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and Development Program Report No. 121

Pilot Scale Testing of Membrane Desalination System Utilizing Novel Two-Phase Cleaning Technology



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Prepared for Reclamation Under Agreement No. 03-FC-81-0922

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1. Executive Summary

Novaflex Technologies (Novaflex) has developed a novel two-phase cleaning technology that will allow improved economics for treating high total dissolved solids (TDS) dairy wastewater by using spiral-wound reverse osmosis (RO) membranes. The investigators have defined the fouling method and the two-phase flow process parameters for cleaning the RO membranes and have also optimized cleaning frequency using simulated dairy wastewater. Test results indicate that we are able to achieve the design output of the RO skid without need for additional microfiltration or ultrafiltration pre-treatment steps. In the demonstration phase of the study, Novaflex has confirmed the cleaning protocols of spiral RO membranes using actual dairy wastewater obtained from Readington Farms, New Jersey.

Novaflex's two-phase cleaning requires a far shorter period of time and less chemical/water consumption compared to conventional cleaning. Conventional cleaning normally consumes 4-8 hours, whereas the Novaflex process takes less than 30 minutes, with minimal use of liquids. There are also significant savings during the rinse cycle. While the Novaflex process uses approximately 1 gallon of water to rinse the three housings, conventional cleaning consumes 50 gallons. Additionally, pre-filtration with 30-micron filter tests was satisfactory.

Cleaning membranes fouled with actual wastewater proved to be more difficult than cleaning membranes fouled with the simulated wastewater due to higher TDS and total suspended solids in actual wastewater, but the ability to restore and sustain flux with the two-phase cleaning matched that of conventional methods. The conventional cleaning was particularly ineffective and impractical with the actual dairy wastewater, showing very little or no improvement after cleaning.

Economic assessment of an RO membrane system for a medium-sized dairy plant utilizing Novaflex's two-phase cleaning technology showed that the treatment cost per 1,000 gallons can be reduced from \$130 to \$59, which means an annual savings of 54.44 percent (%). This percent savings will increase after the cost to build the treatment system is fully amortized over 7 years. After 7 years, the cost of treating 1,000 gallons of wastewater will decrease to \$9, which translates into an annual savings of approximately 93.35%. Since a commercialized system with two-phase cleaning technology is scalable to any plant's wastewater production, it is clear that these results would be applicable to other industries.

2. Background and Introduction

Novaflex Technologies (Novaflex) has developed and patented a membrane cleaning process in which an air stream is mixed with a small amount of a special cleaning solution to form a two-phase mixture with high air-to-liquid ratio in the range of 50:1 to 6000:1, which, when injected into membrane feeding channels at velocities greater than 30 feet per second, achieves effective cleaning in a short period of time (less than 10 minutes) [1-7]. The two-phase flow proven to be effective includes liquid droplets that impact the surface of the membrane at high velocities and create localized high shear stresses that effectively remove foulants from the surface without causing damage to the membrane layer, as demonstrated in previous studies [1-7]. This new cleaning process achieves dramatic cleaning results compared to conventional liquid flushing. The two-phase cleaning process employs a small volume of safe cleaning agents to achieve effective cleaning. The two-phase cleaning process has been successfully applied to several membrane types and module configurations, including spiral-wound reverse osmosis (RO) membranes where surface cleaning is sufficient to restore the flux and avoid deterioration of pressure drop during prolonged operation – see Final Report [8]. Since rapid cleaning with minimal waste stream generation can be accomplished with this new process, more frequent membrane cleaning may be used to keep the average operation flux at higher levels during water treatment.

The means for economically and efficiently managing RO membrane cleaning during water treatment processes will be dramatically improved by implementing this new cleaning process, and a demonstration of the applicability of this technology at the pilot-scale is therefore necessary. A water treatment system, including built-in, two-phase flow cleaning capability, would solve some aspects of the fundamental limitations in cleaning due to fouling of RO membranes when influent brackish water with low quality is used. RO membrane fouling normally results in reduced flux, increased energy consumption, reduced efficiency, and increased operating costs, and this must be compensated for with high capital costs by including excess RO membrane area and oversized systems in the original design. It has been well documented that the overall RO purification costs are driven by the membrane surface area required, its corresponding equipment system, the associated pre-treatment required to protect or optimize RO process, as well as the operating and maintenance costs (energy, consumables, labor, etc.). Fouling drives up all of these cost elements and, therefore, becomes the major limitation in the wider use of membranes in many water purification applications.

High total dissolved solids (TDS) and high organic content hamper the use of RO separation and make it very costly, or even impractical. The wastewater used during this study was obtained from an industrial source that has high levels of TDS and organics generated by dairy processing operations. This wastewater is impossible to reuse, cannot be discharged, and requires high-cost treatment. The membrane process developed during this study, including the use of the two-phase cleaning, allows the production of drinking quality water and, therefore, allows water to be reused and recycled in this industrial operation. Furthermore, other water sources of natural origin can be processed according to the protocols developed during this study following the same process parameters.

Membrane fouling is a limiting factor in using RO separation processes in many water treatment or industrial applications and is, therefore, considered a significant factor affecting the availability of water resources. Current approaches for using RO membranes are based on preventing fouling in the first place by pre-treating influent streams by either chemical or physical means. Chemical means include the addition of antiscaling agents and biocides in the feed stream, where they retard the formation of biofouling and inorganic scale. Physical pre-treatments usually comprise flocculation processes and additional membrane separation steps based on either microfiltration (MF) or ultrafiltration (UF).

RO membrane foulants may be grossly classified as: biofilm, inorganic silt and particles, organic foulants, and inorganic scaling from precipitated salts, or a combinations of these. Scale formation is attributed to the concentration polarization that leads to precipitation of salts on the surface of RO membranes. As the level of salts and biological contaminants in the feed stream increases, pre-treatment is required to prevent significant fouling and to ensure that the process is cost effective. Therefore, economic limitations of current plant designs may be primarily due to fouling, and new plant designs and processes are needed to make the use of RO membrane separation more effective.

The cleaning technology developed during this study enables a smaller RO treatment system to be used in water treatment when the influent water source is difficult to use with current technologies. Conventional membrane cleaning methods, such as liquid circulation, may not be effective in achieving flux recovery, and frequent cleaning or membrane replacement makes such conventional processes impractical.

The major objectives of the pilot scale testing study were to:

1. Demonstrate, through simulated operations, that the use of the two-phase cleaning technology will result in higher average flux rates and improved efficiency in treating saline and impaired water (where RO is very difficult or impractical to use) to produce potable (or better) quality standards.

2. Evaluate alternative RO pre-treatment in addition to submersed microfiltration/membrane bioreactor (MF/MBR) or ultrafiltration/membrane bioreactor (UF/MBR).
3. Determine the most cost-effective hardware design needed to deliver the two-phase clean-in-place (CIP) membrane cycle to multiple pressure vessels in an RO skid.
4. Evaluate operational and utility tradeoffs and determine optimal cleaning cycles, protocols, operating conditions, cleaning frequencies, and cleaning costs.
5. Determine the minimum air requirements for the two-phase cleaning and define the practical limits on the number of elements and vessels that can be cleaned-in-place with a cost-effective compressor or blower.
6. Develop and validate a process, protocol, and design to be scaled up to a 15,000-gallons-per-day (gpd) wastewater plant pending for a New Jersey dairy packaging facility to purify mineralized (high TDS), chemically and biologically impaired water to drinking water quality to be reused in other parts of the dairy packaging operation.

The ability to effectively restore membrane performance in a short period of time with minimal consumption of chemicals and water has significant, positive economic implications for producing usable water, both from the initial membrane treatment of saline or brackish water, as well as from the treatment of high TDS or otherwise impaired wastewater for reuse. Many applications can utilize the RO water treatment system technology that incorporates cleaning with the optimized two-phase flow process, especially in combination with an appropriate pre-treatment technology, with or without the bioreactor, depending on the quality of the feed water used.

The two-phase cleaning process will allow significantly improved operating performance and affordability of water purification/desalting technologies. The outcome of the project study can be applied to any application employing membrane purification of water for potable use and will also apply to a wide array of potential reuse and recycling processes. Furthermore, the small system designed and used in this study is expected to be ideal for small communities where 5,000 to 20,000 gpd of potable water is sufficient. This system can also be used for disaster relief, in remote areas, or in military operations in locales where good water is not available.

The primary objective of the Desalination and Water Purification Research and Development Program, to develop more economical methods to produce drinking

water or usable water, can be met at both ends of the water use cycle, from the initial or primary treatment from surface or well source, to the recovery and reuse of wastewater. When design and economic questions are addressed, this new cleaning technology, as incorporated in an integrated water treatment system, will make RO membrane purification processes affordable in many more applications and in many more geographic locations.

3. Conclusions and Recommendations

Over repeated fouling and cleaning cycles, the Novaflux two-phase cleaning process can restore and sustain flux in a real-world application. The basic parameters developed in the simulated wastewater testing proved effective in the case of real wastewater, independent of the type of pre-filtration that was used and the severity of the wastewater that was being used to foul the membranes.

Novaflux's two-phase cleaning matches or surpasses conventional cleaning in a far shorter period of time and with less chemical/water consumption. Conventional cleaning can take anywhere from 4-8 hours, whereas the Novaflux process takes less than 30 minutes. Conventional cleaning requires the use of a larger volume of cleaning chemistry, in excess of twice as much (30 gallons versus 15 gallons of diluted solution) to clean versus the Novaflux process. There are also significant savings during the rinse cycle. The Novaflux process takes only minutes and uses approximately 1 gallon of water to rinse the three housings, whereas the conventional rinse can take upwards of 50 gallons and close to an hour to complete a similarly effective rinse cycle as the two-phase rinsing.

Pre-filtration, using UF, 5-micron, and 30-micron pre-filters, was tested. It was a very positive and promising result that the 30-micron pre-filtration tests were no worse (and in fact were better in the limited comparison) than the 5-micron and UF pre-filtration tests. This shows that the Novaflux process can be effective even at the minimum level of pre-filtration with very high TDS and total suspended solids (TSS) wastewater.

Cleaning membranes fouled with actual wastewater proved to be more difficult than cleaning membranes fouled with the simulated wastewater due to higher TDS and TSS in actual wastewater, but the ability to restore and sustain flux matched the conventional methods in both cases. The conventional cleaning was particularly ineffective with the actual wastewater, showing very little or no improvement after cleaning.

The air consumption necessary for effective cleaning was far less than initially anticipated. Novaflux began with the notion that upwards of 200 standard cubic feet per minute (scfm) would be needed to clean each housing (two elements), but after many tests to optimize this parameter, it was determined that effective cleaning occurred in the 50-70 scfm range, significantly less than expected and far more practical. This number could be reduced even further in future tests. The current skid could be cleaned (each of the three vessels simultaneously in parallel) in less than 30 minutes with a supply of air less than 200 scfm.

Liquid (alkaline cleaning solution)-to-air ratio used during two-phase cleaning is most effective in the range of 1:250-1:500, with a pH in the range of 11.5-12.0.

4. System Description

The pilot RO system to study the two-phase flow cleaning was designed to produce 4,000 gpd of water meeting the U.S. Environmental Protection Agency secondary drinking water standard for TDS from a high TDS feed. Its operational features include three pressure vessels, referred to as V1, V2, and V3, each accommodating two 4-inch by 40-inch RO membrane elements. These vessels are connected in series to maintain a concentrated flow during fouling. One 7.5-horsepower (HP) feed pump, capable of 500 pounds per square inch (psi), was used to provide adequate feed wastewater to these three vessels, run in series, at manufacturer-specified and standard operating pressure to achieve the targeted higher recoveries. The designed operating pressures were maintained below 300 psi. The system was designed to operate at recoveries from as low as 30% up to 75% by varying pump feed and recirculation flow rates. It could also simulate the performance of membrane elements in the lead end of a large system or the last elements in the concentrate end of a large system, as well as any of the elements in between. The permeate quality was achieved with a single pass of the water through membrane.

A fourth vessel, V4, accommodating four 4-inch by 40-inch RO membrane elements, was installed so that it could be fouled in series with the first three vessels or cleaned independently without interfering with or being interfered by the operation of the first three vessels.

Cleaning was performed by both conventional liquid flushing, which was used to set a baseline, and by two-phase cleaning. Each vessel was cleaned separately. A complete cleaning cycle with up to 18 cleaning steps for each vessel could be programmed by the operator. This cleaning cycle was repeated for each of the three vessels in sequence. The cleaning cycle could also be repeated for the fourth vessel.

The membrane elements selected for use in the pilot RO system are characterized as thin film and high flux, and have excellent sodium chloride rejection. These elements are designed for brackish water desalination. They were considered the best fit with the design goal of the pilot RO system.

The schematic and a photograph of the system designed and developed during this study are shown in figures 1a and 1b.

4.1 Major System Features and Functions

The system was designed to treat real-life wastewater using the first three vessels and to perform an off-line parameter study using the fourth vessel. The main

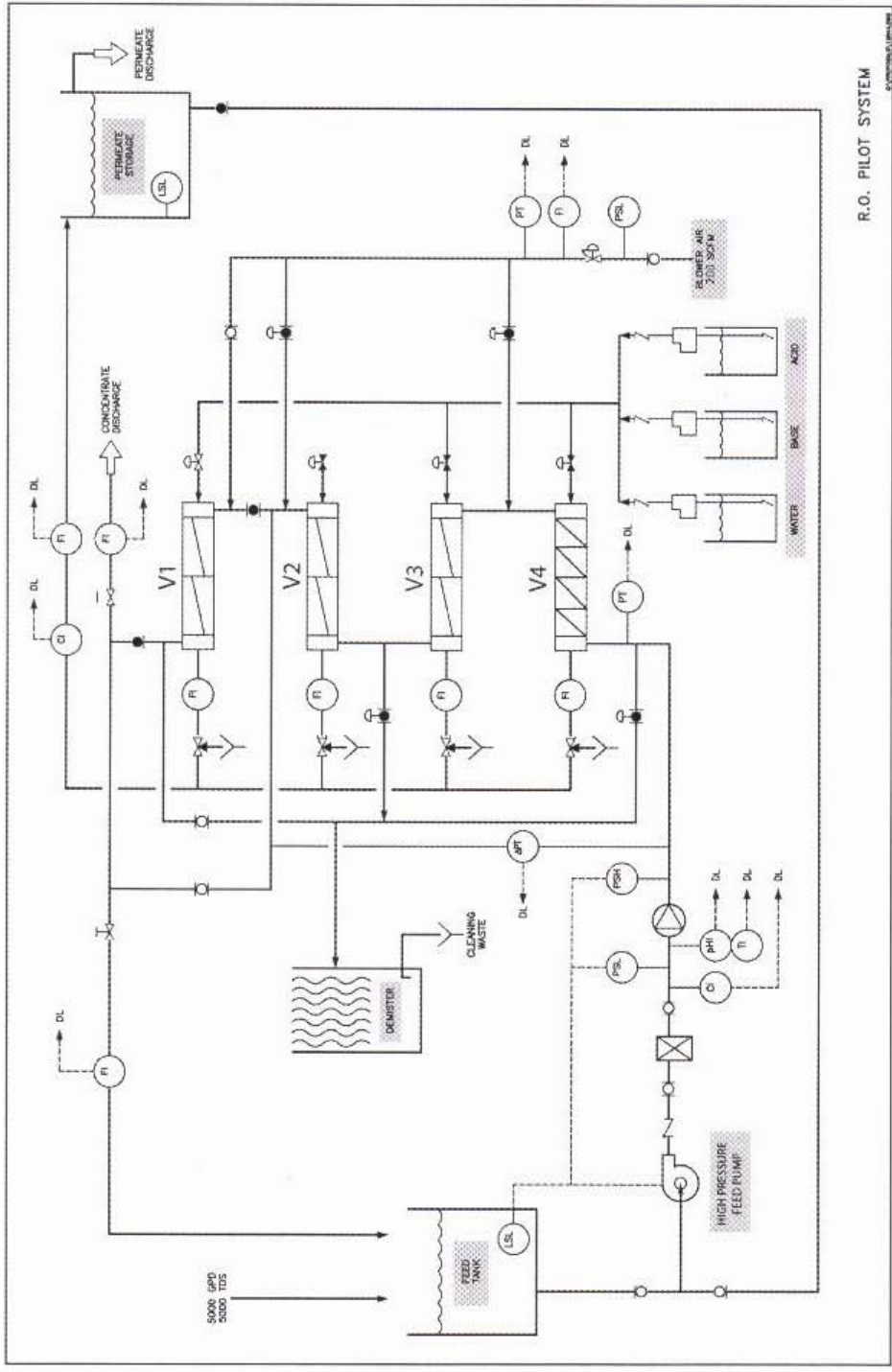


Figure 1a. A schematic of the 4,000-gpd dairy wastewater system designed and developed during this study.

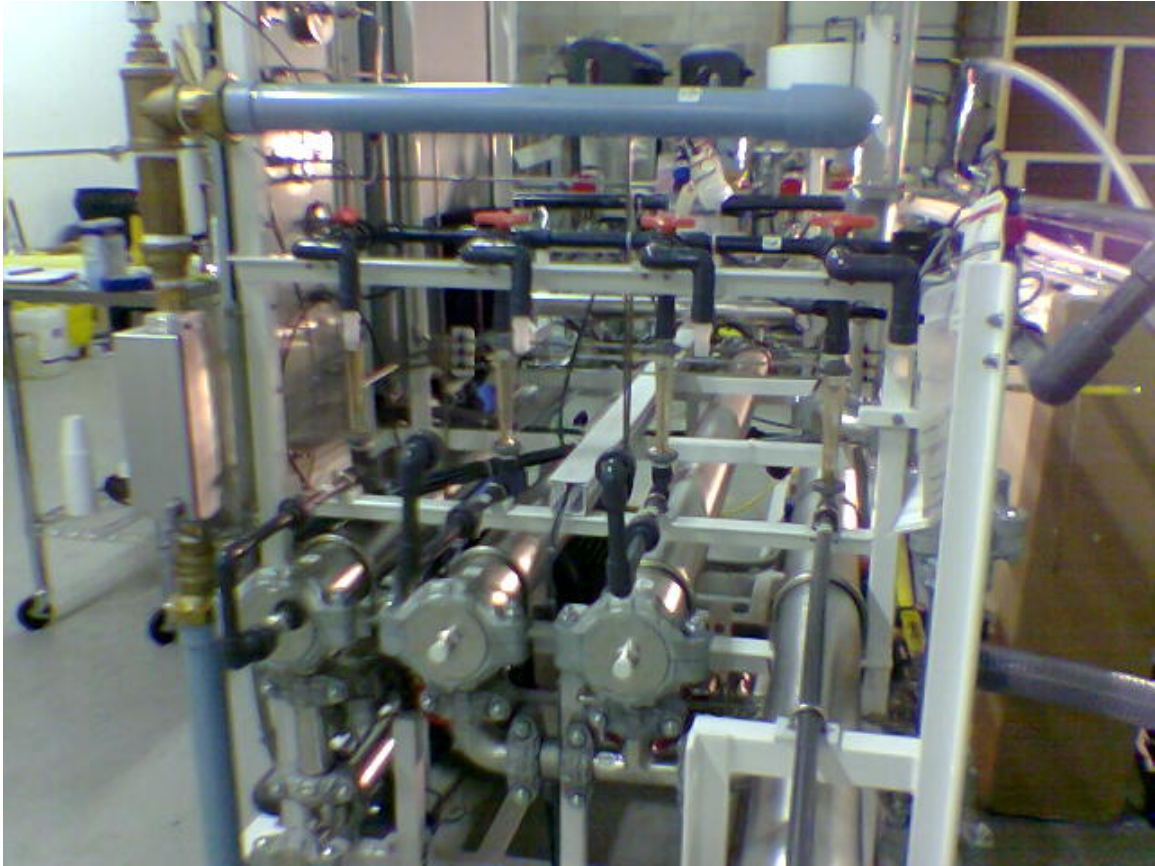


Figure 1b. The 4,000-gpd dairy wastewater system designed and developed during this study.

structure of the skid included a four RO vessel system, a two-phase cleaning module with associated cleaning solution connections, and a control panel with a programmable logic controller (PLC) program. They are briefly discussed as follows.

4.1.1 RO System

1. The RO system consists of four stainless steel vessels integrated with supporting instruments, pumps, piping, etc. The design included:
 - a. Six GE Osmonics AG4040 RO elements, low-fouling design with rejection to enable less than 500 TDS product even when operating at 75% recovery with concentrate recirculation.
 - b. Three 4-inch-diameter stainless steel vessels, each designed to house two 40-inch-long RO membranes with a three-way permeate valve that was used to sample the flows.

- c. A fourth vessel with some isolation valves was used to enable operation in series with the RO system or was operated off-line in cleaning mode.
 - d. A 7.5-hp feed pump, capable of 500 psi, to provide adequate operating pressure at higher recoveries. In this study, the expected operating pressures were below 300 psi. A variable speed drive to control the pump speed and operating pressure was integrated in the design.
 - e. A pre-filter housing and filter element rated at 30 microns.
 - f. Piping and valves to interconnect all the equipment components.
 - g. One structural frame assembly to support the equipment, piping, instruments, and electrical devices.
2. A feed tank and a permeate storage tank were connected with associated valves.
 3. Instruments to provide 11 electrical analog signals for automatic data collection were used, including feed and permeate conductivities, feed pH and temperature, permeate, concentrate, recirculation liquid flow rates, airflow rates, feed and concentrate liquid pressures, and air pressures.

4.1.2 Two-Phase Cleaning Module

1. Each vessel was equipped with a two-phase cleaning module. A fine spray nozzle was installed to provide proper liquid droplets during cleaning and rinsing.
2. An air inlet and outlet piping system and associated valves (2-inch, 1,000-psi rated) with automatic operators were used to enable cleaning of each vessel. Vessel V2 was cleaned in the forward direction (as of the feed flow), and vessels V1 and V3 were cleaned in the reverse flow direction.
3. An alkaline cleaning solution drum, an acidic cleaning solution drum, and a rinse water drum were also included. Each drum was equipped with a diaphragm-metering pump.
4. A demister was used to separate cleaning liquid from exhaust air for discharge.

4.1.3 Control Panel

1. The PLC control system includes discrete inputs and outputs for all valves, pumps, and analog inputs for data collection. It also included an operator interface panel that enabled the operator to modify the PLC program without the need for reprogramming, and to collect data of analog values.
2. Two displays were installed on the panel to show operating conditions during fouling and cleaning. The parameters displayed include temperatures, pressures, liquid pH, liquid conductivities, flux, return flow rates, pre-filter pressure drops, and operation steps.

4.2 Operating and Cleaning Processes

The processes designed to operate the system during fouling and cleaning are briefly discussed below. A more detailed description is given in a later section of this report when fouling and cleaning are discussed (see section 5).

4.2.1 Fouling Process – Normal Operation

The wastewater contained in the feed tank was delivered by a booster pump to the high-pressure feed pump, which, in turn, fed the wastewater into vessel V1, then into vessel V2, and, finally, into vessel V3, all connected in series. In the event that vessel V4 was needed for a particular experiment, the feed coming out from vessel V3 could be fed into vessel V4, or the wastewater could be independently fed directly into vessel V4 for an off-line study. During the fouling process, the RO product was directed to the permeate storage tank and the return flow (concentrate) was delivered back to the feed tank.

4.2.2 Cleaning Process

During cleaning, air from the facility's air source was heated to 50 degrees Celsius (°C) and supplied at a rate up to 200 scfm to the system. At the skid, the air was mixed with an alkaline or acid cleaning solution that had been heated to 50 °C. The solutions were heated and stored in separate drums and were delivered to the two-phase flow generation module via a diaphragm pump. Each vessel had a two-phase flow generation module installed at one end. Once the cleaning steps were programmed, each vessel could be cleaned in series, from vessel V1 to vessel V4. Certain vessels could be programmed out of an experiment if necessary. The exhaust was discharged to the demister, where the cleaning liquid and air were separated and directed to the drain and the exterior of the facility, respectively. The rinse step was an exact repeat of the process used for cleaning, except that instead of cleaning solution being mixed with air, RO water was

mixed with air to create two-phase rinsing. The two-phase rinse exhaust was similarly discharged and separated by the demister. The RO water used for rinsing was stored in a separate drum and was not heated.

Conventional cleaning was performed by circulating heated (50 °C) cleaning solution from the feed tank, through the vessels, and returning it back to the feed tank. The conventional cleaning utilized the same circulation loop that was used during fouling, except that the pressures were much lower, and both the permeate (if any were produced) and the concentrate were returned to the feed tank.

5. System Calibration Using Two-Phase Flow with Air, Water, and Simulated Milk Solution

5.1 Importance of Set Air Pressure on Air Consumption and Velocity

The investigators briefly ran the system with six membranes in series in RO operation mode. There were two elements in series in each of the three vessels, V1, V2, and V3. With tap water, feed recovery was 75%, with 99% rejection for 2 hours within the specs for these polyamide thin film composite membranes (GE Osmonics AG4040). RO feed pump settings were adjusted to obtain around 10-gpm feed to keep permeate flow below the maximum recommended rate. The pressure drop (ΔP) was under 2 psi for two membranes in each vessel (and under 2 psi for four membranes in V4, the off-line vessel). The 2-inch air supply was then connected and the regulator was set to 10 psi to test the operation of the two-phase CIP program with “clean,” new, wet elements installed. The investigators measured the one-phase airflow through each vessel with the onboard flow meter versus air pressure up to the 20-psi ΔP recommended limit. We did not feed any liquid. The initial cleaning program was set up to clean each vessel sequentially. The air consumption rates versus set air pressures without the application of liquid for these four vessels are given in figure 2. These measurements were necessary to set a baseline and to determine the flow characteristics of each vessel. This also enabled us to understand the actual air or two-phase flow velocity incurred inside the membrane during cleaning. The air or two-phase flow velocity was determined by the consumption of air used.

Due to system design characteristics, flow in V1 was consistently higher than that in V2 and V3. V4, which could be run with either two or four elements, was in the same range as V2 and V3 when two elements were installed, and with 4 elements installed in V4, the investigators could see a predictable decrease in flow. The air consumed was reduced with the application of liquid to the airflow. Air velocities were calculated using the diameter of the equivalent cross section of the membrane element and the flow rate. As an example, figure 3 shows the velocity profiles for each vessel from the entrance section, middle section, and end section of the membrane element for conditions at 30-psi set pressure and 1-liter-per-minute liquid flow rate (as indicated by 50 strokes per minute of the metering pump in figure 3). It is clear that the air velocity was higher in vessel V1 than in the other vessels. The air velocity increased through the vessel due to air expansion. As the air traveled through the vessel, the pressure dropped and the

specific volume of air increased. As a result, the air velocity increased due to the increase in the total volumetric flow rate.

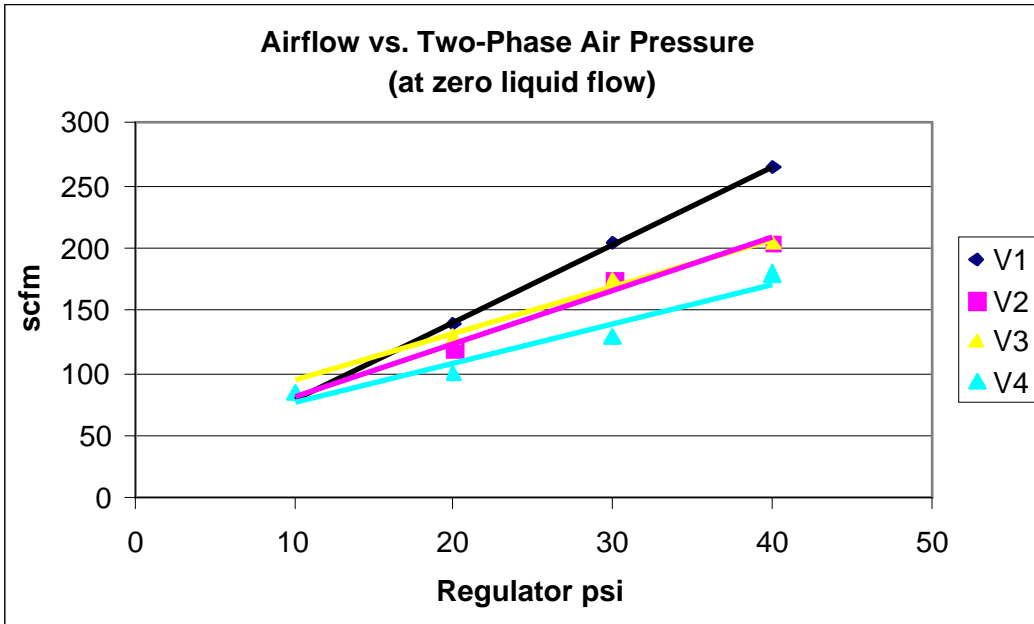


Figure 2. Airflow versus two-phase air pressure (at zero liquid flow).

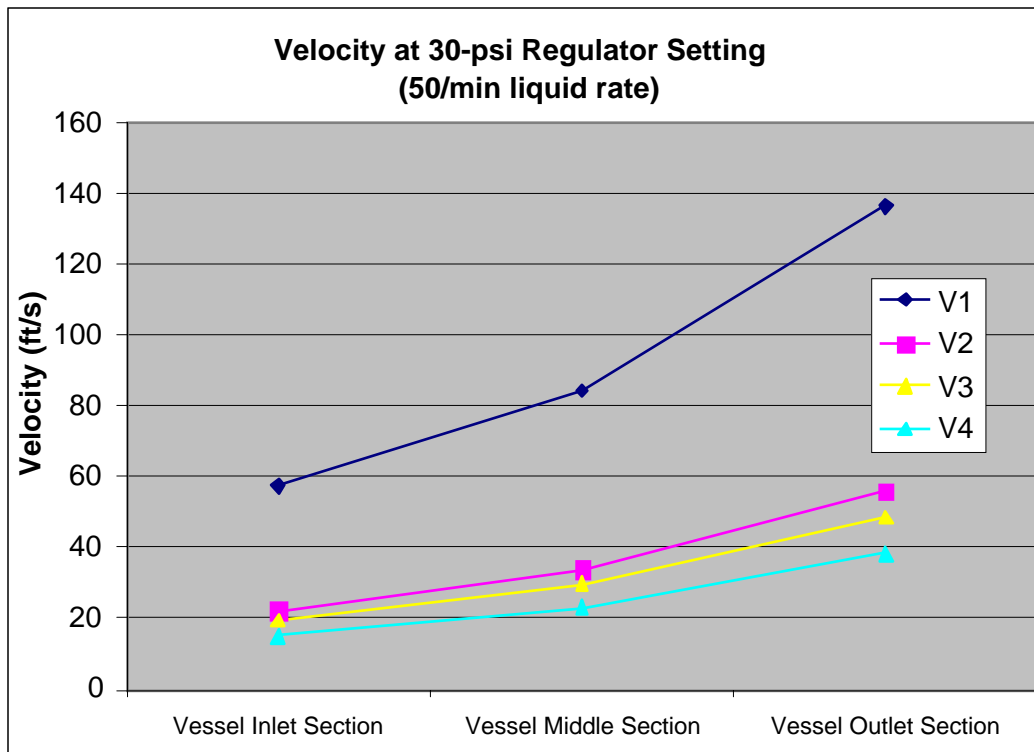


Figure 3. Two-phase flow velocities in four vessels as a function of vessel length.

5.2 Influence of Feed TDS and Temperature on Permeate Flow Rate

Novaflex studied permeate flow over time versus temperature for different feed TDS values to define cleaning parameters and to confirm the effect of TDS on recovery and permeate flow. This was done with just one two-element vessel (V1). Rather than relying exclusively on manufacturer data, we wanted to obtain data with our own instruments. A batch of “clean” feed water with measured TDS was fed to the RO skid. Permeate and concentrate were both returned to the feed tank. The feed water temperature increased over time from the heat input from the feed pump. The RO operation log manually recorded feed pressures, conductivity, pH, temperature, permeate flow and its conductivity, as well as concentrate flow and conductivity from the instruments. Conductivity, TDS, and pH were also periodically measured manually with a hand-held tester. One tank was used for clear water or permeate, and one was used for “waste” water. This enabled us to keep TSS limited to one tank. Results of flow versus temperature at three TDS feed levels are shown in figure 4. This data allowed the investigators to define cleaning parameters and to confirm the effect of TDS and temperature on permeate flow rates. The data provided a guideline for testing the pilot RO system with tap water (TDS = 100 parts per million [ppm]), simulated milk solution (TDS = 2,200 ppm), or actual dairy wastewater (TDS = 5,000 ppm). Initially, we tested at 150-psi feed pressure. These results were compared with the set of results obtained from tests run when the feed was set at 225 psi (figure 5). This data was used to define the range of operating pressures for the simulated milk solution (TDS = 2,200 ppm).

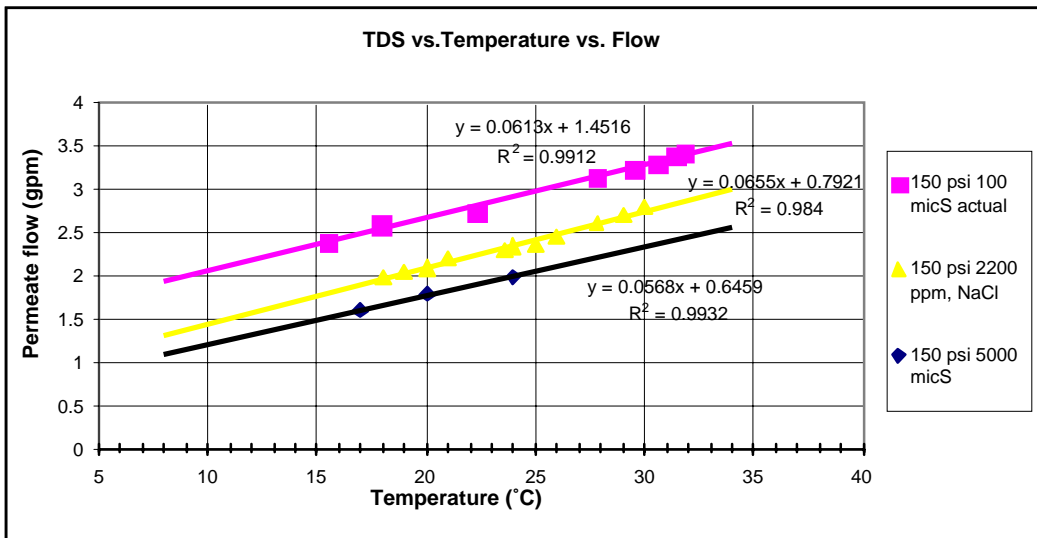


Figure 4. Flow versus temperature at 150 psi for three different TDS values.

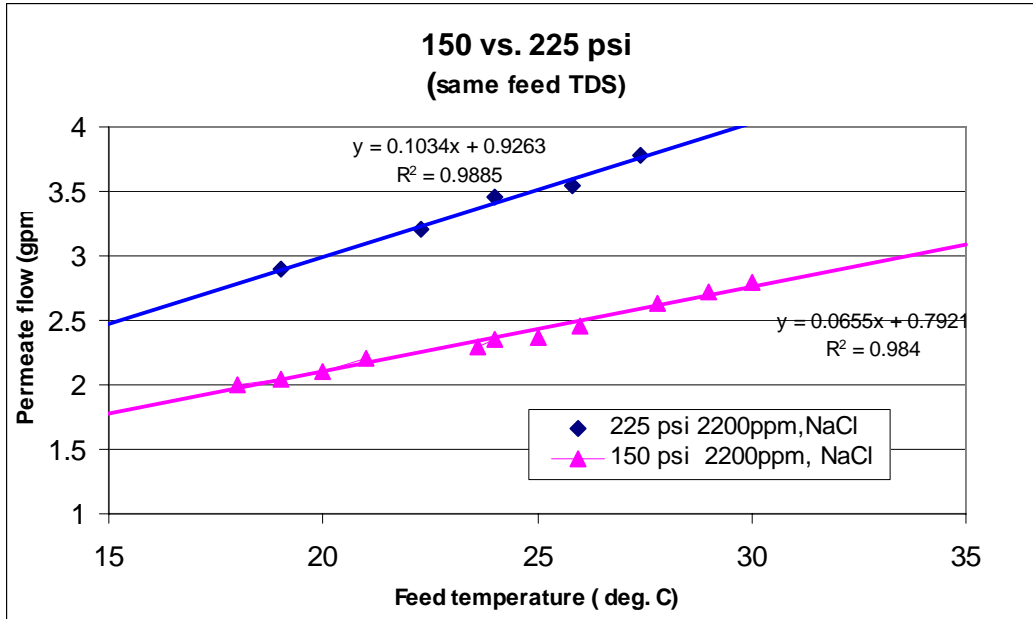


Figure 5. Flow comparison at 150- and 225-psi pressure (TDS = 2,200 ppm).

5.3 Influence of TSS on Fouling Time

In addition to dissolved solids, the investigators also fouled the RO membranes by feeding powdered whole milk at concentrations of 0.25%, 0.3%, and 0.5% (without MF or UF) to determine fouling rates and to set up a standard fouling method for experiments. As with the clear water runs, a series of data was generated by making a batch of wastewater, feeding the RO, recycling all flow (permeate and concentrate) to the feed tank, and tracking data over time (figure 6).

5.4 Preliminary Results of Membrane Fouling and Cleaning Using Simulated Milk Solution

Initial work was conducted with two elements in vessel V1. After conducting the above preliminary fouling work and limited cleaning, the elements in V1 were replaced with two clean elements to repeat some baseline work. This was necessary to confirm that this set of two elements performs in a way similar to that of the first two elements. The two elements were then fouled with 0.3% milk powder at 2,200 ppm TDS from sodium chloride (NaCl).

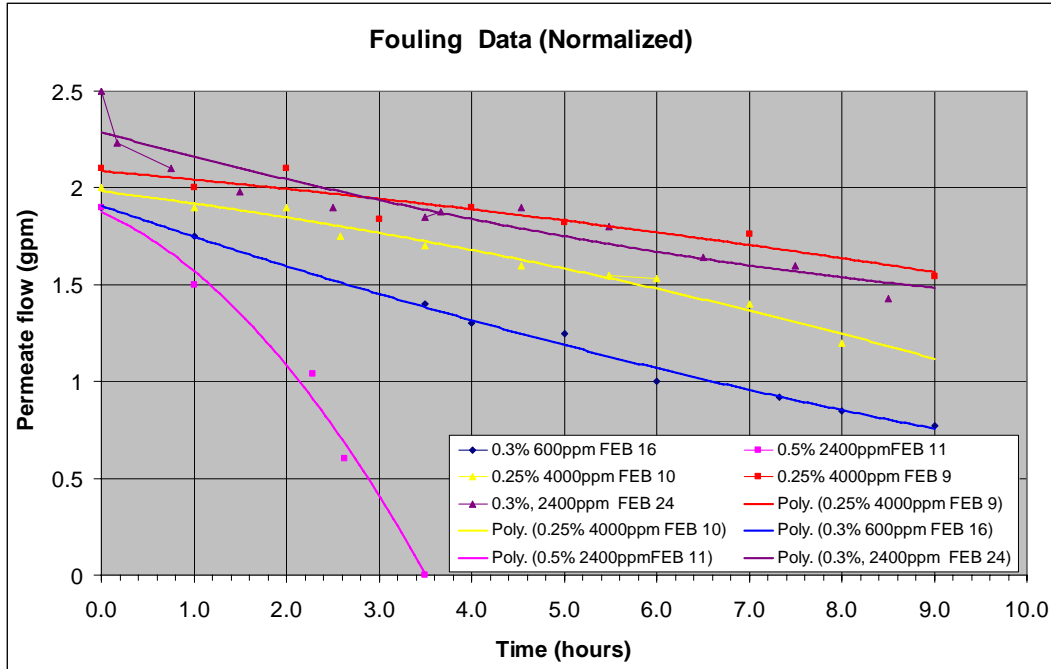


Figure 6. Fouling data as a function of time.

A two-phase cleaning process was used after each fouling run. The results were mixed due to lack of cleaning agent temperature in some of the cleaning runs. The cleaning on the first set of elements was done with sodium hydroxide (NaOH) and surfactant. The cleaning formulation on the second set of elements contained ethylenediaminetetraacetic acid (EDTA), in addition to the NaOH and surfactant. In this initial development stage, the following results were achieved and were used as a base for the future study.

With the first set of elements, Novaflux obtained good initial results. Ten-minute two-phase at 900 milliliters per minute at pH 11.5 increased clean water permeate flow from 1.6 to 2.4 gpm. The highest permeate flow we had before fouling was 2 gpm. Rinsing was complete in under 3 minutes at 1.8 liters per minute.

After fouling for about 30 hours, permeate flow was down to under 1.2 gpm with simulated milk waste feed. The investigators switched back to clean, warm tap water feed, at 32 °C, 250-ppm TDS. The permeate was still below 2.0 gpm after adjusting for feed temperature.

The investigators heated water in the CIP tank to 50 °C, pumped it to the RO skid chemical tank, added the caustic and surfactants, and then mixed with the drill mixer. The temperature remained over 45 °C during cleaning. The two-phase cycle pumping sequence was adjusted to conduct a 10-minute two-phase alkaline cleaning at 900 milliliters per minute, followed by a 3-minute, two-phase, cold RO water rinse at 1.8 liters per minute. We ran air at 40 psi at the regulator,

which resulted in 20 psi at the vessel V1 inlet and about a 15 psi ΔP between the inlet and outlet of V1. Initial airflow was 220 scfm, increasing to 240 scfm over the 10 minutes of cleaning. This dropped to 230 scfm during rinsing as the liquid feed was doubled.

After the two-phase rinsing, the system was rinsed by running the RO with 30 gallons of tap water in recirculation mode. The initial concentrate pH was 6.8, which indicated that the rinsing was complete during the 3-minute two-phase. At 10-gpm feed rate, 30 gallons in the tank “turns over” every 3 minutes for rapid mixing. The RO operation quickly stabilized in under 2 minutes, producing 2.2 gpm of 7.5 micro Siemens (μS) permeate from 600 μS feed at 28 °C. The feed pressure was down to 142 psi and was adjusted to 150 psi to normalize flow. The permeate flow increased to 2.4 gpm at 25.6 °C, which is a 20% increase over the clean water baseline (before fouling) and a 50% increase over fouled flow rate due to cleaning. Summaries of the results are plotted in figure 7.

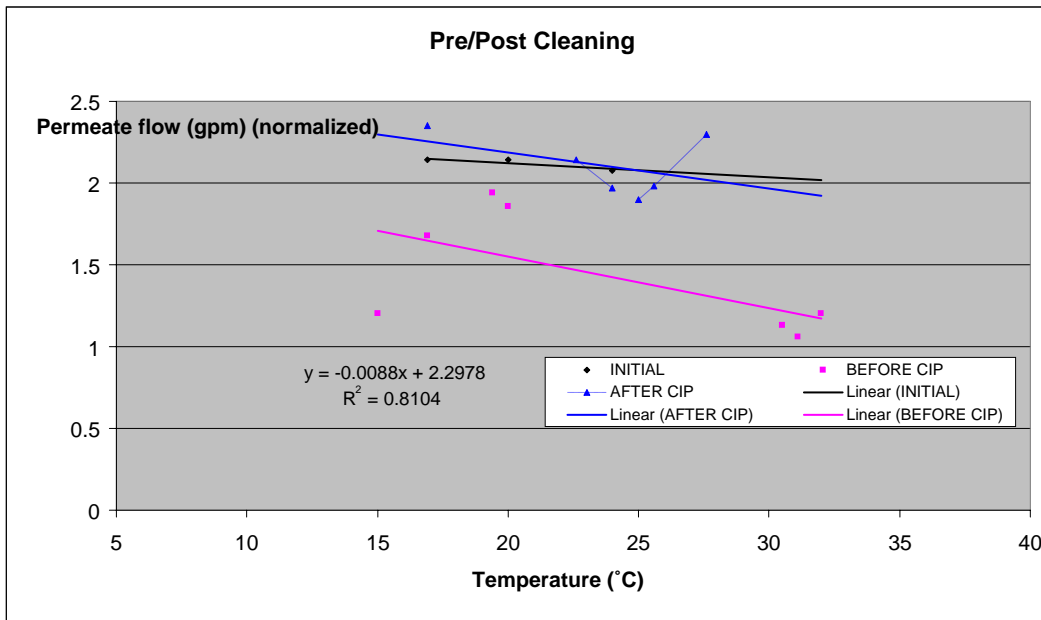


Figure 7. Cleaning results as a function of temperature.

Two subsequent cleanings were not as complete since the post-cleaning permeate flow was only 85% of “new” elements after three 5-minute two-phase cycles with the first set of elements. Our data seemed to indicate some progressive fouling and a need for hotter cleaning conditions.

Subsequently, two new elements were installed and a new clear water baseline was run, fouled to under 1.0 gpm with protein, and cleaned. EDTA was added to the same base cleaning agents (11.7 pH). A single 10-minute cycle increased the flux from 50% to 86%.

6. Fouling Method and Two-phase Cleaning Process Parameters with Simulated Dairy Wastewater

The main approach to demonstrating the effectiveness of the two-phase cleaning technology in cleaning spiral-wound RO membranes has been to use simulated high TDS dairy wastewater to foul such membranes and to conduct cleaning studies to define the two-phase flow cleaning capabilities and limitations. During this study, the method of preparing the simulated dairy wastewater, as well as of fouling the RO elements of the RO skid, was developed. In addition, the method of applying the two-phase flow to clean the RO membranes was also developed.

6.1 Fouling Studies with Simulated Dairy Wastewater

The RO-membrane skid included three pressure vessels, namely, V1, V2, and V3. Each pressure vessel holds two 4-inch RO elements. During the fouling step, all six elements were fouled simultaneously. The foulant used was simulated dairy wastewater containing 1% whole milk powder (26% butterfat), with a TDS value of approximately 2,000 ppm. The TDS was further adjusted with table salt to simulate typical dairy wastewater properties. A typical fouling run normally takes 6-8 hours of RO operation. During our study, the concentrate and the product were returned to the feed tank during fouling. The flux-temperature graph was used to determine the completion of the fouling step. Performing the required temperature corrections for the flux valves was necessary to ensure that fouling was complete. When the flux-temperature curve leveled off, the fouling was deemed complete. Some fouling processes with various milk concentrations are shown in Figure 8. The elements were fouled one time with each solution, and the TDS level for the first four runs of figure 8 was 300 ppm. During fouling, a booster pump and a feed pump were used to circulate the simulated dairy wastewater from the feed tank to the RO vessels, as shown in figure 9.

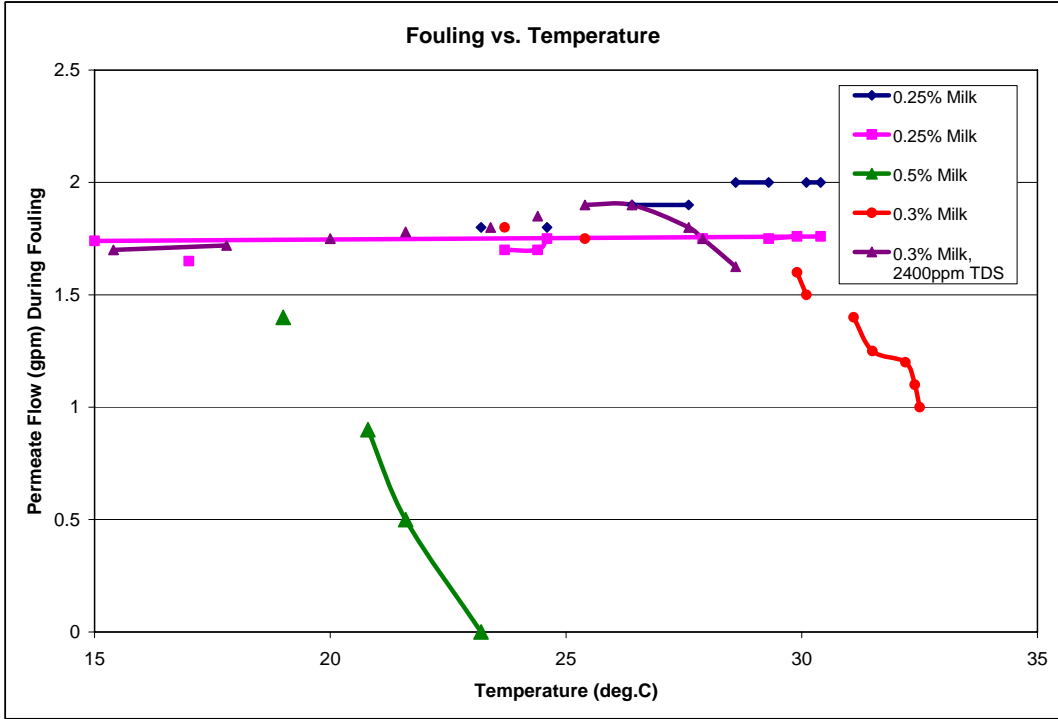


Figure 8. Fouling processes with various milk concentrations.

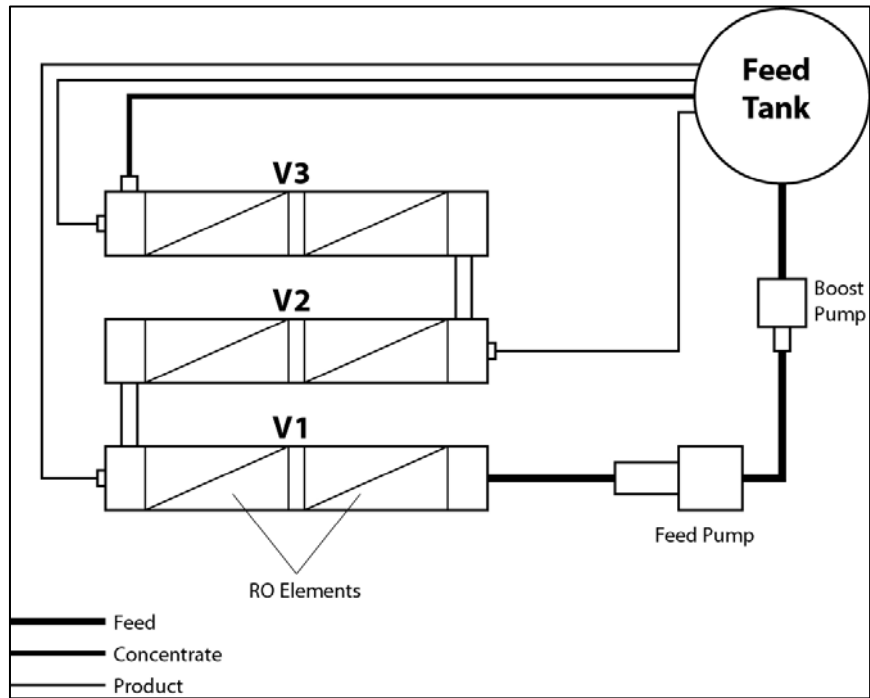


Figure 9. Connection of the system during fouling.

6.2 Two-Phase Cleaning Studies

In earlier experiments, fouled RO membranes from the three pressure vessels were cleaned by placing them in V1. The choice to clean all elements in V1 was made so that each test could be executed under the exact same two-phase dynamic conditions, and to ensure that the data collected was meaningful. The two-phase cleaning was performed as follows.

Once the fouling run was completed, the elements in vessels V2 and V3 were removed from their pressure vessels. The two elements that were fouled in V1 remained in V1 and were used for the first cleaning run. After completing the cleaning run, the membranes were rinsed and the flux measurements were made. The system was then shut down, and the cleaned elements were removed from V1. They were replaced by the elements that were fouled in either V2 or V3, and cleaning, rinsing, and flux measurements were performed. Once all the RO elements were cleaned, rinsed, and tested for flux and rejection ratio, all six elements were replaced in vessels V1, V2, and V3, and the next fouling run commenced. Since RO elements do not foul to the same degree in each vessel, it was important to keep track of which elements were fouled in which vessels. This was done by tracking the RO elements' serial numbers.

Figure 10 shows the system configuration used for cleaning a single vessel. The two-phase flow was created by mixing a cleaning liquid (either basic or acidic cleaning solution) with an air stream at the feed inlet of the vessel and directing the resulting mixture to the RO vessel to perform the cleaning. The exhaust was directed to a demister, where the air and liquid were safely separated for discharge.

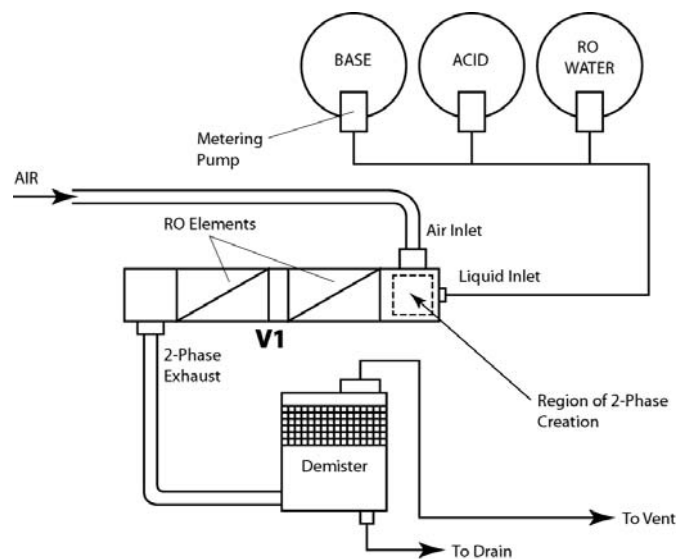


Figure 10. System configuration used for single vessel cleaning.

In the following experiments, all six RO elements were cleaned in their respective vessels after fouling was completed. The two-phase cleaning and rinsing were done sequentially (i.e., V1, then V2, then V3) without removing the membranes from their respective vessels. The flux was then measured as a system, representing the total flux of all six RO elements.

6.3 Simulated Dairy Wastewater Preparation

Typically, one batch of simulated wastewater was made and used for 1 week. The feed tank was first filled with 50 gallons of city water. To this water, 4.1 pounds of whole milk powder (26% butterfat) was added and then mixed thoroughly. As discussed above, the use of 1% milk simulated typical dairy wastewater feed. At that point, the TDS was adjusted to the target value of 2,000 ppm with table salt, and the fouling run commenced.

6.4 Cleaning Cycle Description

This section describes the cleaning of a single pressure vessel. The first step in this process was to perform a quick conventional rinse with RO water. This rinse step generally lasted 30-60 seconds and was designed to remove any bulk foulant accumulated in the feeding channels of the RO elements. Once this short pre-rinse was completed, the main two-phase cleaning cycle commenced. The first step in the cleaning cycle was an air purge. The air purge empties residual water from the RO element. This air purge step lasted for 30 seconds. This was followed by the alkaline two-phase cleaning step, which normally took 20 minutes. The alkaline step was then followed by a 3-minute two-phase rinse step using a mixture of air and RO water. When necessary, an additional two-phase acid cleaning step was used to dissolve calcium milk deposits. The latter two-phase acid cleaning step normally took about 10-15 minutes and was always followed by a 3-minute two-phase rinse using a mixture of air and RO water, as described above. The final step of the cleaning cycle was a 30-second air purge. All cleaning and rinsing steps described above were based on the two-phase flow process.

6.5 Cleaning Frequency and Cleaning Intensity to Optimize the Two-Phase Flow Cleaning Parameters

During the present study, a protocol for flux measurement was first established. The feed water for the flux measurement was RO water. The set feed pressure was 150 psi. The concentrate was directed into the waste tank or directly to the

drain. The product was directed back to the RO tank. The system was left to stabilize for about 5 minutes. When the product and concentrate flows stabilized, and the product conductivity was in the proper range, flux measurements were taken. The flux measurements were recorded automatically by the skid and displayed on the view panel. The measurements were also taken visually with an inline flowmeter. In addition to the product and concentrate flows, the feed conductivity, product conductivity, feed TDS, product TDS, feed pH, product pH, and temperature were also recorded. The temperature was a key measurement because the flux must be normalized to 25 °C. The maximum operating temperature for the AG membranes used in this program was 50 °C, and all the experiments were done below this temperature.

The following includes a summary of the two-phase cleaning studies.

6.5.1 Effect of the Two-Phase Mixture Temperature

To properly clean RO membrane elements fouled with simulated dairy wastewater, the use of higher temperatures was needed to achieve better results, since optimal lipids dissolution requires temperatures above their melting point. However, this was difficult to achieve without heating the air during the two-phase cleaning process. An air heater was designed and constructed during this task to heat 200 scfm air to 50 °C. The air heater was effective in controlling the temperature of the two-phase mixture in the range of 45-55 °C.

6.5.2 Effect of Cleaning Time

The data showed that the optimal two-phase cleaning time was between 15 and 20 minutes. Our experiments showed that the 10-minute cleaning cycle was insufficient to recover the full flux, and that the 30-minute cycle was too long to use with the two-phase technology. The 20-minute runs were satisfactory and produced consistent results. Towards the end of the testing phase of these studies, it was clear that the cleaning time could be reduced to 15 minutes without compromising the results. The average post-cleaning flux results in gallons per minute are summarized in figure 11, where a significant improvement in average flux is indicated when the cleaning time interval was increased from 10 minutes to 20 minutes. There was only a small incremental increase in flux when the cleaning time was extended to 30 minutes.

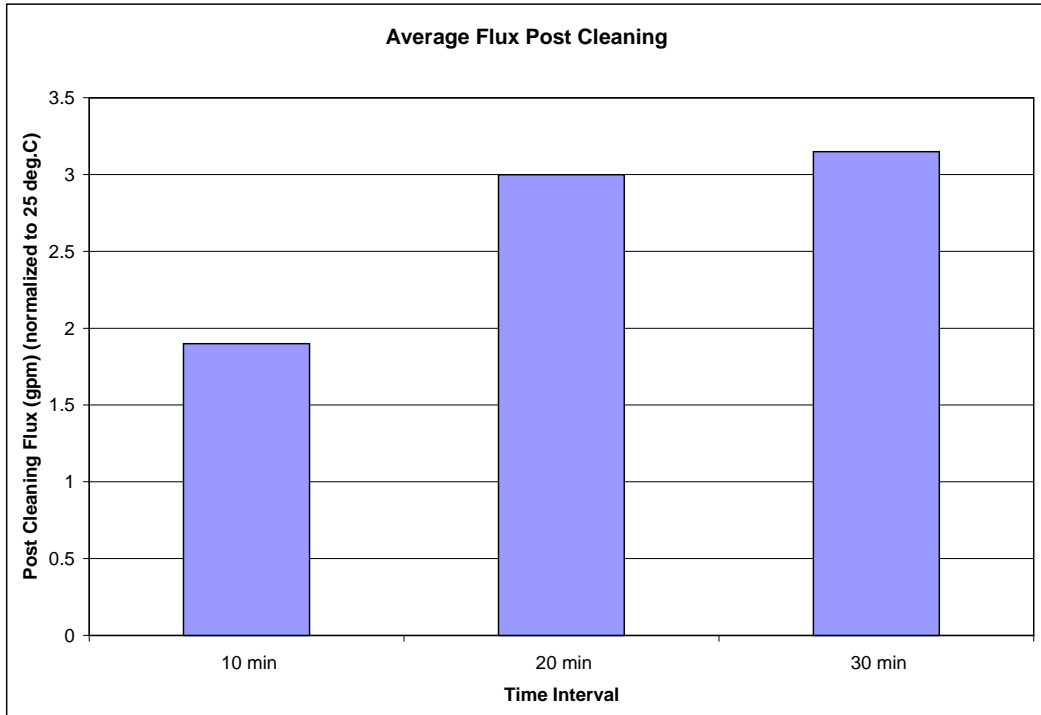


Figure 11. Flux versus two-phase cleaning time for a single two-element pressure vessel, normalized to 25 °C.

6.5.3 Effect of the Liquid-to-Air Ratio

The liquid-to-air (l/a) ratio of the two-phase mixture should be selected to maximize the two-phase fluid dynamics necessary for optimal cleaning. To achieve optimal cleaning, the velocity must also be sufficient to generate the shear stress needed to remove foulants from the feeding channels. The maximum airflow rate available for use in a single vessel is 240 scfm due to system configuration and compressor capacity. The liquid rate was adjusted to achieve a l/a ratio in the range of 1/1500 to 1/2000, based on prior experience. The liquid flow rate needed to achieve 1/1500 to 1/2000 was approximately 850 milliliters per minute. First, several tests were run at a standard l/a ratio of 1/1500, and then the airflow rate was varied. The results showed little difference between cleaning efficacy at 50, 100, and 150 scfm. In fact, each set of tests showed a slight and steady flux decline with respect to the baseline set by the conventional liquid-only cleaning. The next step was to vary the l/a ratio at a constant airflow rate. An airflow rate of 100 scfm was used first. It became clear from the results that the l/a ratio is critical in cleaning this type of fouling. A l/a ratio in the range of 1/250-1/500 was ultimately found to be most effective at both 50- and 100-scfm airflow rates. In conclusion, a two-phase flow at about 1/500 l/a ratio and 50 scfm (air) achieves effective cleaning of dairy-fouled RO membranes (see figure 12).

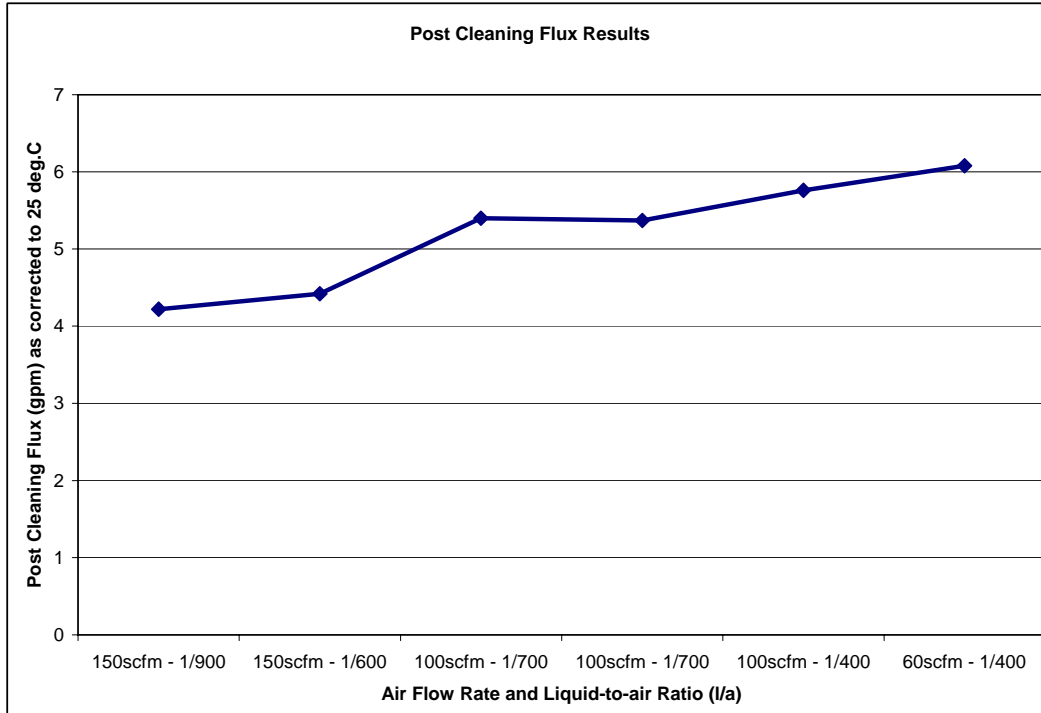


Figure 12. Flux results as corrected to 25 °C for various liquid-to-air ratios using the two-phase cleaning.

6.5.4 Effect of Cleaning Solutions Chemistry

One key variable in the chemistry of the cleaning liquid is the type of surfactant and its concentration. The investigators tried three different surfactants at three different concentrations. The amount of EDTA was held constant at 1% (by weight). The pH was adjusted to 12.0 with NaOH. This is the upper limit of the pH range allowable for the elements used. The final surfactant selected was a low-foaming anionic surfactant at 0.1% concentration.

6.5.5 Definition of the Cycle Design

The following conventional cycle (liquid-only cleaning) was used: an alkaline cleaning followed by a water rinse, then acid cleaning followed by a water rinse, and, finally, another alkaline cleaning followed by a final water rinse. The two-phase flow process was able to achieve equivalent cleaning with only one alkaline cleaning step. An acid cleaning may periodically be needed to descale the system.

6.5.6 Definition of the Rinse Cycle

The post-cleaning water rinse of the system was validated by checking the effluent rinse water for TDS and conductivity. In the two-phase cleaning cycle, the two-phase rinse was executed at twice the flow rate of the two-phase cleaning (i.e., approximately 1.6 liters per minute). From the samples taken online during rinsing, the rinse time was determined to be complete in about 2 minutes. A 3-minute RO water rinse was used throughout our experiments.

6.6 Results with Simulated Dairy Wastewater

The initial cleaning experiments were limited to testing selected chemistries at different cleaning times. Temperature was held constant, and airflow rate was regulated at 180 scfm for all the tests. The flow rates of alkaline, acid, and water were also held constant: 850 milliliters per minute for alkaline and acid, and 1,600 milliliters per minute for rinse water. Elements were fouled in all three vessels and, upon completion of the fouling, were taken out and cleaned in V1. Each set (two elements) was cleaned for 10 minutes, tested for flux, cleaned for another 10 minutes, again tested for flux, and then cleaned for a final 10-minute interval and tested for the last time for flux. A significant increase in flux was observed when the cleaning time interval was increased from 10 minutes to 20 minutes, while only a small improvement in flux was observed when the cleaning time was increased to 30 minutes, as shown in figure 11.

Further cleaning experiments were conducted to understand the effect of the l/a ratio. Initial results with respect to minimum airflow requirements were also obtained. During this phase of testing, the system was fouled and cleaned with all six elements in place. The results generated from six experiments with various l/a ratios are shown in figure 12, where the total flux, as corrected to 25 °C, was improved from 4.2 gpm to 6.1 gpm when the l/a ratio was varied from 1/1000 to 1/400. The RO elements were normally fouled to a flux value of about 2.5 gpm or less prior to cleaning. Apparently, a “wetter” (l/a = 1:400) two-phase mixture may result in better cleaning, even with the airflow rate reduced down to 60 scfm per each two-element vessel.

Many conventional cleaning steps were executed during the course of this study. This was periodically done to restore the membranes to their best possible condition and to reset the baseline, allowing Novaflux to arrive at valid conclusions regarding the two-phase cleaning process. The optimized final parameters for the two-phase cleaning have been more effective in restoring membrane flux than conventional liquid-only cleaning. Comparisons of the two-phase cleaning and the conventional cleaning are shown in figure 13, where the conventional cleaning usually requires about 8 hours of liquid circulation using an

alkaline solution at 50 °C, while the two-phase cleaning only requires a total of about 45 minutes of two-phase cleaning at 50 °C and 9 minutes of two-phase RO water rinsing at room temperature. The average flux results were calculated based on three experiments for each l/a ratio of the two-phase cleaning and the conventional cleaning. As depicted in figure 12, the two-phase cleaning may further outperform conventional cleaning when the l/a ratio is finalized (table 1).

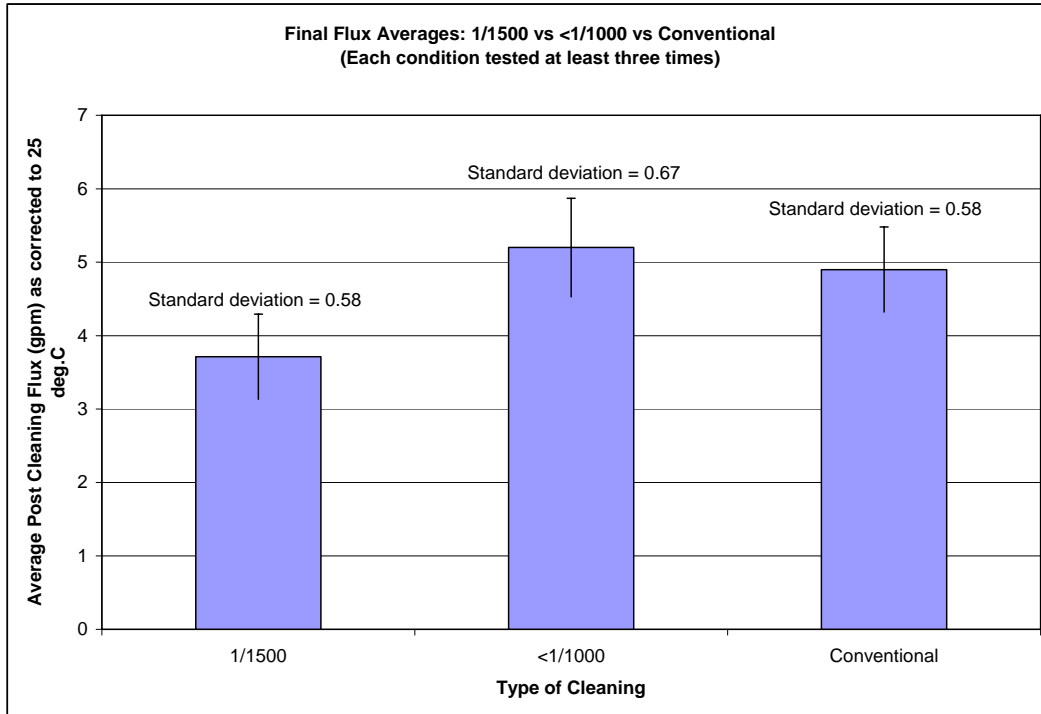


Figure 13. Comparisons of two-phase cleaning and conventional cleaning.

An account of the results of this study is summarized in table 1. In the initial stage of this study, the conventional cleaning was applied intermittently to restore the flux back to a level near 5 gpm to prevent further deterioration of the RO membrane's performance.

Table 1. Summary of Simulated Dairy Wastewater Results

Test No.	Cleaning¹	l/a	Clean Water Flux After Fouling (gpm) at 25 °C	Post-Clean Water Flux (gpm) at 25 °C
1	Conventional	NA	2.50	5.70
2	Two-phase	NA	3.90	5.24
3	Two-phase	NA	1.65	4.00
4	Two-phase	NA	3.85	4.14
5	Conventional	NA	2.50	4.58
6	Two-phase	1/1800	3.00	3.82
7	Two-phase	1/1800	2.60	3.45
8	Two-phase	1/1800	1.27	3.34
9	Conventional	NA	2.50	4.10
10	Two-phase	1/1800	NA	4.68
11	Two-phase	1/1800	NA	4.47
12	Two-phase	1/1800	2.20	3.84
13	Two-phase	1/1800	2.48	3.96
14	Two-phase	1/1800	2.97	3.40
15	Two-phase	1/1800	1.83	4.22
16	Two-phase	1/1800	2.14	4.42
17	Two-phase	1/1000	2.48	5.40
18	Two-phase	1/1000	3.30	5.37
19	Two-phase	1/800	3.61	5.76
20	Two-phase	1/400	2.45	6.08

¹ Alkaline cleaning solution was used in all the tests.
Note: NA = not applicable.

7. Two-Phase Cleaning with Actual Dairy Wastewater

7.1 Wastewater Source/Delivery/Methodology

Wastewater was brought to Novaflux's Hillsborough facility from Readington Farms, 12 Mill Road, Whitehouse, New Jersey, via pickup truck, utilizing a 250-gallon tote that was supplied by Readington Farms. The wastewater was taken directly from the untreated wastewater holding tank at Readington Farms and consisted of 4% grey water (water from kitchen, shower, and toilet) and 96% dairy waste. The dairy waste included all of the water and chemistry that was used to clean the dairy system of Readington Farms, including all of its components and pipelines. The wastewater was brought to the Hillsborough facility at least twice per week to ensure that testing was being done with wastewater that was as close to the real world situation as possible. Upon arrival at the Hillsborough facility, the wastewater was pumped into a 500-gallon holding tank with a high- pressure, high-volume pump. Once the wastewater was in the 500-gallon holding tank, Ventocil-IB was added to the wastewater as a disinfectant at a concentration of 100-500 ppm. The entire volume was recirculated via a low-pressure, lower flow transfer pump connected to the holding tank for approximately 10 minutes to ensure the Ventocil-IB had been fully integrated into the wastewater. The wastewater was then transferred using the same pump to the RO skid feed tank. Once the wastewater was in the feed tank, the fouling run was initiated and the pH was monitored until it reached equilibrium. At this point, NaOH was used to buffer the pH up to the 6.5 to 7.0 range, and the fouling run proceeded as normal. The initial pH before buffering was generally in the 5.0-6.0 range. The TDS ranged from 2,500 to 4,000 milligrams per liter (mg/L) over the course of testing, but the majority of the tests were in the 2,500-3,000 mg/L range.

7.2 Fouling

Fouling was performed in the same manner as with the simulated wastewater, as described in section 5.1, except that, in this case, the temperature was controlled. This was done via a heat exchanger coil installed inside the feed tank. Due to the cold ambient temperature during experimentation, the starting temperature of the feed water was usually cold enough (8-14 °C) so that, by the time the fouling run was complete (6 hours), the temperature had not even made it to the normal RO operating temperature of 25 °C. In only two cases did the temperature exceed this level and require the use of the heat exchanger. The cool

temperatures posed little problem, and all flux measurements were corrected to the 25 °C level for analysis.

7.3 Pre-filters

Three different types of pre-filters were tested. The majority of the tests were completed with a 30-micron pre-filter. A smaller amount of testing was done with a 5-micron pre-filter, and even fewer tests were completed with UF pre-filtration. The preliminary results indicated that 30-micron pre-filtration led to the best results, but due to the counterintuitive nature of that result, it is assumed that over the course of long periods of time, 5-micron pre-filters would be as good as or better than 30-micron pre-filters, and UF pre-filters would be as good as or better than both 5- and 30-micron pre-filters. The conclusion is that Novaflux’s two-phase cleaning is successful with even the minimum amount of pre-filtration that would typically be used for this type of wastewater.

7.4 Results with Actual Dairy Wastewater

7.4.1 Fouling Data

Figure 14 shows the flux as a function of time for the first 13 runs of fouling with actual dairy wastewater.

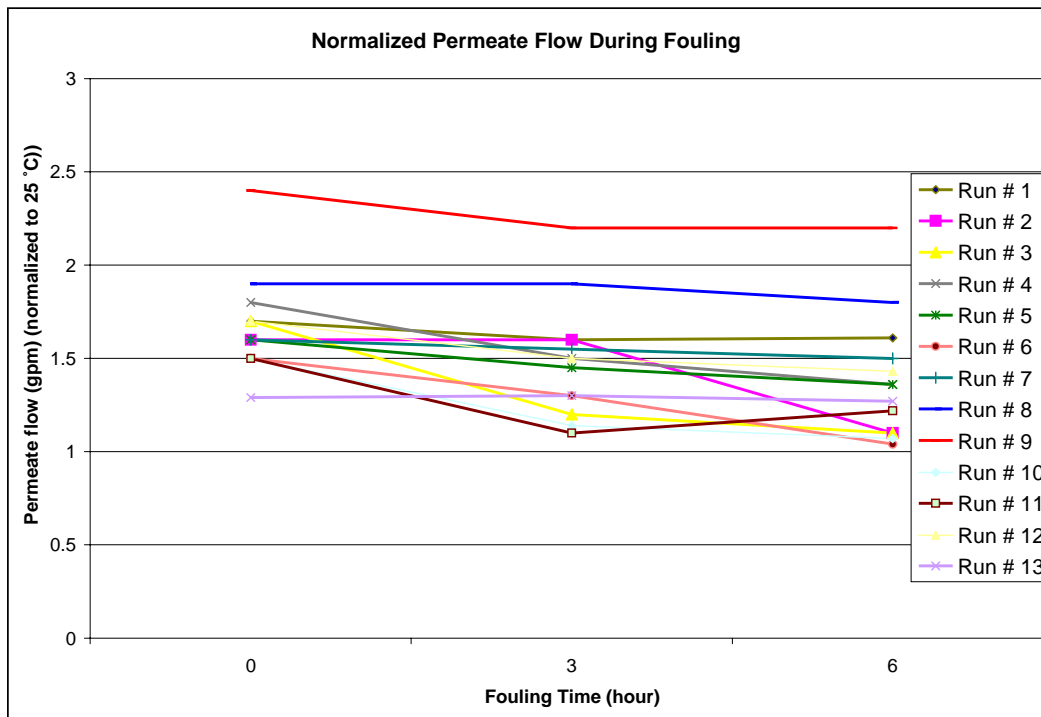


Figure 14. Normalized permeate flow rate during fouling as a function of time.

7.4.2 Two-Phase Cleaning for Membranes Fouled with Actual Dairy Wastewater

Table 2 shows data for the first 13 fouling and cleaning runs with actual wastewater. The first 10 runs were performed with a 30-micron pre-filter, and the final three runs were performed with a 5-micron pre-filter. The fouling time is the amount of time the membranes were subject to wastewater. Final fouling flux is the flux of the membranes with wastewater at the end of the fouling run, at the 6-hour mark. Post-fouling flux is the flux of the membranes after the wastewater has been rinsed out with RO water and equilibrium has been reached. The flux reading was taken at approximately the 5- to 10-minute mark. Post-cleaning flux is the measurement of the flux after two-phase cleaning and rinsing have been completed. This measurement was performed in the exact same manner as the post-fouling flux measurement.

Table 2. Flux Data for the First 13 Fouling and Cleaning Runs with Actual Dairy Wastewater

Fouling Run	Fouling Time (hrs)	Final Fouling Flux (wastewater) (gpm)	Post-Fouling Flux (RO water) (gpm)	Post-Cleaning Flux (RO water) (gpm)	Percent Recovery (%)
1	6.75	1.61	2.40	6.30	163
2	5.00	1.10	1.34	4.20	213
3	5.50	1.10	1.34	3.44	156
4	6.00	1.36	1.52	2.14	40
5	6.00	1.36	1.52	3.38	122
6	6.00	1.04	1.50	3.46	130
7	5.00	1.50	1.93	4.00	107
8	6.00	1.80	2.00	3.92	96
9	5.00	2.20	2.70	3.86	43
10	6.00	1.07	1.45	3.62	149
¹ 11	6.00	1.22	1.12	3.40	203
¹ 12	6.00	1.43	1.49	2.90	94
¹ 13	6.00	1.27	1.26	3.14	149
Averages:			1.66	3.67	121

¹ 5-micron prefilter instead of 30-micron pre-filter.

Final fouling flux – the flux with wastewater at the end of the fouling cycle.

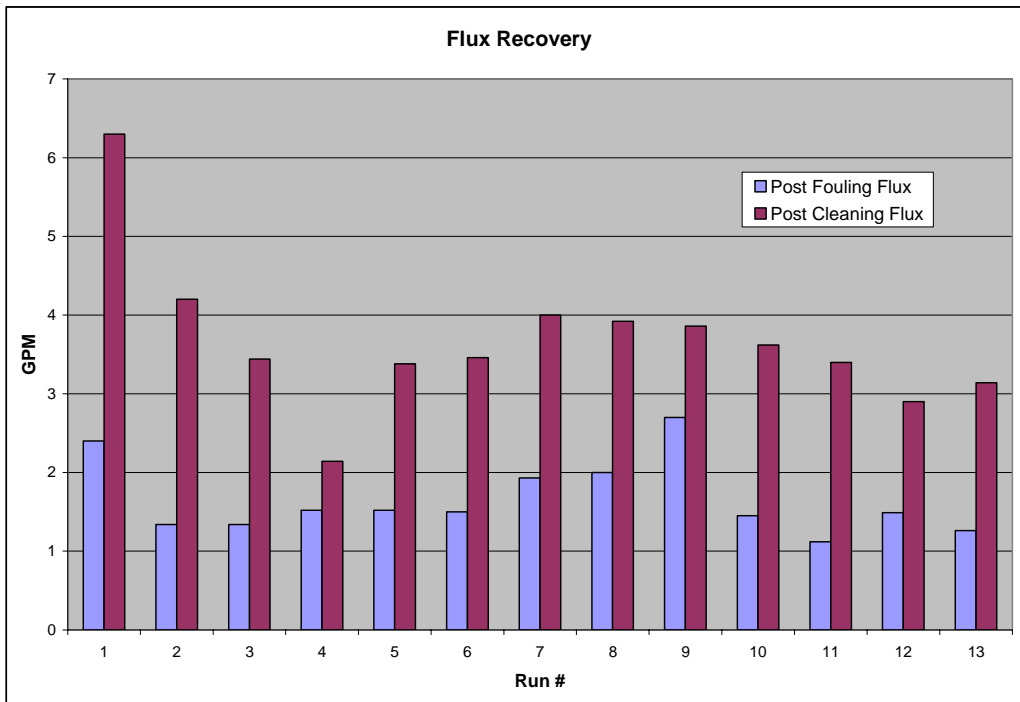
Post-fouling flux – the flux with RO water after fouling.

Post-cleaning flux – the flux with RO water after cleaning.

The data here support the claim that the Novaflux two-phase cleaning process restores and sustains flux as good as or better than conventional cleaning in a shorter period of time. The precipitous drop from 6.3 gpm down to 2.14 gpm (see table 2 – fouling runs 1 to 4) might have been alarming if, over the course of the next few cleanings, the flux did not come back to acceptable levels. There is

another drop towards the end of the testing, but again, when testing was completed, the flux was being restored to average. The membranes that were used for this test were subject to many hundreds of cycles over the course of more than 1 year. The assumption is that there was a considerable amount of irreversible fouling and that addition of the actual wastewater to the system might have caused a certain type of fouling that took more than one cycle to clean. Over the course of many cycles, we are confident that the 4.0-gpm design flow rate could be sustained, and in the case of a new set of membranes, a much higher level could be sustained. The two-phase flow has the added effect of not only cleaning what has been deposited on the membranes in the most recent fouling cycle, but it also possesses the ability to clean built up foulants from previous cycles. In light of this, the claim that a flux level of 2.8 gpm (equivalent to 4,000-gpd designed value) could be sustained might be a conservative one. It is likely that over many hundreds of cycles, the average flux would slowly increase to a point where the system would be operating at its maximum capacity.

Figure 15 compares the initial flux to the final flux after the two-phase cleaning for each of the 13 runs. Figure 16 compares the average initial flux to the average final flux. The standard deviations would decrease considerably over time.



Runs # 1-10: 30-micron pre-filter; Runs # 11-13: 5-micron pre-filter

Figure 15. Flux recovery after the application of the two-phase flow cleaning.

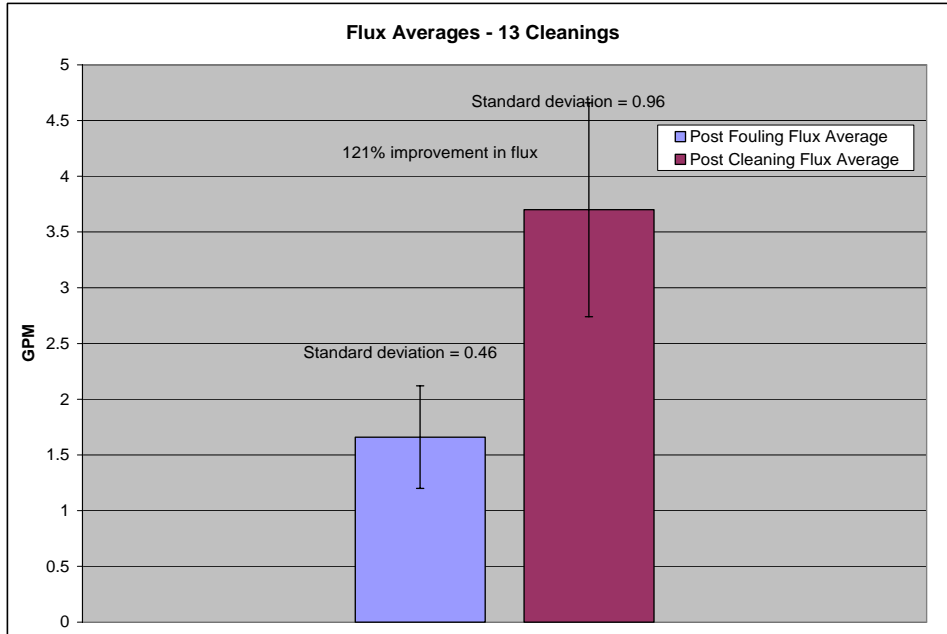


Figure 16. Average flux before and after the two-phase cleaning for the first 13 runs of the membranes fouled with actual dairy wastewater.

7.4.3 Average TDS and Rejection Rate

Table 3 shows the TDS data for each fouling run. Over the time periods we operated, there is no evidence of membrane damage due to the Novaflux two-phase cleaning. The rejection rates here are the averages over the course of the 6-hour fouling run, but the initial values and final values are where they should be and the average value is well within acceptable bounds for each of the 13 runs.

Table 3. TDS and Rejection Rates for Each of the First 13 Runs with Actual Dairy Wastewater

Fouling Run	Average Feed TDS	Feed pH	Average Product TDS	Average Rejection Rate	Flow Rate Recovery (%)
1	3,443	6.68	44	98.7	163
2	3,883	6.7	28	99.3	213
3	3,847	6.6	27	99.3	156
4	3,092	6.8	19	99.4	40
5	2,866	6.5	20	99.2	122
6	3,276	7.1	22	99.3	130
7	2,420	6.5	21	99.1	107
8	2,244	7.5	29	98.8	96
9	2,150	5.5	38	98.2	43
10	2,831	6.7	27	98.8	149
11	2,703	6.7	30	98.9	203
12	2,470	6.6	28	98.9	94
13	2,226	6.6	26	98.9	149

8. Economic Assessment and Commercialization Analysis of an RO Membrane System Utilizing Novaflux's Two-Phase Cleaning Technology

8.1 Introduction

This section provides an economic assessment of employing an RO treatment system using the two-phase flow cleaning technology. The pilot system designed and developed during this study provided the basis for reasonably estimating the cost associated with using an RO treatment system with two-phase cleaning technology as an alternative. Data from prior tests and industry data about the dairy industry and conventional RO cleaning systems were used in the analysis. With this information, Novaflux was able to determine the economic impact of a commercial RO treatment system with two-phase cleaning technology. For the purposes of this analysis, Readington Farms was used as an example of a dairy plant subject to wastewater problems. Readington Farms is a medium-sized dairy plant with common wastewater disposal restrictions, and it must absorb related costs associated with disposal.

A typical Northeast medium-sized dairy plant that produces 50,000 gallons of wastewater per day is forced to transport, by truck, one-third of its daily wastewater production. Based on this, Novaflux considered a commercial RO system which has the capacity to treat about 16,667 gallons of wastewater per day. This data was compared with the current cost of transporting excess wastewater by truck. Because of the high TDS and the high load in dairy wastewater, it is impractical to treat such low water quality with RO membranes and perform the cleaning with conventional liquid circulation methods. Therefore, no cost analysis to compare this option with the RO and Novaflux process was made.

Some general data about the dairy industry is provided below to present an overview of how dairy plants are categorized and segmented throughout the industry. It is important to understand the relative cost associated with dairy plant wastewater disposal and the impact of “municipal support” on local treatment plants. A dairy plant’s municipal support will significantly impact any economic assessment and analysis.

The result of this assessment and analysis shows that the treatment cost per 1,000 gallons can be reduced from \$130 to \$59, which means an annual savings

for the medium-sized dairy plant of 54.44%. This percent savings will increase after the cost to build the treatment system is fully amortized over 7 years. After 7 years, the cost of treating 1,000 gallons of wastewater will decrease to \$9, which translates into an annual savings of approximately 93.35%. Since a commercialized system with two-phase cleaning technology is scalable to any plant's wastewater production, it is clear that these results would be applicable to other industries.

8.2 Overview

The current U.S. dairy industry includes approximately 1,200 plants. Plant size is categorized as small, medium, and large based upon the number of cattle supplying the plant. Small dairy plants have less than 200 active milk-producing cattle, medium-sized plants have 200-500, and large-sized plants have over 500. For the purposes of this economic analysis, Novaflux has segmented plants in each category by the availability of municipal services, as follows:

1. Full Municipal Support (FS): The dairy plant has adequate access to operating water needs and is permitted to dispose of wastewater to a local municipal water treatment plant.
2. Partial Municipal Support (PS): The dairy plant does not have adequate access to operating water needs and is restricted by volume to dispose of wastewater to a local municipal water treatment plant. This requires the plant to transport some wastewater, via a vehicle other than a pipeline, out of the plant.
3. No Municipal Support (NS): The dairy plant does not have adequate access to operating water needs and cannot dispose of wastewater to a local municipal water treatment plant. This demands that the plant transport all of its wastewater, via a vehicle other than a pipeline, out of the plant. This type of plant is usually located in a remote area.

8.3 Impact of Municipal Support on Wastewater Costs

In this economic analysis, it is important to factor in the level of municipal service available to a dairy plant because the cost significantly differs for each category. To simplify the comparison between these categories, we will use data obtained through third-party sources about medium-sized plants in the dairy industry and compare it to the actual data Novaflux generated from its pilot system trials. The

plant considered in the study was a medium-sized PS dairy plant. Table 4 displays cost assumptions used per 1,000 gallons of wastewater treatment/disposal.

Table 4. Cost Assumptions Used per 1,000 Gallons of Wastewater

Type of Plant	Operating Water (\$/1,000 gallons)	Wastewater (municipal) (\$/1,000 gallons)	Wastewater (transported) (\$/1,000 gallons)
FS	10.00	8.00	—
PS	20.00	27.00	130.00
NS	40.00	—	200.00

The wastewater production characteristics of the medium-sized dairy plant are given in table 5. The total annual cost of wastewater production for the medium-sized plant exceeds \$1.1 million. The breakdown of this cost is given in table 5 and is based upon the plant spending: (1) \$27 per 1,000 gallons of wastewater to treat and discharge into a local municipal treatment plant, and (2) an average of \$130 per 1,000 gallons of wastewater to transport by truck (see table 5).

Table 5. Volume and Cost of 50,000 Gallons of Wastewater (per day) from Dairy Plant

	Daily		Annually	
	Volume (gallons)	Cost (\$)	Volume (gallons)	Cost (\$)
Wastewater treated onsite and discharged to municipal treatment plant	33,000	900	12,167,000	328,500
Wastewater transported out of the dairy plant ¹	17,000	2200	6,083,000	790,800
Total wastewater volume and cost	50,000	3,000	18,250,000	1,119,300

¹ This is the fraction used in the RO system economic analysis.

To treat its wastewater production (33,333 gpd), the dairy plant would require a two-phase flow RO treatment system. Based upon the capacity of RO membranes, this system would require a minimum of 26 4-inch RO membranes (see table 6). These membranes would be contained within vessels; the size of the vessels and, therefore, the number of membranes per vessel, could vary to conform to the space available within the dairy plant.

Table 6. Size and Throughput of the Pilot RO and Two-Phase Flow Commercial RO Systems

	Pilot RO System (present study)	Two-Phase Flow (TPF) Commercial RO System
Number of vessels	3	13
Membranes per vessel	2	2
Total membranes	6	26
Daily throughput (gallons)	4,000	17,333
Annual throughput (gallons)	1,460,000	6,326,667

The pilot RO system (pilot system) developed during this demonstration study uses 4-inch RO membranes; therefore, for the purposes of this economic analysis, we will use a commercial system model for the two-phase flow RO treatment system comprised of 4-inch RO membranes, as well to minimize variability (see table 6).

8.4 Cost Comparison of Two-Phase Versus Conventional RO Treatment Systems in Dairy Plants

Since dairy plants operate 365 days a year, the two-phase flow RO treatment system will need excess capacity to allow for maintenance and replacement of membranes. In actual use, as membranes need replacing, or general maintenance is required, this system would allow one vessel to be shut down or taken offline but would still be able to treat the daily 16,667 gallons of wastewater that is currently transported out of the dairy plant to the treatment plants. The difference in the costs of the commercial system utilizing two-phase cleaning versus conventional cleaning would be as follows (see table 7).

Table 7. System Cost Analysis

	TPF Commercial RO System¹	Conventional Cleaning RO System (if applicable)²	Price Advantage	%
System cost	\$600,000	\$500,000	-\$100,000	-20.00%
Annual operation cost	\$222,000	\$246,000	\$24,000	9.76%

¹ Estimated cost based on new design including compressor cost.

² The cost does not include allocation for pre-treatment stages.

System cost includes purchase price and installation and does not include any interest or financing charges. A cost comparison between the commercial RO system with a two-phase cleaning capability versus an RO system with a conventional cleaning capability is estimated to be 20% higher. Annual operation cost, which includes utilities and projected maintenance, for the commercial

system with two-phase cleaning would be almost 10% less. Annual costs for both the commercial RO system and the conventional RO system do not include the cost of cleaning membranes. This cost is derived in table 9. Amortizing the cost of these systems over 7 years would result in an annual cost, as shown in table 8.

Table 8. Adjusted Annual Cost

	TPF Commercial RO System	Conventional Cleaning RO System (if applicable)¹	Price Advantage	%
Amortized annual cost	\$85,714	\$71,429	-\$14,286	-20.00%
Annual operation cost	\$222,000	\$246,000	\$24,000	9.76%
Total adjusted annual cost	\$307,714	\$317,429	\$9,714	3.06%

¹ The cost does not include allocation for pre-treatment stages.

8.4.1 Labor Cost

The variability of labor costs, and how they are accounted for and utilized in current day-to-day activity, is beyond the scope of this project and will, therefore, not be factored into this analysis. We will apply the following assumptions regarding labor:

1. As described above, membrane failure rate will be 20% using conventional cleaning versus 10% using two-phase cleaning technology. Therefore, conventional cleaning should be twice the labor cost of two-phase cleaning but will be assumed to be equal for this analysis.
2. The labor costs incurred by the dairy plant to maintain existing storage tanks and transfer systems to fill trucks will be equal to the cost associated with replacing membranes and to maintaining the commercial system.

8.4.2 Cost to Clean RO Membranes

As previously mentioned, one clear advantage the two-phase cleaning has over conventional cleaning is the reduction in chemistry and water consumed during membrane cleaning. The estimated difference in cost per membrane is shown in table 9.

Table 9. Cleaning Cost Comparison per Membrane – Analysis

	TPF Commercial RO System	Conventional Cleaning RO System (if applicable)¹	Price Advantage	%
Cleaning chemistry (gallons)	\$2.60	\$5.20	\$2.60	50.00%
Clean water for cleaning (gallons)	\$0.15	\$0.30	\$0.15	50.00%
Clean water for rinsing (gallons)	\$0.02	\$0.68	\$0.66	97.04%
Total	\$2.77	\$6.18	\$3.41	55.14%

¹ The cost does not include allocation for pre-treatment stages.

8.5 Economic Benefit

Using the annual cost to own and operate a TPF commercial RO system, and assuming that the system will treat 6,083,333 gallons of wastewater annually compared to trucking out this wastewater, the economic benefit will be as shown in tables 10 and 11.

Table 10. Cost Savings with Capital Cost

	Current	TPF Commercial RO System	Savings	%
Adjusted annual cost	\$790,833	\$307,714		
Annual treatment cost		\$52,575		
Total	\$790,833	\$360,289	\$430,544	54.44%
Per 1,000 gallons	\$130	\$59	\$71	54.44%

Table 11. Cost Savings without Capital Cost

	Current	TPF Commercial RO System	Savings	%
Adjusted annual cost	\$790,833			
Annual treatment cost		\$52,575		
Total	\$790,833	\$52,575	\$738,258	93.35%
Per 1,000 gallons	\$130	\$9	\$121	93.35%

9. Conclusions and Recommendations

Based on pilot testing, we have shown that over repeated fouling and cleaning cycles, the Novaflux two-phase cleaning process can restore and sustain flux of RO membranes. The basic cleaning process parameters developed in the simulated wastewater testing proved effective in the case of real wastewater, independent of the type of prefiltration that was used and the severity of the wastewater that was being used to foul the membranes.

Novaflux's two-phase cleaning process matches or surpasses conventional cleaning strategies in flux restoration, while using less chemical/water consumption, and the cleaning process is much faster. Conventional cleaning can take anywhere from 4 to 8 hours, whereas the Novaflux process takes less than 30 minutes. Conventional cleaning requires the use of a larger volume of cleaning chemistry, in excess of twice as much (30 gallons versus 15 gallons of diluted solution) to clean versus the Novaflux process. There are also significant savings during the rinse cycle. The Novaflux process takes only a few minutes and uses approximately 1 gallon of water to rinse the three housings, whereas the conventional rinse can take upwards of 50 gallons and close to 1 hour to complete a similarly effective rinse cycle as the two-phase rinsing.

Prefiltration prior to RO, using UF, 5-micron, and 30-micron prefilters, was tested. The 30-micron prefiltration tests were comparable to the 5-micron and UF prefiltration tests. This shows that the Novaflux process can be effective, even at the minimum level of prefiltration with very high TDS and TSS wastewater.

The air consumption necessary for effective cleaning was far less than initially anticipated. Novaflux began with the notion that upwards of 200 scfm would be needed to clean each housing (two elements), but after many tests to optimize this parameter, it was determined that effective cleaning occurred in the 50- to 70-scfm range, significantly less than expected and far more practical. This number could be reduced even further in future tests. The current skid could be cleaned (each of the three vessels simultaneously in parallel) in less than 30 minutes with a supply of air less than 200 scfm. Furthermore, liquid- (alkaline cleaning solution) to-air ratio used during two-phase cleaning is most effective in the range of 1:250-1:500, with a pH in the range of 11.5-12.0.

10. References

1. Labib, M.E., and C.-Y. Lai, Cleaning method for removing biofilm and debris from lines and tubing, U.S. Patent No. 6,027,572 (February 22, 2000).
2. Labib, M.E., and C.-Y. Lai, Cleaning composition and apparatus for removing biofilm and debris from lines and tubing and method therefore, U.S. Patent No. 6,326,340 (December 4, 2001).
3. Labib, M.E., C.-Y. Lai, P. Materna, and G.L. Mahon, Method of cleaning passageways using a mixed phase flow of gas and a liquid, U.S. Patent No. 6,454,871 (September 24, 2002).
4. Labib, M.E., and C.-Y. Lai, Cleaning composition and apparatus for removing biofilm and debris from lines and tubing and method therefore, U.S. Patent No. 6,619,302 (September 16, 2003).
5. Labib, M.E., C.-Y. Lai, P. Materna, and G.L. Mahon, Method of cleaning passageways using a mixed phase flow of a gas and a liquid, U.S. Patent No. 6,857,436 (February 22, 2005).
6. Tabani, Y. and M.E. Labib, Method for cleaning hollow tubing and fibers, U.S. Patent No. 6,945,257 (September 20, 2005).
7. Labib, M.E., C.-Y. Lai, and Y. Tabani, Apparatus and method for cleaning pipelines, tubing and membranes using two-phase flow, U.S. Patent Application No. 20040007255 (January 15, 2004).
8. Labib, M.E., R. (C.-Y.) Lai, S.A. Weitzel, and Y. Tabani, Removal of biofilm and other foulants from spiral wound reverse osmosis membranes, final report submitted to the Bureau of Reclamation, Agreement No. 01-FC-81-0740, (June 2003).