0.2% solution of poly (ethylene glycol), 15,000 Mw through regenerated cellulose UF membrane, 10,000 MWCO using a mylar coated bimorph PVDF piezoelectric transducer.	No enhancement of permeate flux was observed.
7	Filtration of a solution of 0.5% poly (ethylene glycol), 10,000 Mw, through regenerated cellulose UF membrane, 10,000 MWCO assisted by a polymorph PVDF piezoelectric transducer driven by a pulse generator.	No enhancement of permeate flux was observed.

result. Because of the weak mechanical properties of PVDF, we believe that the electromechanical conversion is insufficient to enhance the permeate flux during ultrafiltration.

In the attempt to overcome this drawback bimorph and polymorph membranes were used. The polymorph PVDF films consisted of four PVDF perforated films, joined to each other by conducting silver ink. Leads connected to different constituent membranes were joined so that the surfaces of PVDF films joined together by silver ink had the same polarity as shown in Figure 11. The polymorph film was encapsulated in a thin coating of RTV. The bimorph was similarly made of two PVDF films.

However, the results of ultrafiltration tests with polymorph PVDF films were not encouraging either (Experiments # 6 and 7).

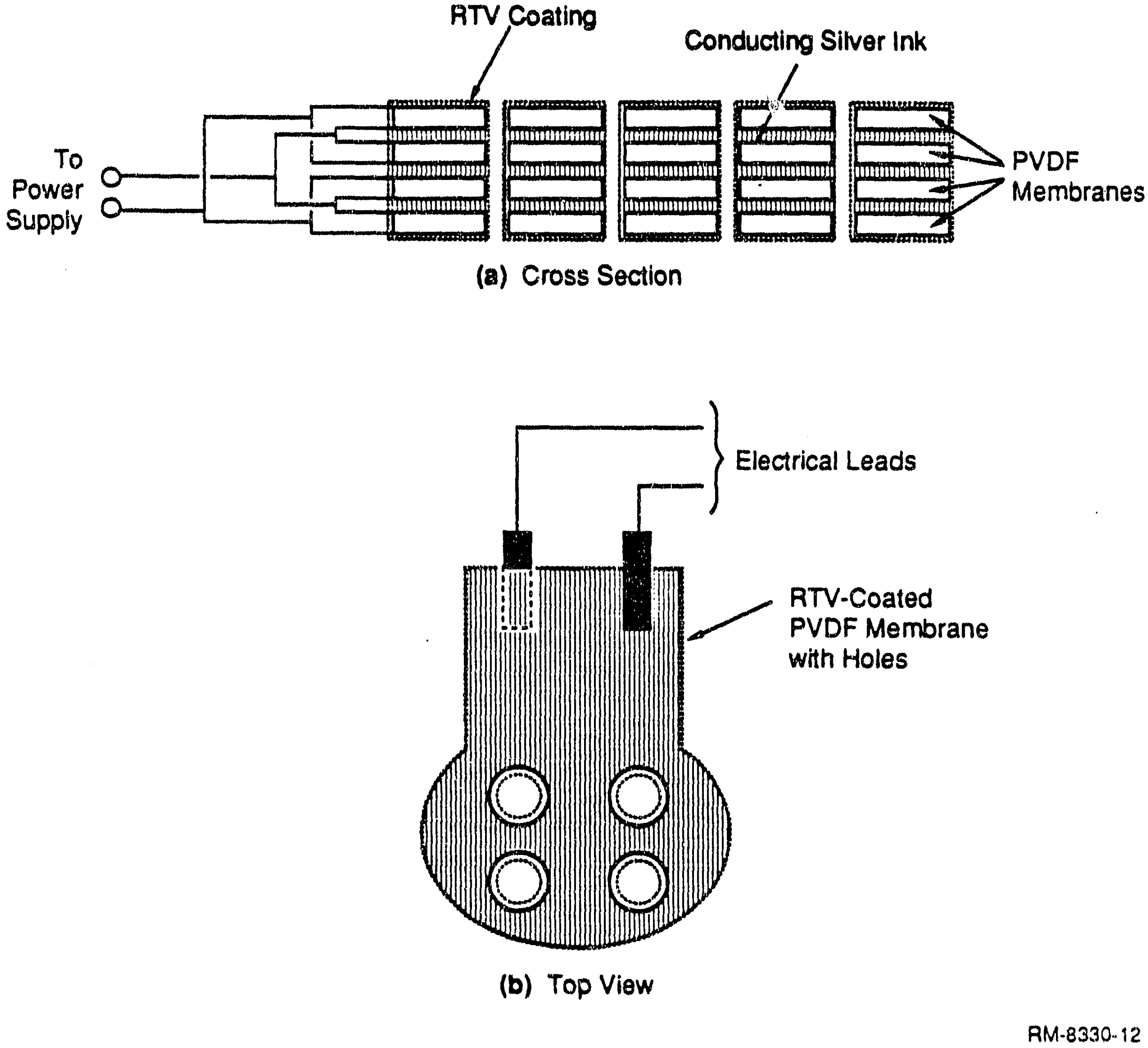

Figure 11. Schematic representation of the PVDF polymorph membrane with holes.

FURTHER DEVELOPMENT

We have demonstrated the feasibility of piezoelectrically assisted ultrafiltration as a cost effective means to reduce fouling and enhance the flux through ultrafiltration membranes. PZT discs are very effective piezoelectric drivers that presumably promote local turbulence next to UF membranes. Because of their small size and ease of adaptability to ultrafiltration apparatus, PZT transducers may be feasible for use in spiral wound membrane modules.

In our future work we will continue the development of laminated membranes fabricated from commercially available UF membranes and piezoelectric materials. We will seek to develop a second generation of piezoelectric drivers for ultrafiltration based on porous piezoelectric ceramic microfilters. The piezoelectric elements will be studied to optimize their vibration amplitude and frequency. Tests at different frequencies will be carried out by changing the size of the piezoelectric element to assess the effect of frequency on the permeate flux enhancement. Although preliminary experiments can be carried out by driving the piezoelectric elements off resonance, such experiments lead to excessive heat generation and inefficient use of power. Ultimately, the experiments will have to be carried out by changing the disc size.

Detailed long term flat sheet ultrafiltration experiments with the newly developed piezoelectric membranes, both laminated and porous, will be carried out. We will characterize the permeation behavior of prototypical piezoelectric driver configurations with a range of flows, and with different UF membranes. Continuous and pulse piezoelectrically enhanced ultrafiltrations will be conducted and compared on model feed solutions, such poly(ethylene glycol) and dextran with polysulfone and regenerated cellulose. We will also configure the piezoelectric elements in a manner prototypical of that required for incorporation into a spiral module.

Economic evaluations will be also carried out to calculate the process economics for piezoelectrically assisted ultrafiltration of whey protein and oil/solvent mixtures in deasphalting operations.

Appendix

ECONOMIC EVALUATION OF SOLVENT RECOVERY IN DEASPHALTING OPERATIONS WITH PIEZOELECTRICALLY ENHANCED ULTRAFILTRATION MEMBRANES

ECONOMIC EVALUATION OF SOLVENT RECOVERY IN DEASPHALTING OPERATIONS WITH PIEZOELECTRICALLY ENHANCED ULTRAFILTRATION MEMBRANES

To assess the economic viability of our piezoelectric (PZ) technology, we have considered the recovery of solvent from an oil deasphalting operation. We mentioned this application in our original proposal as one offering good potential for national energy savings. Much of our information on this process will be drawn from a report recently submitted to EG&G–Idaho (Gottschlich and Roberts, 1990).

Deasphalting of oil is a widely-practiced process in refining. In traditional deasphalting, solvent recovery is done by evaporating the solvent (Figure A-1). The energy consumed in the evaporation exceeds 1000 Btu/lb of deasphalted oil (DAO; thermal energy equivalent; Table A-1). The capital investment for this facility is $4.3 million (Table A-2). The "discounted cash flow" cost of deasphalting oil by this process is 1 cent/lb of deasphalted oil (DAO; Table A-3).

Ultrafiltration (UF) membranes, in principal, can recover the solvent without a phase change, thereby saving substantial energy (Figure A-2). Membranes are not feasible to perform the complete separation, and some assistance by conventional evaporation is required. The energy consumed by this "hybrid" process is 449 Btu/lb of DAO (Table A-4), a savings of 924 Btu/lb of DAO. If this savings were realized by the commercial sector, the national energy savings would be 28 trillion Btu/yr (based on 1985 deasphalting capacity in U.S. of 283,000 barrels/day).

The installation of a membrane unit is, however, rather capital intensive, and the total required capital cost of this process is nearly double that of the traditional process (Table A-5). As a consequence, the total processing cost decreases to only 0.876 cents/lb DAO (Table A-6). This rather minor decrease in the cost versus the traditional process is probably not enough to convince

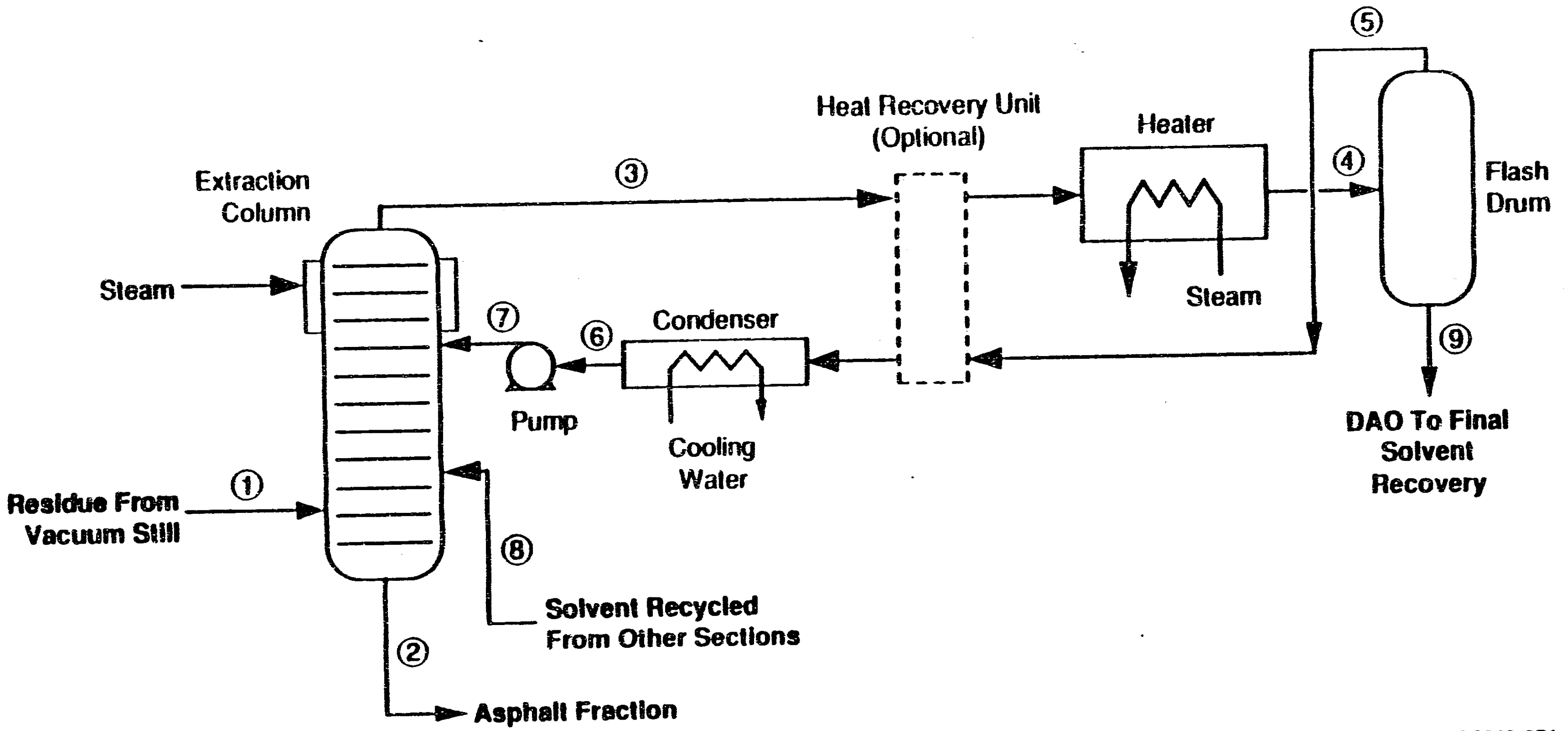

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Figure A-1. Conventional process for primary solvent recovery.

Table A-1
UTILITY REQUIREMENTS FOR SOLVENT RECOVERY IN SOLVENT DEASPHALTING WITH EVAPORATION TECHNOLOGY ALONE
(Basis: 95% DAO, 100,000 lb DAO/hr)

	Utility				
	Steam				
	Extraction Tower	Evap Heater	Water	Electricity	Total
Mass flow (lb/lb DAO)	0.18	1.20	43.5	---	---
Energy flow (Btu/lb DAO)	150	991	-1,085	9.91	---
Thermal energy equivalent* (Btu/lb DAO)	177	1,166	---	29.7	1,373

Source: Gottschlich and Roberts (1990).
* Thermal energy equivalents are based on an electric power generation efficiency of 33% and a steam generation efficiency of 85%.

Table A-2
CAPITAL INVESTMENT FOR SOLVENT RECOVERY IN SOLVENT DEASPHALTING WITH EVAPORATION TECHNOLOGY ALONE
(Basis: 95% DAO, 100,000 lb DAO/hr)

Process Unit	Installed Cost ($1,000)
Extraction tower	$ 1,410
Process heater	414
Flash drum	736
Condenser	1,084
Evaporation pump	122
General services	565
TOTAL	$ 4,332

Source: Gottschlich and Roberts (1990).

Table A-3
SOLVENT DEASPHALTING WITH EVAPORATION TECHNOLOGY: ESTIMATED ANNUAL OPERATING COSTS AND PROCESSING COST
(Basis: 95% DAO, 100,000 lb DAO/hr)

	Thousands of Dollars per Year	Cents per Pound of DAO	Percent of Total
Maintenance materials	$ 87	0.011	1.1
Labor			
Operating labor	$ 39	0.005	0.5
Supervision	22	0.003	0.3
Maintenance labor	108	0.014	1.4
Benefits	60	0.008	0.8
Total labor	$ 229	0.029	2.9
Utilities			
Steam	$ 5,460	0.691	68.9
Electric power	115	0.015	1.5
Cooling water	234	0.030	3.0
Total utilities	$ 5,808	0.736	73.3
Fixed costs			
Corporate costs	$ 159	0.020	2.0
General administrative expenses	34	0.004	0.4
Property taxes and insurance	234	0.014	1.4
Total fixed costs	$ 301	0.038	3.8
Total annual operating costs	**$ 6,425**	**0.814**	**81.0**
Capital-related charges and income tax	$ 1,503	0.190	19.0
Total processing cost	**$ 7,928**	**1.004**	**100.0**

Source: Gottschlich and Roberts (1990).

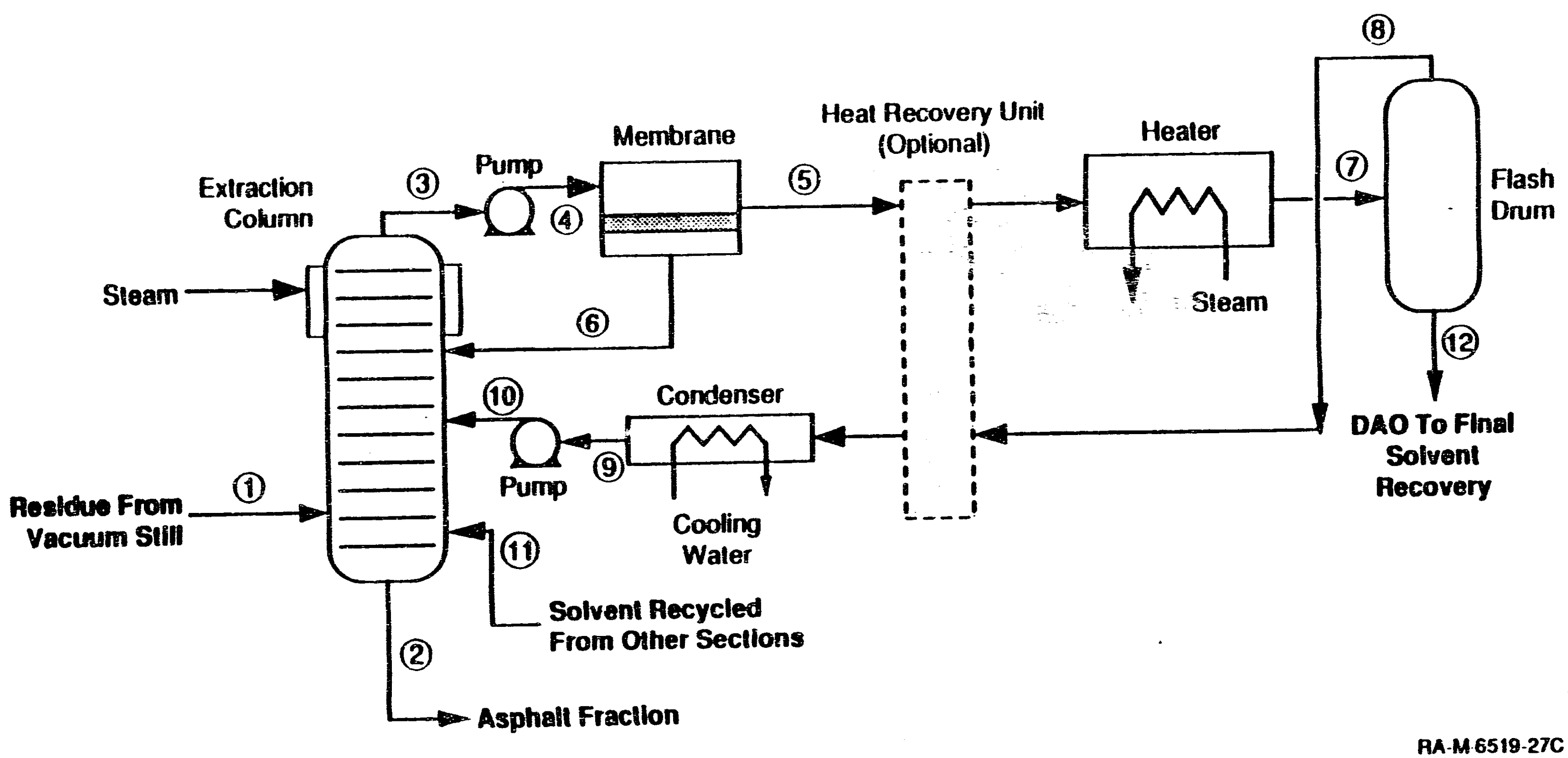

Figure A-2. Membrane/evaporation hybrid process for primary solvent recovery.

Table A-4
UTILITY REQUIREMENTS FOR SOLVENT RECOVERY IN SOLVENT DEASPHALTING WITH MEMBRANE/EVAPORATION HYBRID TECHNOLOGY
(Basis: ϕ_m = 0.5; 95% DAO; 100,000 lb DAO/hr)

	Utility					
	Steam			Electricity		
	Extraction Tower	Evap Heater	Water	Memb Pump	Evap Pump	Total
Mass flow (lb/lb DAO)	0.044	0.337	11.2	---	---	---
Energy flow (Btu/lb DAO)	36.4	278	-279	23.6	2.86	---
Thermal energy equivalent (Btu/lb DAO)	42.8	327	---	70.8	8.58	449

Source: Gottschlich and Roberts (1990).

Table A-5
CAPITAL INVESTMENT FOR SOLVENT RECOVERY IN SOLVENT DEASPHALTING WITH MEMBRANE/EVAPORATION HYBRID TECHNOLOGY
(Basis: ϕ_m = 0.5, 95% DAO; 100,000 lb DAO/hr)

Process Unit	Installed Cost ($1,000)
Membrane unit*	$ 3,863
Membrane pump	296
Extraction tower	2,035
Process heater	146
Flash drum	591
Condenser	344
Evaporation pump	38
General services	1,097
TOTAL	$ 8,409

Source: Gottschlich and Roberts (1990).
* Membrane replacement = $1,610,000

Table A-6
SOLVENT DEASPHALTING WITH MEMBRANE/EVAPORATION HYBRID TECHNOLOGY: ESTIMATED ANNUAL OPERATING COSTS AND REVENUE REQUIREMENTS
(Basis: ϕ_m = 0.5; 95% DAO, 100,000 lbs DAO/hr)

	Thousands of Dollars per Year	Cents per Pound of DAO	Percent of Total
Maintenance materials	$ 168	0.021	12.4
Labor			
Operating labor	$ 39	0.005	0.6
Supervision	37	0.005	0.5
Maintenance labor	210	0.027	3.0
Benefits	101	0.013	1.5
Total labor	$ 388	0.049	5.6
Utilities			
Steam	$ 1,504	0.190	21.8
Electric power	307	0.039	4.4
Cooling water	60	0.008	0.9
Total utilities	$ 1,871	0.237	27.1
Fixed costs			
Corporate costs	$ 138	0.020	2.0
General administrative expenses	57	0.007	0.8
Property taxes and insurance	210	0.027	3.0
Total fixed costs	$ 406	0.051	5.9
Total annual operating costs	**$ 2,833**	**0.359**	**41.0**
Capital-related charges and income tax	$ 4,082	0.517	59.0
Total processing cost	**$ 6,914**	**0.876**	**100.0**

Source: Gottschlich and Roberts (1990).

industry to switch technologies. Keller (1990) has observed that industry rarely spends capital to conserve energy and often spends energy if it saves on capital. Therefore, to realize the substantial energy savings potential of ultrafiltration, a lower capital cost for the membrane system would be highly desirable.

Our PZ membrane system lowers the capital cost of using membranes. It does so because it maintains a higher flux than in a conventional membrane, and we therefore need less membrane to get a given job done. The capital cost per unit area of our PZ membrane will likely exceed that of a conventional membrane. We have sought consultation with an industrial membrane supplier (Koch Membrane Systems) on the issue of capital cost. Dr. Jamie Monet, director of research at Koch Membrane Systems, advised us that our raw materials cost would be typically a 20% increase in today's raw material cost. In addition, he advised us that unless we make our driver film thinner, we will get 25% less membrane area in a module housing. Raw materials cost is only a portion of the overall module cost, so a 20% raw materials cost increase does not increase the module cost by 20%. However, Dr. Monet felt that overall, there would be a 50% cost increase for a complete UF system. We will use this 50% figure in our economic analysis. In addition, we have done our analysis with a factor of 2 (100%) capital cost increase to assess a *worst case* scenario.

Our PZ driver consumes energy, and therefore, the energy consumption with our PZ membranes exceeds that for a conventional membrane.* We take our energy consumption to be the same *per unit of DAO* as in our annual report dated November 1990. This assumption *over* estimates the power consumption because an alternative logical assumption is to take the power *per*

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* Another option for taking advantage of the PZ membrane is to reduce the pump feed pressure so that the same flux is achieved as with a conventional membrane. This approach saves energy, but the energy consumed by the UF feed pump is so trivial compared with the solvent evaporator that it is of no economic consequence to reduce the feed pump pressure.

unit of membrane area the same as in our annual report. However, the energy consumption is so small that our *over* estimation does not materially influence our analysis and is appropriate given the need to address quickly DOE's questions about the economic attractiveness of this process.

We take our flux enhancement to be a factor of 8 because this increase has been observed with our PZT disks (pg. 9 of annual report). With these factors, the energy consumption of the PZ membrane hybrid system rises slightly to 463 Btu/lb (Table A-7), and even with a 50% capital cost increase for our PZ membrane system, the total system capital cost decreases substantially to $4,800 (Table A-8). The total processing cost now drops to 0.574 cents/lb of DAO (Table A-9). We have done similar calculations for the factor of 2 (worst case) capital cost increase scenario, as noted in Table A-10.

The PZ membrane process is the most economical of all three processes *whether the membrane capital cost increase is 50% or even 100%* (factor of 2; Table A-10). Significantly, the capital cost of the entire process with our PZ membrane is only 11% to 17% above a process with *no membranes at all.* Further, the processing cost with respect to traditional technology is 40% to 50% less, a strong commercial incentive. Therefore, the PZ membrane makes it much more likely that industry will adopt energy-saving membrane technology. In this *one* application, the energy saving potential is about 910 Btu/lb DAO or 27 trillion Btu/yr nationwide. The electricity consumed by the piezoelectric portion of the PZ membrane comes to 4.7 Btu of electricity/lb of DAO ($1.377 \cdot 10^{-3}$ kWh/lb). At an electricity cost of 5¢/kWh, the additional electricity cost of running the PZ membrane adds $\$6.9 \cdot 10^{-5}$/lb of DAO. This figure is completely insignificant which is why (at least in this case) the best way to use the PZ membrane is to reduce the required membrane capital cost so that the intrinsic energy saving potential of ultrafiltration is commercially attractive.

Table A-7
UTILITY REQUIREMENTS FOR SOLVENT DEASPHALTING WITH PIEZOELECTRIC MEMBRANE HYBRID TECHNOLOGY
(Basis: ϕm = 0.5; 95% DAO; 100,000 lb DAO/hr)

	Utility						
	Steam			Electricity			
	Extraction Tower	Evap Heater	Water	Memb Pump	PZ Driver	Evap Pump	Total
Mass flow (lb/lb DAO)	0.044	0.337	11.2	---	---	---	---
Energy flow (Btu/lb DAO)	36.4	278	-279	23.6	4.7	2.86	---
Thermal energy equivalent (Btu/lb DAO)	42.8	327	---	70.8	14.1	8.58	463

Table A-8
CAPITAL INVESTMENT FOR SOLVENT DEASPHALTING WITH PIEZOELECTRIC MEMBRANE HYBRID TECHNOLOGY WITH 50% MEMBRANE CAPITAL COST INCREASE
(Basis: ϕm = 0.5, 95% DAO; 100,000 lb DAO/hr)

Process Unit	Installed Cost ($1,000)
Membrane unit*	$ 724
Membrane pump	296
Extraction tower	2,035
Process heater	146
Flash drum	591
Condenser	344
Evaporation pump	38
General services	626
TOTAL	$ 4,800

* Membrane replacement = $302,000

Table A-9
SOLVENT DEASPHALTING WITH PIEZOELECTRIC MEMBRANE HYBRID TECHNOLOGY: ESTIMATED ANNUAL OPERATING COSTS AND REVENUE REQUIREMENTS WITH 50% MEMBRANE CAPITAL COST INCREASE
(Basis: ϕ_m = 0.5; 95% DAO, 100,000 lbs DAO/hr)

	Thousands of Dollars per Year	Cents per Pound of DAO	Percent of Total
Maintenance materials	$ 96	0.012	2.1
Labor			
Operating labor	$ 39	0.005	0.9
Supervision	28	0.004	0.6
Maintenance labor	120	0.015	2.7
Benefits	63	0.008	1.4
Total labor	$ 250	0.032	5.5
Utilities			
Steam	$ 1,504	0.190	33.2
Electric power	361	0.046	8.0
Cooling water	60	0.008	1.3
Total utilities	$ 1,925	0.244	42.5
Fixed costs			
Corporate costs	$ 79	0.010	1.7
General administrative expenses	33	0.004	0.7
Property taxes and insurance	120	0.015	2.7
Total fixed costs	$ 231	0.029	5.1
Total annual operating costs	$ 2,502	0.317	55.3
Capital-related charges and income tax	$ 2,026	0.257	44.7
Total processing cost	$ 4,528	0.574	100.0

Table A-10
COMPARISON OF THREE PROCESSES FOR SOLVENT RECOVERY IN DEASPHALTING OPERATIONS

	Process			
			PZ Membrane Hybrid with Membrane Capital Cost Increase	
	Traditional	Conventional Membrane Hybrid	50%	100%
Energy (Btu/lb DAO)	1,373	449	463	463
Capital Cost (thousands of $)	4,332	8,409	4,800	5,078
Overall Processing Cost (¢/lb DAO)	1.004	0.876	0.574	0.593

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Gottschlich, D. E., and D. L. Roberts, "Energy Minimization of Separation Processes Using Conventional/Membrane Hybrid Systems," Final Report to EG&G–Idaho, Subcontract C88-101848, September 28, 1990.

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