# REUSE OF ELECTROPLATING WASTEWATER. AN INVESTIGATION INTO FACTORS AFFECTING RELIABILITY OF REVERSE OSMOSIS

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# ABSTRACT

Membrane filtration processes such as ultrafiltration and reverse osmosis have great potential in significantly reducing water consumption in metal finishing industries by allowing recycling of treated electroplating effluent that is currently discharged. This research attempted to identify potential foulants in electroplating wastewaters and to study the effect of operating conditions on the performance of selected reverse osmosis membranes. Total dissolved solids of the electroplating waste from a local facility ranged from 3600 to 18,000 ppm. The primary contributors were sodium and sulfate ions which varied from 950 to 7600 ppm and 2200 to 14,200 ppm respectively. The pH of the waste varied from 8.0 to 9.3.

With the Dow-FilmTec SW30 membrane, flux with model 10,000 ppm sodium sulfate was about 50 liters per square meter per hour (LMH) at a pressure of 500 psi and 30°C. It reduced to 20 LMH at 5% total solids. With the real waste stream, flux was 8-15 LMH under similar conditions. On-site trials in a continuous feed-and-bleed mode at 3X showed a 75% drop in flux over 7 days without membrane cleaning. Permeate quality was consistently excellent with less than 100 ppm total solids in the permeate.

# 1. INTRODUCTION

The metal finishing effluent guidelines of 1983 (40 CFR part 433) set definite limits on harmful pollutants that could be discharged. Manufacturing facilities that do electroplating, electroless plating, anodizing, coating (chromating, phosphatizing, and coloring), chemical etching and milling, and printed circuit board manufacture are covered by these guidelines. If a facility engages in any of these six processes, then discharges from other regulated operations in that facility are also subject to these metal finishing guidelines. These forty other unit operations include metal working, organic coating, cutting and welding, paint stripping and several other processes. Permitting authorities, normally the state or publicly owned treatment works (POTWs), use these guidelines as a basis for permitting and setting limits on heavy metals, oil and grease and total toxic organics. Federal law requires that the limits set be at least as stringent as the USEPA guidelines. As a result of the effluent guidelines, most industries installed various waste treatment systems to remove pollutants and meet the end-of-pipe guideline discharge standards. This typically involves pH-adjustment and oil-skimming, followed by lime and settle treatment. The treated water is discharged and the precipitated waste is compacted in a filter press for disposal to landfill. The lime and coagulants used in this process add considerable bulk to the landfill waste.

"The Common Sense Initiative", a sector-based, EPA-led initiative, was launched to explore industry-specific strategies for environmental protection. This program is designed to promote a smarter, cleaner and cheaper environmental performance, employing a non-adversarial consensus process that tests new ideas and approaches. One result of the CSI in metal finishing was "The Strategic Goals Program" that created a set of voluntary National Performance goals for the industry. The goals are divided into two segments. The first is a facility-based goal and the second is an industry-based goal. One facility and industry goal directly addresses water usage. Using 1992 as a base year, the facility-based goal is to achieve a 50% reduction in water purchased and used by the year 2002. The industry goal is for 80% of facilities nationwide to achieve these goals.

Membrane-based filtration processes such as ultrafiltration (UF) and reverse osmosis (RO) are increasingly being recognized as important technologies to help achieve these goals in electroplating and other metal finishing industries (Cheryan and Rajagopalan 1998). In particular, they could reduce water consumption by at least 50% and potentially as much as 90% by allowing recycling of treated electroplating effluent that is currently discharged.

Cushnie (1994), in a survey of electroplating facilities nationwide, reported that only 1.3% of the respondents used any form of membrane technology, such as reverse osmosis (RO). Extrapolation of the survey results suggest that only 170 companies out of 13,500 plants in the country may be using any form of membrane technology. Many respondents thought that membrane fouling in the form of biofouling by algae, colloidal fouling and scaling as significant problems with the technology. Fouling decreases the productivity of the system, increases maintenance costs and in general lowers the reliability of the technology. This uncertainty with respect to performance, varying quality of wastewater, and perceived cost of treatment are factors that have limited the use of these technologies.

Fouling of membranes can be controlled by proper pretreatment. However, the specific components of the feed stream that cause the fouling must be identified if a cost-effective pretreatment is to be implemented. The determination of fouling potential of electroplating wastewater effluent, identification of foulants and appropriate pretreatment is the subject of this proposal. The results of this project are expected to have broad applicability to a number of electroplating facilities that might want to consider the use of membranes for recycling effluent wastewater.

### 1.1. Literature Review

Several studies have been conducted on the use of RO for recycling rinse waters in electroplating. Its use for concentrating nickel (Ni) in rinse waters is widely accepted as a successful application (Cushnie, 1994). However, it appears that RO has not been as successful so far in treating mixed electroplating waste. In one study (Schoeman et al., 1992), a mixed electroplating effluent (mixture of acid and alkaline streams) with a pH of 4.5 was treated by RO after clarification by ultrafiltration. RO was done at 600 psi using tubular RO elements. Flux was steady at 38 liters/m$^2$.h (LMH) even at the relatively high volume recovery of 96%. The rejection of conductivity was 96% with rejections of cadmium (Cd), nickel (Ni) and zinc (Zn) at 96%, 71%, and 97% respectively (Table 1). Copper removal was not very effective and iron levels were reported to be 40 mg/L. However, the tests were conducted for a short period and no data on fouling potential on a long term basis was reported.

*Table 1. RO of mixed electroplating waste (Schoeman et al., 1992)*

| **Constituent (mg/L)** | **Feed** | **Permeate** | **Concentrate** | **Rejection (%)** | **Tap water** (Pretoria, S. Africa) |
|---|---|---|---|---|---|
| Sodium | 181 | 28 | 629 | 84.5 | 21.5 |
| Potassium | 5 | 1 | 32 | 80.0 | 2.25 |
| Calcium | 56 | 3 | 252 | 94.6 | 33.25 |
| Magnesium | 19 | 2 | 92 | 89.5 | 9.5 |
| Nitrate+Nitrite | 4.9 | 1.8 | 14.4 | 63.3 | 0.5 |
| Sulfate | 618 | 53 | 1544 | 91.4 | 29.5 |
| Chloride | 221 | 49 | 1050 | 77.8 | 20.75 |
| COD | 96 | 40 | 305 | 58.3 | 12.75 |
| TDS | 1368 | 188 | 6252 | 82.3 | 197 |
| Chromium (total) | <0.2 | < 0.2 | | | |
| Iron | 39.5 | 0.55 | 56.25 | 98.6 | 0.09 |
| Cadmium | 10.1 | 0.4 | 30 | 96.2 | |
| Copper | 3 | 2.3 | 6.1 | 25.0 | 0.025 |
| Nickel | 20.8 | 6 | 82 | 71.2 | 0.025 |
| Zinc | 100 | 3.25 | 430 | 96.7 | 0.083 |
| Conductivity (ms/m) | 174 | 24.8 | 660 | 85.7 | <70 |

The above study is an interesting example in two aspects. It utilized ultrafiltration as a primary clarification step followed by RO. Second, it proposed using chemical treatment of the concentrate after RO. The first step presumably removed all suspended solids and emulsified oil, and perhaps also surfactants to a limited degree. It is likely that organic contaminants were also removed during UF treatment by partitioning into the oil phase, though this was not explicitly discussed in that work. The molecular weight cut-off (MWCO) of the UF membrane was not mentioned though the membrane material was identified as polysulfone.

Chemical treatment following RO is an interesting approach since it avoids the problems of increased TDS loading in the feed water as might occur if it is neutralized with lime or sodium hydroxide. However, it is not clear whether preconcentration by RO would increase the difficulty of subsequent treatment and cause problems achieving the required compliance levels. The low rejection of copper and nickel point to the probability of organic complexes.

Another study (Chai et al., 1997) focused on RO of the first rinse from a copper electroplating line. The feed was composed of copper sulfate, sulfuric acid and gelatin and had a pH of 0.63. The feed was pretreated with a 1 μm cartridge filter, pH adjusted to 4, and ultrafiltered with 30,000 MWCO polysulfone membrane. The RO system used was a thin film composite membrane. These studies were primarily on a laboratory scale and short term. They reported a 98.8% rejection of copper. This is to be contrasted with the previous study where copper rejections were very low. This points out that the system performance is highly dependent on the matrix of the feed water.

Sato et al (1977) also studied treatment of electroplating wastewater with RO. The wastewater was a combination of alkaline cyanides and acidic chromium. It was pretreated before filtration and further post-treated. They report significant problems with pretreatment due to fluctuations in the feed water quality. A similar observation was reported by Kremen et al. (1977) in the treatment of brass mill waste. They however reported resolving the problem by careful monitoring and operation of the dual media filter.

It is clear from the above that the feed matrix can significantly impact both membrane performance and fouling. The effect of chelating agents on ion rejection as well as interaction effects of ions in a multi-solute situation is much more difficult to control. One possible approach is to segregate constituents causing such problems for separate treatment. Potential methods of pretreatment for lowering fouling is discussed below.

## 1.2. Pretreatment

Fouling of RO membranes can occur in several ways (Barger and Carnahan, 1991). These include colloidal fouling, scaling or precipitation of sparingly soluble salts (Brucilovsky et al., 1992), biological fouling, and pore restriction by adsorption. All of the above cause decreases in flux and productivity and also affect salt rejection.

The potential for colloidal fouling of RO membranes has traditionally been measured by parameters termed variously as silt density index (SDI), fouling index (FI) or plugging potential (Walton 1987). Multimedia filters and cartridge filtration have long been used as pretreatment for reducing the SDI. In many cases, the potential for colloid formation is also minimized by changing the solution environment, e.g., lowering pH. However, these traditional technologies are prone to process upsets and can be particularly unreliable in electroplating facilities where batch discharges are very common. More recently, novel RO module designs such as ROChem™ high-pressure disk tube technology (Peters 1991; Rautenbach et al. 1996) have demonstrated the ability to tolerate high SDI feed waters very well.

The use of microfiltration (MF) or ultrafiltration (UF) is another technique that has recently gained currency as a method of pretreatment (Cheryan 1998), especially in seawater desalination (Bou-Hamad et al., 1997; Rautenbach, et al., 1997; Rosberg, 1997; Wilf and Klinko, 1998). The primary advantage of MF and UF is that the quality of the permeate from these operations is relatively insensitive to variations in feed quality. However, this approach is not without its own problems. The microfilters and ultrafilters are also themselves susceptible to fouling. High capital and operating cost of the final system make the proposition less attractive. Coagulation as a pretreatment for reducing fouling of microfilters has been shown to very successful (Abdessemed, 1998). Coagulation is effective at reducing both colloidal fouling as well as lowering dissolved organic carbon, both of which alleviate fouling problems downstream. The recent introduction of immersed filtration systems have allowed dramatic reductions in operational costs by lowering energy requirement, decreased complexity of operation, and reduced capital costs (Cote et al. 1998; El Hani Bouhabila 1998). The combined use of existing coagulation systems in electroplating plants and the use of immersed filtration appear very promising as a pretreatment to RO.

The use of calcium hydroxide and chloride for coagulation and pH adjustment as well as introduction of other cations such as aluminum (aluminum chloride) and iron (ferric chloride) can cause additional problems of scale formation and hydroxide precipitation on the RO membrane. Typical approaches to limit the adverse impact include the use of anti-scalants and pH adjustment (Butt et al., 1995). An intriguing possibility is the use of seeded crystallization of the feedwater to promote external growth of crystals and colloids. This may further decrease the cost of chemicals in the RO process.

Biological fouling is another common problem in RO. However, microfiltration or ultrafiltration will minimize this problem to a great extent. Finally, organic fouling of RO can occur, although it is possible it could be less of a problem after coagulation.

Thus, the overall objective was to study the factors that affect the reverse osmosis of electroplating effluent streams. The specific objectives were:

(1). To survey and document effluent treatment practices in an elecroplating facility and fluctuations in water quality. This was done to provide information on day-to-day variations in treated effluent quality.

(2). To design a reverse osmosis system for pilot testing at an electroplating facility.

(3). To study and document the performance of the above system on a long-term basis. This will include collecting data on productivity, salt rejection, identification of foulants and developing cleaning methods.

# 2. METHODS AND MATERIALS

## 2.1. Electroplating waste

The electroplating waste was obtained from the Guardian West plant of Flex-n-Gate, an electroplating facility in the Urbana, Illinois. This plant electroplates bumpers for several automobile manufacturers including Toyota and General Motors. The major operations being performed are stamping, forming, cleaning, buffing, nickel plating and chromium plating. There are various chemistries being used that are typical of such facilities. At the start of this project, the wastewater treatment consisted of neutralization and precipitation.

An automatic sampling (ISCO 6700 FR) system was set-up at the Guardian West plant to characterize the variability of the effluent stream. Two sets of samples were collected during October and November 2000.

## 2.2. Membrane equipment

Two types of equipment were used for membrane studies:

### *2.2.1. Bench-top equipment*

These were dead-end stirred cells which used flat-sheet membrane disks or "coupons" and used gas from a compressed gas cylinder to provide the pressure for filtration. A schematic of the cells is shown in Figure 1. One of the cells was used only for ultrafiltration experiments (Model 502 from Amicon Corporation, now part of Millipore, Bedford, MA). It was made of plastic and could withstand a pressure of about 75 psi. The cell holds a flat-sheet membrane disc of 62mm diameter with an effective area of 28.7 $cm^2$. The reservoir containing the feed/retentate was continuously stirred with a magnetic stirrer and kept at the required temperature by immersing the whole cell in a water bath. Permeate was collected continuously while retentate samples were collected by releasing the pressure and taking an aliquot as needed. For diafiltration experiments, fresh DI water was added to the retentate in the cell and re-pressurized.

The other dead-end stirred cell was the Sepa ST (Osmonics, Minnetonka, MN) and was used for reverse osmosis experiments. It was made of stainless steel and could withstand pressures up to 1500 psi. It could accommodate flat-sheet disks/coupons of 5 cm diameter (15.2 $cm^2$). The cell had a magnetic stirrer which rotated at about 300 RPM and had a provision for taking samples of retentate under pressure as shown in Figure 1.

### *2.2.2. Pilot plant equipment*

A schematic of the pilot plant reverse osmosis system is shown in Figure 2. It consisted of a feed tank with a cooling coil, a high-pressure pump and a membrane module with associated fittings such as pressure gauges, valves and flow meters in the retentate and permeate lines. Two similar systems were designed and fabricated during the course of this project. In one system, the feed tank was a 20-gallon square cross-sectioned tank and the pump was a high-pressure Wanner D-10 model fitted with a variable speed drive that had a rated capacity of 10 gpm at 1000 psi. In this system, cooling was provided only with the cooling coil in the feed tank and there was no external heat exchanger. The module was mounted vertically in the initial series of tests in our laboratory, but mounted horizontally on a frame when this system was taken to the Guardian West plant for the on-site tests.

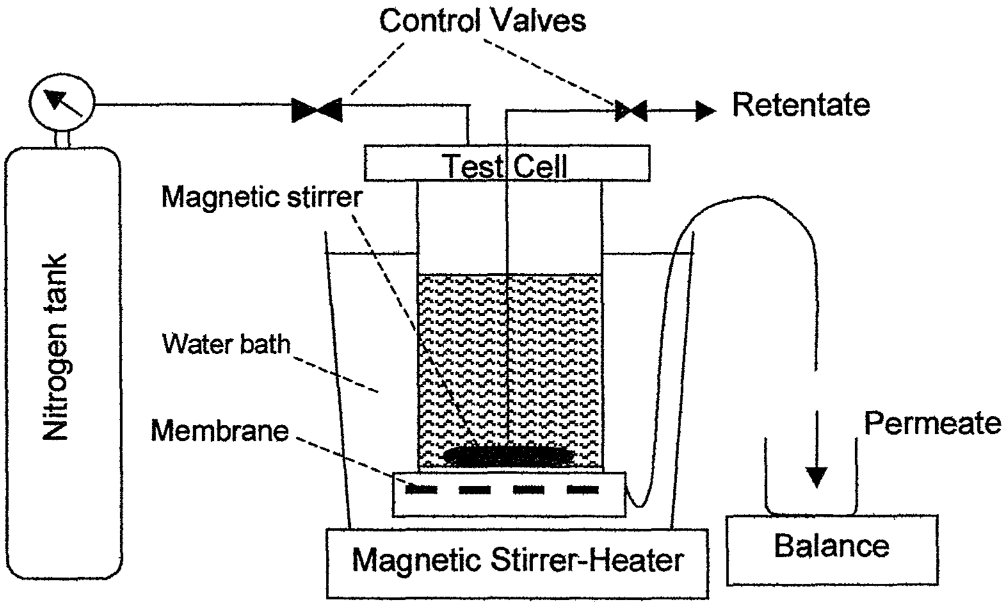


*Figure 1: Bench-top stirred cell. The Amicon UF cell did not have the retentate outlet*

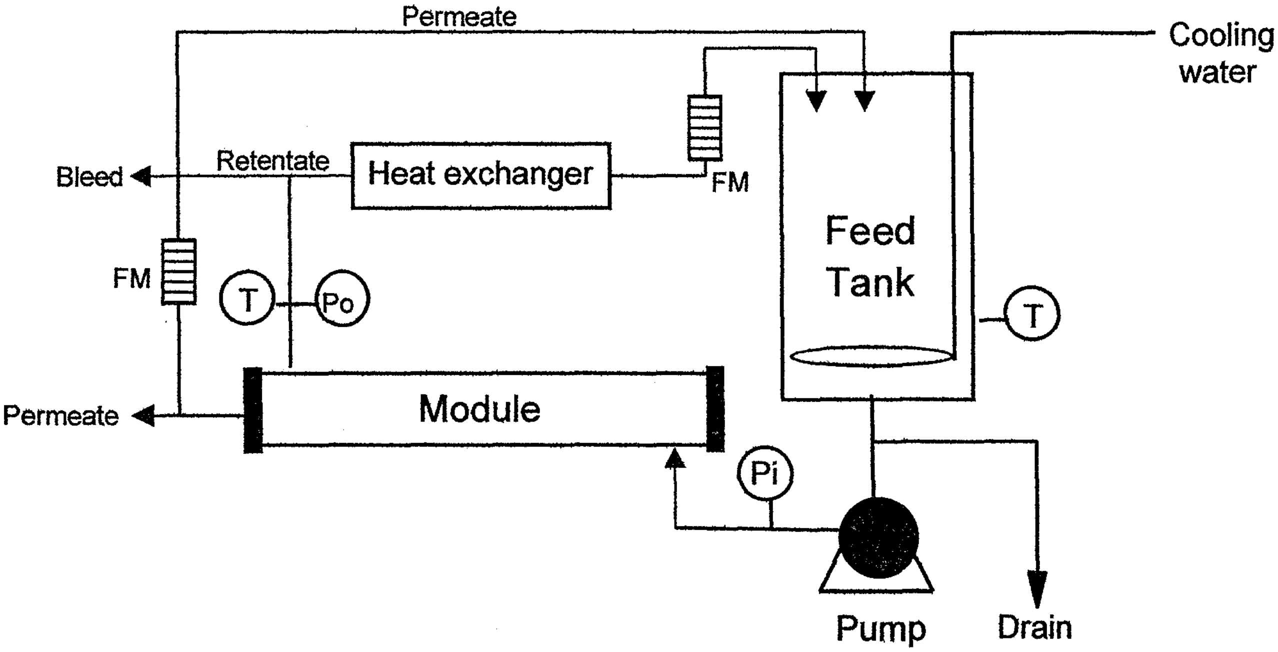


*Figure 2: Pilot plant system for reverse osmosis. Pi = inlet pressure, Po = outlet pressure, FM = flowmeter, T = temperature.*

In the other system, the feed tank was either a cylindrical 30-gallon tank or a 100-gallon tank, depending on the volume processed, and the pump was a D-35 Wanner pump with a variable speed drive rated at 35 gpm at 1000 psi. This system had both a cooling coil fitted in the tank as well as a shell-and-tube heat exchanger connected to a refrigerated water supply at 1-2°C. The module was mounted horizontally on a frame with this system. In all other aspects, the systems were identical.

The cooling coil was made of ¼" 316 stainless steel and constructed to fit in the feed tank and connected to hot and cold water to control temperature. Two pressure vessels to house the 2540 RO module were obtained. One was made of 316 SS, rated at 1000 psi and constructed by PTI-AFI, Brooklyn Park, MN (cost $1100). High pressure triclamps for use with this system were obtained from Holland Applied Technologies Inc., Burr Ridge, IL. The other pressure vessel was a PVC CodeLine unit made by Pentair Water Treatment group capable of 1000 psi obtained through Home Reverse Osmosis, Peru, IL (cost $250). Fittings for this module (1/4" NPT) were obtained from St. Louis Valve and Fittings, St. Louis, MO. Swagelok fittings for the rest of the piping were obtained from St. Louis Valve and Fittings, St. Louis, MO. Triclamp fittings were obtained from Holland Technologies, Burr Ridge, IL. Thermcouples and flowmeters were obtained from Omega Engineering.

Table 2 shows specifications of the FilmTec SW30-2540 reverse osmosis module obtained from Home Reverse Osmosis, Peru, IL (cost $155). For the trials described in this report, the module was loaded into the SS housing with the brine seal at the downstream end. Fluxes were measured either with the permeate flow meter or with a stopwatch and graduated cylinder.

The following instrumentation on the unit was calibrated:

(a) System thermometer fitted in the feed line just after the pump, against a standard glass thermometer,

(b) Retentate flowmeter (Omega HFL6110A, rated at 0-10 gpm) against a stop watch and graduated cylinder. The Omega flowmeter calibration is shown in Figure 3. All cross flow data mentioned in this report are actual flow rates.

(c) Feed tank (height of water in the tank versus tank volume): Figure 4.

(d) Conductivity meter (Hanna Instruments, Model HI8733) that was used to measure salt concentrations: Figure 5.

Pressure-flux relationships were studied in the total recycle mode, i.e., both permeate and retentate were recycled back to the feed tank to keep concentration of feed constant. The concentration runs were conducted by removing the permeate from the system and weighing the permeate to measure the volume concentration ratio.

### 2.3. Definition of terms

Flux (J) = volume of permeate per unit time per unit membrane area. Typical units are liters per square meter per hour (LMH) or gallons per square foot per day (GFD).

*Table 2. Specifications of the FilmTec SW30 reverse osmosis module*

| | |
|---|---|
| Manufacturer | Dow/FilmTec, Edina, MN |
| Configuration | Spiral-wound with fiberglass outerwrap and U-cup brine seal |
| Membrane material | Polyamide (SW30, for sea water desalination) |
| Diameter | 2.4” |
| Length | 37.8” |
| Permeate tube diameter | 0.75” |
| Membrane area | 29.5 ft$^2$ (2.75 m$^2$) |
| Spacer height | 30-mil |
| Maximum feed turbidity | 1 NTU |
| Maximum feed silt density index | 5 |
| Maximum pressure | 1000 psi (6.9 MPa) |
| Recommended maximum cross flow | 6 gallons per minute (22 L/min) (1.4 m$^3$/h) |
| Maximum pressure drop | 10 psi |
| Maximum temperature | 113°F (45°C) |
| Recommended pH range | 2-11 |
| Recommended cleaning procedure | pH 1-12, 30 min |
| Maximum chlorine exposure | <0.1 ppm |
| Recommended storage solution | 1.5% (w/w) sodium metabisulfite |
| Product water flow rate with 32000 ppm NaCl, 800 psi, 25°C, pH 8 | 540 gpd (83 L/h) (30 LMH) |

$$\text{Transmembrane pressure (TMP or P)} = (0.5)\,(Pi + Po) \qquad (1)$$

$$\text{Pressure drop } (\Delta P) = (Pi - Po) \qquad (2)$$

where Pi = inlet pressure (psi) and Po = outlet pressure (psi)

$$\text{Rejection (R)} = (1 - Cp/Cr) \times 100 \qquad (3)$$

where Cp = concentration of solute in permeate, and Cr = concentration of solute in retentate

$$\text{Concentration factor (X)} = \text{Initial volume of feed/retentate volume} \qquad (4)$$

where retentate volume = feed volume – permeate volume

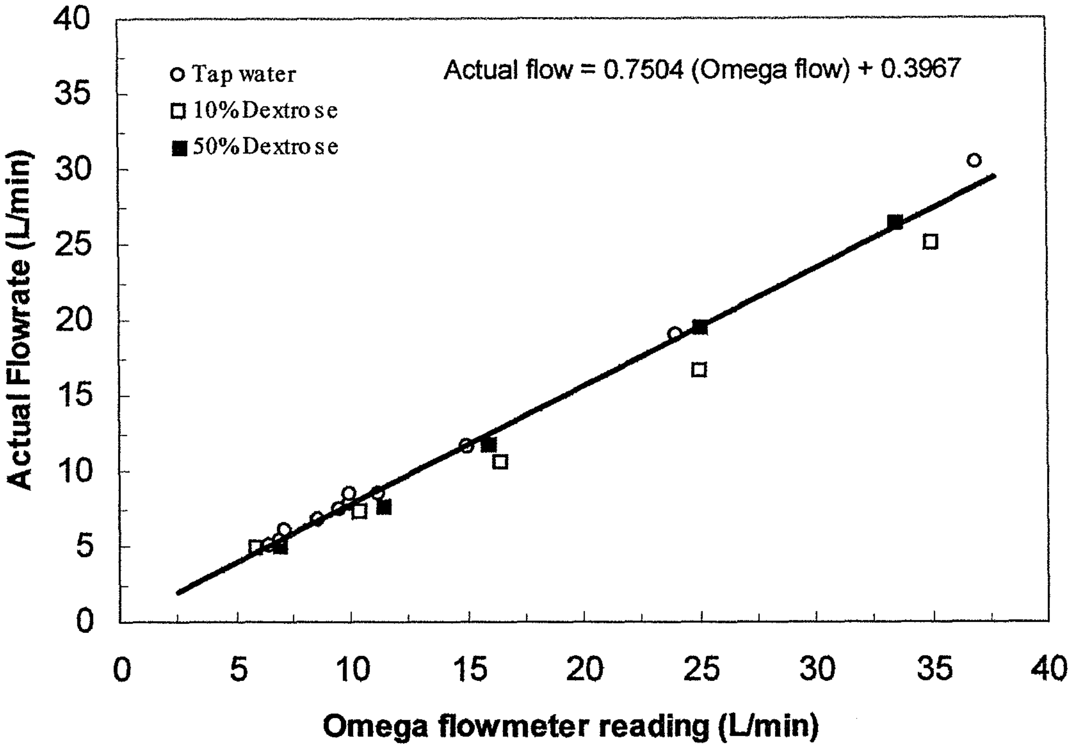


*Figure 3. Calibration of Omega flowmeter in retentate line*

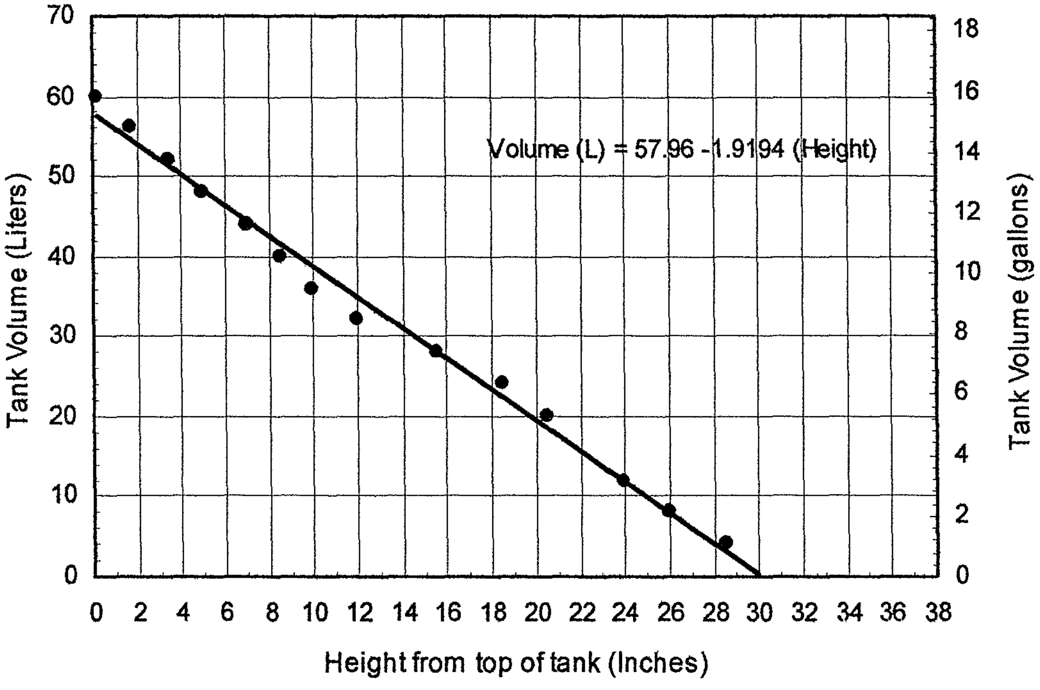
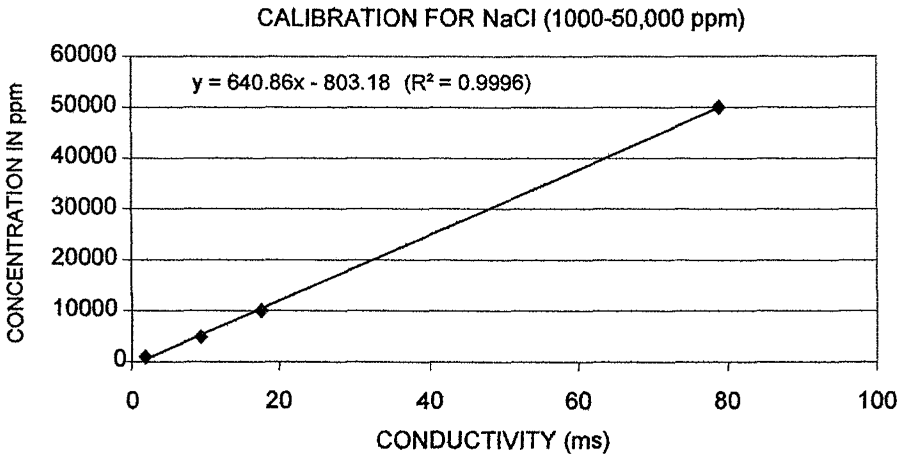
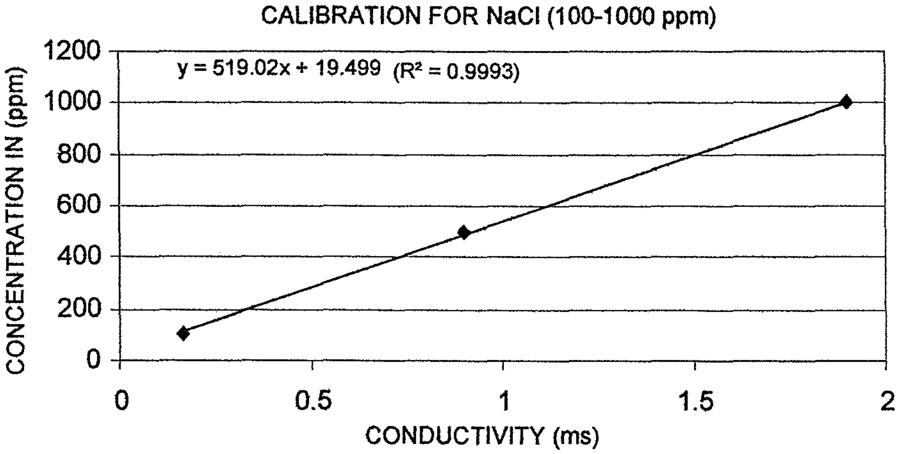


*Figure 4. Calibration of 15-gallon square cross-sectioned feed tank of system*





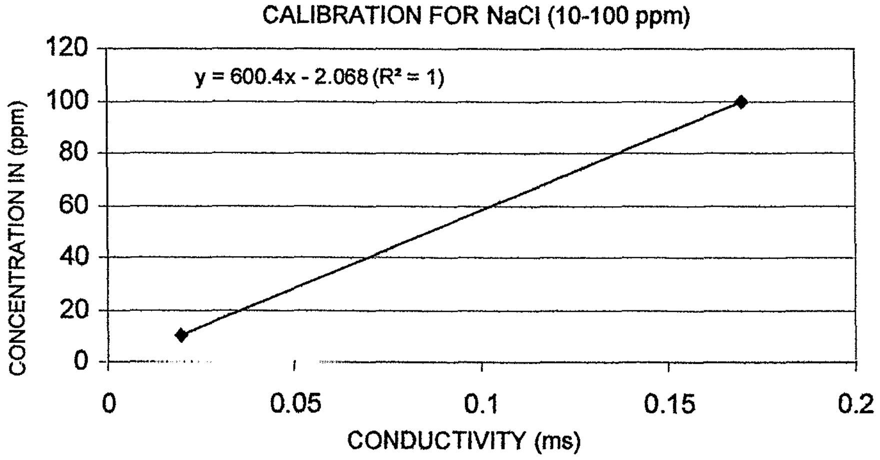


*Figure 5. Calibration of Cole-Parmer conductivity meter*

# 3. RESULTS AND DISCUSSION

## 3.1. Process map and effluent characteristics

The overall schematic of the plant is shown in Figure 6. The sequence of steps includes metal sheet rolling, cutting, stamping, welding, buffing/grinding, prewashing, cleaning, plating and washing.

Figure 7 and Table 3 show the process map of the plating operation and the wastewater treatment operation. The analysis of the waste water are shown in Tables 4-7. The primary observations resulting from the analysis are summarized below:

(a) The total dissolved solids (TDS) of the effluent is quite high with an average of 13,000 mg/L and a range of 10,000-18,000 mg/L for the samples collected in November. As a reference point, the TDS of seawater is commonly taken as 30,000 mg/L. The immediate consequence of the high TDS is that the highest recovery using conventional single stage reverse osmosis (RO) equipment, under ideal conditions, would be limited to about 80% (the equivalent of a volume concentration factor of 4).

(b) The primary contributors to TDS are sodium and sulfate ions in the effluent. The presence of sulfate ions in the system is traced to the various acid rinses and plating baths used in the facility and is expected. The high level of sodium is believed to be from caustic rinses, alkaline cleaners as well as caustic addition at the waste water treatment plant.

(c) The analytical data (Tables 4-7) suggest that barium sulfate concentrations exceed solubility limits greatly. If it is present as part of the total suspended solids (TSS), it may not pose a problem as SS can be removed by ultrafiltration. However, fouling by calcium carbonate is definitely possible in this effluent stream. The carbonate levels in the effluent were arrived at by subtracting the total anions from total cations to represent total carbonates. Depending on the pH, these can exist as bicarbonate, carbonate and/or carbon dioxide. Fluctuations in pH (as is evident in the analytical data) as well as the higher concentrations during RO could result in severe fouling and limit recoveries.

One approach to controlling such fouling is to increase the pH of the effluent to force the total carbonate to exist primarily as carbonate and force it to precipitate and then be removed by filtration. This is similar to current water softening approaches. A second approach is to do the opposite and lower carbonate by acidification to convert it to bicarbonates. Both approaches are feasible and could be used at Guardian West. If the effluent is ultrafiltered prior to pH adjustment, the acid-adjusted effluent could then be sent for RO.

(d) The variation in effluent quality is very high as seen in the data presented in Tables 4-7. This will have a substantial impact on the operating characteristics of the RO system, especially in terms of productivity and fouling. The site should offer ample opportunity to study the impact of such fluctuations on the robustness of RO operation

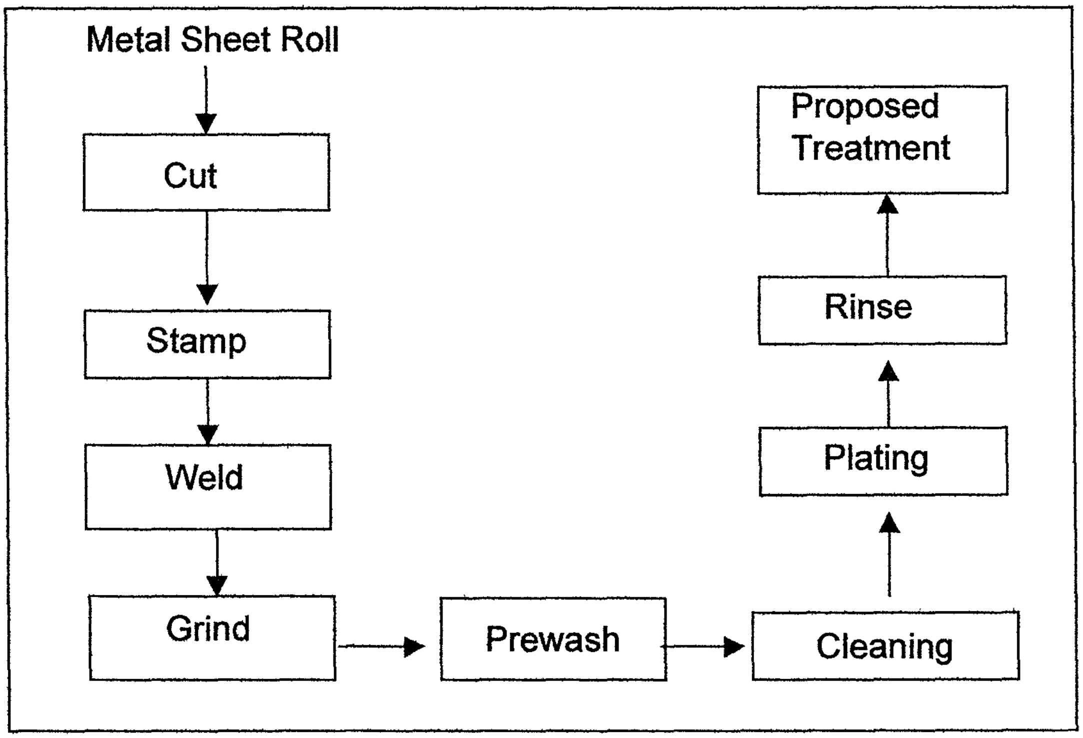


*Figure 6. Schematic of plant operations at Guardian West*

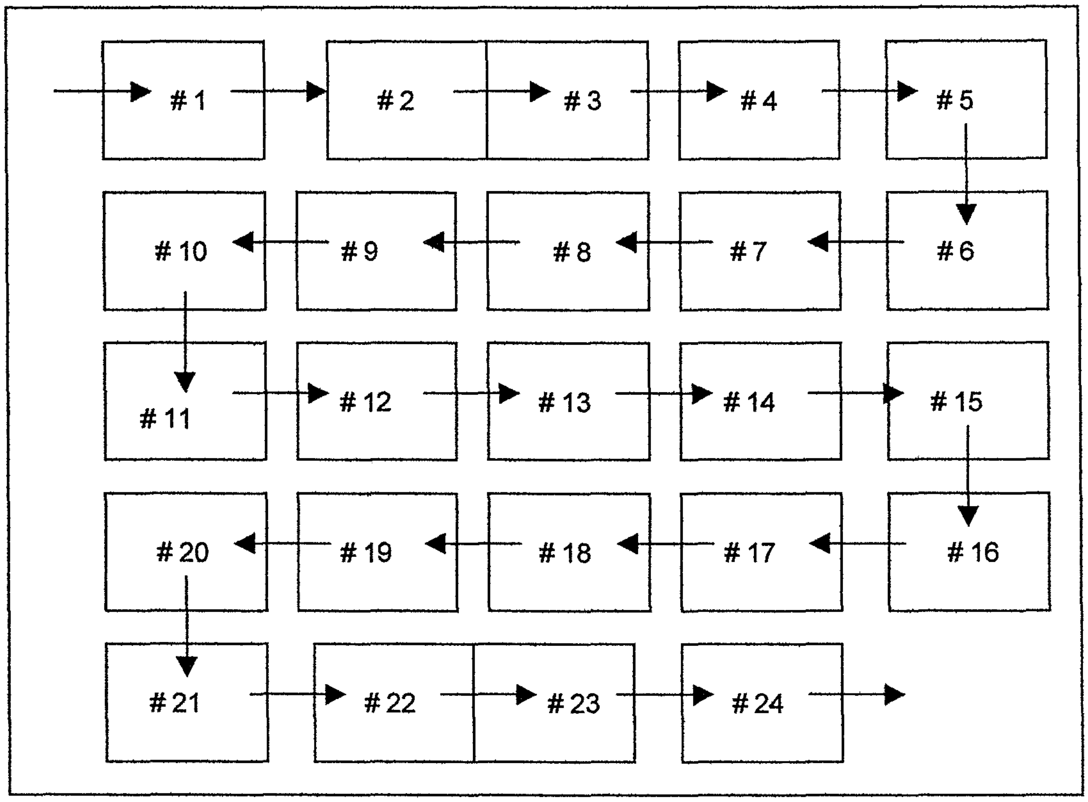


*Figure 7: Process map of the plating and wastewater treatment operations.*

*Table 3. Process map of Guardian West electroplating line*

| Tank # | DESCRIPTION | MATERIALS | CONC. (oz/gal) | TEMPERATURE (°F) | DUMP FREQUENCY | VOLUME (gallons) |
|---|---|---|---|---|---|---|
| 1 | SOAK | ENPREP 146 | 10 | 190 | 2 weeks | 3650 |
| 2 | SPRAY | | | | Weekly | |
| 3 | ELECTROCLEAN | ENPREP 270 | 12 | 160 | 3 weeks | 4230 |
| 4 | CHROME STRIP | ENSTRIP 486 | 8 | 140 | 6 months | 4230 |
| 5 | RINSE | | | | weekly | |
| 6 | ACID | MURIATIC | | | | 2260 |
| 7 | RINSE | | | | weekly | |
| 8 | REPLATE ACID | Sulfuric Acid | 10% | | 2 weeks | 2260 |
| 9 | NEUTRALIZE | | | | weekly | |
| 10 | ELECTROCLEAN | ENPREP 270/271 | 12 | 160 | 3 weeks | 2300 |
| 11 | RP ELECTRO | ENPREP 274 | 12 | 160 | 3 weeks | 2300 |
| 12 | CF RINSES | | | | weekly | |
| 13 | ACID | Muriatic | 15% | | 2 weeks | 2260 |
| 14 | CF RINSES | | | | weekly | |
| 15 | NICKEL STRIKE | Ni sulfate, Ni chloride<br>Boric acid | | 90 | | 2500 |
| 16 | RINSE SHUTTLE | | | | weekly | |

………Table 3 continued next page

*Table 3 (continued):*

| | | | | | | |
|---|---|---|---|---|---|---|
| 17 | SEMI-BRIGHT NI | Ni sulfate, Ni chloride<br>Boric acid | | 135 | | 10,800 |
| 18 | SEMI-BRIGHT NI | Same as above | | 135 | | 10,800 |
| 19 | SEMI-BRIGHT | Same as above | | 135 | | 10,800 |
| 20 | TRI-NI | Ni sulfate, Ni chloride<br>62A<br>Tri-Ni | | 135 | | 2650 |
| 21 | BRIGHT NI | Ni sulfate, Ni chloride,<br>Boric acid<br>61<br>63<br>62A<br>66E | | 135 | | 10,800 |
| 22 | DUR-NI | Nickel sulfate<br>Nickel chloride<br>Boric acid<br>DN 618<br>DN Enhancer 63 | | 135 | | 2500 |
| 23 | RINSE | | | | | |
| 24 | CF RINSES | | | | weekly | |
| 25 | PRE-DIP | Chromic acid<br>Sulfuric acid | | | 3 months | |
| 26 | CHROME | Sulfuric acid | | 115 | Recycled | |
| 27 | CF RINSES | | | | weekly | |
| 28 | HOT RINSE | | | 190 | weekly | |

*Table 4. Major anions in Guardian West waste stream*

| Date | Fluoride (mg/L) | Chloride (mg/L) | Nitrate (mg/L) | Sulfate (mg/L) |
|---|---|---|---|---|
| Oct 17-1 | < 100 | 430 | < 100 | 3,700 |
| Oct 17-2 | < 100 | 1100 | < 100 | 5,600 |
| Oct 17-3 | < 100 | 1400 | < 100 | 4,900 |
| Oct 18-1 | < 100 | 930 | < 100 | 5,900 |
| Oct 18-2 | < 100 | 570 | < 100 | 6,100 |
| Oct 18-3 | < 100 | 400 | < 100 | 6,200 |
| Oct 18-4 | < 100 | 390 | < 100 | 6,700 |
| Oct 19-1 | < 100 | 480 | < 100 | 7,300 |
| Oct 19-2 | < 100 | 510 | < 100 | 6,900 |
| Oct 19-3 | < 100 | 470 | < 100 | 7,500 |
| Nov 6-1 | < 13 | 348 | < 13 | 7,230 |
| Nov 6-2 | < 13 | 368 | < 13 | 9,870 |
| Nov 7-1 | < 13 | 424 | < 13 | 7,640 |
| Nov 7-2 | < 13 | 472 | < 13 | 9,480 |
| Nov 8-1 | < 13 | 356 | < 13 | 9,340 |
| Nov 8-2 | < 13 | 366 | < 13 | 14,200 |
| Nov 9-1 | < 13 | 272 | < 13 | 6,840 |
| Nov 9-2 | < 13 | 470 | < 13 | 13,400 |
| Nov 10-1 | < 13 | 444 | < 13 | 8,210 |

*Table 5. Major cations in Guardian West waste stream*

| Date | Sodium (mg/L) | Potassium (mg/L) | Magnesium (mg/L) | Calcium (mg/L) | Barium (mg/L) |
|---|---|---|---|---|---|
| Oct 17-1 | 2100 | 51 | 1.9 | 6.1 | 0.017 |
| Oct 17-2 | 3570 | 87 | 4.0 | 61 | 0.051 |
| Oct 17-3 | 2970 | 78 | 2.5 | 53 | 0.044 |
| Oct 18-1 | 3640 | 87 | 4.7 | 42 | 0.034 |
| Oct 18-2 | 3490 | 92 | 7.8 | 36 | 0.038 |
| Oct 18-3 | 3270 | 213 | 9.0 | 26 | 0.038 |
| Oct 18-4 | 3840 | 246 | 16 | 33 | 0.050 |
| Oct 19-1 | 3800 | 135 | 30 | 31 | 0.077 |
| Oct 19-2 | 3730 | 87 | 51 | 33 | 0.089 |
| Oct 19-3 | 3640 | 205 | 36 | 27 | 0.075 |
| Nov 6-1 | 4960 | 32 | 3.8 | 14 | 0.023 |
| Nov 6-2 | 4480 | 36 | 37 | 28 | 0.059 |
| Nov 7-1 | 4270 | 36 | 16 | 39 | 0.041 |
| Nov 7-2 | 5270 | 32 | 8.8 | 44 | 0.054 |
| Nov 8-1 | 5480 | 34 | 15 | 18 | 0.043 |
| Nov 8-2 | 7610 | 64 | 42 | 50 | 0.073 |
| Nov 9-1 | 3790 | 33 | 3.9 | 8.7 | 0.015 |
| Nov 9-2 | 6140 | 39 | 18 | 41 | 0.049 |
| Nov 10-1 | 4970 | 32 | 2.1 | 13 | 0.024 |

*Table 6. Ion balance and possible carbonate concentration*

| Date | Total Cations (meq) | Total Anions (meq) | Carbonate (meq) (By difference) |
|---|---|---|---|
| Oct 17-1 | 93.07 | 89.20 | 3.87 |
| Oct 17-2 | 160.82 | 147.65 | 13.17 |
| Oct 17-3 | 134.00 | 141.52 | -7.52 |
| Oct 18-1 | 162.97 | 149.11 | 13.86 |
| Oct 18-2 | 156.52 | 143.14 | 13.39 |
| Oct 18-3 | 149.66 | 140.43 | 9.23 |
| Oct 18-4 | 176.26 | 150.57 | 25.69 |
| Oct 19-1 | 172.68 | 165.60 | 7.07 |
| Oct 19-2 | 170.23 | 158.12 | 12.11 |
| Oct 19-3 | 167.79 | 169.49 | -1.70 |
| Nov 6-1 | 217.48 | 160.43 | 57.05 |
| Nov 6-2 | 200.14 | 215.98 | -15.84 |
| Nov 7-1 | 189.83 | 171.11 | 18.72 |
| Nov 7-2 | 232.87 | 210.78 | 22.09 |
| Nov 8-1 | 241.26 | 204.61 | 36.65 |
| Nov 8-2 | 338.45 | 306.14 | 32.30 |
| Nov 9-1 | 166.38 | 150.15 | 16.23 |
| Nov 9-2 | 271.48 | 292.40 | -20.92 |
| Nov 10-1 | 217.73 | 183.54 | 34.19 |

*Table 7. Total organic carbon (TOC) and Chemical Oxygen Demand (COD) in electroplating effluent from Guardian West*

| **Date** | **COD (mg/L)** | **TOC (mg/L)** |
|---|---|---|
| Oct 17-1 | 396 | 166 |
| Oct 17-2 | 464 | 109 |
| Oct 17-3 | 672 | 500 |
| Oct 18-1 | 464 | 180 |
| Oct 18-2 | 712 | 91 |
| Oct 18-3 | 432 | 470 |
| Oct 18-4 | 553 | 550 |
| Oct 19-1 | 589 | 330 |
| Oct 19-2 | 613 | 140 |
| Oct 19-3 | 746 | 420 |
| Nov 6-1 | 494 | 130 |
| Nov 6-2 | 389 | 105 |
| Nov 7-1 | 652 | 189 |
| Nov 7-2 | 362 | 101 |
| Nov 8-1 | 415 | 122 |
| Nov 8-2 | 757 | 201 |
| Nov 9-1 | 389 | 94 |
| Nov 9-2 | 573 | 157 |
| Nov 10-1 | 468 | 136 |

(e) A substantial quantity of total organic carbon (TOC) appears to be present in the effluent and could be an important factor in fouling.

### 3.2. Preliminary bench-top membrane experiments

Bench top experiments were performed three times with effluent collected from the plant site. A considerable variation in effluent quality was observed. Prefiltration with Whatman #1 filter paper (with an equivalent average pore size of 11 microns) resulted in darkening of the filter paper by the suspended solids. This type of pretreatment may be necessary prior to any membrane step to minimize membrane fouling. Flux data are summarized in Figures 8-11. A summary of the results of this study is given below:

(a) Ultrafiltration of the Whatman-filtered effluent with polyethersulfone (PES) membranes (Koch HFK-131, Osmonics HO83) and polyvinylidene difluoride (PVDF) membranes (PTI AF30B, Koch HFM180) showed little or no effect on total solids (TS), conductivity or pH between the feed, permeate and retentate (Table 8)

(b) Reverse osmosis of the Whatman-filtered effluent at 50°C and 600 psi with Osmonics AG, FilmTec SW30 and SW30HR membranes gave good results. Permeate quality was good. Compared to a feed TS of 9.1 g/L, permeate TS was 0.7 g/L with AG and ~0 g/L with FilmTec membranes. pH of feed was 9.0, while permeates were 7.5-7.8. Conductivity of feed was 6220 μmho, while permeate conductivities were 600 μmho (AG), 130 μmho (SW30) and 180 μmho (SW30HR).

(c) Flux at 50°C and 600 psi with RO membranes were 90 LMH for AG, 40 LMH for SW30 and 15 LMH for SW30HR (Figure 11).

*Table 8. Ultrafiltration of electroplating waste: effect on total solids, pH and conductivity*

| | PTI-AF30B | Osmonics HO83 | Koch HFM-180 | Koch HFK-131 |
|---|---|---|---|---|
| Total solids (g/L) | | | | |
| Feed | 6.1 | 6.1 | 6.1 | 6.1 |
| Retentate | 6.0 | 6.0 | 6.0 | 6.0 |
| Permeate | 6.0 | 6.0 | 6.0 | 6.0 |
| .pH | | | | |
| Feed | 9.11 | 9.11 | 9.11 | 9.11 |
| Retentate | 9.13 | 9.18 | 9.13 | 9.23 |
| Permeate | 9.11 | 9.10 | 9.18 | 9.11 |
| Conductivity (μmhos) | | | | |
| Feed | 7430 | 7430 | 7430 | 7430 |
| Retentate | 7560 | 7210 | 6790 | 6790 |
| Permeate | 7300 | 7100 | 6610 | 6270 |

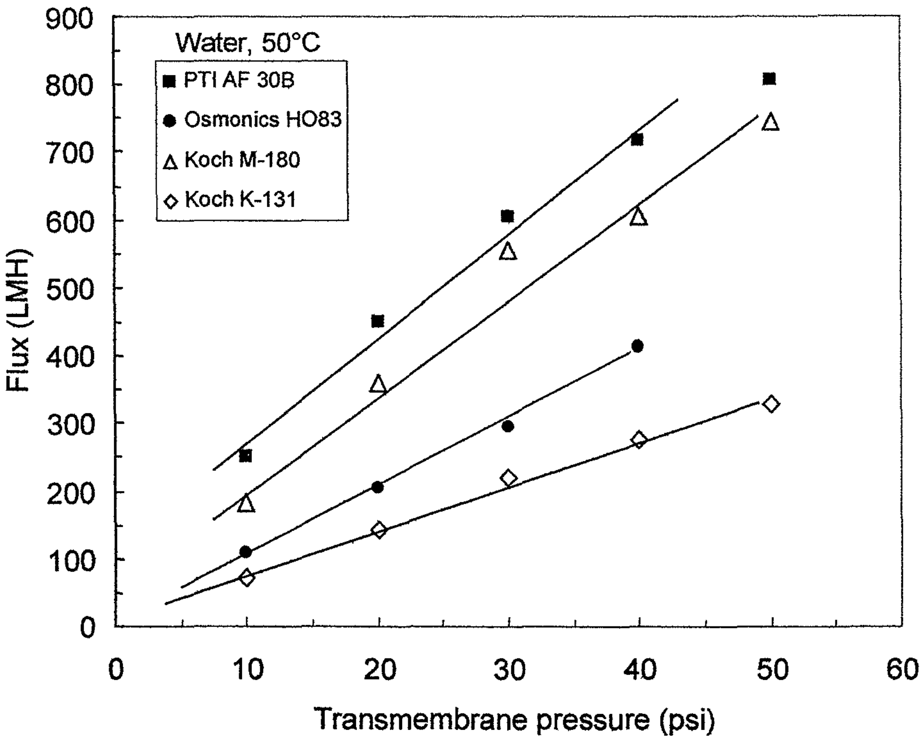


*Figure 8. Water flux of UF membranes at 50°C*

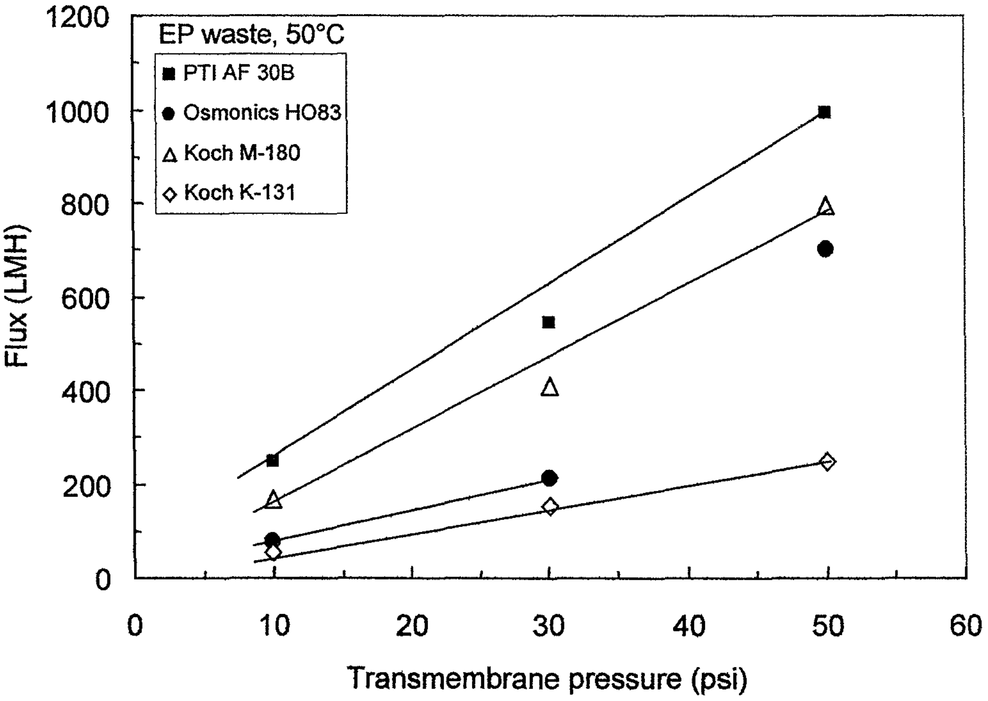


*Figure 9. Flux with EP waste of UF membranes at 50°C*

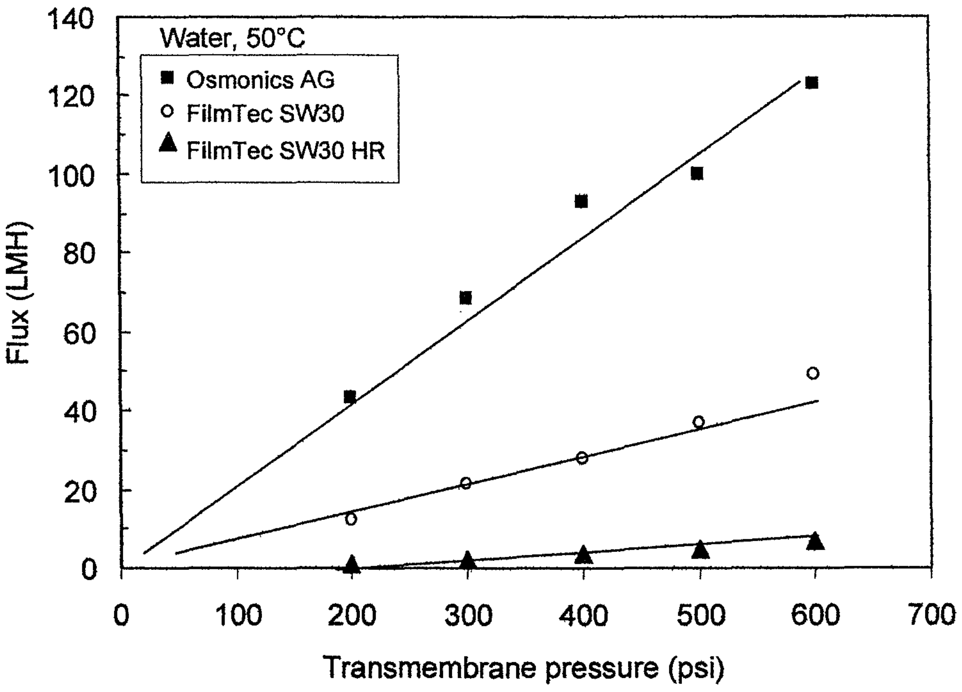


*Figure 10. Pressure vs. water flux for RO membranes at 50°C*

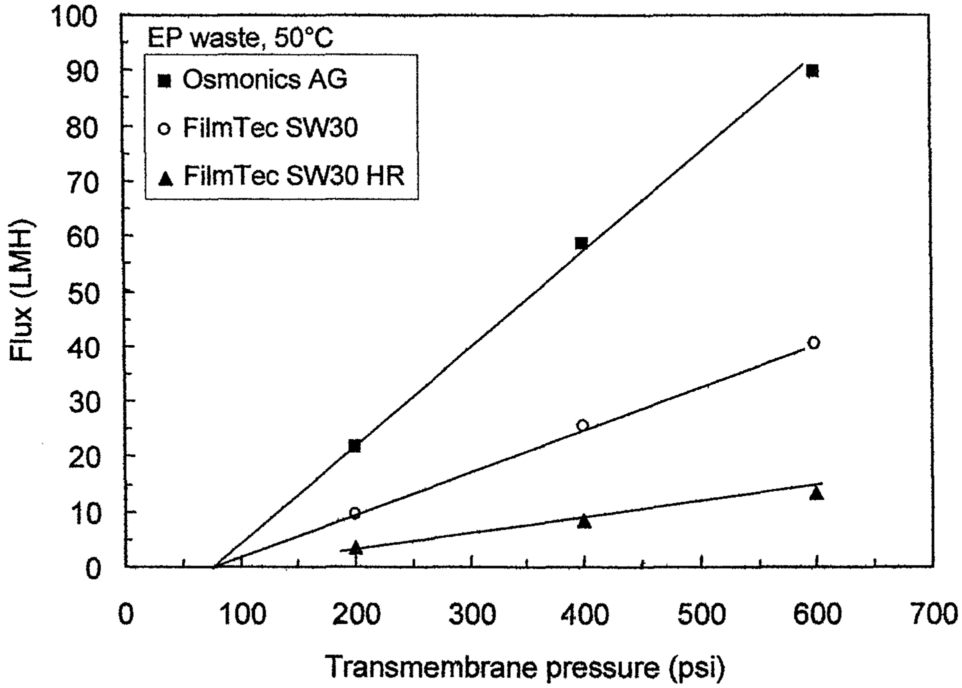


*Figure 11. Pressure vs. EP waste flux for RO membranes at 50°C*

Based on the above studies, the FilmTec SW30 reverse osmosis membrane was selected over the FilmTec SW30HR and Osmonics AG membranes for further testing. Even though the flux of the Osmonics AG membrane is substantially higher, it comes at the expenses of a poorer rejection of solids. On the other hand, the FilmTec SW30 membrane has excellent rejection, good flux and is easily available. Pilot scale testing will proceed with a FilmTec SW30-2540 spiral-wound module.

### 3.3. Preliminary pilot scale experiments

Prior to conducting trials with the electroplating effluent, it is important to characterize a reverse osmosis membrane module in terms of flux behavior with water, inorganic salts and selected organic compounds. This will serve to not only ensure that the module meets manufacturer's specifications, but also provides a base line for evaluating membrane deterioration with use and the efficiency of cleaning. The following data were obtained:

(1) Water flux with tap water and deionized/distilled/microfiltered water
(2) Flux with sodium chloride solutions using 1000 ppm and 10,000 ppm NaCl in DI water
(3) Flux behavior with dextrose using 0.5% and 5% dextrose solutions

Figure 12 shows the relationship between cross-flow rate (Q) and the pressure drop (ΔP). across the SW2540 module A curved relationship is obtained between ΔP and Q, which is expected for a module operating in turbulent flow. The module manufacturer limits the cross-flow rate to 6 gallons per minute (gpm)/22 Liters per min (L/min) or a pressure drop (ΔP) of 10 psi (Table 2). However, our data in Figure 6 indicates that this particular module reaches the ΔP limit of 10 psi at a flow rate of only 9 L/min. The module was operated mostly 3 gpm (11.35 L/min).

#### *3.3.1. Water flux*

Figure 13 shows water flux of the SW-30 module. As expected, a linear relationship is obtained between TMP and flux. Assuming the flow of permeate through a membrane can be modeled as hydraulic flow through channels, the Hagen-Pouisuelle model can be expressed as:

$$J = A\,(P - \Delta\pi) \qquad (5)$$

where J is the flux in LMH, P is the transmembrane pressure (psi) and $\Delta\pi$ is the osmotic pressure difference across the membrane (psi):

$$\Delta\pi = \pi_r - \pi_p \qquad (6)$$

where $\pi$ is the osmotic pressure and subscripts *r* and *p* refer to retentate and permeate respectively. The membrane permeability coefficient A characterizes the overall resistance to permeate flow through the membrane. It is a function of membrane properties such as pore size distribution, pore density, thickness of the membrane, etc., and feed solution properties, such as viscosity, density, diffusivity, etc.

The osmotic pressure of solutions can be estimated to a first approximation by the van't Hoff equation:

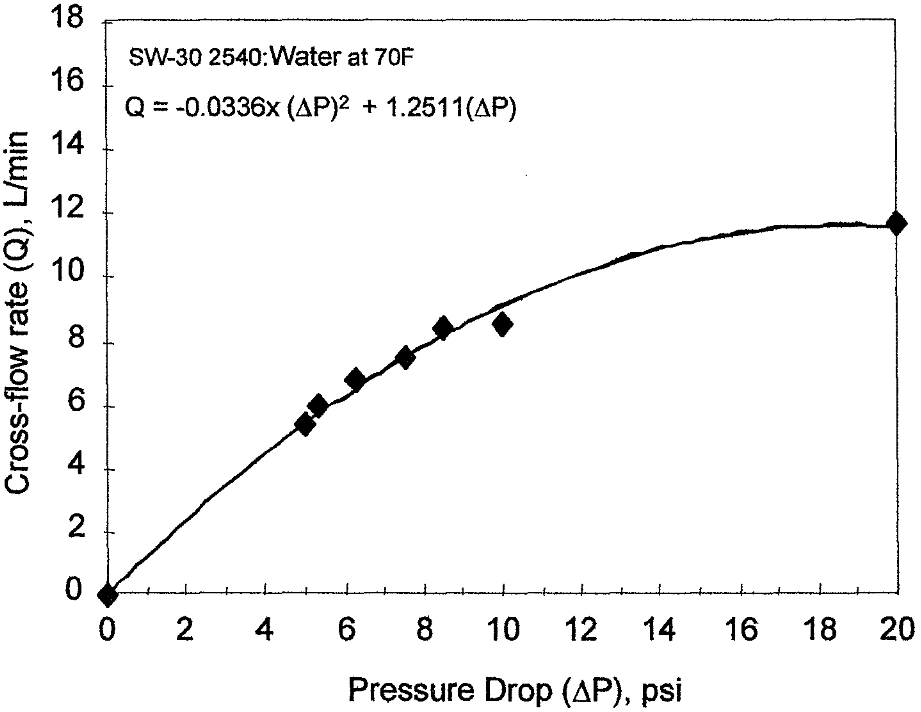


*Figure 12. Correlation between pressure drop and cross-flow rate for 2540 spiral module*

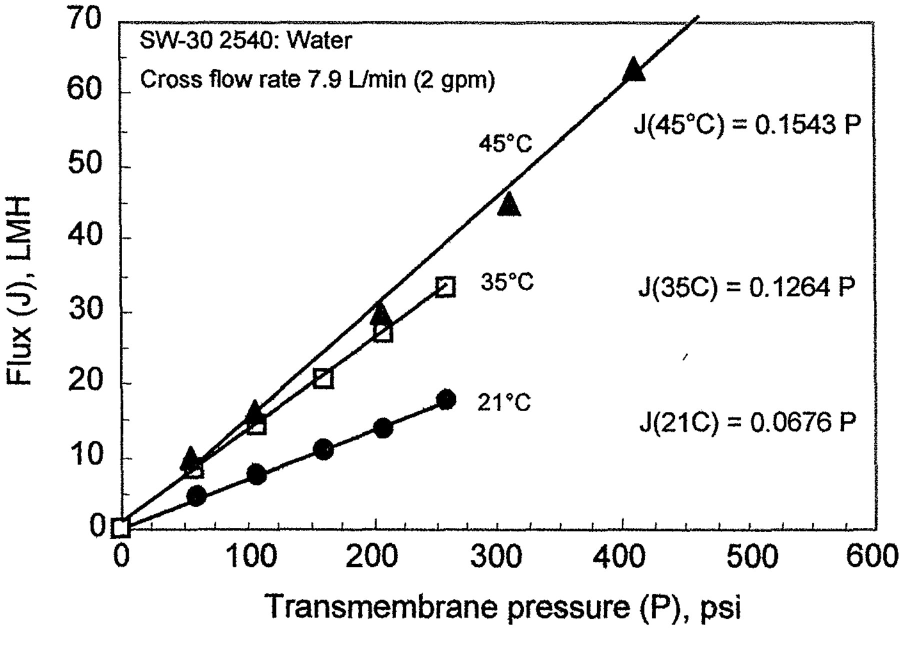


*Figure 13. Water flux of new SW30 2540 spiral module. Data at 21°C and 35°C obtained with tap water, 45°C data with DI water*

$$\pi = iCRT/M \tag{7}$$

where i = number of ions (for ionized solutes). For NaCl, i = 2, for dextrose, i = 1
C = concentration of the solute, g/L
R = universal gas constant, 0.08206 atm-L/gmole-°K
T = absolute temperature, °K
M = molecular weight of solute. For NaCl, M = 58.5, for dextrose, M = 180
$\pi$ = osmotic pressure, atm

From equations (3), (6) and (7):

$$\Delta\pi = R.\ \pi_r \tag{8}$$

Equation (5) becomes $$J = A\,(P - R\,\pi_r) \tag{9}$$

Since A is known or can be easily determined for a membrane, flux can be predicted if the osmotic pressure of the feed solution and membrane rejection of the feed component(s) are known.

With pure water as the feed, $\pi_r$ and $\pi_p$ are zero. The flux models for water at various temperatures were determined from the slopes of the lines in Figure 13 (J is in LMH and P is in psi):

At 69°F (21°C) $$J = 0.0676\ P \tag{10}$$
At 97°F (35°C) $$J = 0.1264\ P \tag{11}$$
At 113°F (45°C) $$J = 0.1543\ P \tag{12}$$

The main reason for the increase in the coefficient A with temperature is due to a decrease in viscosity of the permeate with increase in temperature. For example, the viscosity of water at the above three temperatures are 1 cp, 0.7cp and 0.6 cp, respectively.

### *3.3.2. Salt solutions*

Figures 14-17 show data obtained with a NaCl solution at a feed concentration of ~0.1% (~1000 ppm) NaCl. Figure 14 shows flux-pressure data at a cross-flow of 2 gpm and 45°C. Salt concentration in the permeate is low (5-20 ppm) and decreases at higher pressures (Figure 15). This is probably a dilution effect: the flow of water through the membrane pores increases much faster than the passage of the salt through the membrane. This is because the mechanism of transport of water is probably convective flow while salt transports through the membrane by a diffusion mechanism. This results in an increase in salt rejection with TMP (Figure 15).

The RO flux model (equation 9) can be used to predict the flux. For a feed solution of 987 ppm NaCl at 45°C, the osmotic pressure can be calculated using equation (7):

$$\begin{aligned}\pi &= 2\ (0.987)(0.08206)\ (318.15)/58.5\\ &= 0.881\ \text{atm} = 12.8\ \text{psi}\end{aligned}$$

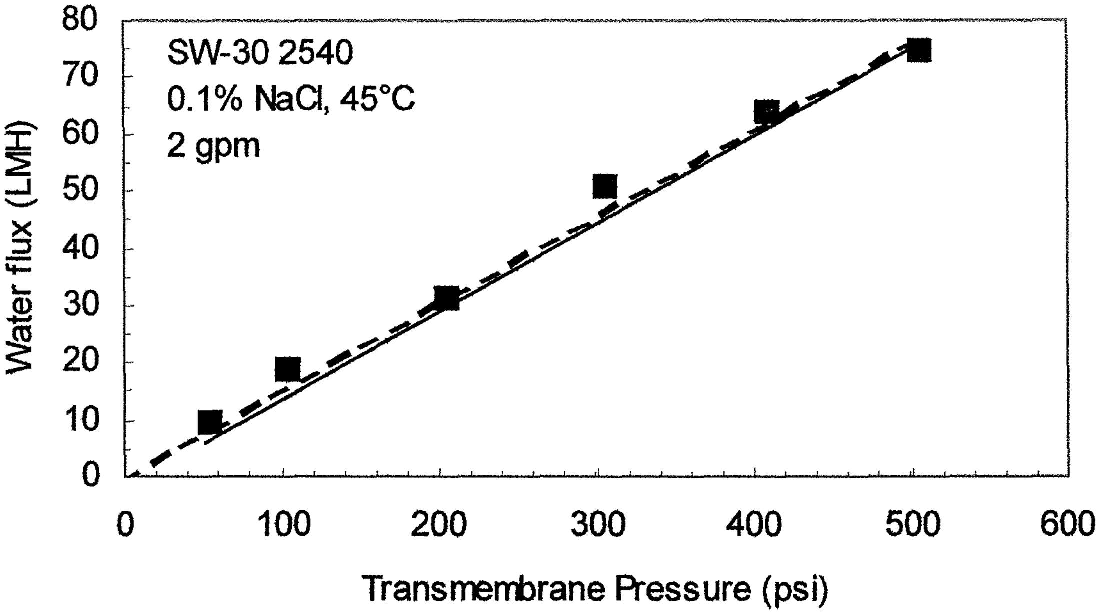


*Figure 14. Flux of water (broken line) and salt (full line) in SW-30 module. Feed was 987 ppm NaCl solution. Full line is model according to equation 13, points are experimental data.*

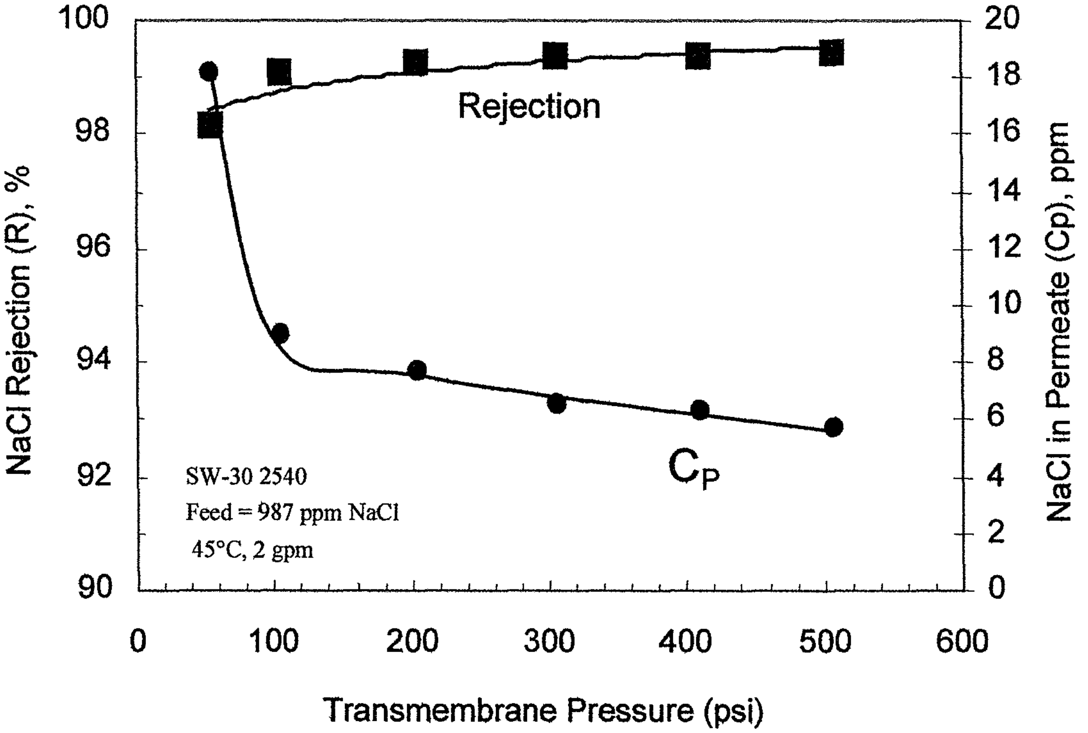


*Figure 15. Salt rejection and passage. Same experiment as Figure 14.*

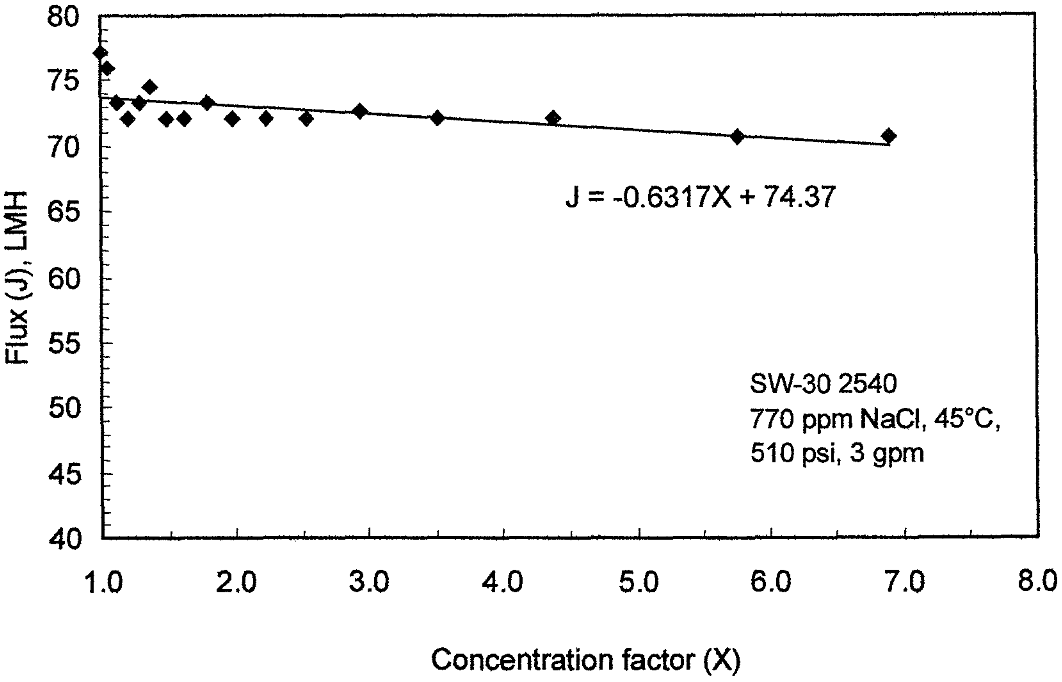


*Figure 16. Effect of concentration on flux. Feed was 770 ppm NaCl solution. Temp = 113°F (45°C), cross flow = 2 gpm, TMP = 510 psi*

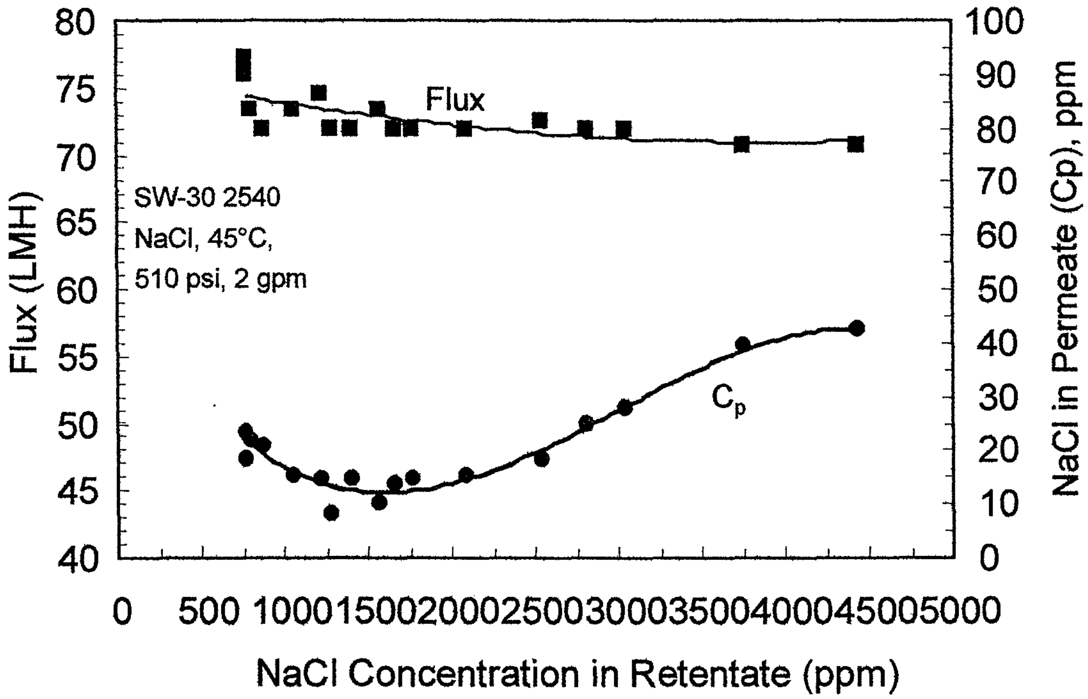


*Figure 17. Effect of concentration on flux and salt passage through membrane. Same experiment as Figure 16.*

Thus, for the 987 ppm NaCl feed solution shown in Figures 14 and 15, equation (9) becomes:

$$J = 0.1543\,[P - (12.8R)] \qquad (13)$$

From Figure 15, the average rejection is about 99%, so the value of $R = 0.99$ can be used in equation (13). Figure 8 shows that equation (13) predicts the flux with dilute salt solutions quite well.

The effect of concentration of salt is shown in Figures 16 and 17. The initial feed was 770 ppm NaCl and the operating pressure was 510 psi. After steady state conditions had been achieved in the total recycle mode (~15 minutes), the concentration mode was started by removing permeate and weighing it to keep track of concentration factor (X). An X of 6.9 was achieved at the end of the run, giving a retentate NaCl concentration of 4441 ppm.. The concentration of the salt in the permeate had increased from ~10 ppm initially to ~40 ppm (Figure 17), resulting in a NaCl rejection of 99%.

According to equation (7), the osmotic pressure of the final retentate (4441 ppm NaCl) is 57.6 psi. Applying equation (5):

$$J = 0.1543\,(510 - 57.6) = 69.8 \text{ LMH} \qquad (14)$$

The actual flux was 70 LMH at the end of the run (Figure 17).

Figures 18-21 show data obtained with a feed solution of ~1% (9817 ppm) NaCl. Two cross-flow rates were studied: 2 gpm and 3 gpm. At this higher concentration of feed, the permeate salt levels were much higher (50-500 ppm) resulting in slightly lower average rejections of 98%. However, the trend was the same as observed with the 0.1% NaCl solution: salt in the permeate decreased at higher pressures. Flow rate did not affect the flux (Figure 18) or salt rejection (Figure 19) significantly, indicating that concentration polarization was not rate-controlling in these experiments (or perhaps the range of flow rates studied were not wide enough to display flow rate effects).

According to equation (7), the osmotic pressure of the feed solution (9718 ppm NaCl) is 126.0 psi. Assuming R = 98%, the flux prediction equation (9) can be written as

$$J = 0.1543\,(P - .98 \times 126) = 0.1543\,(P - 123.5) \qquad (15)$$

As shown in Figure 18, equation (15) does a fairly good job of predicting flux except at higher pressures, where there is a deviation from the predicted line. This could indicate the beginning of concentration polarization effects occurring at the higher pressures or the effect of membrane compaction.

Figures 20 and 21 show the effect of concentration on flux and rejection. The feed was 9718 ppm NaCl, and the system was operated at 510 psi, 3 gpm and 45°C. The flux decreases in a

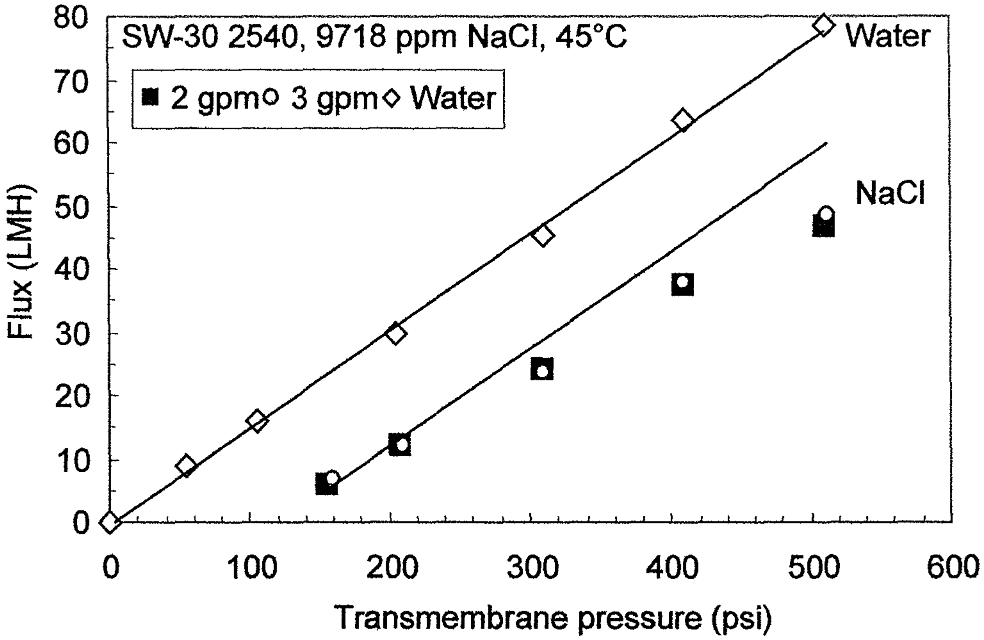


*Figure 18. Flux in SW30 module with a feed of 9718 ppm NaCl solution at cross-flow rates of 2 gpm and 3 gpm. Lines are drawn according to equation 12 for water and equation 15 for salt solutions. Points are experimental data*

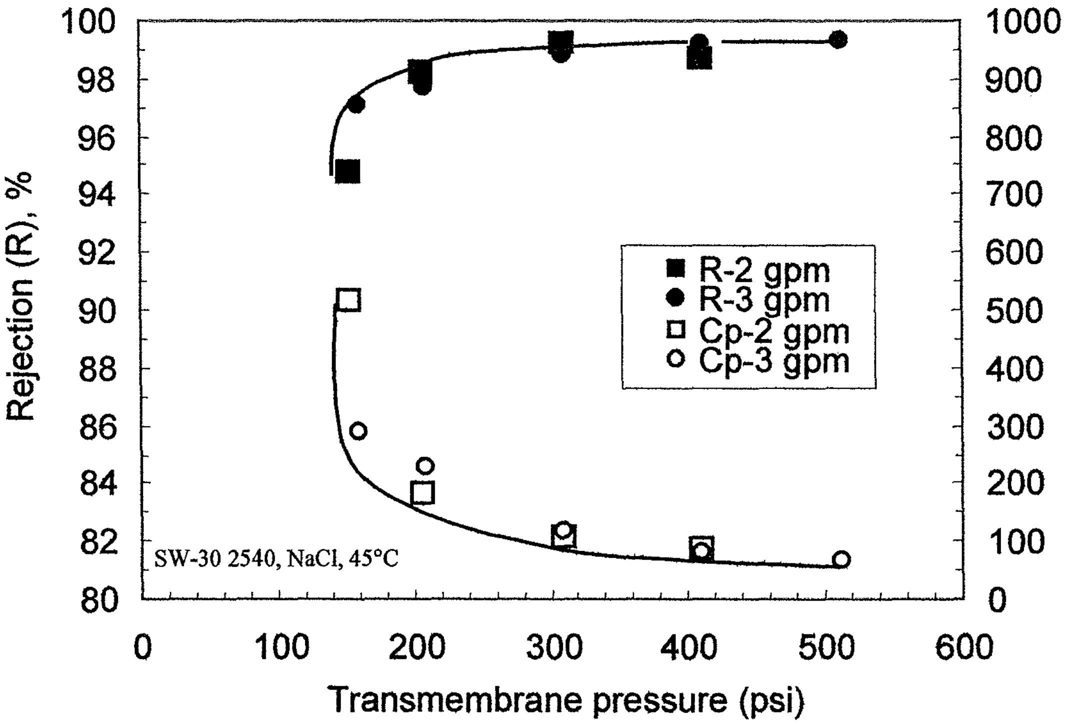


*Figure 19. Salt rejection and passage with 9718 ppm NaCl. Effect of pressure and cross-flow rate. Same experiment as Figure 18.*

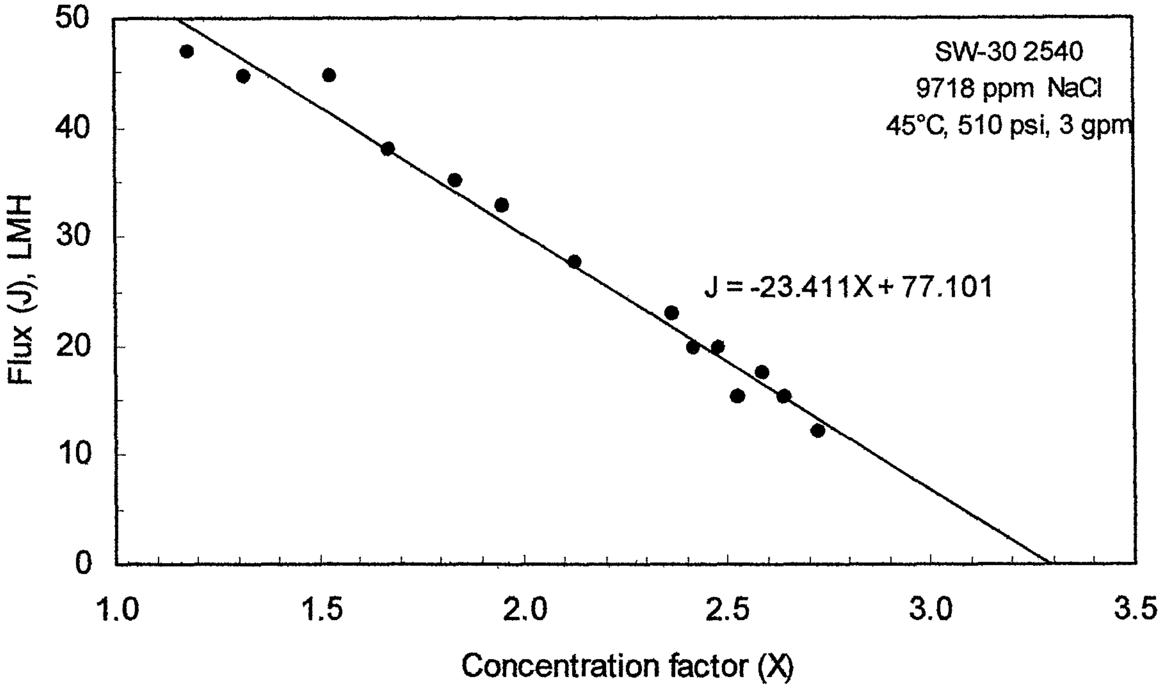


*Figure 20. Effect of concentration on flux. Feed was 9718 ppm NaCl solution. Temp= 113°F (45°C), cross flow = 3 gpm, TMP = 510 psi*

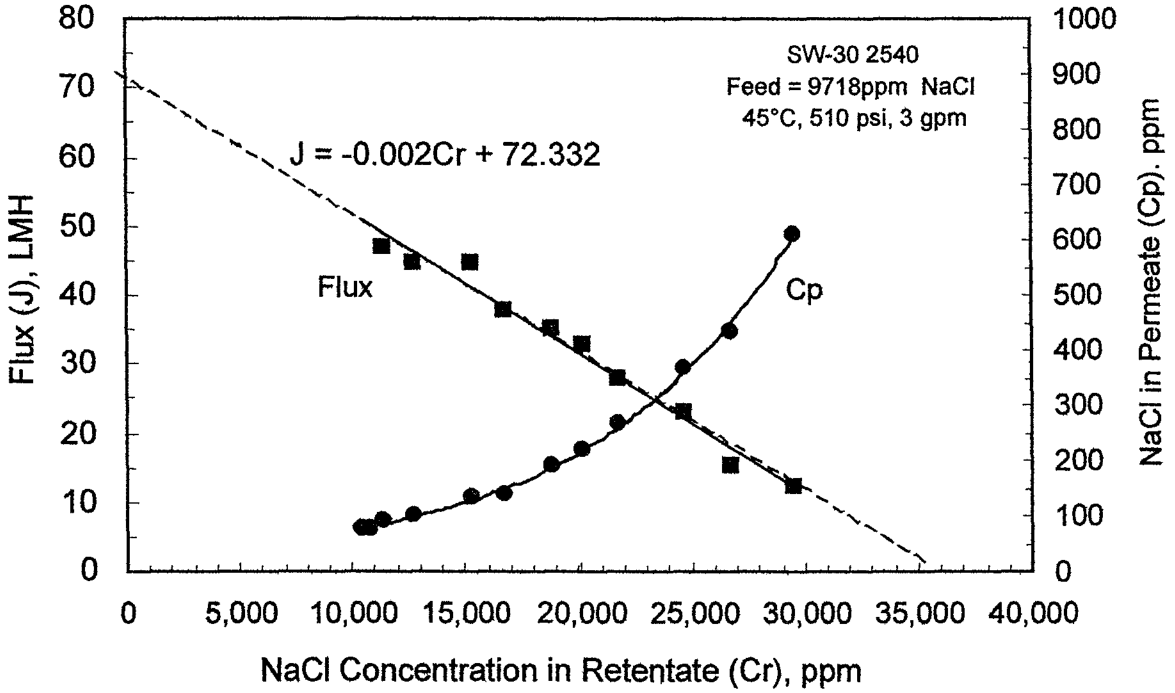


*Figure 21. Effect of concentration on flux and salt passage through membrane. Same experiment as Figure 20.*

linear manner with concentration factor (Figure 20) and with NaCl concentration in the retentate (Figure 21). According to the prediction equation (9), the maximum concentration of salt in the retentate occurs when flux is zero. Or,

$$J = A\,(P - R.\ \pi_r) = 0 \qquad (16)$$

For the run shown in Figures 20 and 21, P = 510 psi and R = 99.3%. Thus equation (16) becomes

$$\pi_r = 510/0.993 = 513.6 \text{ psi} = 35.42 \text{ atm} \qquad (17)$$

From equation (7), the salt concentration at zero flux under these operating conditions ($C_{max}$) is

$$C_{max} = 35.42\ (58.5)/2(0.08206)(318.15) = 39.6 \text{ g/L} = 39{,}600 \text{ ppm}$$

The corresponding $X_{max} = 39600/9718 = 4.07$

The equation for the line in Figure 20 predicts $X_{max} = 77.101/23.411 = 3.29$ at zero flux and $C_{max} = 72.332/.002 = 36166$ ppm, a difference of 8%. The most likely explanation for this discrepancy is errors when using equation (7) which is only valid for dilute solutions. Osmotic pressure is probably higher than that predicted with equation (7).

### *3.3.3. Dextrose solutions*

Dextrose was used a model for organic compounds. The behavior of the SW30 membrane with 0.43% dextrose is shown in Figures 22-25. The osmotic pressure of this feed solution is 9.04 psi and the rejection of dextrose is 100% (Figure 23), and thus the prediction model becomes

$$J = 0.1543\ (P - 9.04) \qquad (18)$$

Equation (19) does a very good job of predicting flux (Figure 22). This feed was then concentrated 9.4X resulting in a final dextrose concentration of 4.25% (Figures 24 and 25).

Figures 26 and 27 show data with a feed concentration of 4.5% dextrose. The data also show how sensitive flux is to temperature. The osmotic pressure of this feed solution is 94.6 psi and the rejection of sugar under these conditions is 100%. Thus the prediction flux equation is

$$J = 0.1543\ (P - 94.6) \qquad (19)$$

Figure 26 shows this model also predicts flux well. Figure 27 shows a concentration run with the 4.5% dextrose feed. Dextrose in the permeate averaged 0.04-0.37%, resulting in rejections of 98.3-99.6%. The data extrapolates to zero flux at X = 4.1. Applying equation (16) at a pressure of 510 psi, R = 99%, $C_{max}$ should be 24%. However, $C_{max}$ from Figure 27 is 4.1 x 4.5 = 18.45%. The most likely explanation for this discrepancy is inaccuracy in estimating osmotic pressure with equation (7) which is only valid for dilute solutions. Osmotic pressure is probably higher than that predicted with equation (7).

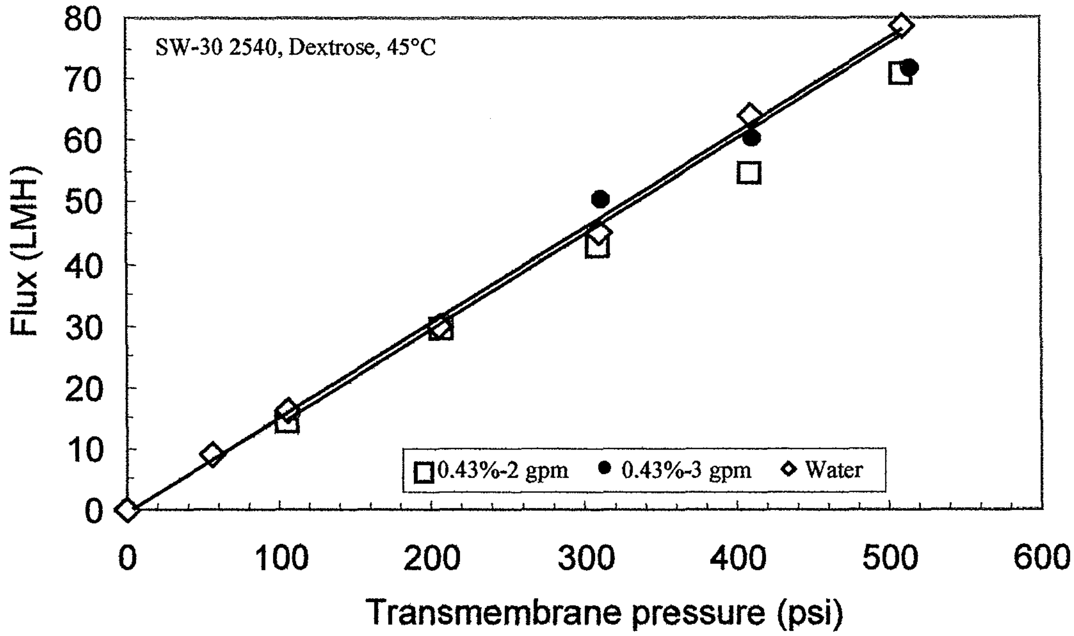


*Figure 22. Flux of 0.43% dextrose solution with SW-30 module at cross-flow rates of 2 gpm and 3 gpm.Lines are model predictions (equation 12 for water and equation 18 for dextrose), points are experimental data*

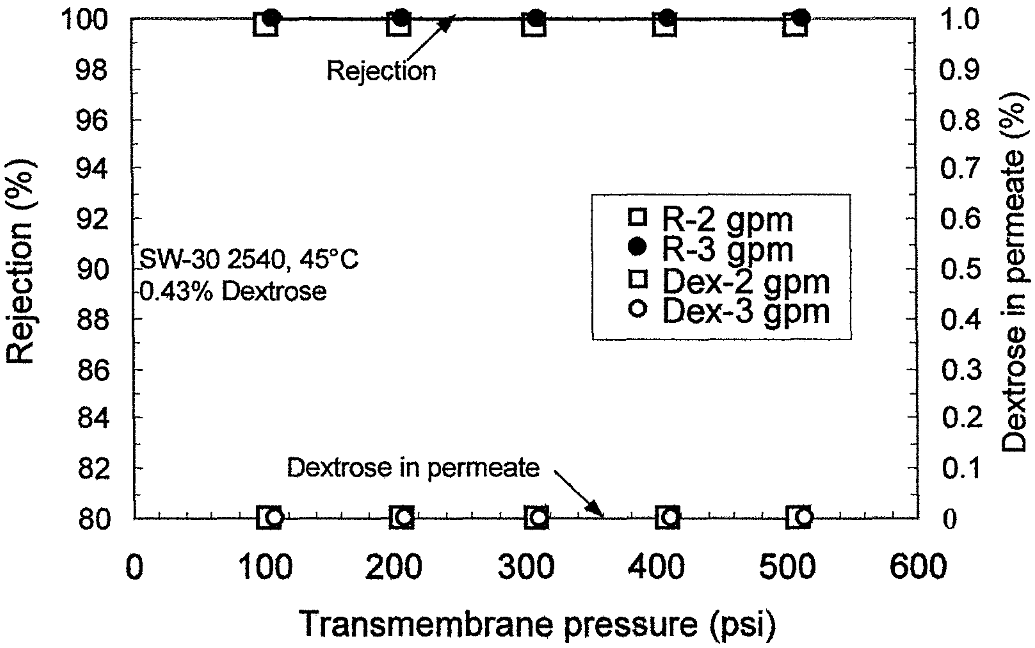


*Figure 23. Dextrose rejection and passage. Same experiment as Figure 22.*

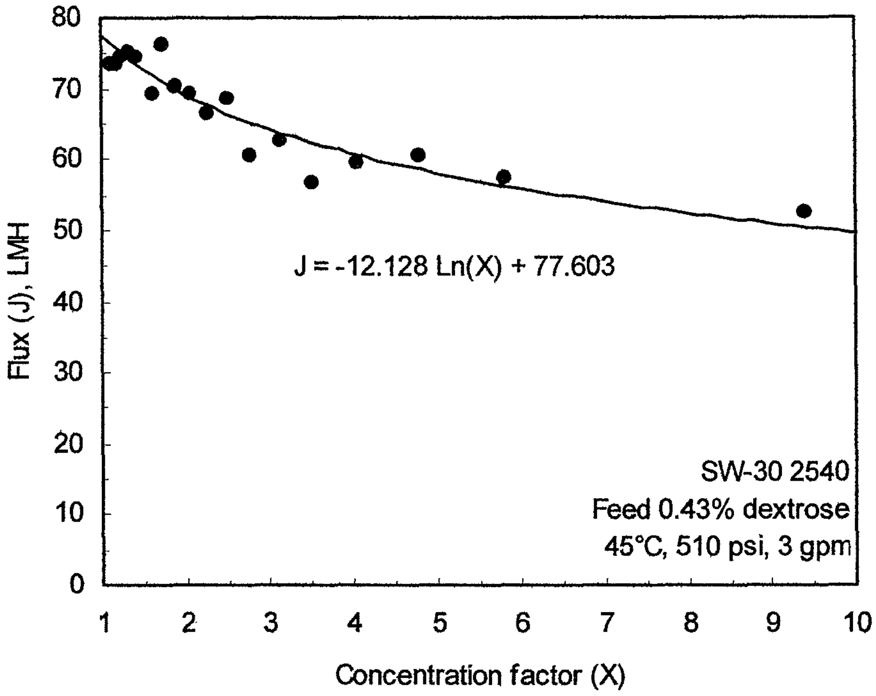


*Figure 24. Effect of concentration on flux. Feed was 0.43% dextrose in DI water. Temp= 113°F (45°C), cross flow = 3 gpm, TMP = 510 psi*

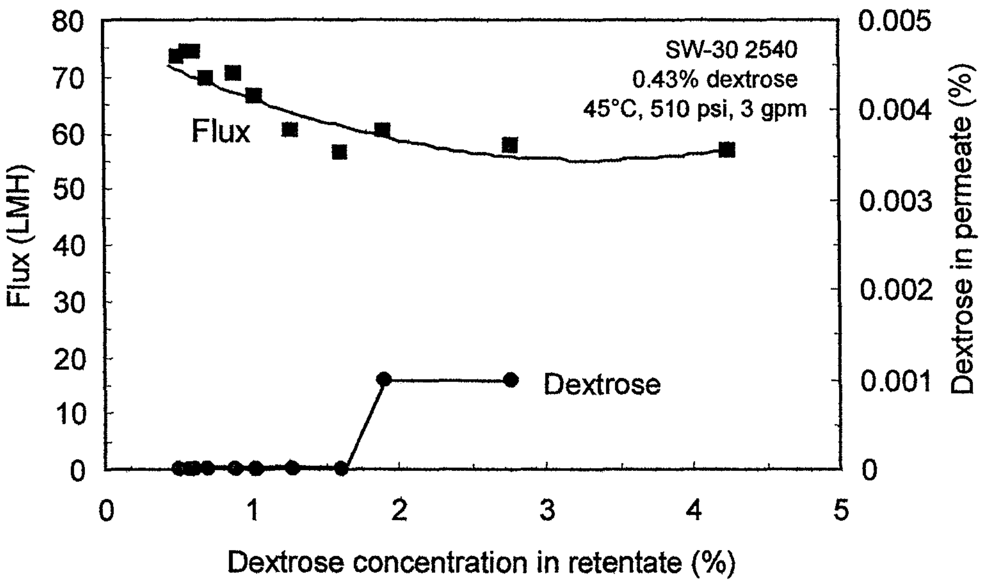


*Figure 25. Effect of concentration on flux and dextrose passage through membrane. Same experiment as Figure 24.*

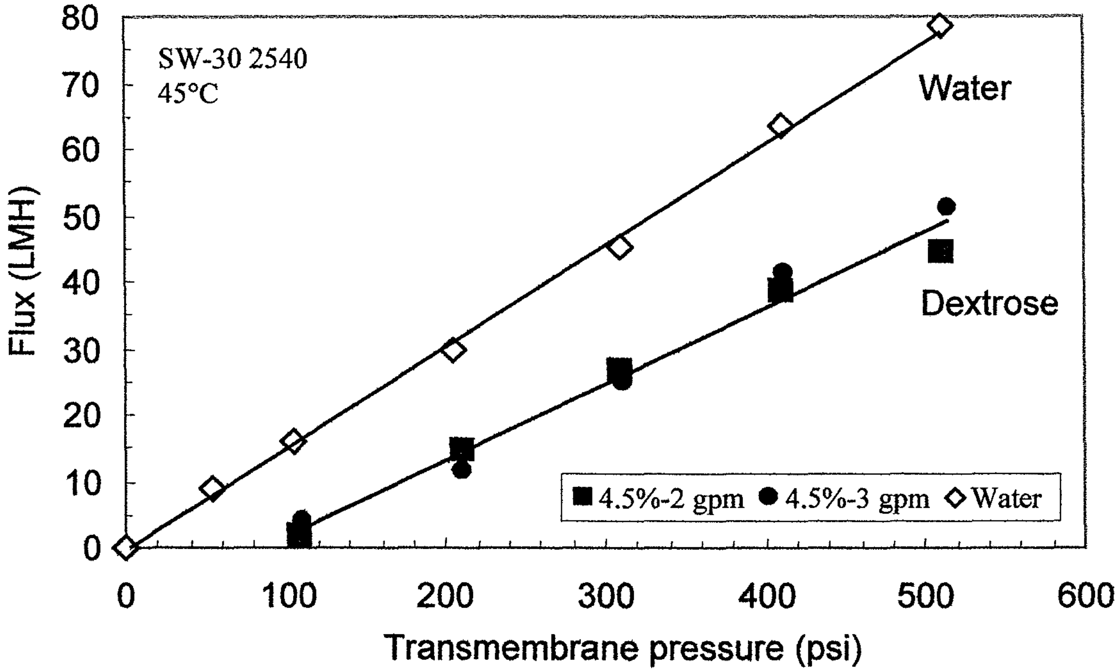


*Figure 26. Flux of 4.5% dextrose solution with SW-30 module at cross-flow rates of 2 gpm and 3 gpm.Lines are model predictions (equation 12 for water and equation 19 for dextrose), points are experimental data*

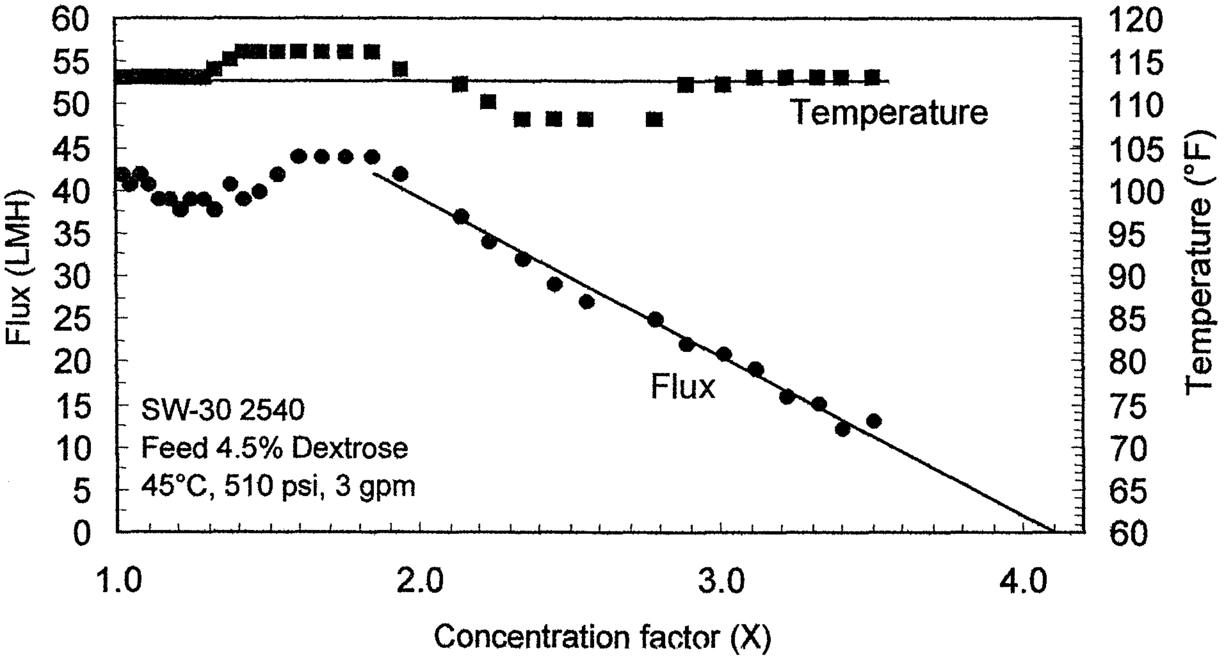


*Figure 27. Effect of concentration on flux. Feed was 4.5% dextrose in DI water. Temp= 113°F (45°C), cross flow = 3 gpm, TMP = 510 psi*

### 3.4. Model electroplating waste solutions

Ion analysis of the wastewater from the plant showed that sodium and sulfate were the predominant ions, while barium and calcium were potential colloidal foulants (Tables 4 and 5). A study was conducted with model solutions of sodium sulfate (section 4.4.1) and with added calcium and barium chlorides (4.4.2) to determine the factors affecting the performance (flux, fouling and rejection) during reverse osmosis. The main variables were transmembrane pressure, pH, temperature and concentration. The stability of the membrane was checked by repeating experiments with 1000 ppm NaCl solutions and DI water at different times.

#### *3.4.1. Sodium sulfate solutions*

Figures 28-31 show data at 45°C (the maximum recommended temperature for this membrane) and at the "normal" (unadjusted) pH of the sodium sulfate solutions (~pH 7) Figure 28 shows membrane fouling by sodium sulfate solutions of ~1% and ~2% concentrations. Higher solids results in lower flux. Fouling is noticeable in the initial period after start-up and flux levels off after 2-4 hours. Data shows good reproducibility between runs. Figure 29 shows the effect of solids concentration and cross-flow rate on flux. Higher cross-flows and lower solids result in higher flux, as expected. Figure 30 shows the effect of sodium sulfate concentration on flux and Figure 31 shows the relationship between concentration factor (X) and total solids of the retentate and the permeate. The membrane shows a small leakage of the solids above 3X, equivalent to about 7% solids in the retentate.

Figures 32-43 show data at 30°C, which is probably a more realistic temperature for commercial operation. Figures 32 and 33 show concentration data for a solution that was initially at ~2% sodium sulfate and Figures 34 and 35 show the data for a 2.5% sodium sulfate feed solution. The appearance of solids in the permeate is again apparent above 2.5X in Figure 35.

Figures 36 and 37 summarize the data from Figures 28-35 in a single plot. The benefit of operating at higher temperature is clearly shown in Figure 36. The consolidated data in Figure 37 seems to indicate that high concentrations (>7% TS) cause a noticeable leakage of solids into the permeate.

Figures 38-43 show two experiments where the pH of the feed solution (1% sodium sulfate) was adjusted to pH 9 by adding 0.05% of sodium hydroxide as needed. Since the solution was unbuffered, it was difficult to keep the pH constant during the run and the best we could do was ± 0.2 pH units. The temperature was maintained at 30°C but occasionally reached as high as 35°C due to inadequate heat exchange capacity (in subsequent work, an in-line shell-and-tube heat exchanger was added in the retentate return line as shown in Figure 2). However, the data is still similar to previous experiments.

Figure 40 shows the time course of the second experiment in which we studied the simultaneous effect of concentration factor and fouling on flux. For the first 3 hours, the system was run with the feed solution in the fouling mode (permeate and retentate recycled to the feed tank to keep solids level constant). It was then operated in the concentration mode to remove about half the initial volume as permeate. It was then operated in the fouling mode for about 1.5 hours before removing more permeate to reach 4X and then operated in the fouling/recycle mode

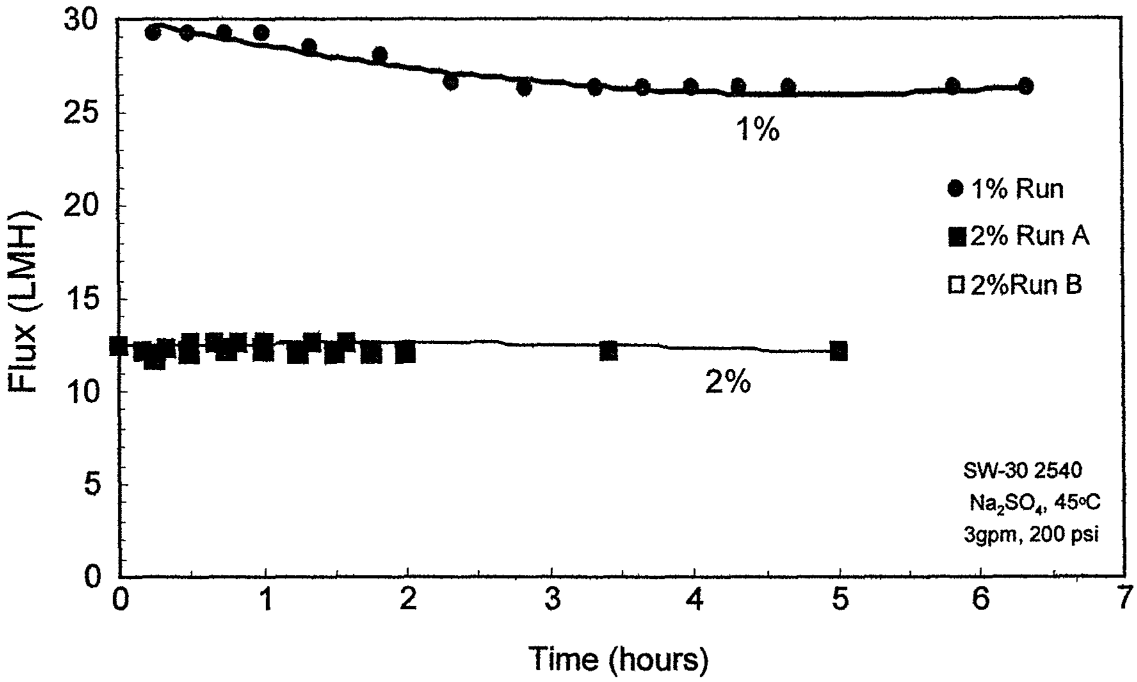


*Figure 28. Fouling of 1% and 2% $Na_2SO_4$ at 45°C. The 2% run was done twice.*

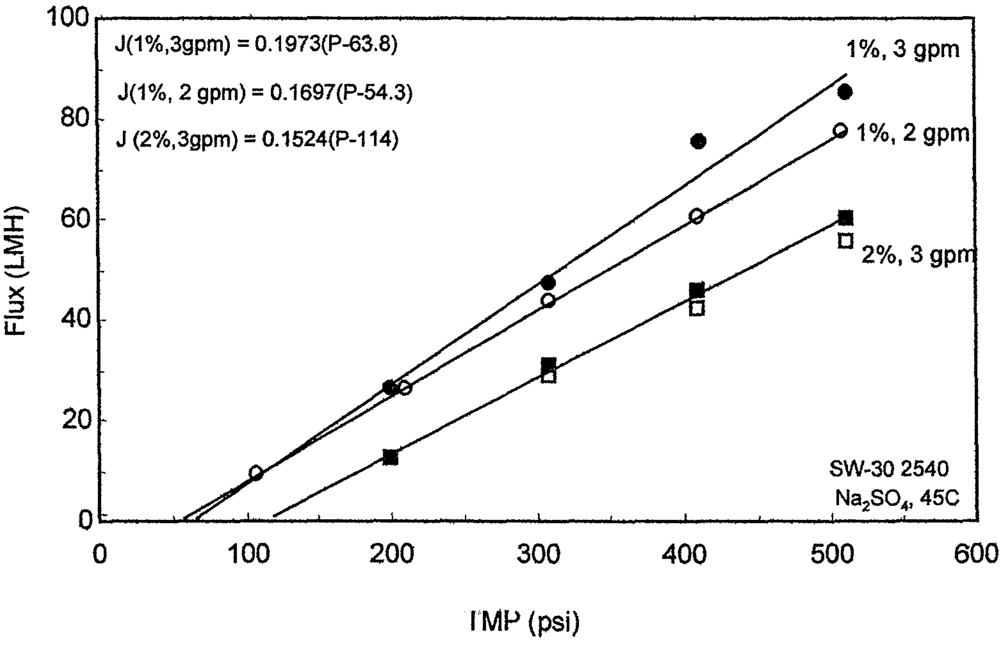


*Figure 29. Effect of transmembrane pressure (TMP) on flux for 1% and 2% $Na_2SO_4$ at 45°C.*

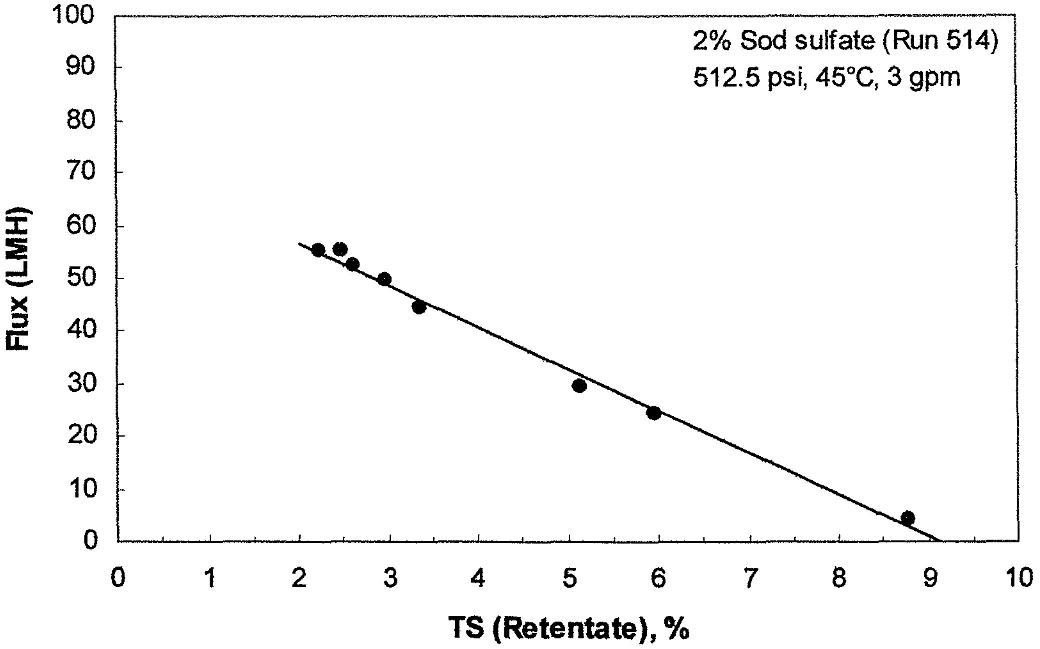


*Figure 30. Concentration of 2% $Na_2SO_4$ at 45°C.*

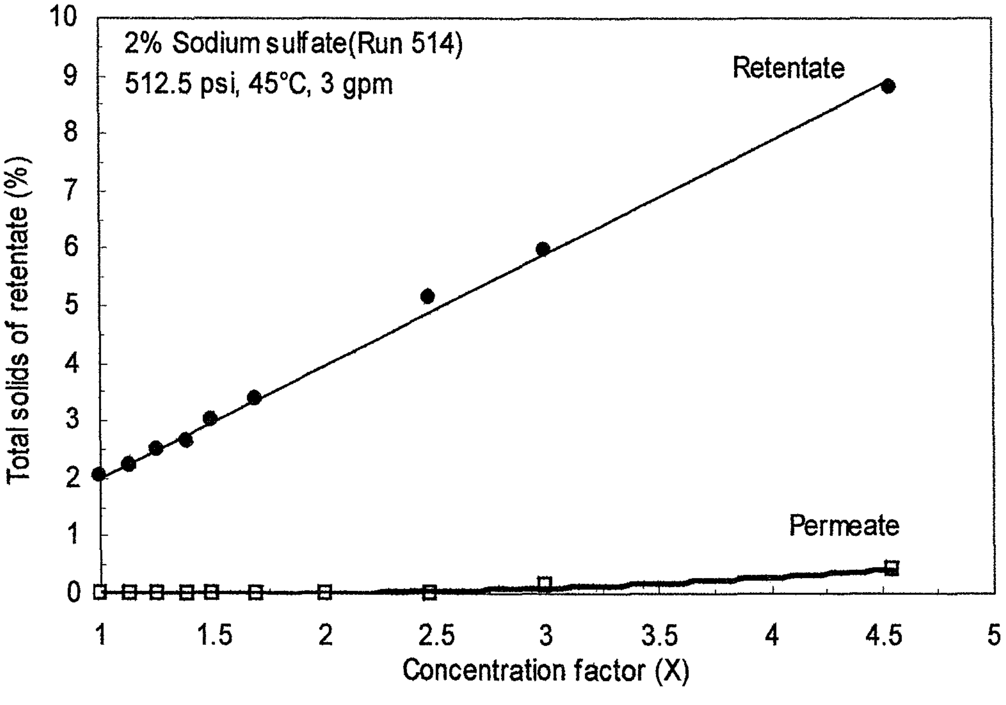


*Figure 31. Concentration of 2% $Na_2SO_4$ at 45°C. Same run as Figure 30*

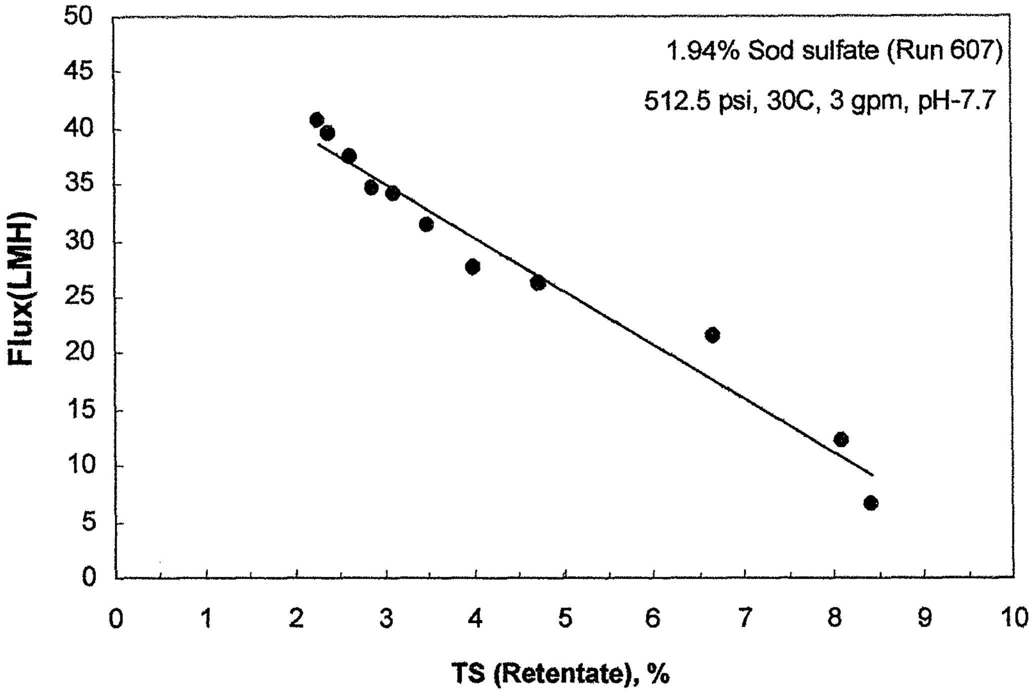


*Figure 32. Concentration of 1.94% $Na_2SO_4$ at 30°C.*

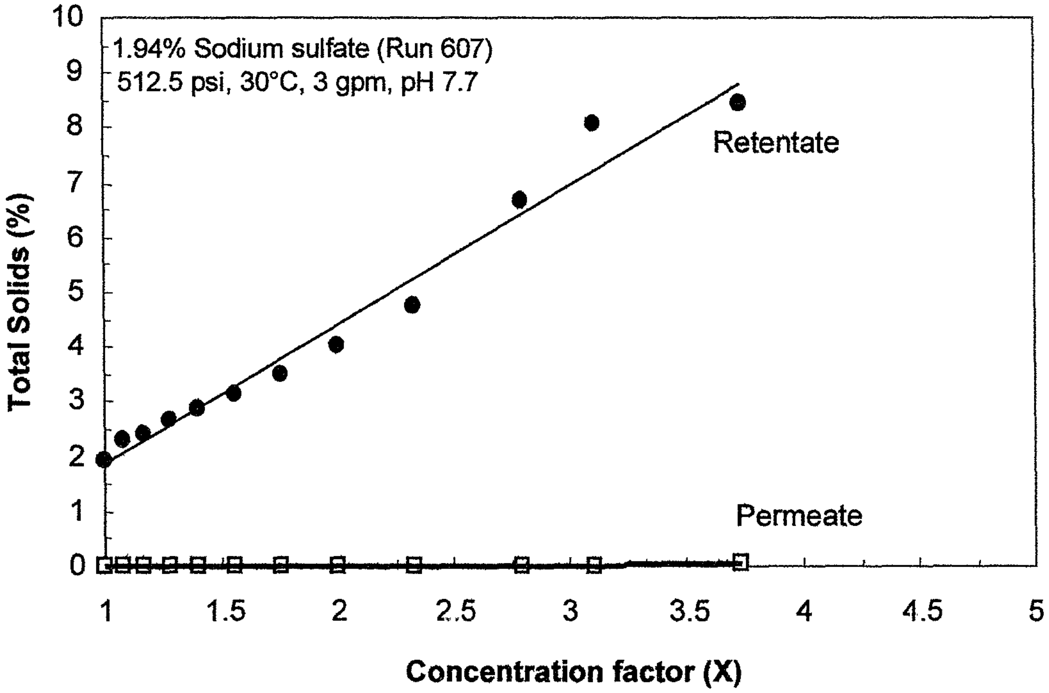


*Figure 33. Concentration of $Na_2SO_4$ solution at 30°C. Same experiment as Figure 32.*

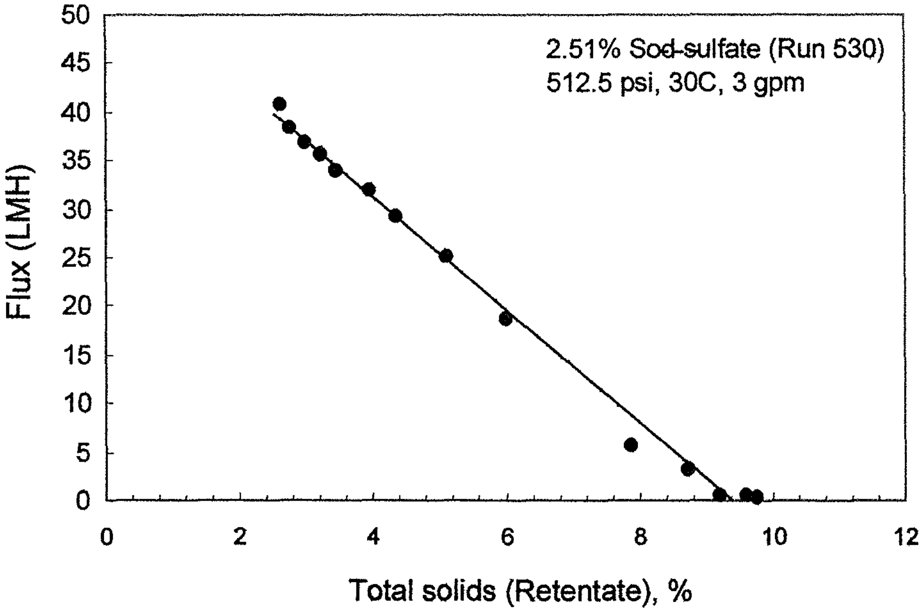


*Figure 34. Concentration of 2.5% $Na_2SO_4$ at 30°C. Retentate samples were taken from retentate outlet valve and not from the feed tank.*

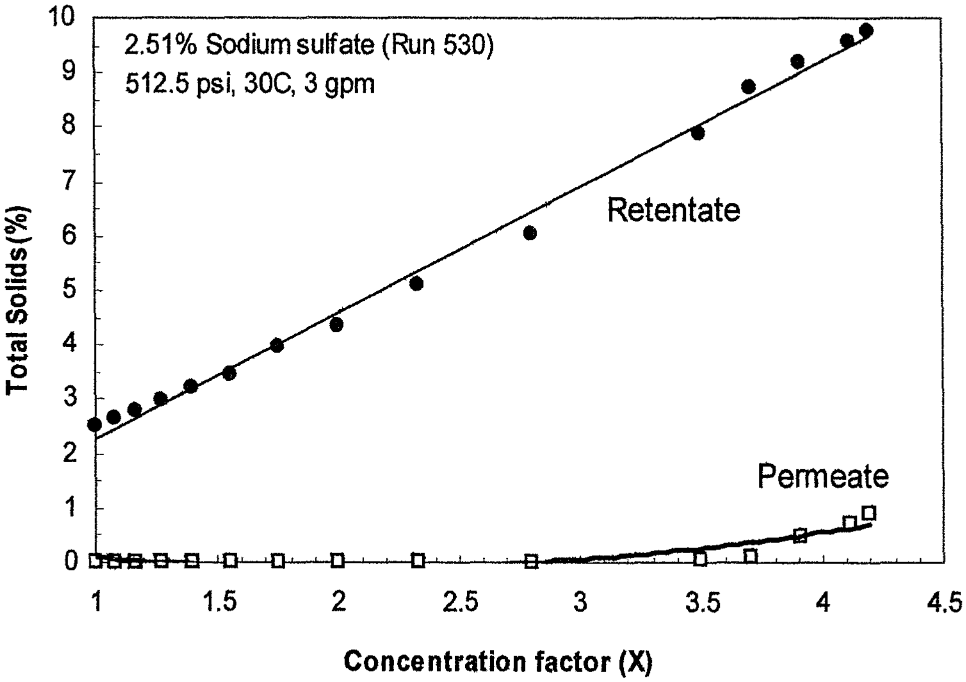


*Figure 35. Concentration of 2.5% $Na_2SO_4$ at 30°C. Same experiment as Figure 34.*

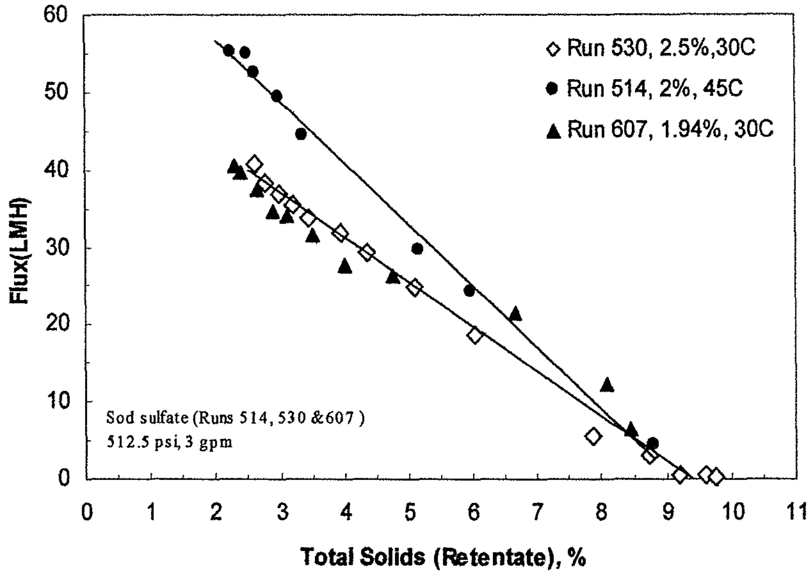


*Figure 36. Flux of 2% and 2.5% $Na_2SO_4$ solutions as a function of retentate total solids*

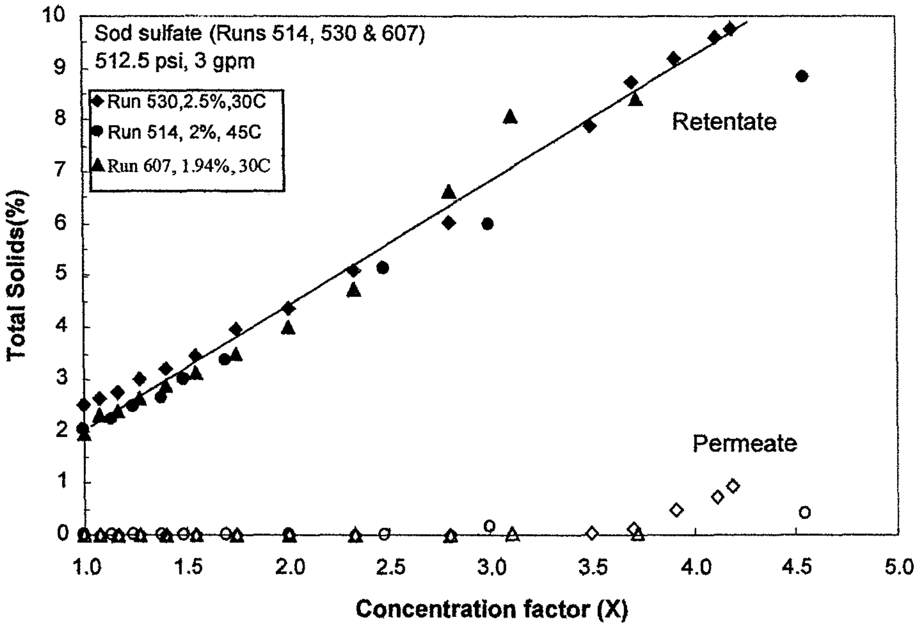


*Figure 37. Concentration of 2% and 2.5% $Na_2SO_4$ solutions. Same experiment as Figure 36.*

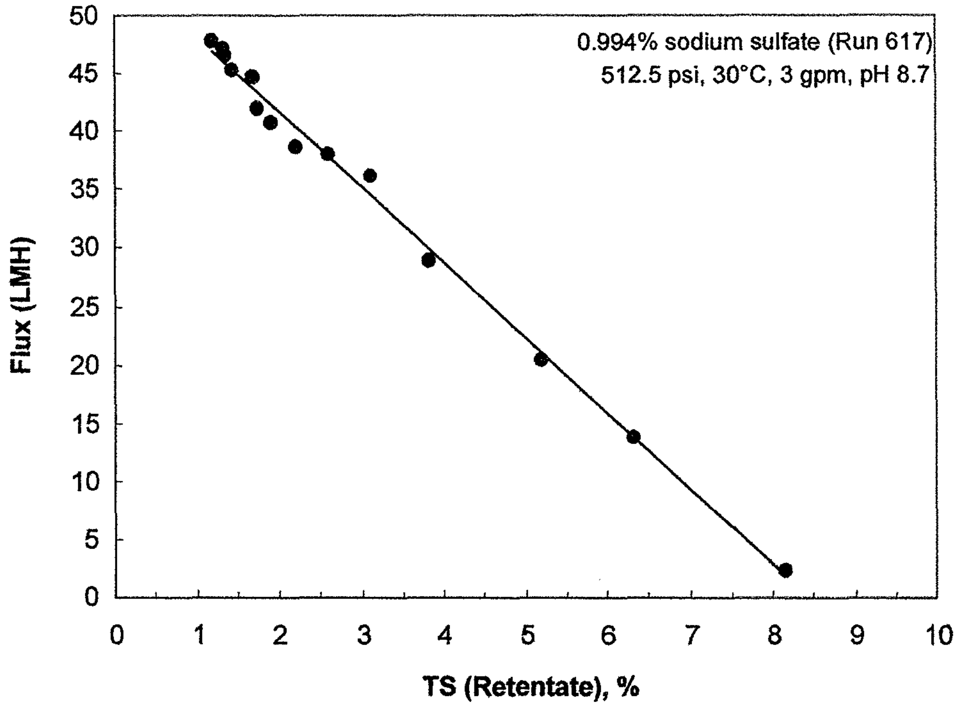


*Figure 38. Concentration of 1% $Na_2SO_4$ at 30°C. Same experiment as Figure 39.*

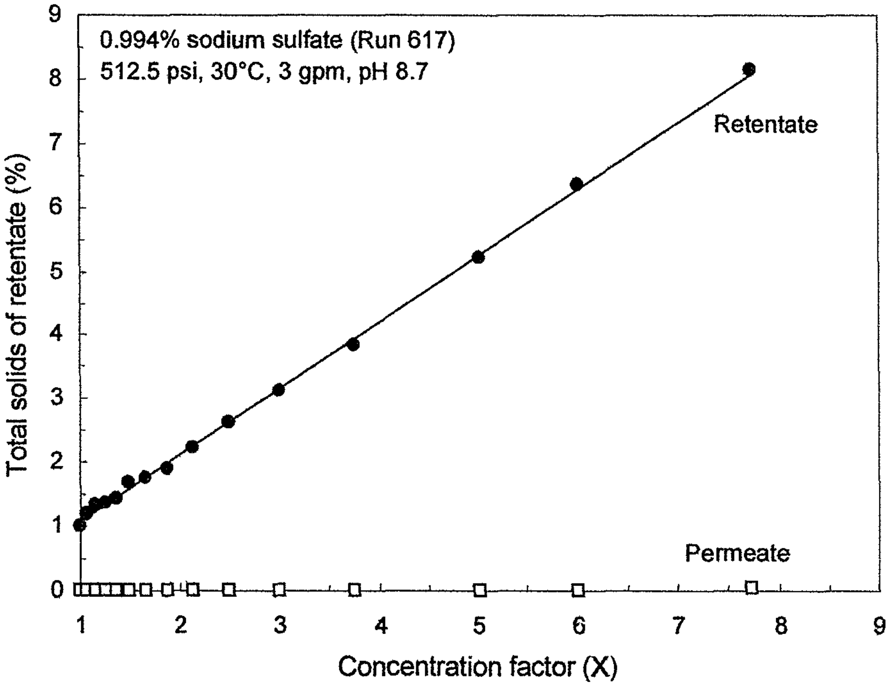


*Figure 39. Concentration of 1% $Na_2SO_4$ at 30° C. Same experiment as Figure 38.*

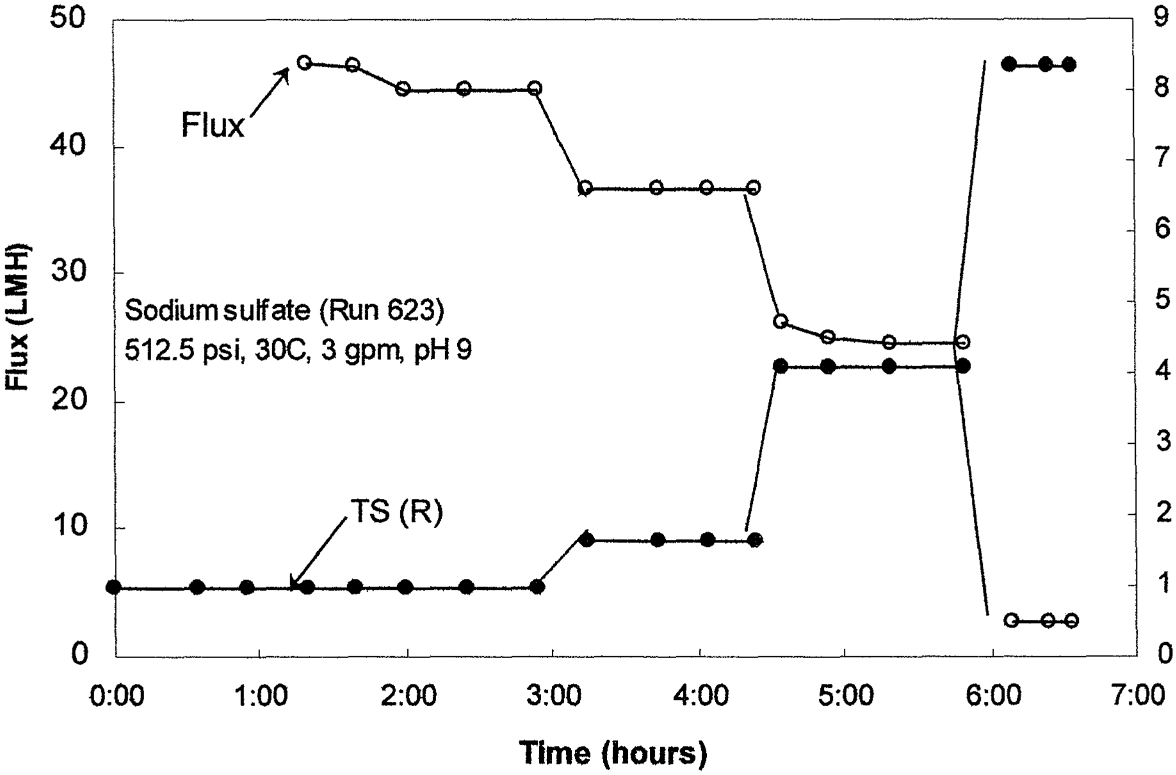


*Figure 40. Effect of concentration and fouling on flux. Feed was 1% $Na_2SO_4$ at 30°C. Additional data for this run shown in Figures 41-43.*

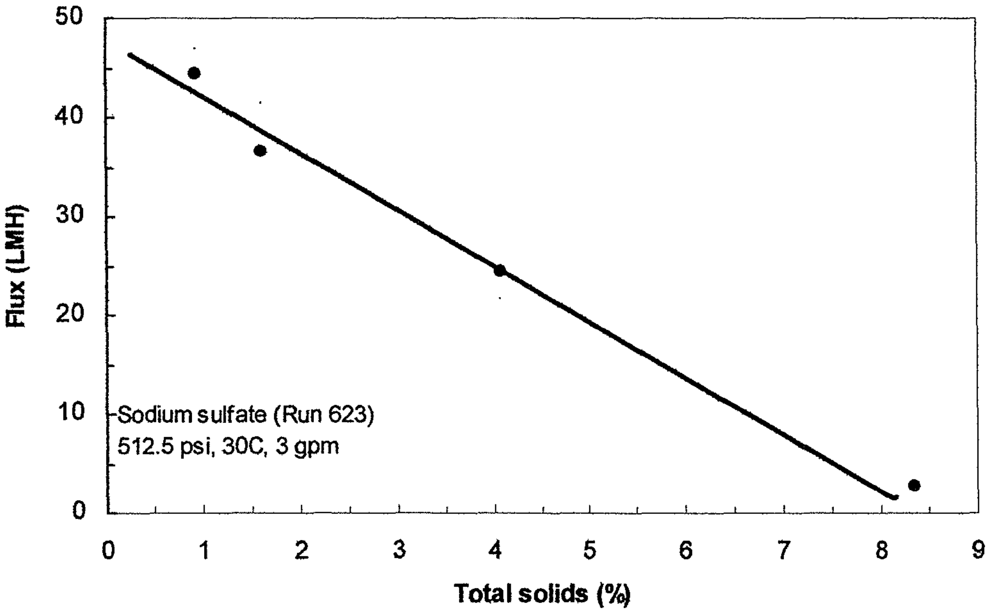


*Figure 41. Concentration of 1% $Na_2SO_4$ at 30°C. Same experiment as Figure 40.*

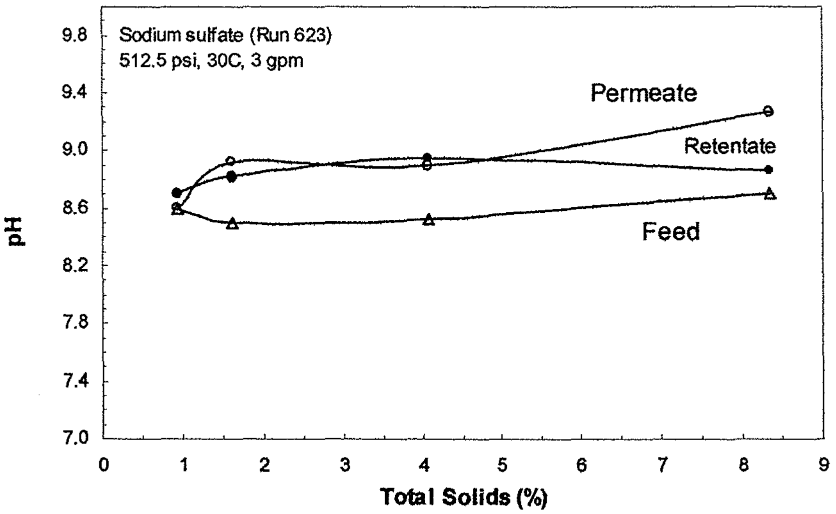


*Figure 42. Relationship between total solids and pH of feed (open triangles), pH of retentate (closed circles) and pH of permeate (open circles). Same experiment as Figures 40, 41 and 43.*

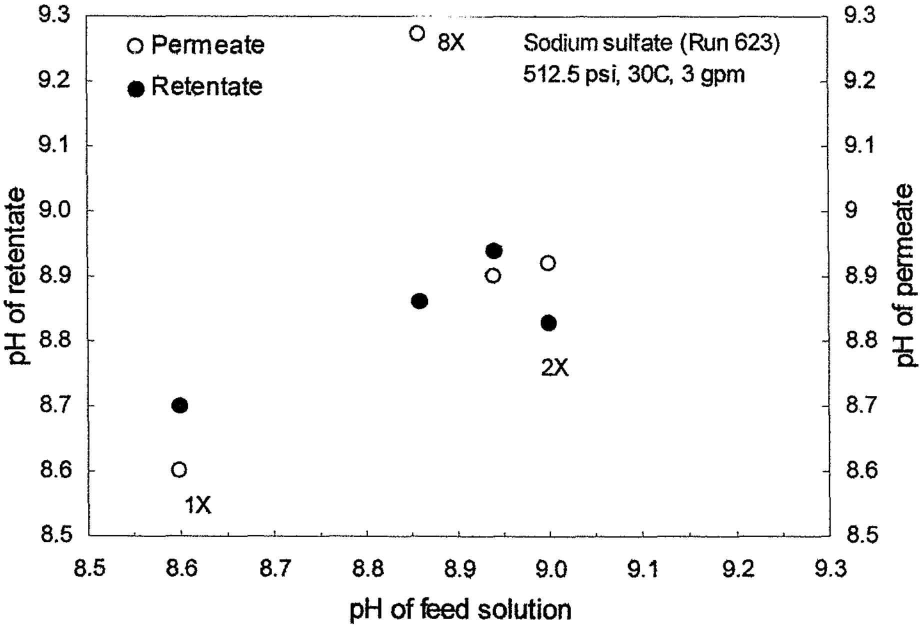


*Figure 43. Relationship between pH of feed, pH of retentate and pH of permeate. Same experiment as Figures 40, 41 and 42.*

again, and so on. There was some initial flux decline during the fouling mode at the higher concentrations (Figure 40), although it is difficult to ascertain whether it is due to specific solute-membrane interactions or due to slight variations in operating conditions and physicochemical properties.

The corresponding steady-state flux is plotted against total solids of the retentate in Figure 41. The pH of the feed, retentate and permeate were monitored during the experiment and are shown in Figure 42. The pH of the permeate was slightly higher than the feed. There appears to be a rough correlation between pH of the feed and pH of the permeate (Figure 43). This pH phenomenon has been observed in subsequent experiments with the real wastewater as will be seen later.

At this point, the module had about 3 months of use. The water flux was tested again and the following flux models were obtained:

At 75°F (24°C) $J = 0.085\ P$ (20)
At 86°F (30°C) $J = 0.106\ P$ (21)
At 97°F (35°C) $J = 0.121\ P$ (22)
At 113°F (45°C) $J = 0.167\ P$ (23)

Water flux showed a slight increase of 8% at 45°C compared to the new membrane. Similarly, salt solution flux increased by 10-20% (data not shown here). This suggests a slight dilation of the pores of the membrane over time but rejection of NaCl was not affected.

### *3.4.2. Effect of added barium and calcium*

The next study was to evaluate the effect of added suspended solids such as barium and calcium salts on the performance (flux, fouling and rejection) during reverse osmosis of sodium sulfate solutions. The barium chloride reacts with the sodium sulfate to form barium sulfate which is a colloidal suspension. Three levels of barium chloride were used to represent the possible range of barium salts expected in the wastewater: 0.1, 5 and 50 mg/L. In all cases, the pH of the feed solution was adjusted to pH 8.0 with dilute NaOH or HCl as needed.

Figures 44-51 show experiments conducted with barium chloride added at various concentrations. Each concentration was a separate run with a fresh sodium sulfate solution with a clean membrane. Fouling rates were minimal with added barium as shown in Figure 44. Only at the highest barium concentration of 50 mg/L did the flux decrease slightly. This is also seen in Figure 45, which is the pressure-flux relationship for this system. Extrapolating to zero flux indicates the osmotic pressure of the feed solution with low levels of added barium was about 60 psi. Figure 46 shows the effect of concentrating the feed solutions on flux. At a pressure of 512.5 psi, the maximum solids concentration of the low-barium feed when flux becomes zero is 512.5/60 = 8.4%. This is close to what was observed in Figure 46.

Figure 47 shows that higher solids in the retentate results in higher solids in the permeate, regardless of barium levels. However, the rejection of solids is still >99.7% even at the highest

Figures 48-51 show the pH of the feed (which was maintained at pH 8.0) and pH of the retentate and permeate during the concentration runs. The pH of the permeate was always higher

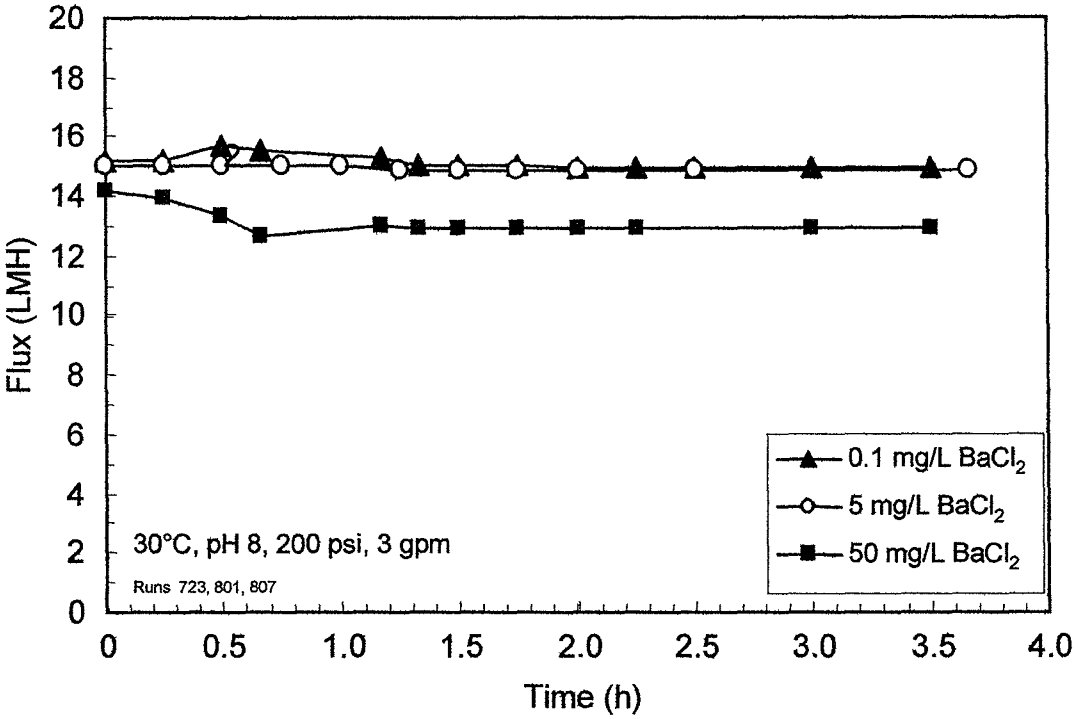


*Figure 44. Fouling of 1% sodium sulfate with added barium chloride at 200 psi, 30°C pH 8 and cross-flow rate of 3 gpm.*

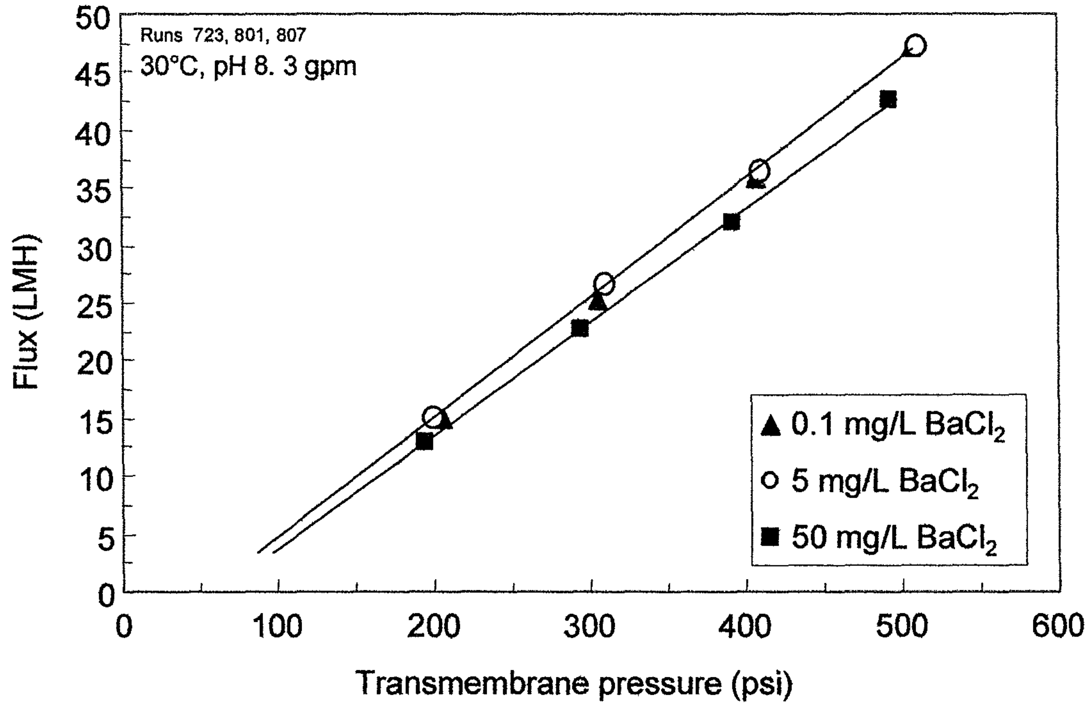


*Figure 45. Flux-pressure relationships for RO of 1% sodium sulfate with added barium chloride at 30°C, pH 8 and cross-flow rate of 3 gpm*

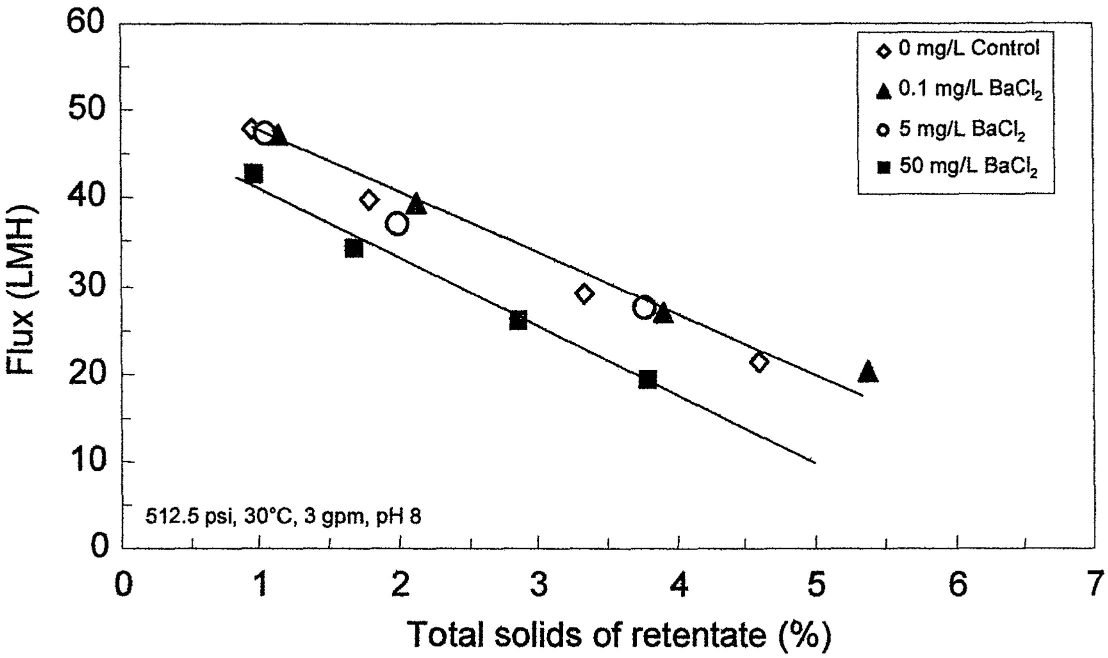


*Figure 46. Concentration of 1% sodium sulfate with added barium chloride by RO at 512.5 psi, 30°C, pH 8 and cross-flow rate of 3 gpm*

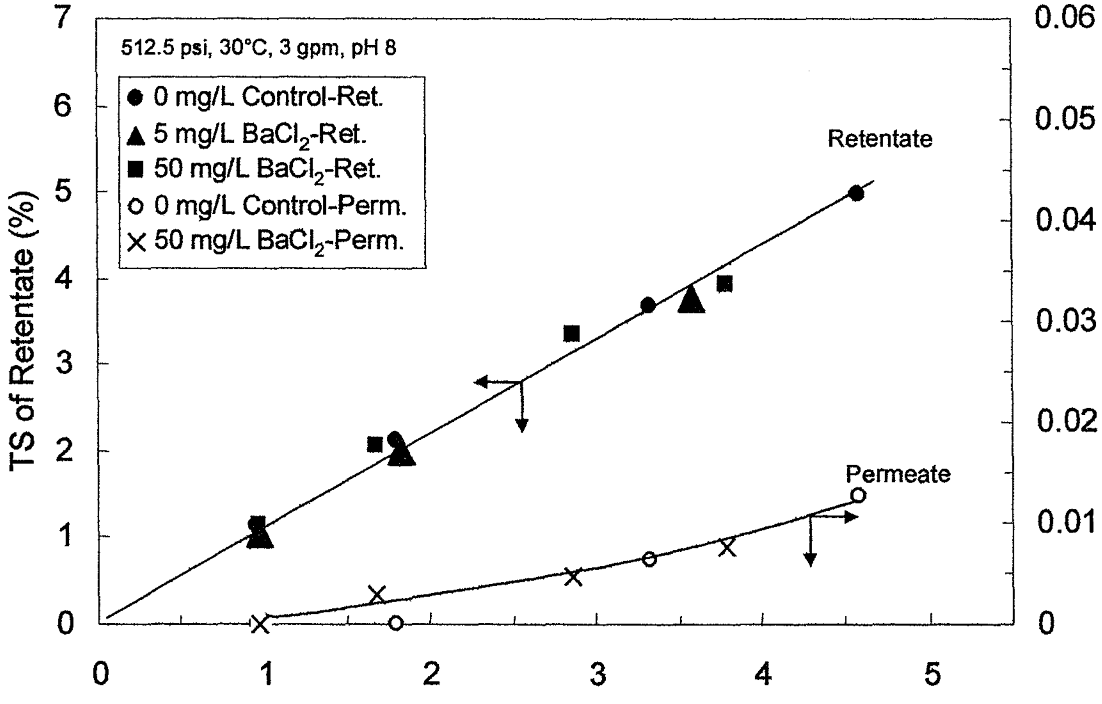


*Figure 47. Relationship between total solids in the feed and total solids in the retentate and permeate. Same experiment as Figure 46.*

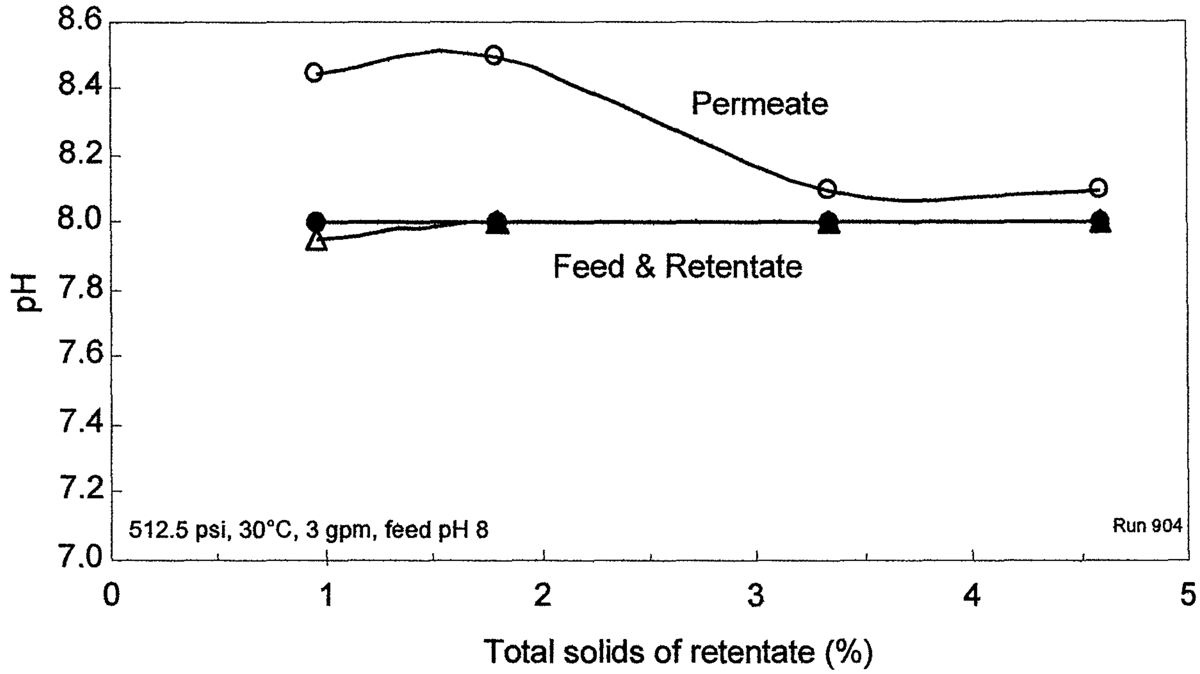


*Figure 48. pH profiles during concentration of 1% sodium sulfate by RO. pH of feed was maintained at pH 8.0. Same experiment shown as "0 mg/L Control" in Figures 46 and 47.*

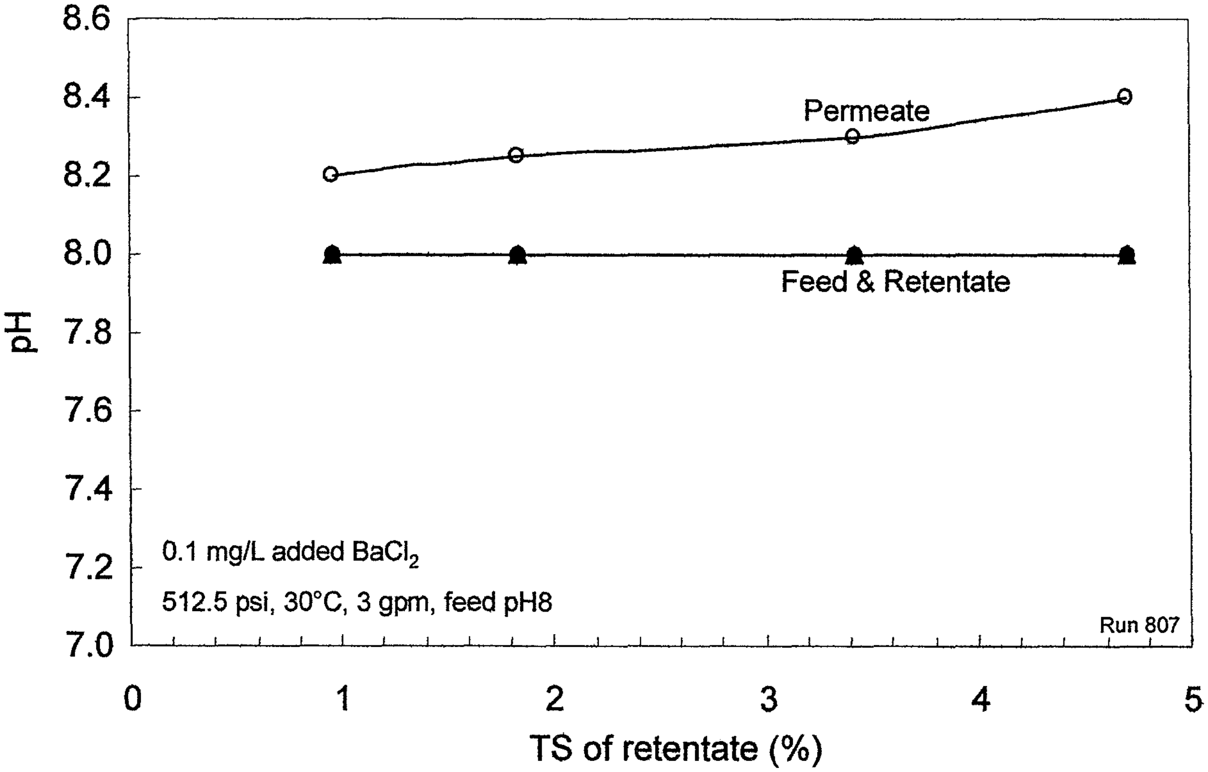


*Figure 49. pH profiles during concentration of 1% sodium sulfate by RO. pH of feed was maintained at pH 8.0. Same experiment shown as "0.1 mg/L added $BaCl_2$" in Figures 46 and 47.*

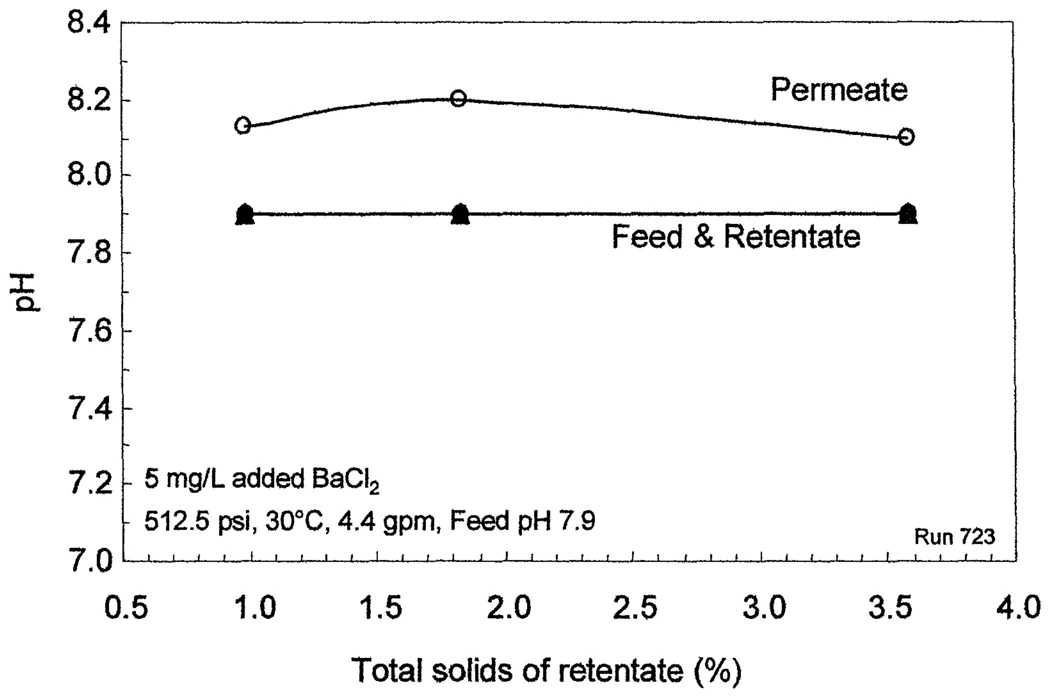


*Figure 50. pH profiles during concentration of 1% sodium sulfate by RO. pH of feed was maintained at pH 7.9. Same experiment shown as "5 mg/L added $BaCl_2$" in Figures 46 and 47.*

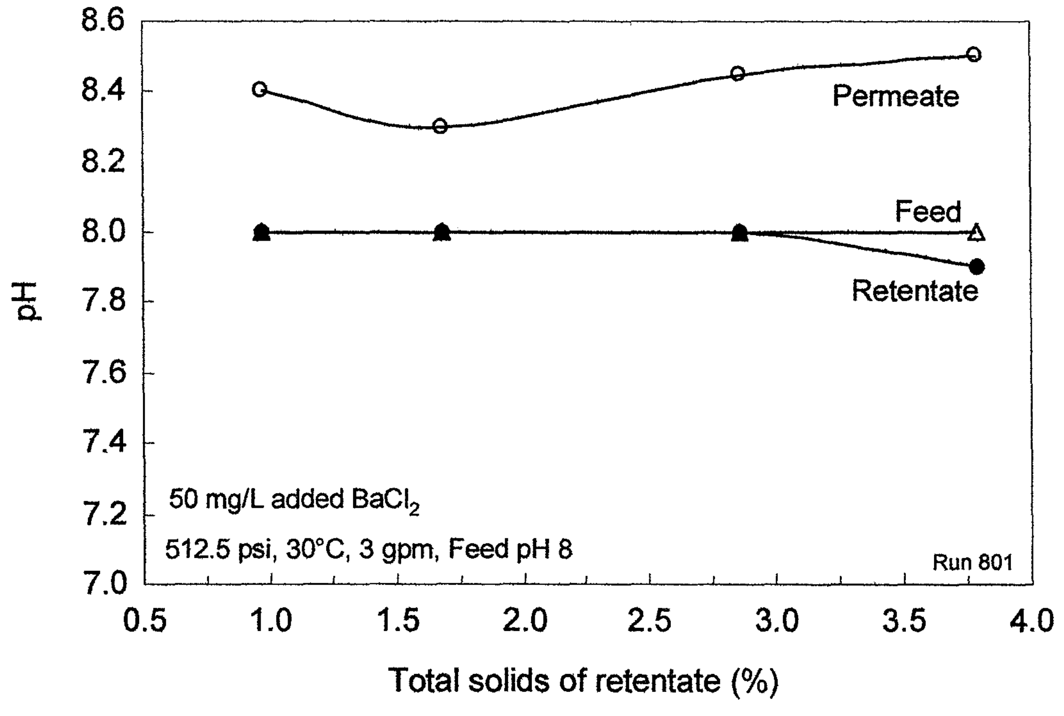


*Figure 51. pH profiles during concentration of 1% sodium sulfate by RO. pH of feed was maintained at pH 8.0. Same experiment shown as "50 mg/L added $BaCl_2$" in Figures 46 and 47.*

than the pH of the feed or retentate by 0.2-0.4 pH units.

Figures 52-56 show data with added calcium chloride at various concentrations. The resulting suspended solids would be calcium sulfate. Figure 52 shows minimal fouling with added calcium even at 55 mg/L calcium chloride. There is little effect on the flux-pressure relationship (Figure 53). Figure 54 shows the maximum concentration of solids possible at the applied pressure of 512.5 psi was about 7.5%. Figure 55 shows a significant loss of solids in the permeate above a feed concentration of 3% TS although the rejection of the solids is still >99.6%. Figure 56 shows the pH profile during the runs. Again the pH of the permeate was higher.

## 3.5. Guardian West electroplating waste streams: Pilot plant studies

The next task was to study the reverse osmosis of real samples from the Guardian West plant in Urbana. Fouling (short term and long term), performance (flux and rejection), effect of concentration of the feed and the effect of bacteria on flux were studied. Several sets of samples were obtained over a period of 6 months for testing in our pilot system in our laboratory. The tests and results are described below.

### *3.5.1. Phase I*

Table 9 lists the experiments conducted in this phase of the project and specifications of the two samples of the feed stream. The two samples were each used for two separate experiments. The samples differed in their total solids and pH which might be expected to have an effect on their reverse osmosis behavior.

Figure 57 shows short-term fouling data obtained in three experiments with the Guardian West feed. Also shown for comparison is the data obtained with a model solution of 1% sodium sulfate at pH 8. The model solution showed the highest flux, even though it had the highest total solids. Run 1124A showed similar flux but it had much lower solids (0.6%) and a higher pH. The other feeds had a similar pH (8.3-8.4) but total solids was higher (0.86%), which resulted in 15-20% lower flux. [The membrane was cleaned between each experiment].

*Table 9. Phase I trials with electroplating waste from Guardian West plant*

| | Run Number | | | |
|---|---|---|---|---|
| | 1002 | 1021 | 1124A | 1124B |
| Bacteria added | No | No | No | Yes |
| Total solids of feed (%) | 0.86 | 0.86 | 0.60 | 0.60 |
| pH of feed | 8.4 | 8.3 | 9.3 | 9.3 |
| Fouling Study | Short term<br>(3 hours) | Short, Long term<br>(3 hours, 1 week) | Short term<br>(3 hours) | Long term<br>(1 week) |
| Performance Study | Yes | No | Yes | No |
| Concentration Study | Yes | No | No | No |

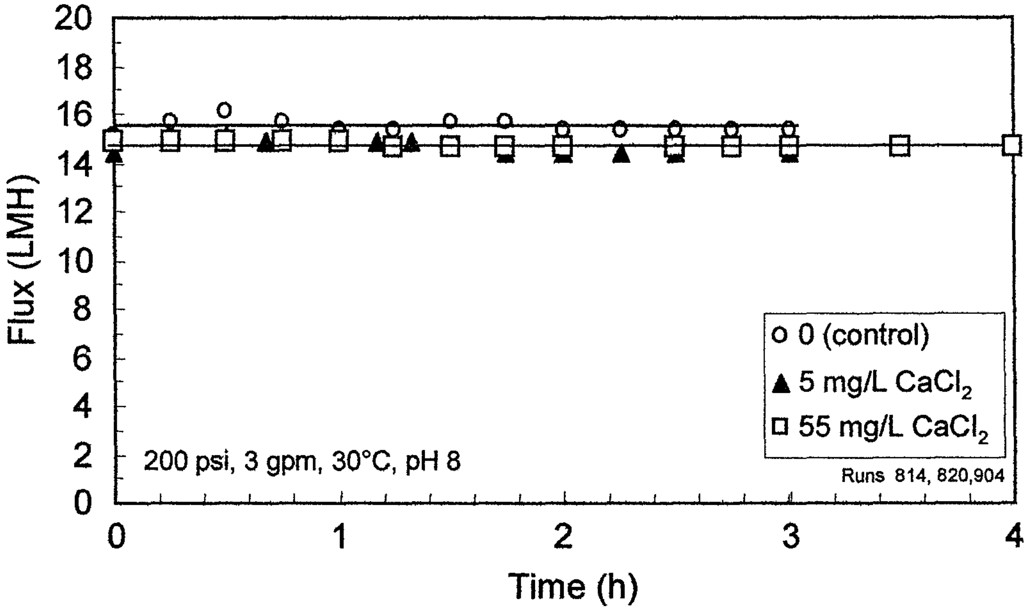


*Figure 52. Fouling of 1% sodium sulfate with added calcium chloride at 200 psi, 30°C pH 8 and cross-flow rate of 3 gpm.*

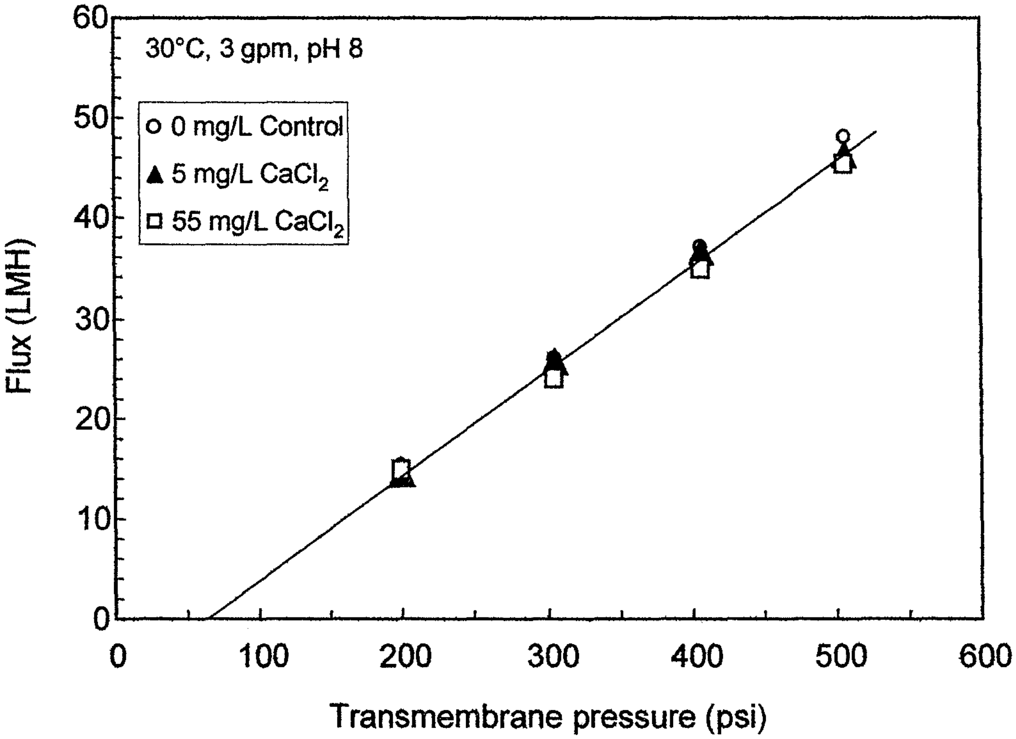


*Figure 53. Flux-pressure relationships for RO of 1% sodium sulfate with added calcium chloride at 30°C, pH 8 and cross-flow rate of 3 gpm*

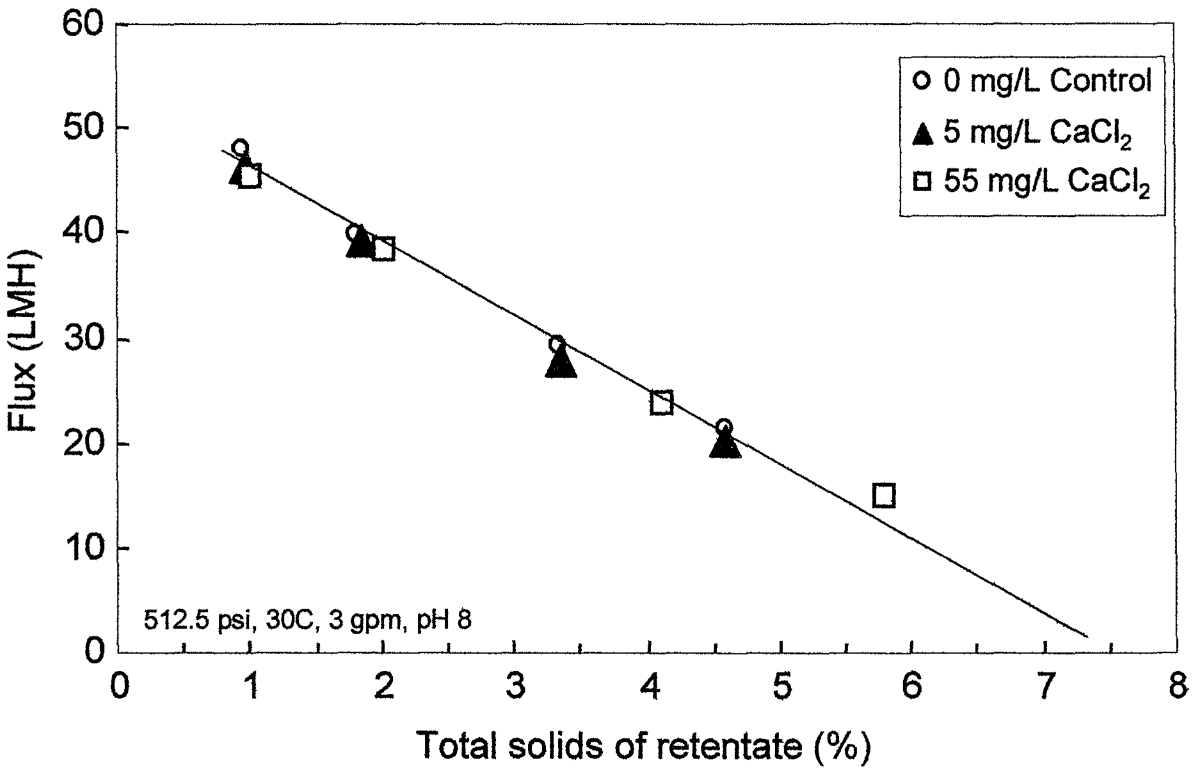


*Figure 54. Concentration of 1% sodium sulfate with added calcium chloride by RO at 512.5 psi, 30°C, pH 8 and cross-flow rate of 3 gpm*

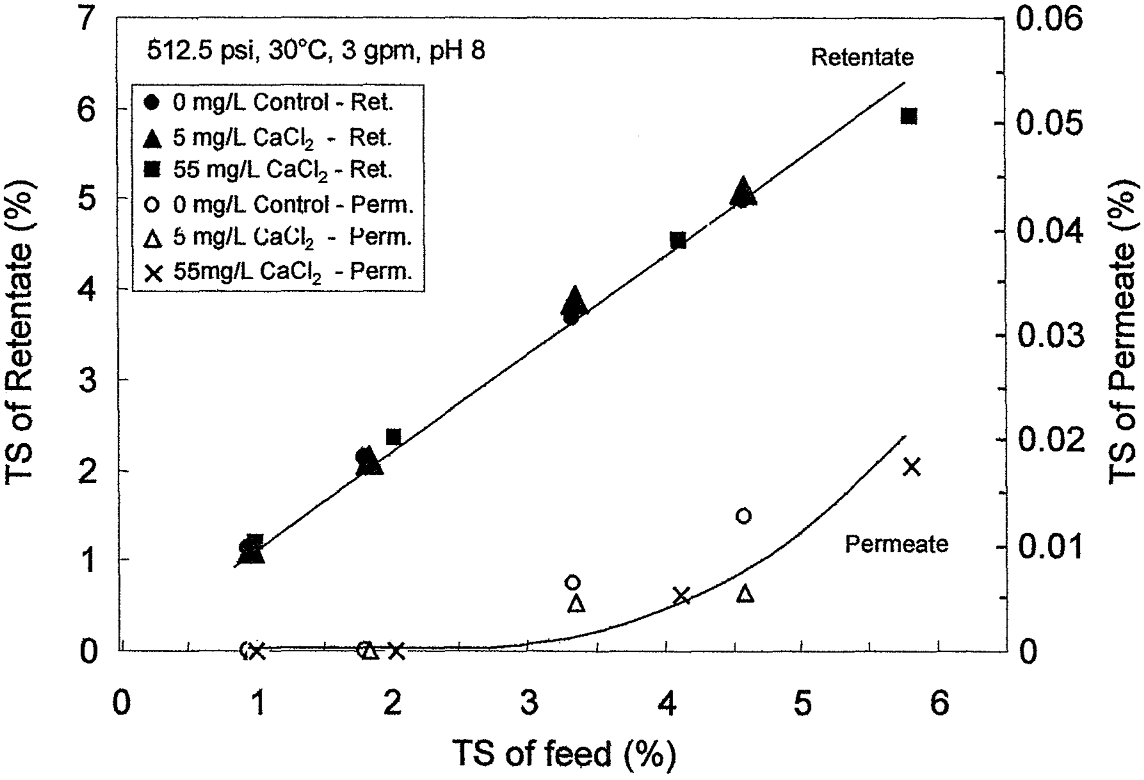


*Figure 55. Relationship between total solids in the feed and total solids in the retentate and permeate. Same experiment as Figure 54.*

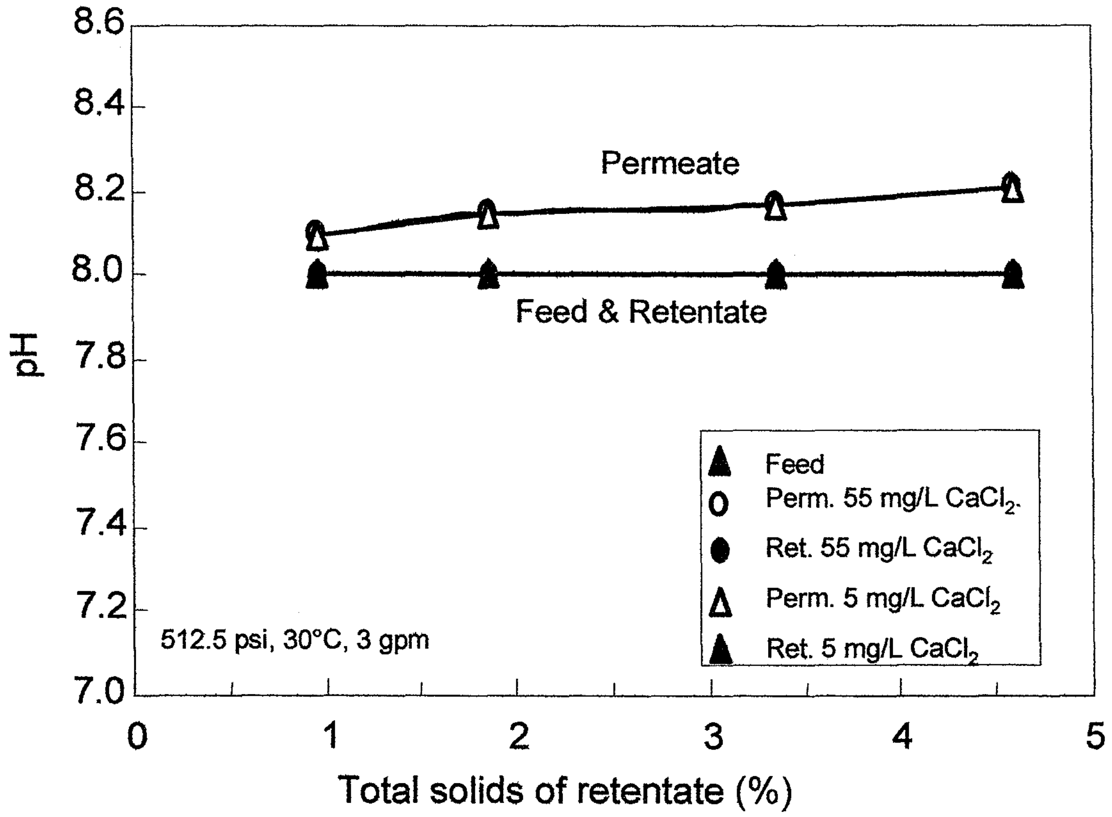


*Figure 56. pH profiles during RO concentration of 1% sodium sulfate with added calcium chloride. pH of feed was maintained at pH 8.0. Same experiment as Figures 54 and 55.*

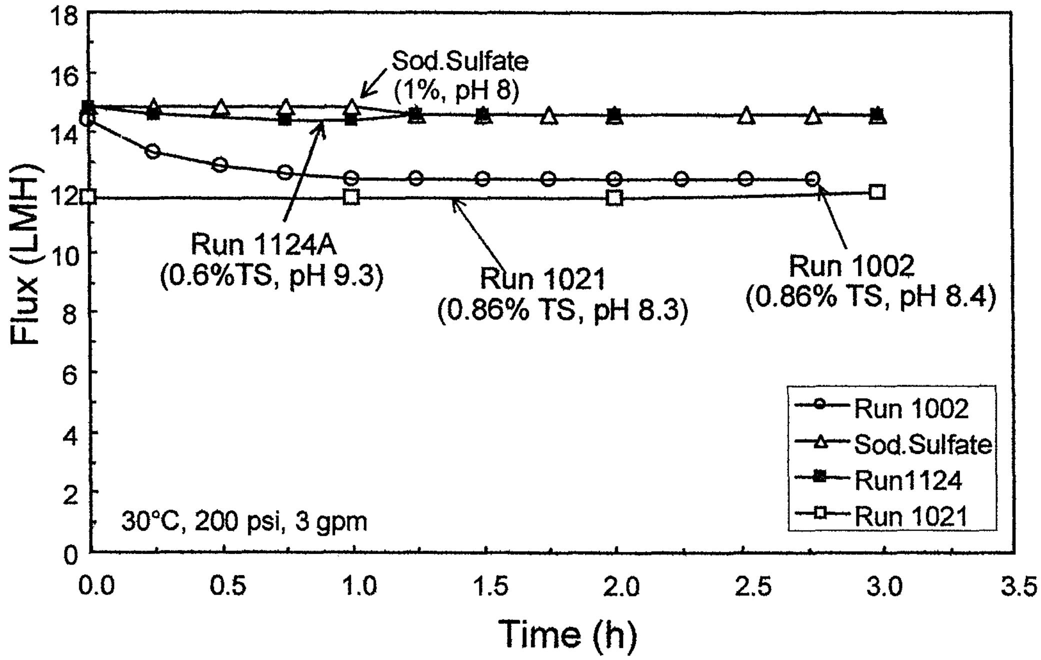


*Figure 57. Fouling of RO membrane by Guardian West streams (no added bacteria). Model sodium sulfate solution is shown for comparison.*

Figure 58 shows performance of the real feeds compared to the model solution. The lower flux with the real feeds is again clearly visible.

Figure 59 shows the flux obtained during concentration of one of the feeds. According to the equation that describes the data, the maximum concentration of solids (where flux is zero) would be 7.3% under these operating conditions. This is quite close to the values we had obtained with model sodium sulfate solutions as shown in the earlier section.

Figure 60 shows the distribution of solids between the retentate and permeate. Rejections of solids were over 99.6% with the real and model feeds. However, as is typical with reverse osmosis, the solids in the permeate increased at higher concentration factors. This phenomenon is similar to that observed with the model sodium sulfate solution.

Figures 61 and 62 show long-term fouling runs where the permeate and retentate were recycled for one week. Flux (Figure 61) remained fairly constant during this trial. The pH (Figure 62) decreased from 8.4 to about 8.0 in the first day and remained steady thereafter. Permeate pH was higher by about half a unit. It also decreased in the first day and remained steady after that.

### *3.5.1.1. Effect of added bacteria*

The effect of added bacteria is shown in Figures 63 and 64. The concentration of added bacteria in the feed was 0.0026%. The pH was not affected by the addition of the bacteria. The flux was higher than feed without the bacteria, but this is probably due to the lower solids in this sample of feed. The pH of the permeate was about 0.5 units higher than the retentate (Figure 64).

Figure 65 shows the effect of bacteria on flux. (The spikes in the flux can be traced to higher temperatures overnight). The data are plotted in a typical fouling log-log plot and the equation that describes the data is given below:

$$J = J_o t^{-b} \tag{24}$$

$J$ is the flux at time $t$ (hours) and $J_o$ is the flux at $t = 1$ hour. The parameters of equation 24 are shown in Table 10. Even though the two feeds were obtained on different days and had different characteristics (solids and pH), there are some conclusions one can draw about the effect of the bacteria vs. the solids on flux. The effect of solids is reflected in the coefficient of the equations (the coefficient is the flux at t = 1 hour). The lower solids feed (Run 1124B) had a coefficient of 17 LMH while the higher solids feed had a coefficient of 13.2 LMH. This difference is probably due to the effect of solids on osmotic pressure. On the other hand, Run 1124B (with the bacteria) had a slope of –0.08, which is higher than the slope of the feed without bacteria (-0.049), even though the latter feed had higher solids. In other words, bacteria apparently cause

*Table 10: Parameters of fouling equation 24. Data shown in Figure 65.*

| **Experiment** | **Total solids (%)** | ***J<sub>o</sub>*** | ***b*** |
|---|---|---|---|
| Run 1021: Without bacteria | 0.86 | 13.2 | 0.049 |
| Run 1124B: With bacteria | 0.60 | 17.0 | 0.080 |

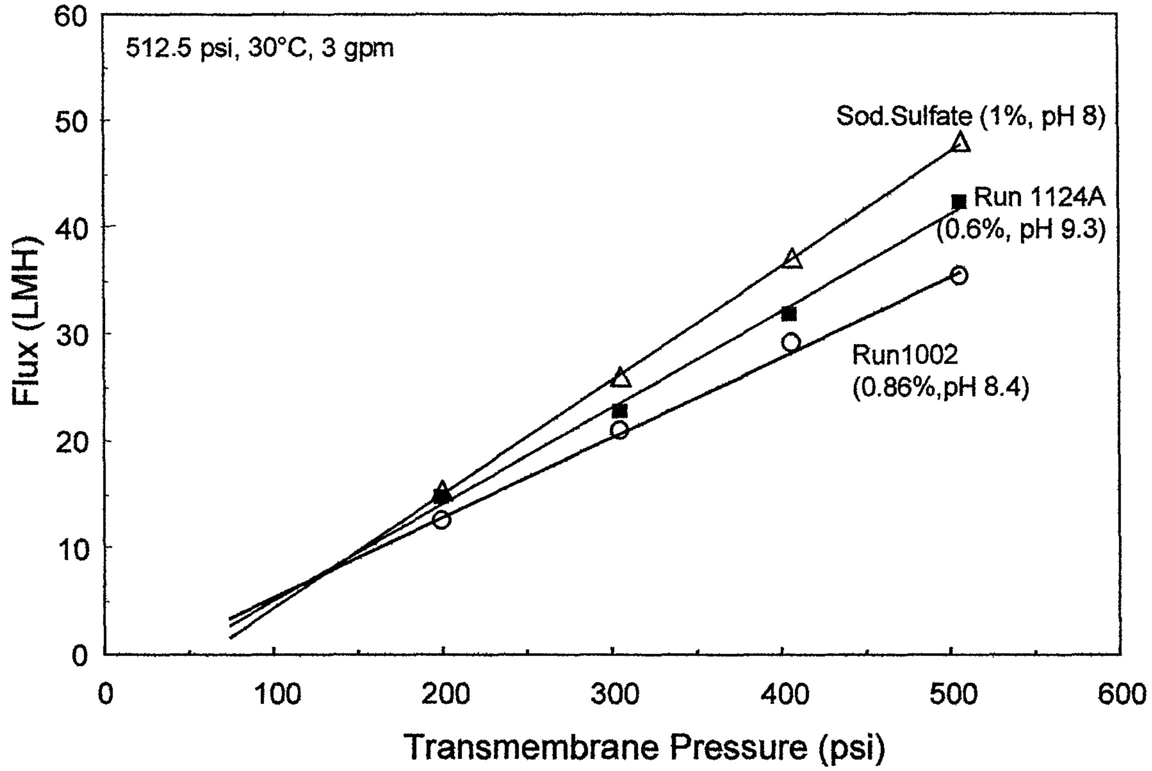


*Figure 58. Performance of RO membrane with Guardian West streams (no added bacteria). Model sodium sulfate solution is shown for comparison.*

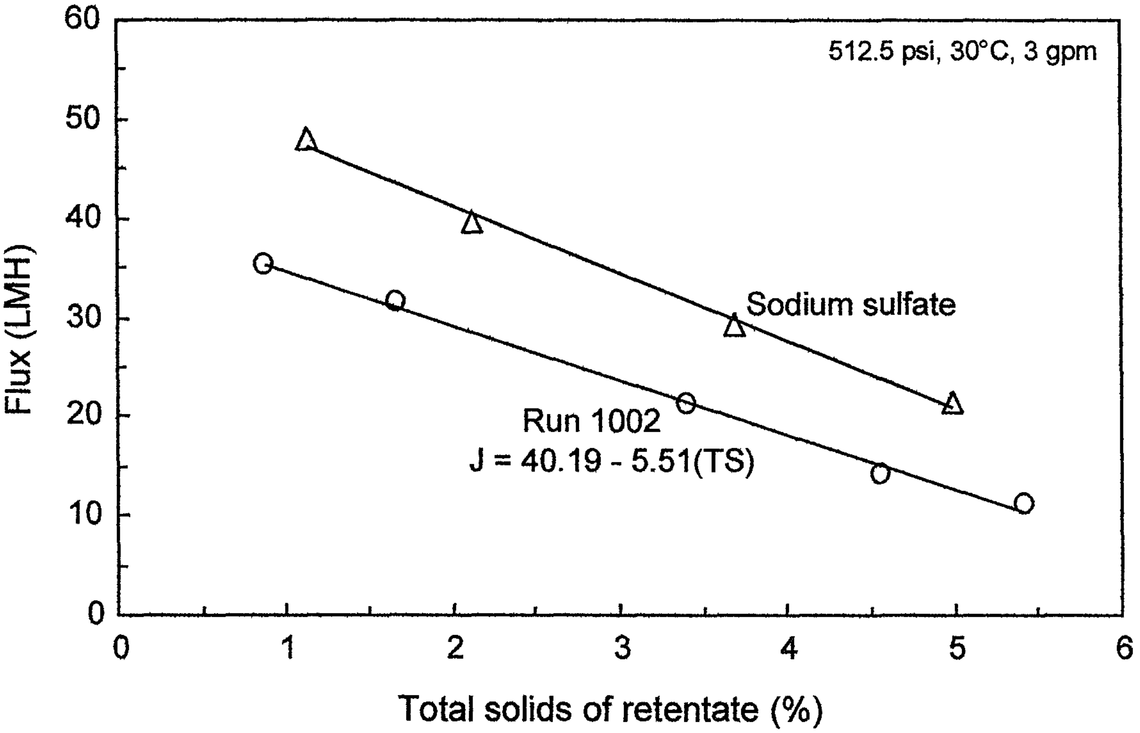


*Figure 59. Concentration of Guardian West stream (Run 1002, no added bacteria). Initial TS = 0.86%; pH = 8.4). Model sodium sulfate (1%, pH 8) shown for comparison*

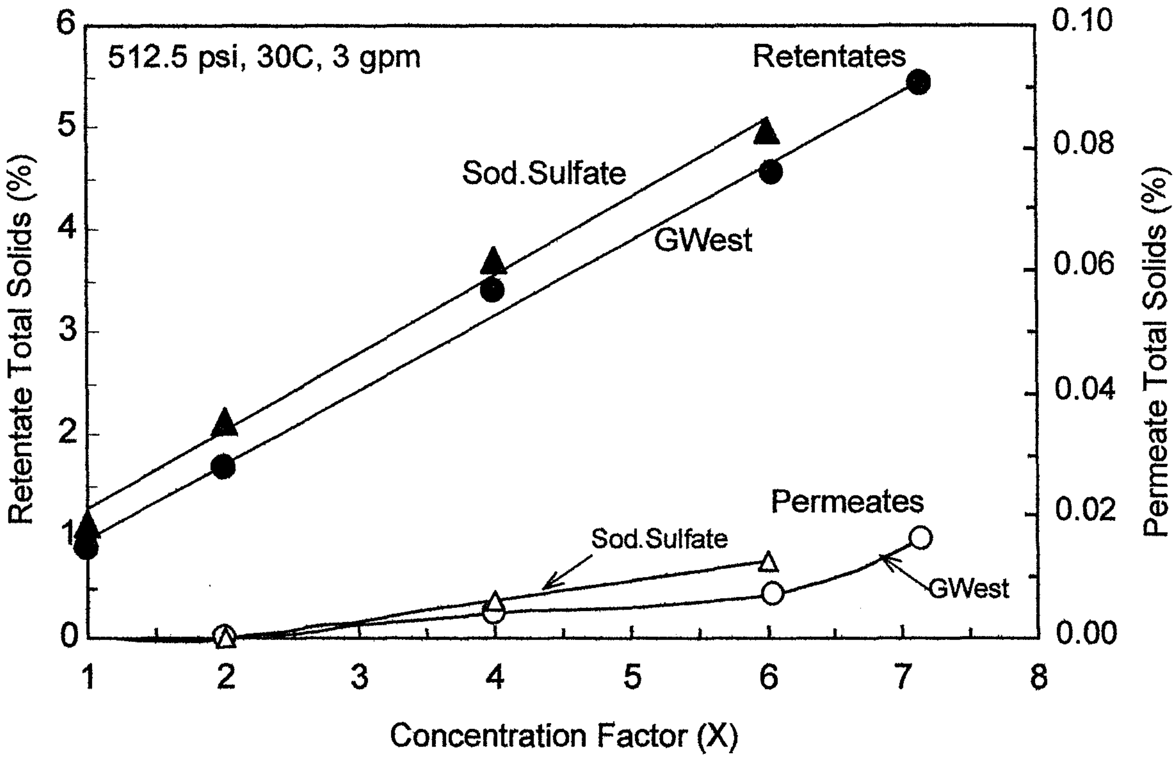


*Figure 60. Concentration of Guardian West stream by RO. Model solution of 1% sodium sulfate is shown for comparison*

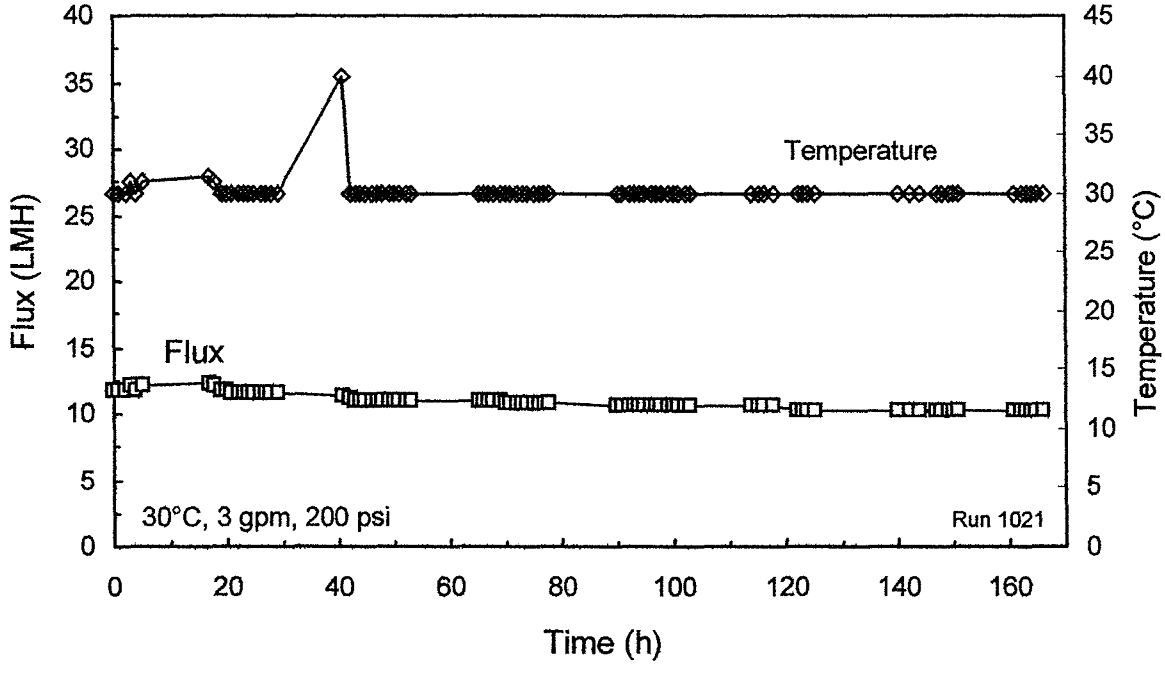


*Figure 61. Long term fouling of RO membrane by Guardian West stream. (Run 1021. Feed TS = 0.86%, pH = 8.3)*

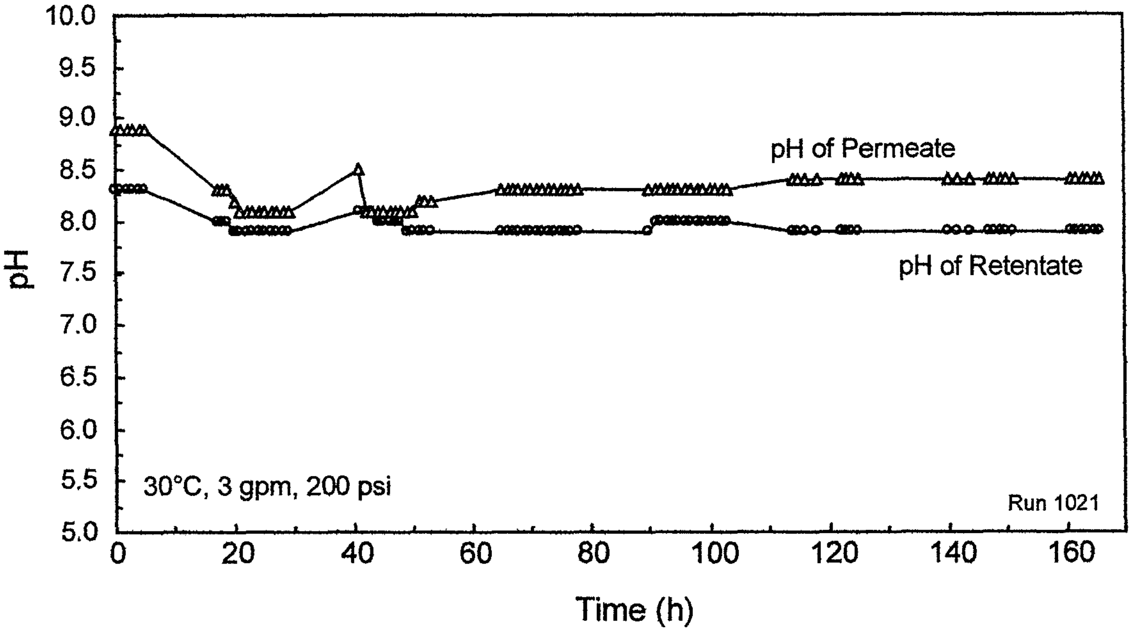


*Figure 62. pH during long term fouling study by Guardian West stream. Same conditions as Figure 61.*

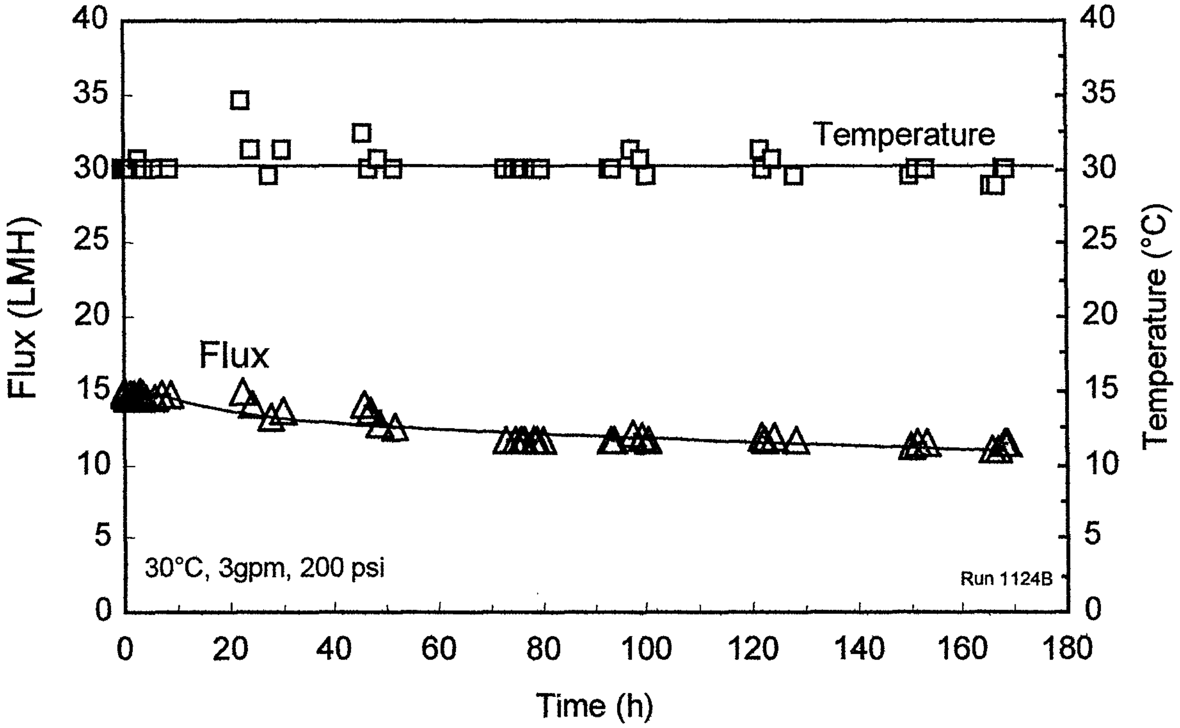


*Figure 63. Long term fouling of RO membrane by Guardian West stream with added bacteria. (Run 1124B. Feed TS = 0.6%, pH = 9.3, bacteria concentration = 0.0026%)*

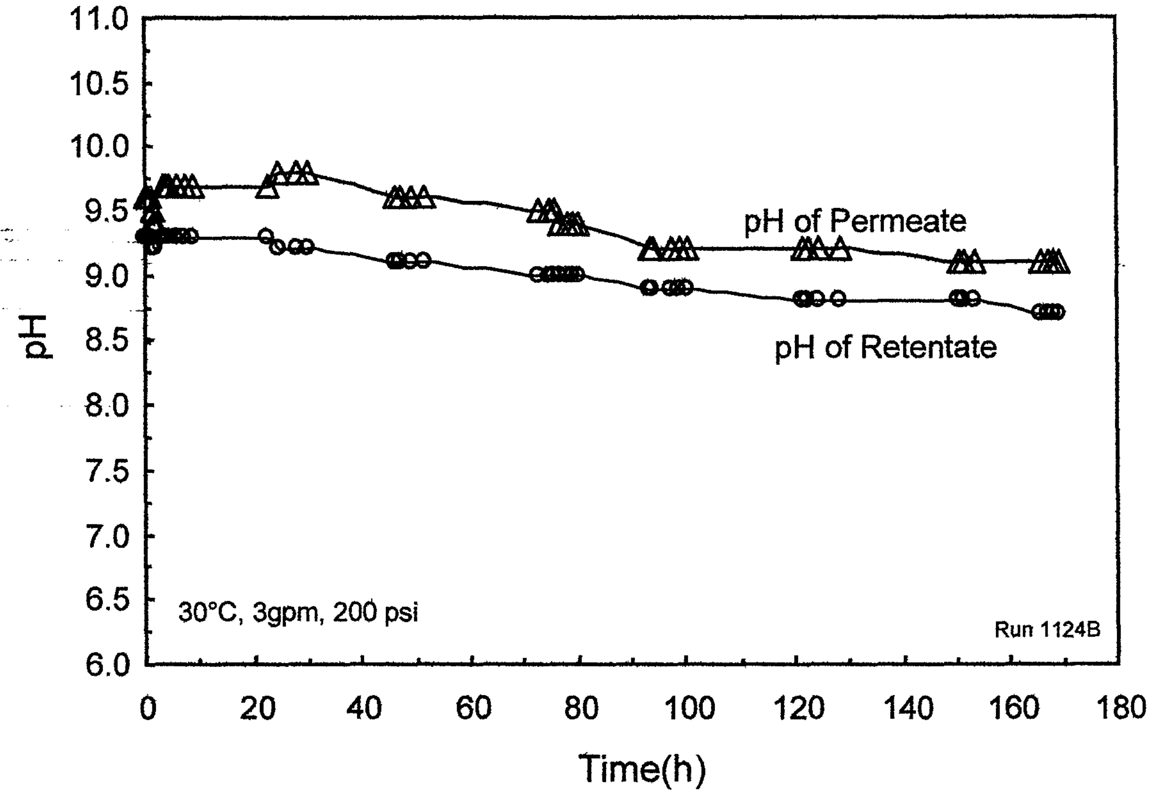


*Figure 64. pH during long term study of fouling by Guardian West stream with added bacteria. Same conditions as Figure 63.*

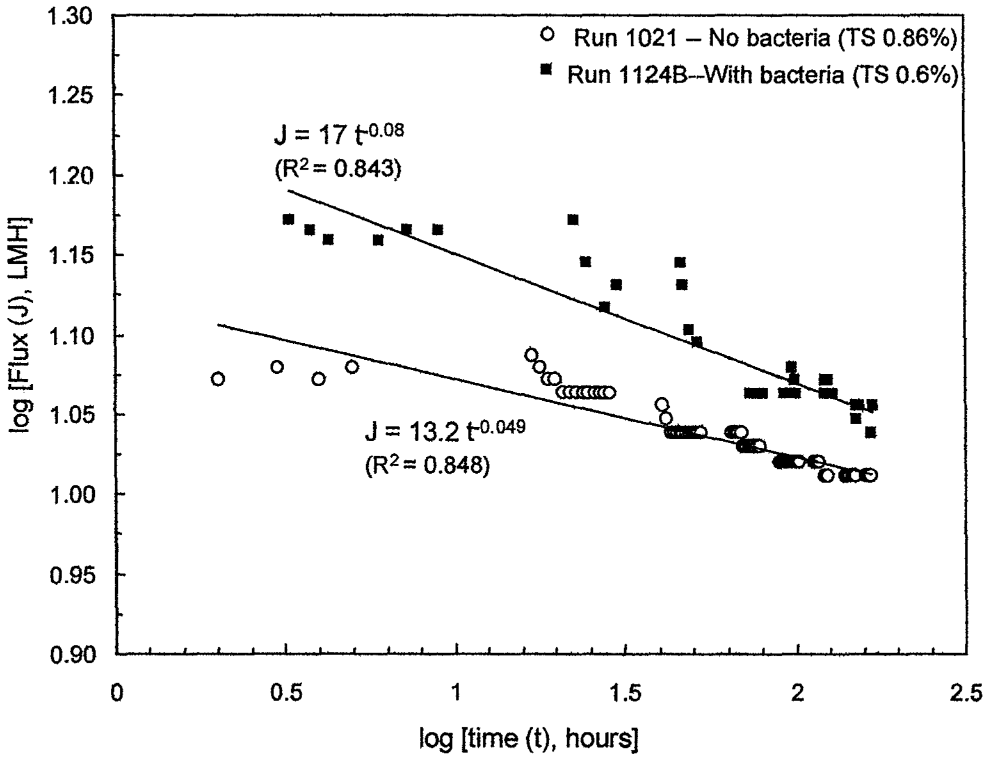


*Fig.65. Comparision of long term fouling of Guardian West streams with and without bacteria*

greater fouling of the membrane.

Cleaning the membrane after conducting experiments with the Guardian West feed required more aggressive techniques. Two cleanings were required with a commercial alkaline cleaner (Ultrasil-10 from Klenzade) with an interval of 3-4 days between cleanings. This successfully recovered the original water flux.

### *3.5.1.2. Ion analysis*

The concentrations of various ions (calcium, sodium, chromium, etc.), TOC and TIC of the samples that were taken during reverse osmosis of the real feed was determined and are shown in Tables 11-13. The major anions shown in Table 11 are much lower than the samples obtained 2 years ago (Table 4). The sodium is also lower but the magnesium is substantially higher (Compare Table 12 with Table 5). The other ions are approximately the same. TOC is also somewhat lower in this latest batch of electroplating waste (compare Table 13 with Table 7).

The permeate quality is consistently excellent in comparison to the feed or retentate. The concentration of ions go down as the pressure increases at the same feed/retentate concentration (at least, in those cases which can be measured, e.g., sodium in Table 12). On the other hand, ion levels in the permeate increase as the concentration increases (see chloride and sulfate in Table 11 and sodium in Table 12). Both phenomena are consistent with known mechanisms of RO transport and models.

### *3.5.2. Phase II*

Table 14 lists experiments conducted in this phase of the study and an analysis of the feed. The three experiments conducted in this period are numbered 209, 310 and 318. Between Phase I (Table 9) and Phase II runs done between October 2002 and March 2003, the solids varied from 0.31% to 0.86% and feed pH varied from pH 7.8 to 9.2. The performance of the membrane will vary with the properties of the feed.

Figure 66 shows short term fouling data conducted at low pressure (200 psi). Flux of all the feeds are in the same range, between 13 and 15 liters per square meter per hour (LMH). Variations in the flux can be partially accounted for by the variations in solids content.

After the short-term fouling study, the pressure was increased to 507.5 psi, keeping the temperature at 30°C and the cross-flow rate at 3 gpm. Figures 67-72 show long-term fouling runs in which the permeate and retentate were recycled, and the effect of added bacteria. In general, bacteria have a small effect on flux and pH. Permeate pH was higher than retentate pH in some runs and the opposite in others. The difference between the pH of the permeate and the retentate was higher in runs without bacteria than with bacteria.

Figures 73-75 show performance of the RO membrane with and without bacteria. In general, addition of the bacteria depressed the flux by about 16%. Figure 76 is a concentration study. At the end of Run 318C, the permeate was removed from the system and the volume of the feed allowed to decrease. The small volume of feed available allowed us to go only to about 4X. At this level of solids in the feed, permeate solids was essentially zero, showing a 100% rejection of solids by this RO membrane. Flux decreased as the solids increased.

*Table 11: Anions in Guardian West electroplating waste stream during RO. Data shown is for Run 1002 shown in Figures 57-59*

| **Sample** | **Chloride (mg/L)** | | | **Nitrate (mg/L)** | | | **Sulfate (mg/L)** | | |
|---|---|---|---|---|---|---|---|---|---|
| | Feed | Ret. | Perm | Feed | Ret. | Perm | Feed | Ret. | Perm |
| Feed (before start of run ) | 200 | | | 15 | | | 5700 | | |
| Feed (at start of short-term fouling ) | 190 | | | <13 | | | 5400 | | |
| Feed (end of short-term fouling) | 190 | | | <13 | | | 5700 | | |
| Pressure excursion at 1X: 200 psi | | 190 | 1 | | <25 | <0.5 | | 5600 | 10 |
| 305 psi | | 210 | 1 | | <25 | 1.1 | | 5900 | 11 |
| 407 psi | | 200 | 1 | | <25 | <0.5 | | 5500 | 7 |
| 507 psi | | 200 | 1 | | <25 | <0.5 | | 5600 | 10 |
| Concentration at 507 psi: 2X | | 390 | 1 | | <13 | <0.5 | | 11000 | 11 |
| 4X | | 730 | 4 | | <25 | <0.5 | | 21000 | 28 |
| 6X | | 1100 | 8 | | <25 | 1 | | 30000 | 53 |
| 7.1X | | 1300 | 12 | | <25 | <0.5 | | 35000 | 86 |

*Table 12: Cations in Guardian West electroplating waste stream during RO. Data shown is for Run 1002 shown in Figures 57-59*

| **Sample** | **Barium (mg/L)** | | | **Calcium (mg/L)** | | | **Chromium (mg/L)** | | |
|---|---|---|---|---|---|---|---|---|---|
| | Feed | Ret. | Perm | Feed | Ret. | Perm | Feed | Ret. | Perm |
| Feed (before start of run ) | 0.011 | | | 29 | | | 0.32 | | |
| Feed (at start of short-term fouling ) | 0.054 | | | 28 | | | < 0.1 | | |
| Feed (end of short-term fouling) | 0.021 | | | 29 | | | 5.0 | | |
| Pressure excursion at 1X: 200 psi | | 0.036 | < 0.002 | | 26 | < 1 | | 0.009 | < 0.002 |
| 305 psi | | 0.037 | < 0.002 | | 28 | < 1 | | 0.009 | < 0.002 |
| 407 psi | | 0.044 | < 0.002 | | 27 | < 1 | | 0.008 | < 0.002 |
| 507 psi | | 0.066 | < 0.002 | | 27 | < 1 | | 0.010 | < 0.002 |
| Concentration at 507 psi: 2X | | 0.042 | < 0.002 | | 54 | < 1 | | 0.015 | < 0.002 |
| 4X | | 0.071 | < 0.002 | | 98 | < 1 | | 0.034 | < 0.002 |
| 6X | | 0.083 | < 0.002 | | 140 | < 1 | | 0.046 | < 0.002 |
| 7.1X | | 0.070 | < 0.002 | | 170 | < 1 | | 0.052 | < 0.002 |

*........Table 12 continued next page*

*Table 12 (continued): Cations in Guardian West electroplating waste stream during RO. Data shown is for Run 1002 shown in Figures 57-59*

| **Sample** | **Copper (mg/L)** | | | **Iron (mg/L)** | | | **Magnesium (mg/L)** | | |
|---|---|---|---|---|---|---|---|---|---|
| | Feed | Ret. | Perm | Feed | Ret. | Perm | Feed | Ret. | Perm |
| Feed (before start of run ) | < 0.1 | | | < 1 | | | 140 | | |
| Feed (at start of short-term fouling ) | < 0.1 | | | < 1 | | | 140 | | |
| Feed (end of short-term fouling) | 0.11 | | | 5.3 | | | 150 | | |
| Pressure excursion at 1X: 200 psi | | 0.003 | < 0.002 | | 0.24 | < 0.02 | | 140 | < 1 |
| 305 psi | | < 0.002 | < 0.002 | | 0.25 | < 0.02 | | 150 | < 1 |
| 407 psi | | < 0.002 | < 0.002 | | 0.21 | < 0.02 | | 160 | < 1 |
| 507 psi | | < 0.002 | < 0.002 | | 0.24 | < 0.02 | | 140 | < 1 |
| Concentration at 507 psi: 2X | | 0.003 | < 0.002 | | 0.44 | < 0.02 | | 260 | < 1 |
| 4X | | 0.007 | < 0.002 | | 0.87 | < 0.02 | | 520 | < 1 |
| 6X | | 0.011 | < 0.002 | | 1.2 | < 0.02 | | 1200 | < 1 |
| 7.1X | | 0.009 | < 0.002 | | 1.2 | < 0.02 | | 1300 | 1 |

........*Table 12 continued next page*

*Table 12 (continued): Cations in Guardian West electroplating waste stream during RO. Data shown is for Run 1002 shown in Figures 57-59*

| **Sample** | **Nickel (mg/L)** | | | **Sodium (mg/L)** | | | **Zinc (mg/L)** | | |
|---|---|---|---|---|---|---|---|---|---|
| | Feed | Ret. | Perm | Feed | Ret. | Perm | Feed | Ret. | Perm |
| Feed (before start of run ) | 0.41 | | | 2400 | | | < 0.1 | | |
| Feed (at start of short-term fouling ) | 0.15 | | | 2300 | | | < 0.1 | | |
| Feed (end of short-term fouling) | 5.6 | | | 2300 | | | 0.24 | | |
| Pressure excursion at 1X: 200 psi | | 0.11 | < 0.002 | | 2400 | 6.7 | | 0.004 | < 0.002 |
| 305 psi | | 0.12 | < 0.002 | | 2600 | 5.9 | | 0.004 | < 0.002 |
| 407 psi | | 0.11 | < 0.002 | | 2800 | 3.9 | | 0.004 | < 0.002 |
| 507 psi | | 0.11 | < 0.002 | | 2800 | 4.2 | | 0.005 | < 0.002 |
| Concentration at 507 psi: 2X | | 0.21 | < 0.002 | | 5700 | 7 | | 0.004 | < 0.002 |
| 4X | | 0.38 | < 0.002 | | 11000 | 15 | | 0.007 | < 0.002 |
| 6X | | 0.54 | < 0.002 | | 16000 | 33 | | 0.009 | < 0.002 |
| 7.1X | | 0.70 | < 0.002 | | 24000 | 49 | | 0.012 | < 0.002 |

*Table 13: Total organic carbon (TOC) and total inorganic carbon (TIC) in Guardian West electroplating waste stream during RO. Data shown is for Run 1002 shown in Figures 57-59*

| **Sample** | **TOC (mg/L)** | | | **TIC (mg/L)** | | |
|---|---|---|---|---|---|---|
| | Feed | Ret. | Perm | Feed | Ret. | Perm |
| Feed (before start of run ) | 69 | | | 19.0 | | |
| Feed (at start of short-term fouling ) | 67 | | | 23.0 | | |
| Feed (end of short-term fouling) | 70 | | | 28.0 | | |
| Pressure excursion at 1X: 200 psi | | 54 | 4.7 | | 11 | <0.5 |
| 305 psi | | 62 | 3.7 | | 15 | <0.5 |
| 407 psi | | 58 | 4.1 | | 9.7 | <0.5 |
| 507 psi | | 62 | 5.7 | | 8.4 | <0.5 |
| Concentration at 507 psi: 2X | | 140 | 11 | | 32 | <0.5 |
| 4X | | 200 | 5 | | 60 | <0.5 |
| 6X | | 330 | 14 | | 77 | 21 |
| 7.1X | | 440 | 15 | | 78 | <0.5 |

*Table 14. Phase II trials of RO of electroplating waste from Guardian West*

| | **Run Number** | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | **209A** | **209B** | **209C** | **310A** | **310B** | **310C** | **318A** | **318B** | **318C** |
| **Bacteria Added** | No | No | Yes | No | No | Yes | No | No | Yes |
| **Total Solids of Feed (%)** | 0.58% | 0.58% | 0.59% | 0.31% | 0.31% | 0.31% | 0.34% | 0.34% | 0.35% |
| **pH of Feed** | 9 | 9 | 8.3 | 8.3 | 8.3 | 7.8 | 7.8 | 7.8 | 7.8 |
| **Fouling Study** | Short term (3 hours) | Long term (92 hours) | Long term (100 hours) | Short term (3 hours) | Long term (92 hours) | Long term (100 hours) | Short term (3 hours) | Long term (92 hours) | Long term (72 hours) |
| **Performance Study** | Yes | No | Yes | Yes | No | Yes | Yes | No | Yes |
| **Concentration Study** | No | No | No | No | No | No | No | No | Yes |

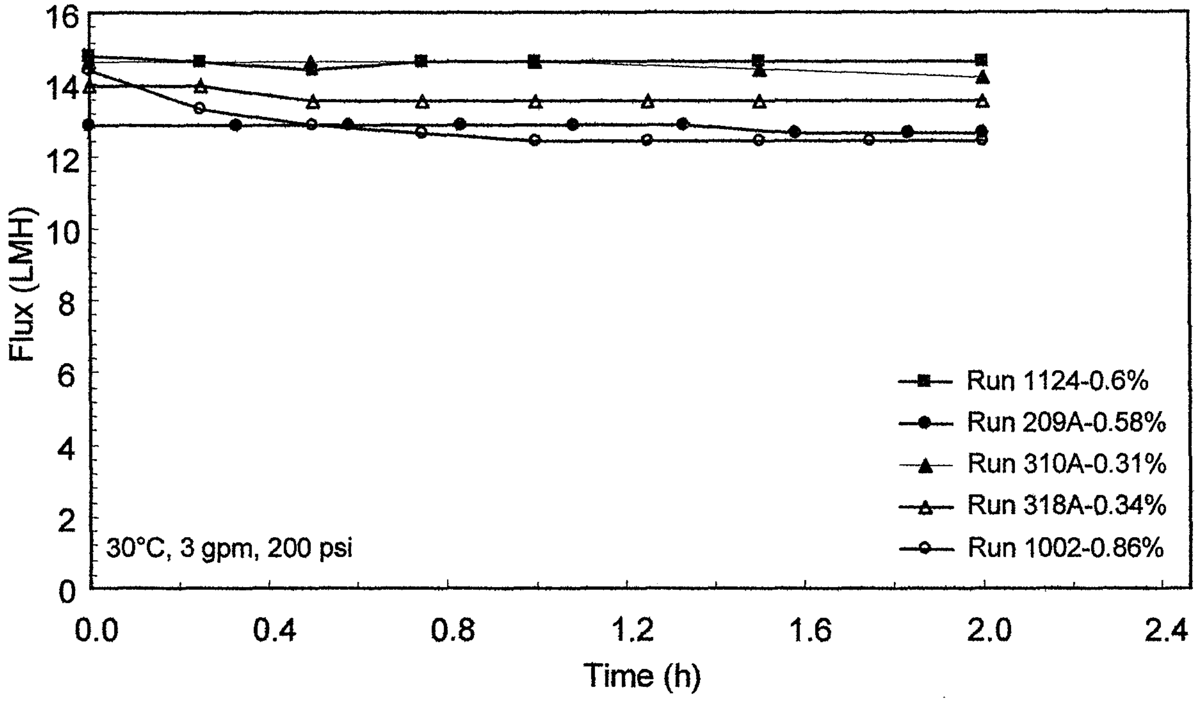


*Figure 66. Comparison of short term low-pressure fouling by Guardian West streams.*

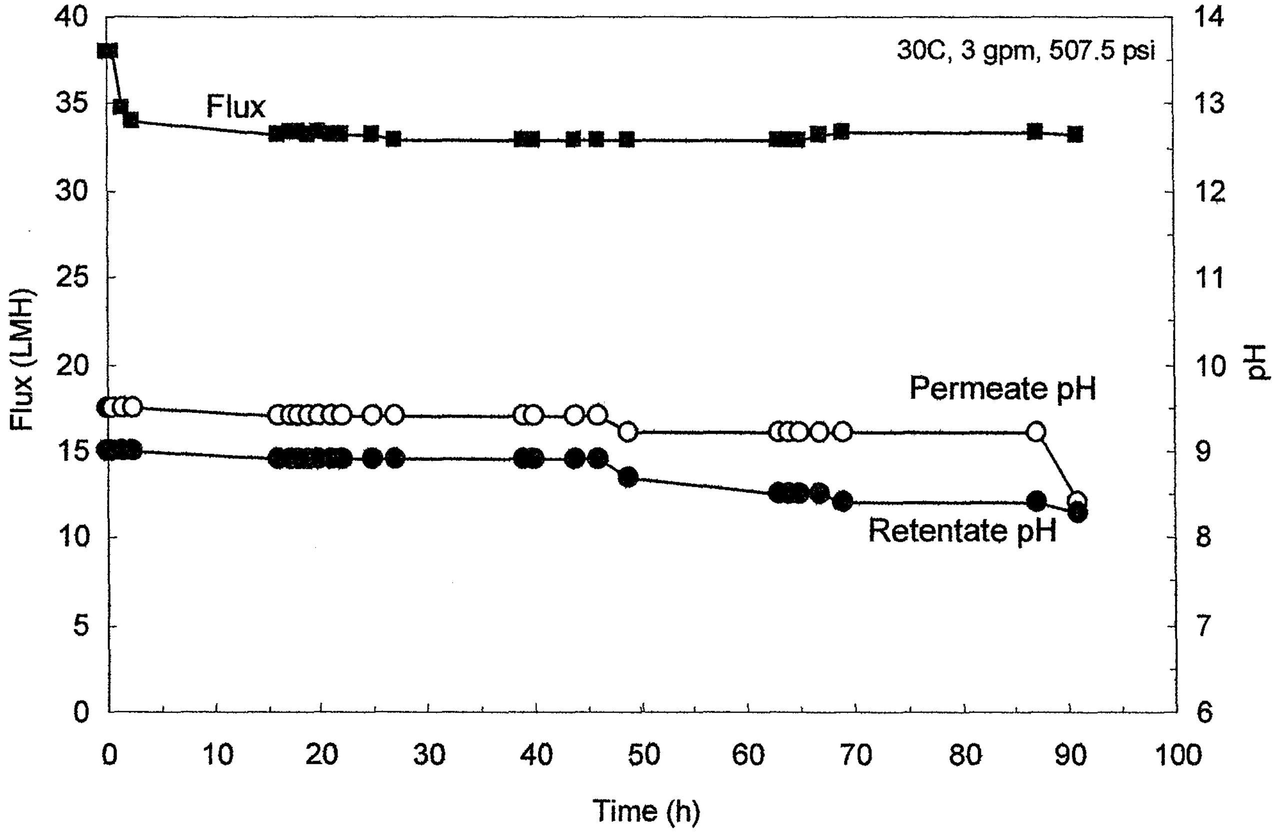


*Figure 67. Long term, high-pressure fouling by Guardian West stream. Run 209B (no bacteria). TS = 0.58%, pH = 9.0*

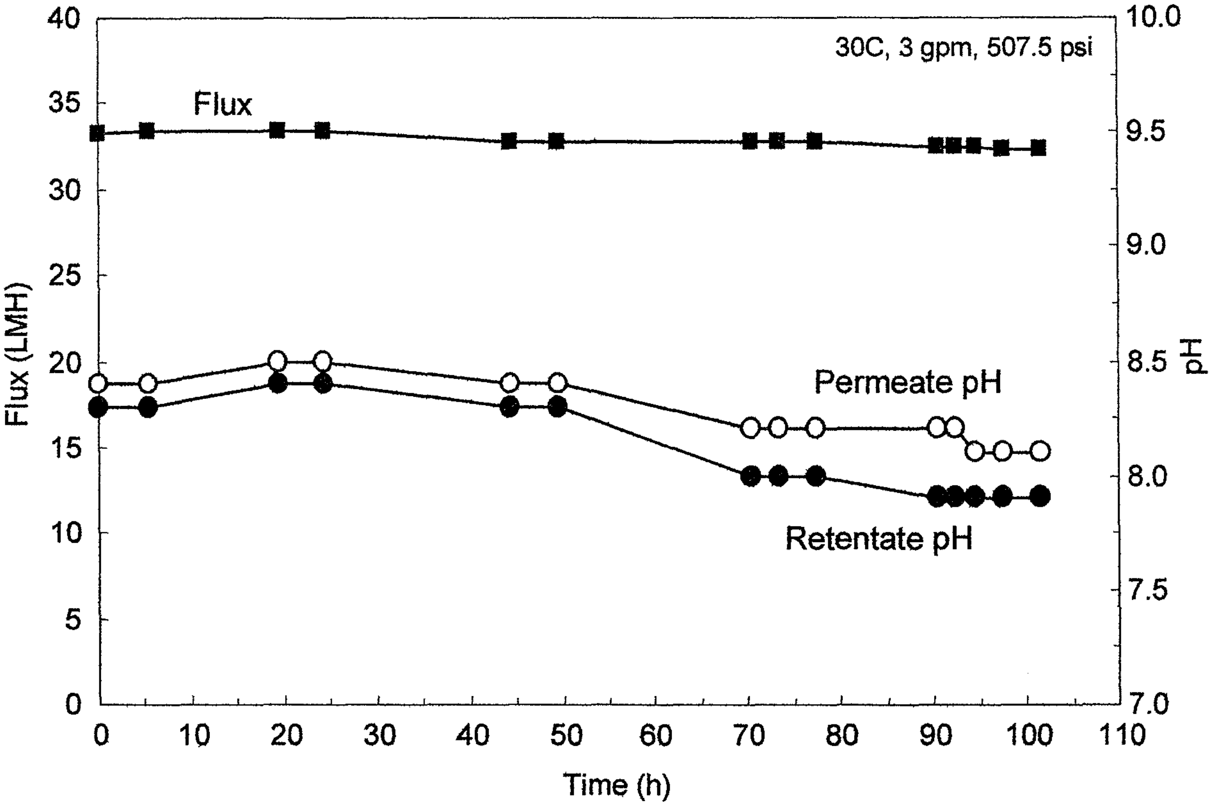


*Figure 68. Long term, high-pressure fouling by Guardian West stream. Run 209C (with bacteria). TS = 0.59%, pH = 8.3*

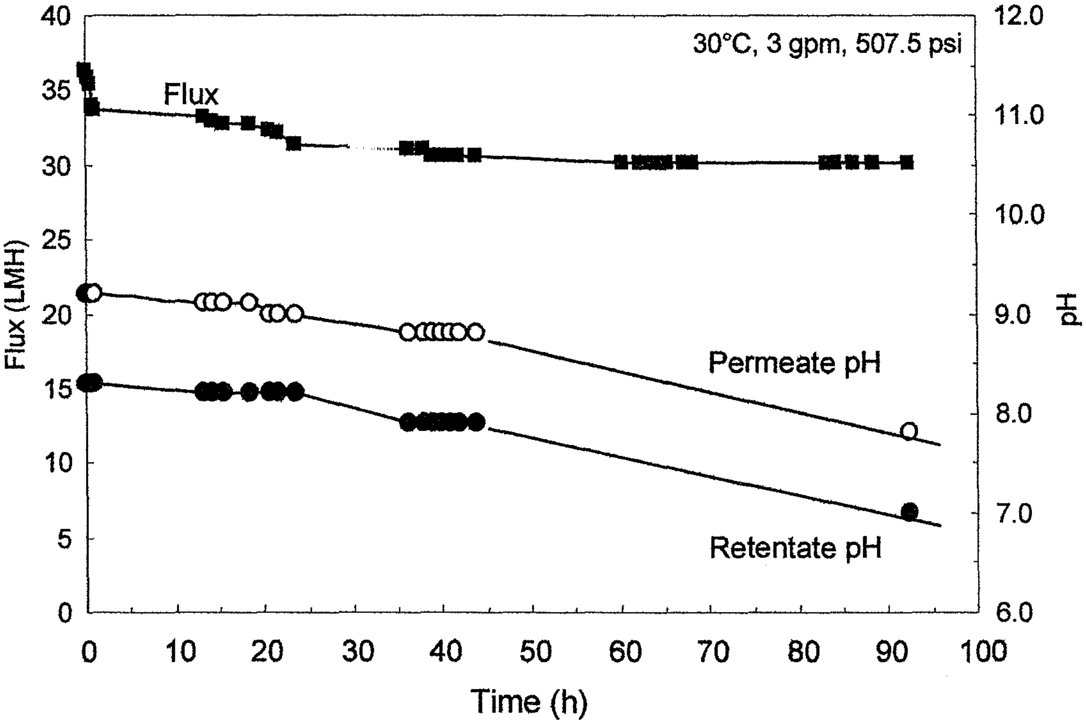


*Figure 69. Long term, high-pressure fouling by Guardian West stream.Run 310B (no bacteria). TS = 0.31%, pH = 8.3*

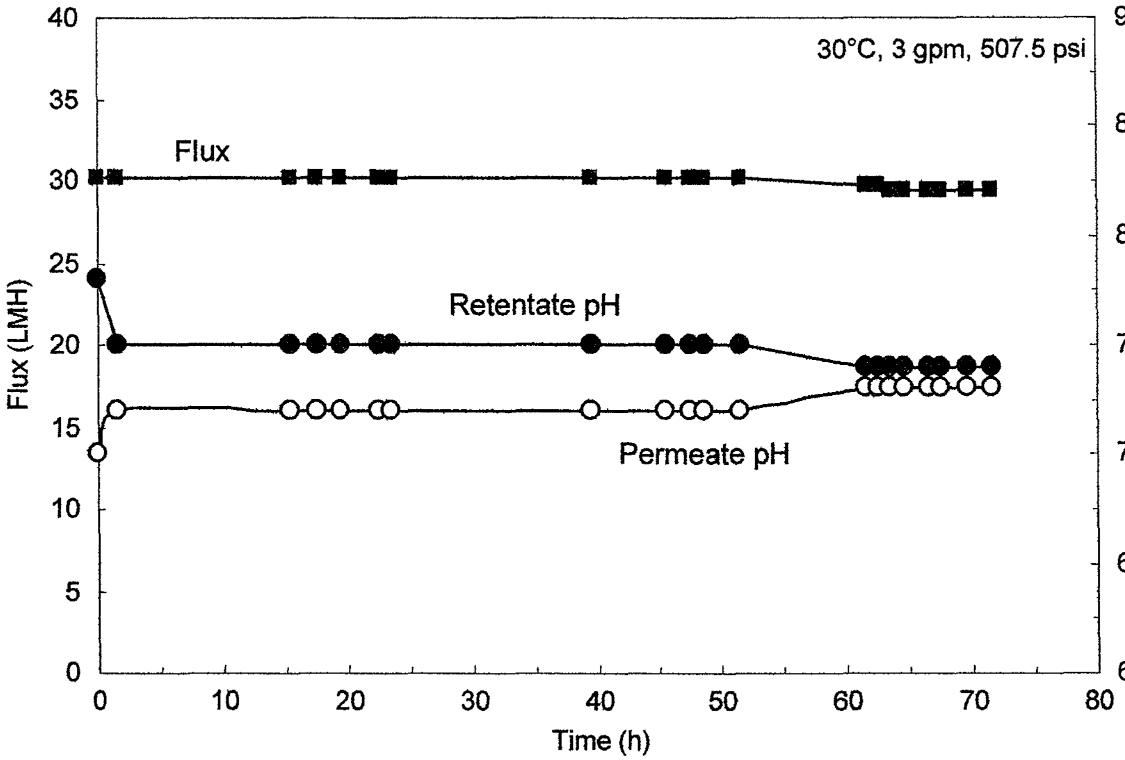


*Figure 70. Long term, high-pressure fouling by Guardian West stream. Run 310C (with bacteria ). TS = 0.31%, pH = 7.8*

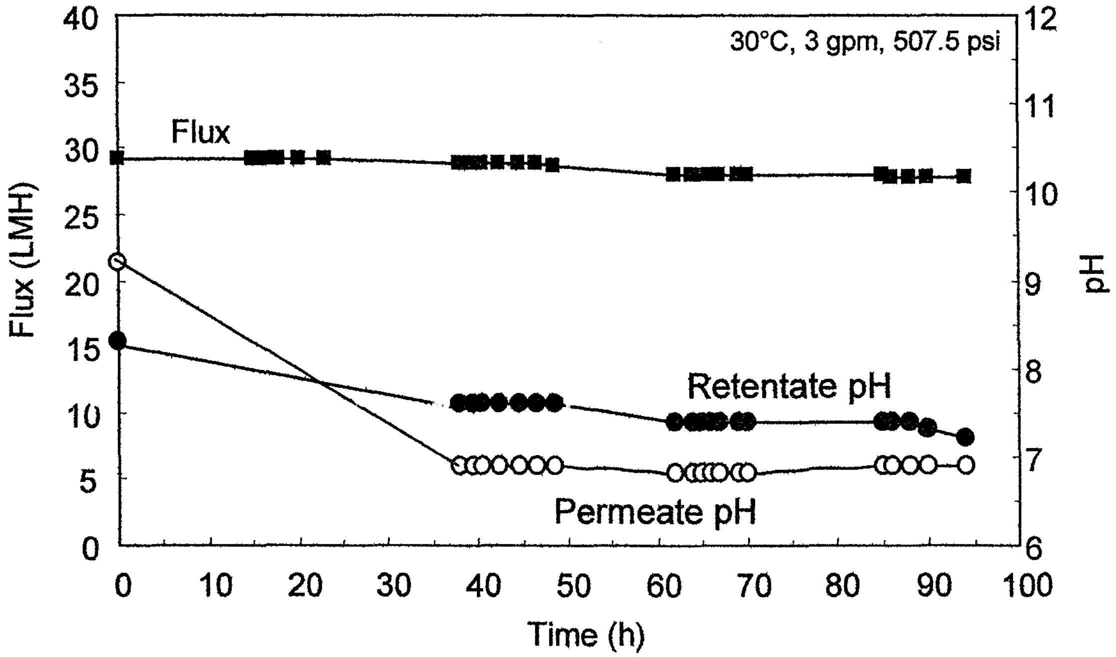


*Figure 71. Long term, high-pressure fouling by Guardian West stream. Run 318B (no bacteria ). TS = 0.31%, pH = 9.2*

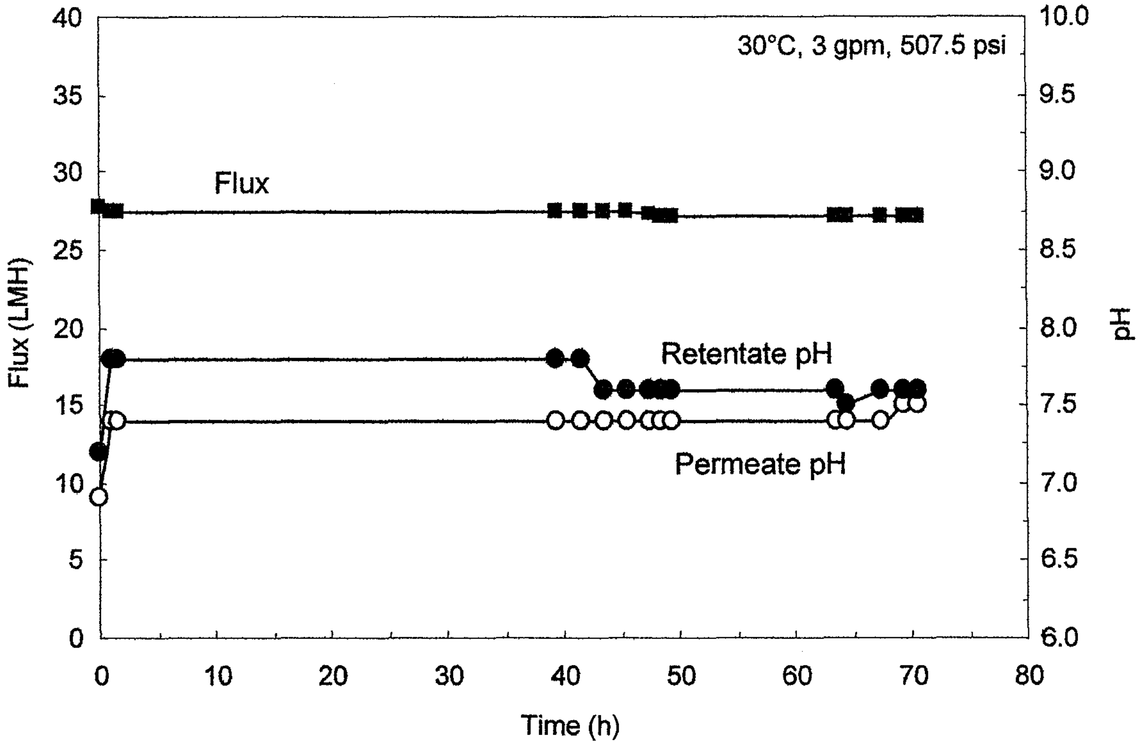


*Figure 72. Long term, high-pressure fouling by Guardian West stream. Run 318C (with bacteria ). TS = 0.31%, pH = 7.2*

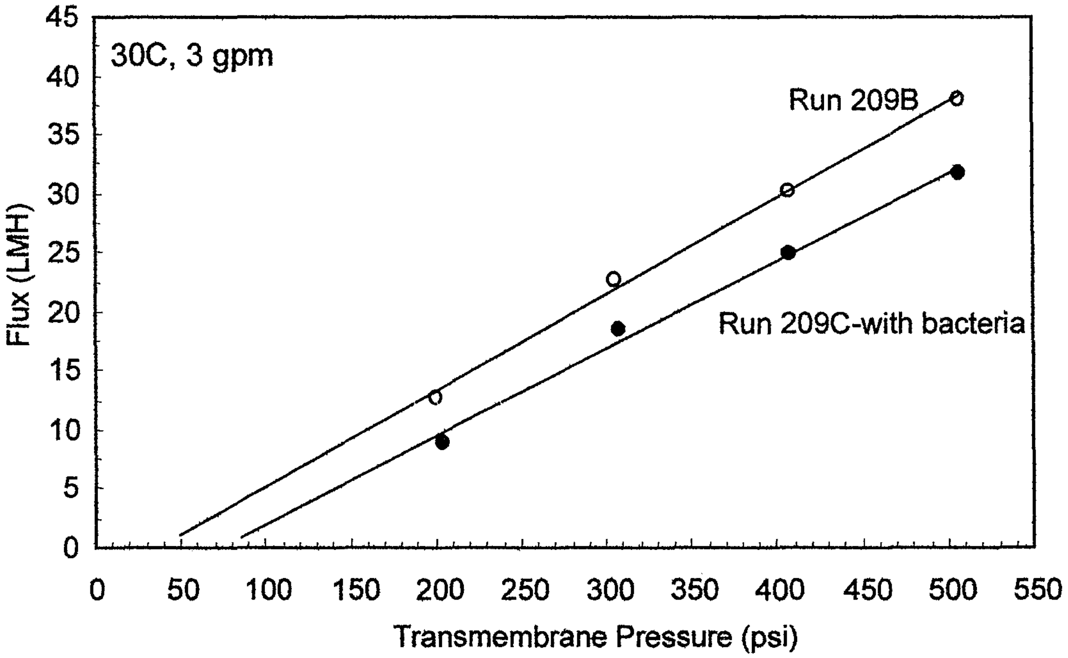


*Figure 73. Effect of bacteria on RO performance with Guardian West stream. Run 209B (no bacteria, 0.58% TS, pH 9); Run 209C (with bacteria, 0.59%TS, pH 8.3)*

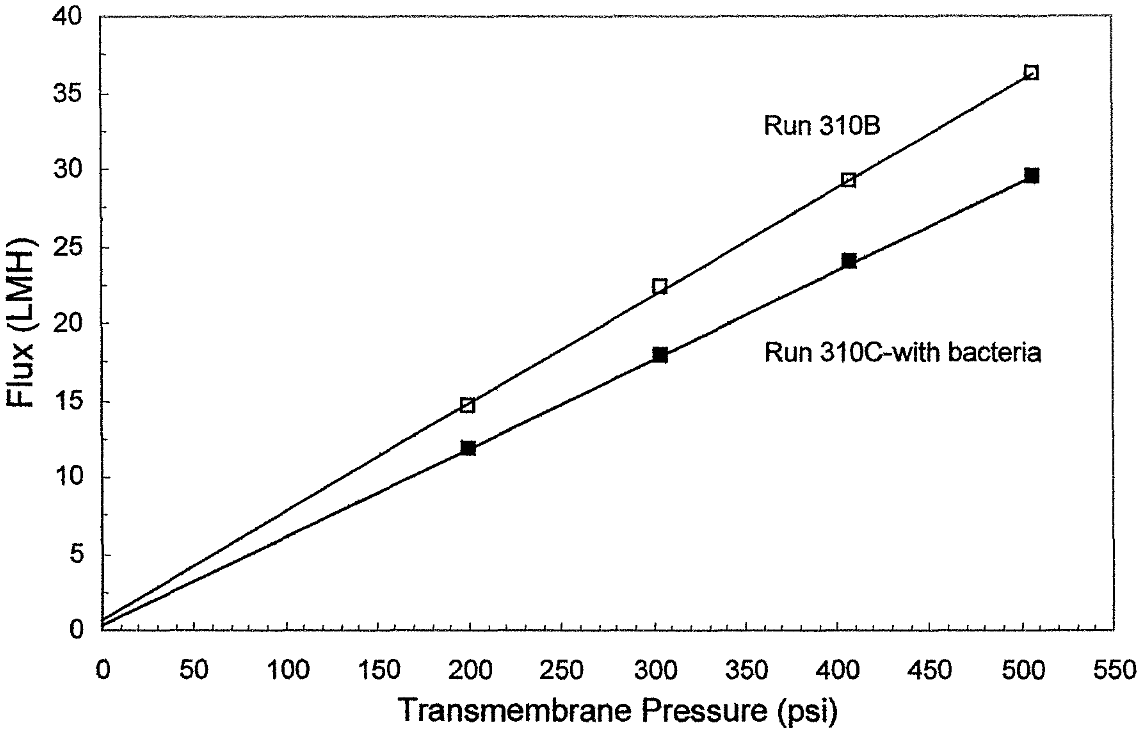


*Figure 74. Effect of bacteria on RO performance with Guardian West stream.*
*Run 310B (no bacteria, 0.31% TS, pH 8.3); Run 310C (with bacteria, 0.31%TS, pH 7.8)*

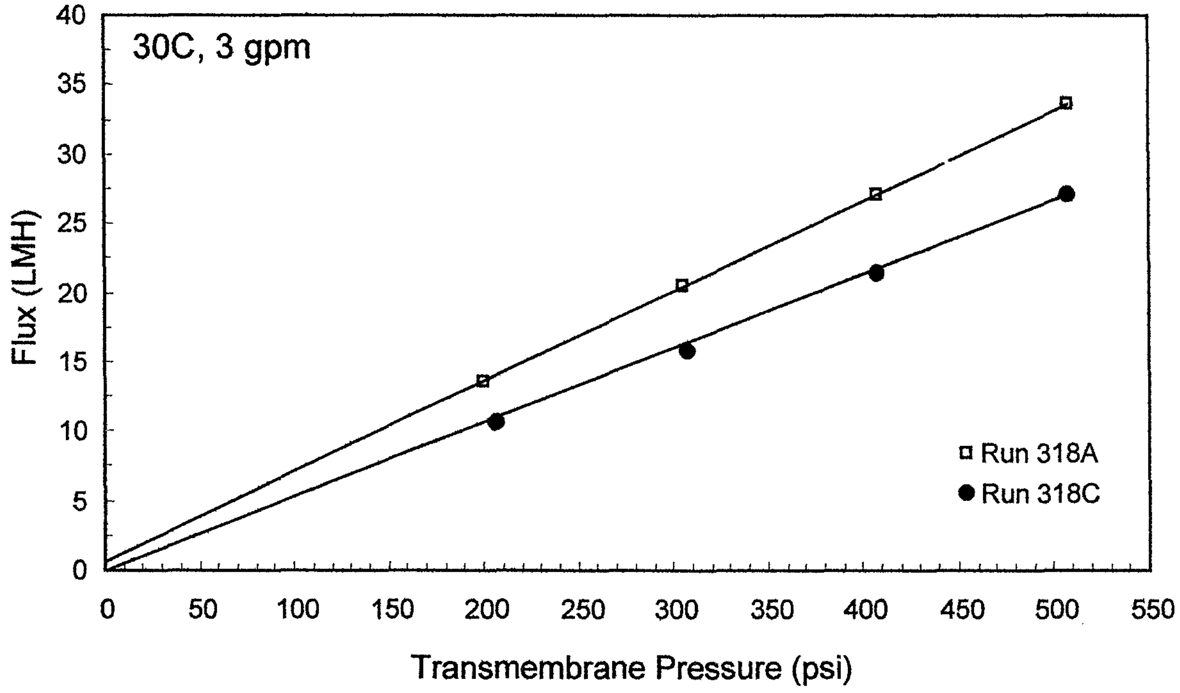


*Figure 75. Effect of bacteria on RO performance with Guardian West stream.*
*Run 318A (no bacteria, 0.34% TS, pH 7.8); Run 318C (with bacteria, 0.46%TS, pH 7.6)*

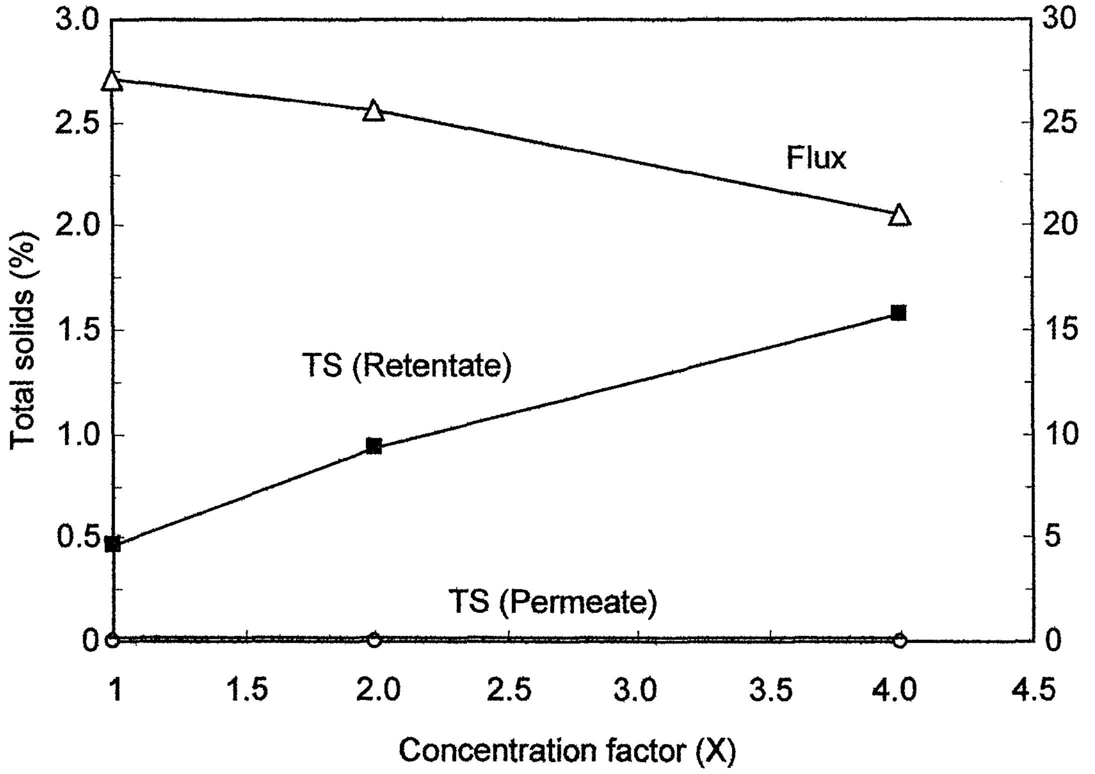


*Figure 76. Concentrating Guardian West stream by RO. Run 318C. Feed (with bacteria). 0.46% TS, pH 7.6.*

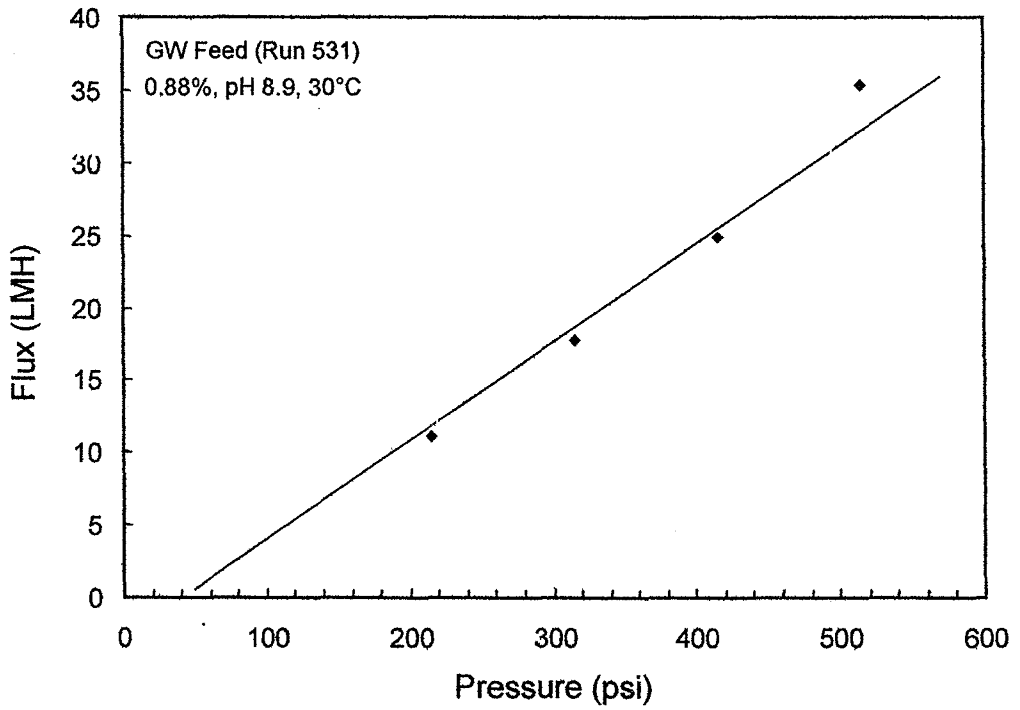


*Figure 77. Effect of pressure on flux of Guardian West feed at a cross-flow rate of 3 gpm (Run 531).*

### *3.5.3. Phase III*

The previous runs had been done in a batch recycle mode of operation. In this phase of the pilot-plant studies, the performance of the reverse osmosis system was studied in a continuous mode of operation at a concentration factor of 3X as a prelude to the final trial in the plant. The feed was electroplating waste from the Guardian West plant in Urbana. Three separate loads of 100-500 gallons each were collected at different times from the plant. Fouling (short term and long term) and performance (flux and rejection) were studied. Table 15 lists the experiments and the total solids and pH of the feeds.

In a continuous feed-and-bleed mode of operation, the permeate is removed from the system and the retentate is bled out from the system at a rate to achieve the desired concentration factor (X). With our pilot plant, typical steady state fluxes at 3X are about 20 liters per square meter per hour (LMH) which results in permeate flows of 54 liters per hour. For X =3, this means a total feed flow of 81 liters per hour. A 24-hour run would require more than 500 gallons per day of feed.

To reduce the volume of feed that must be brought over to our laboratory from the plant, Runs 531 and 604 were operated as pseudo feed-and-bleed runs. Run 531 was run as a "fed-batch" batch recycle. After a short three-hour period of "pre-fouling" at 200 psi (not shown here), the pressure was increased to 515 psi (Figure 77). The feed had an osmotic pressure of 54.7 psi as determined by extrapolation of the flux-pressure data. Fresh feed was concentrated 3X in a batch mode and then the system operated in batch recycle with both permeate and retentate recycled to the feed tank. This is shown in Figure 78. After a certain time period (indicated by the arrows in Figure 78), the retentate was removed and the feed tank emptied to the minimum possible volume. Fresh feed was then added, concentrated to 3X and the system operated in batch recycle mode again. Flux for this run is shown in Figure 78 and the total solids and pH are shown in Figures 79 and 80. There was no trace of solids in the permeate and thus rejection of the solids was 100%.

Run 604 was operated in a similar fed-batch mode, except that the system was operated in a feed-and-bleed mode. After 3 hours of prefouling at 200 psi, the pressure was increased to 515 psi. Flux data is shown in Figure 81. The osmotic pressure of this feed, even though it had almost

*Table 15. Phase III trials with electroplating waste from Guardian West plant*

| | **Run Number** | | |
|---|---|---|---|
| | **531** | **604** | **609** |
| Total solids of feed (%) | 0.88 | 0.86 | 0.86 |
| pH of feed | 8.9 | 9.0 | 8.8 |
| Fouling study | Long-term (76 hours) | Long-term (100 hours) | Long-term (24 hours) |
| Operating mode | Fed-Batch batch recycle at 3X | Fed-Batch feed-and-bleed at 3X | Continuous feed-and-bleed at 3X |
| Performance study | Yes | Yes | No |

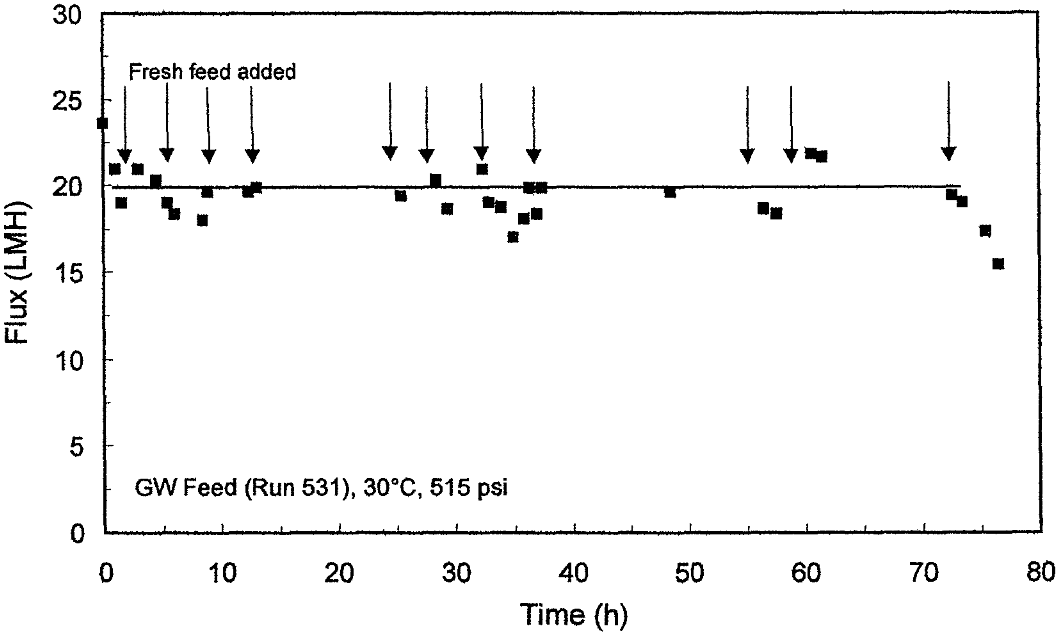


*Figure 78. Flux during "Fed Batch" batch recycle operation at 3X. Arrows show when permeate was removed and fresh feed was added (Run 531 at 515 psi, 3 gpm, 30°C).*

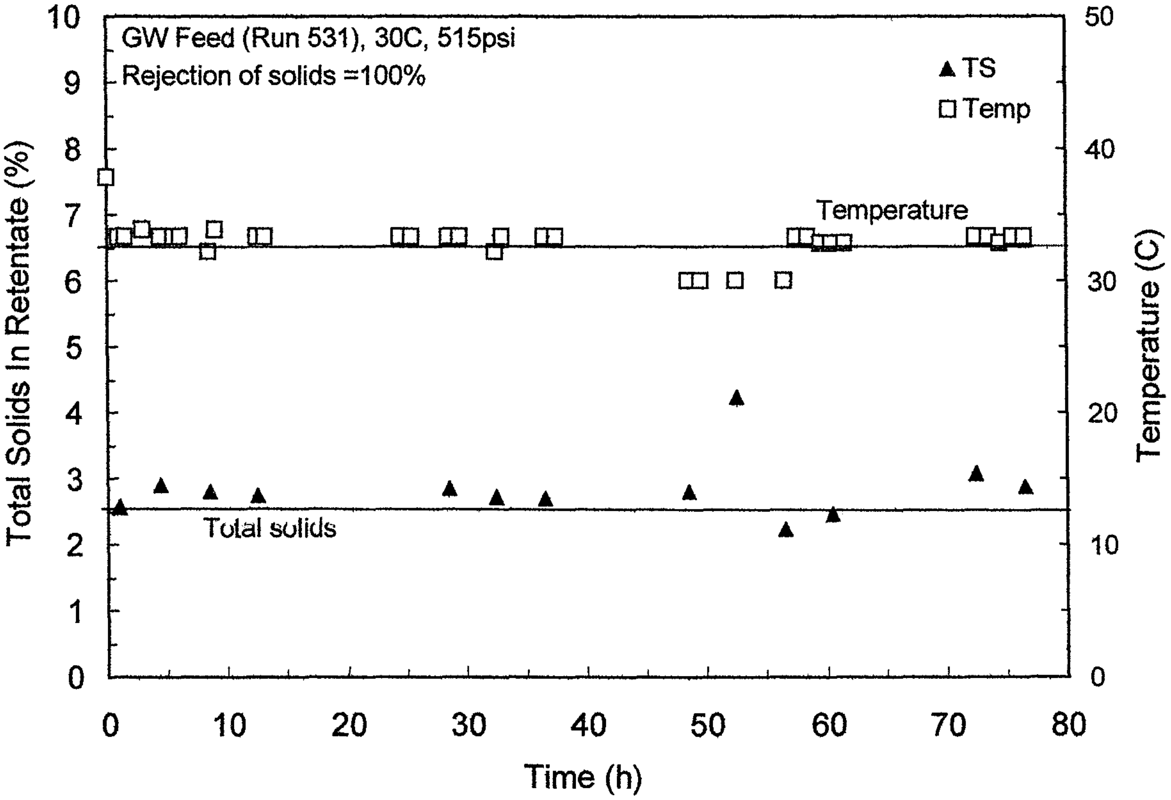


*Figure 79. Total solids and temperature during "Fed Batch" batch recycle operation at 3X. Same run as Figures 78 and 80.*

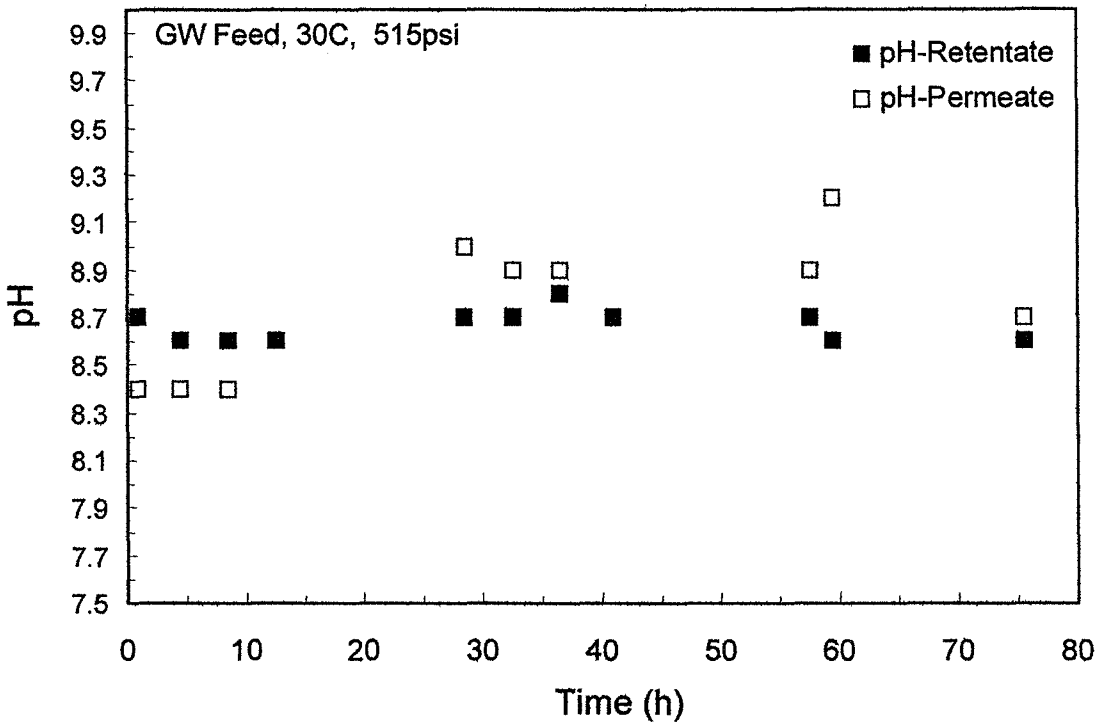


*Figure 80. pH during "Fed Batch" batch recycle operation at 3X. Same run as Figures 78 and 79.*

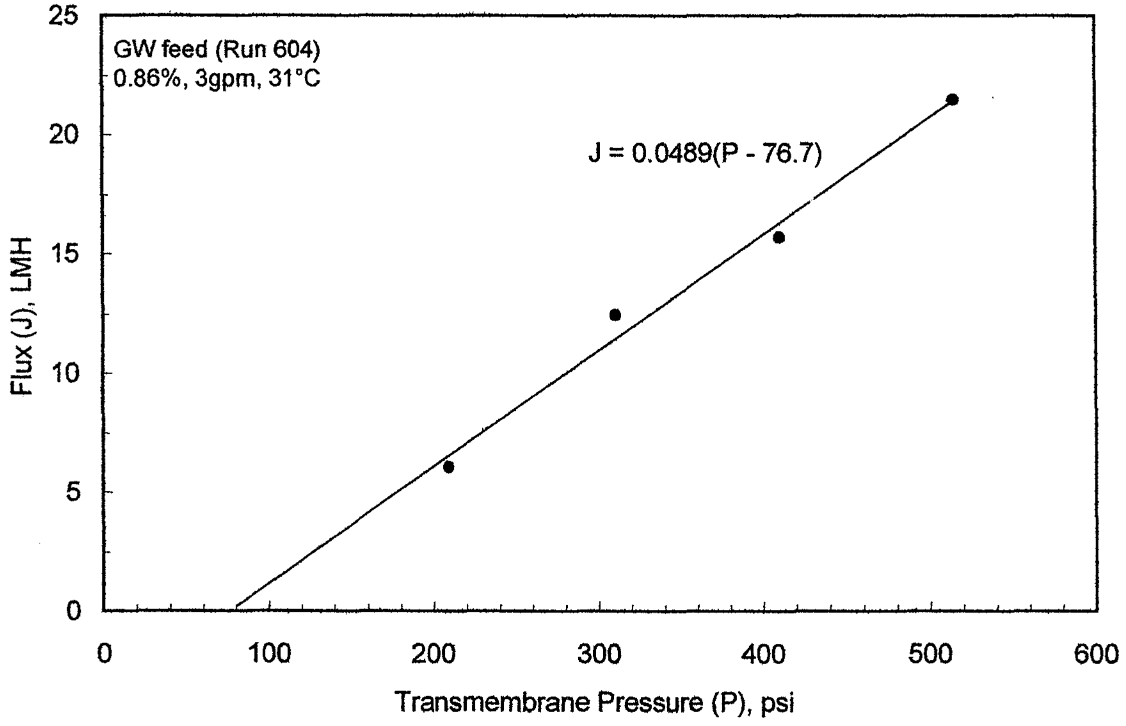


*Figure 81. Effect of pressure on flux of Guardian West feed at a cross-flow rate of 3 gpm (Run 604).*

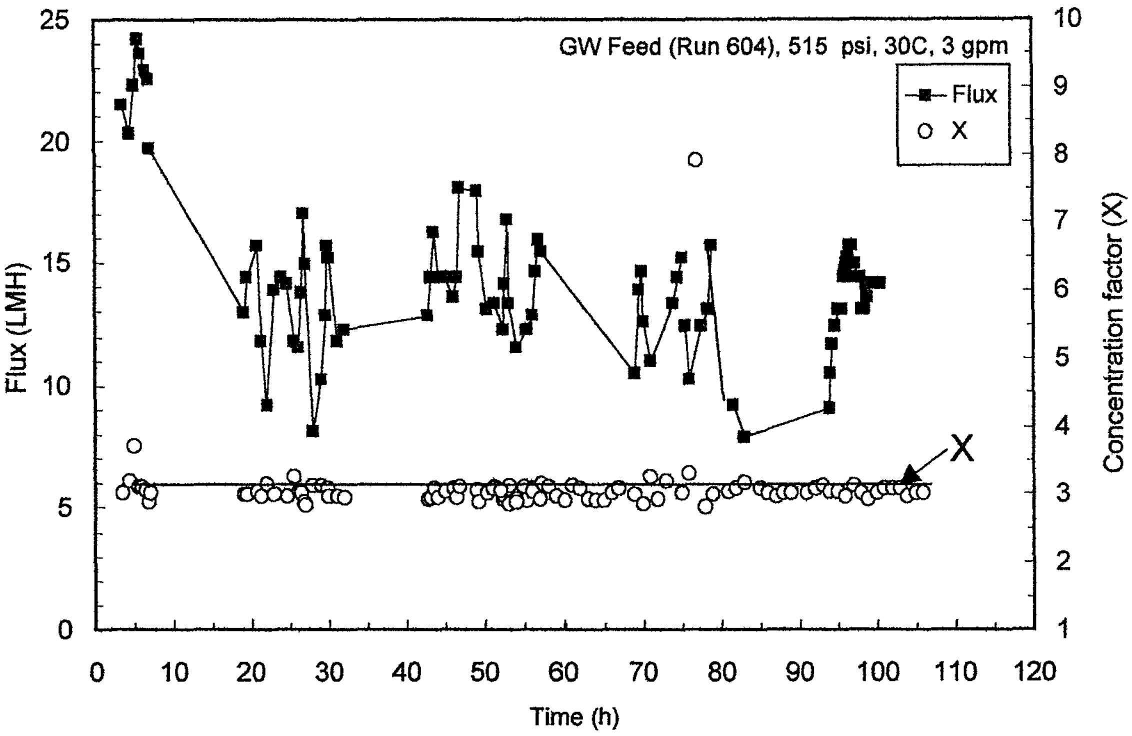


*Figure 82. Flux during "Fed Batch" feed-and-bleed operation at 3X.(Run 604 at 515 psi, 3 gpm, 30°C).*

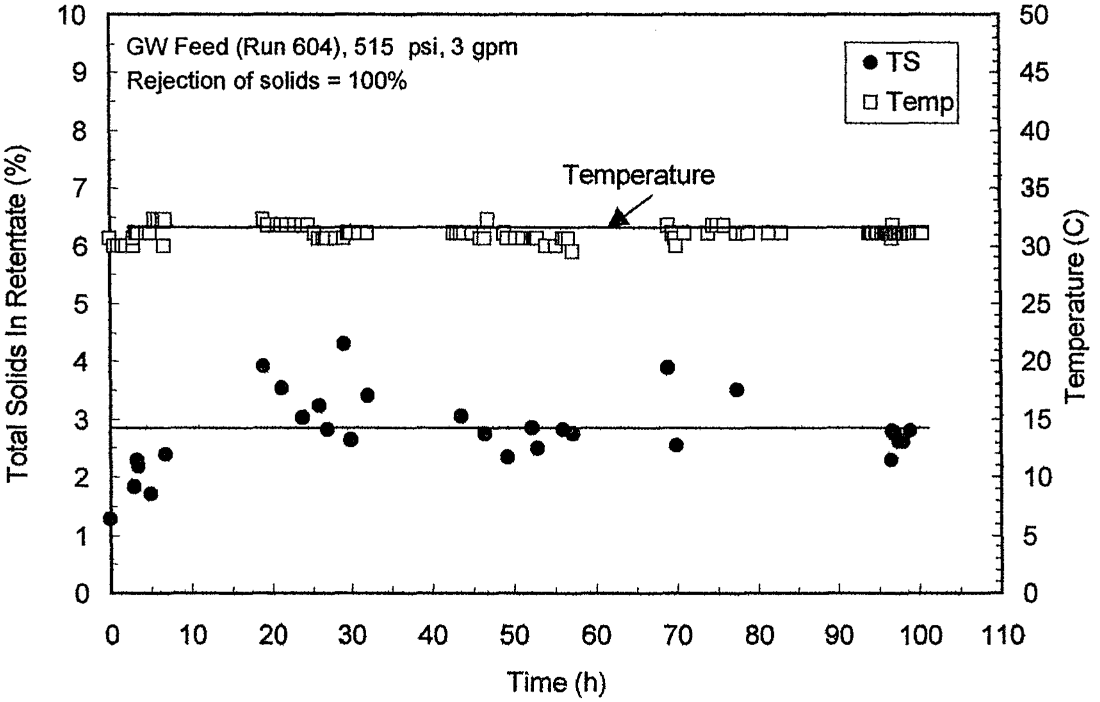


*Figure 83. Total solids and temperature during "Fed Batch" feed-and-bleed at 3X. Same run as Figures 82 and 84.*

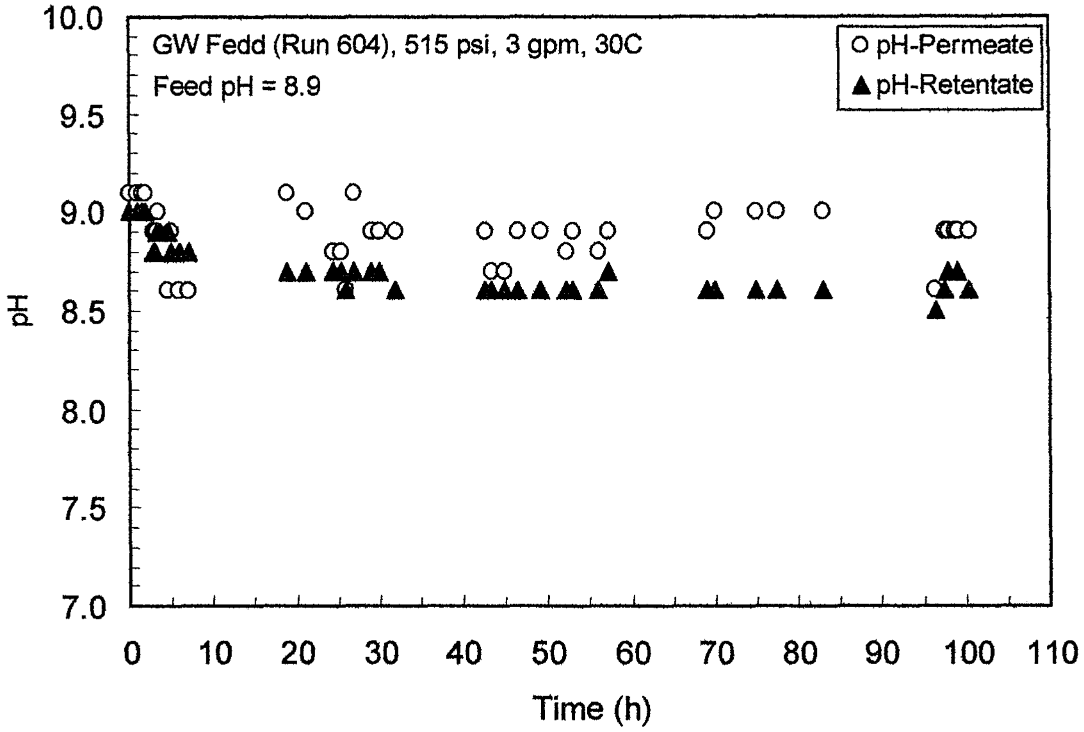


*Figure 84. pH during "Fed Batch" feed-and-bleed at 3X. Same run as Figures 82 and 83.*

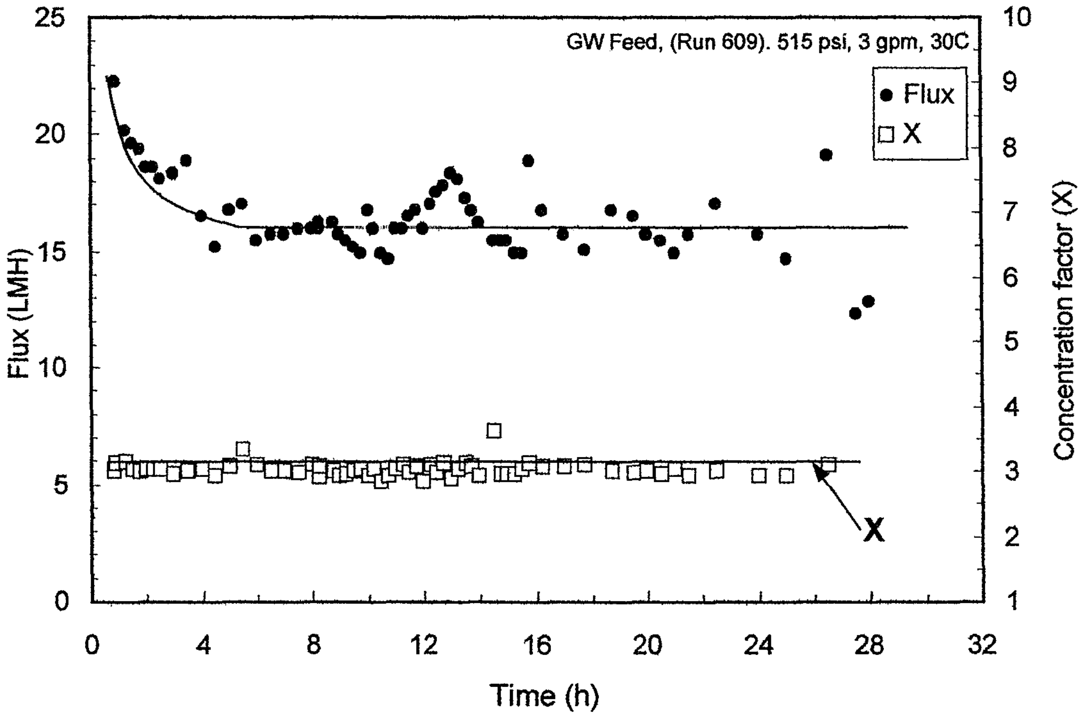


*Figure 85. Flux during continuous feed-and-bleed at 3X with no recycle. (Run 609 at 515 psi, 3 gpm, 30°C).*

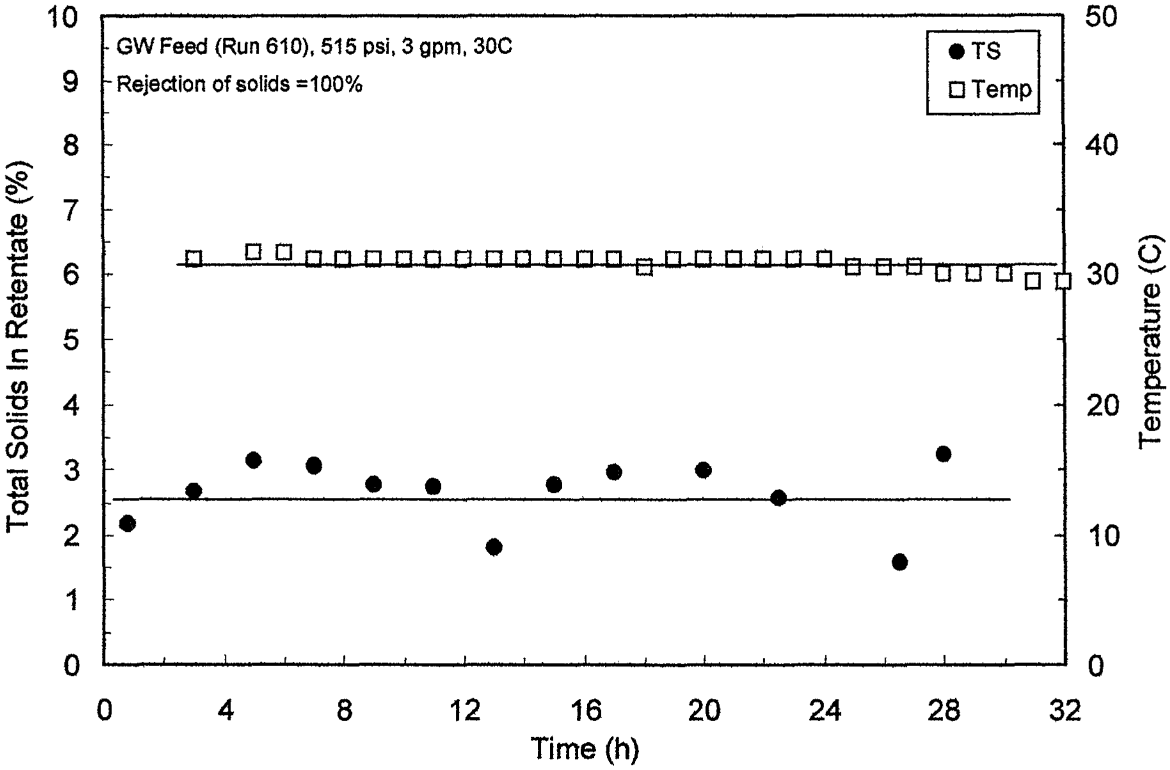


*Figure 86. Total solids and temperature during continuous feed-and-bleed at 3X. Same run as Figures 85 and 87.*

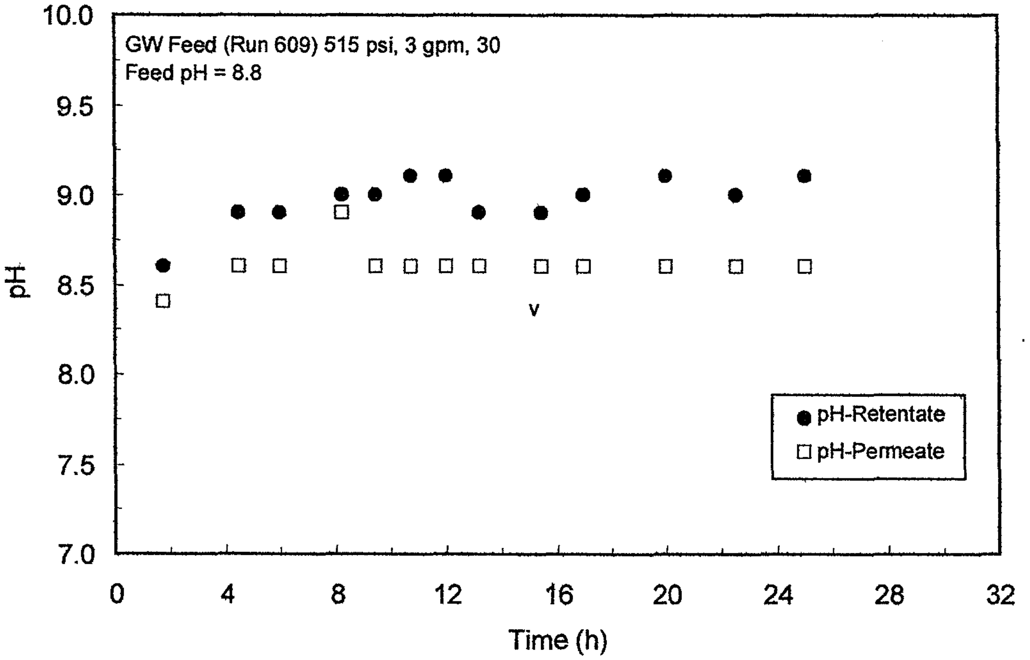


*Figure 87. pH during continuous feed-and-bleed at 3X. Same run as Figures 85 and 86.*

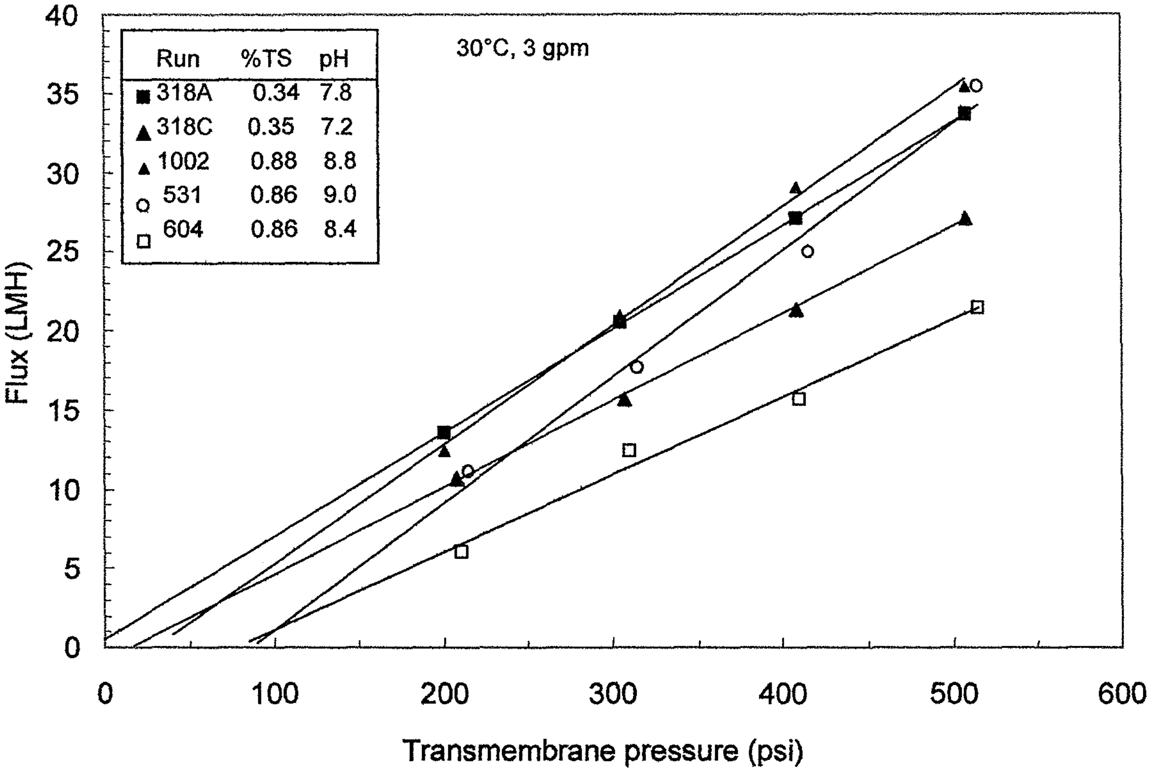


*Figure 88. Comparison of feeds obtained at various times from Guardian West.*

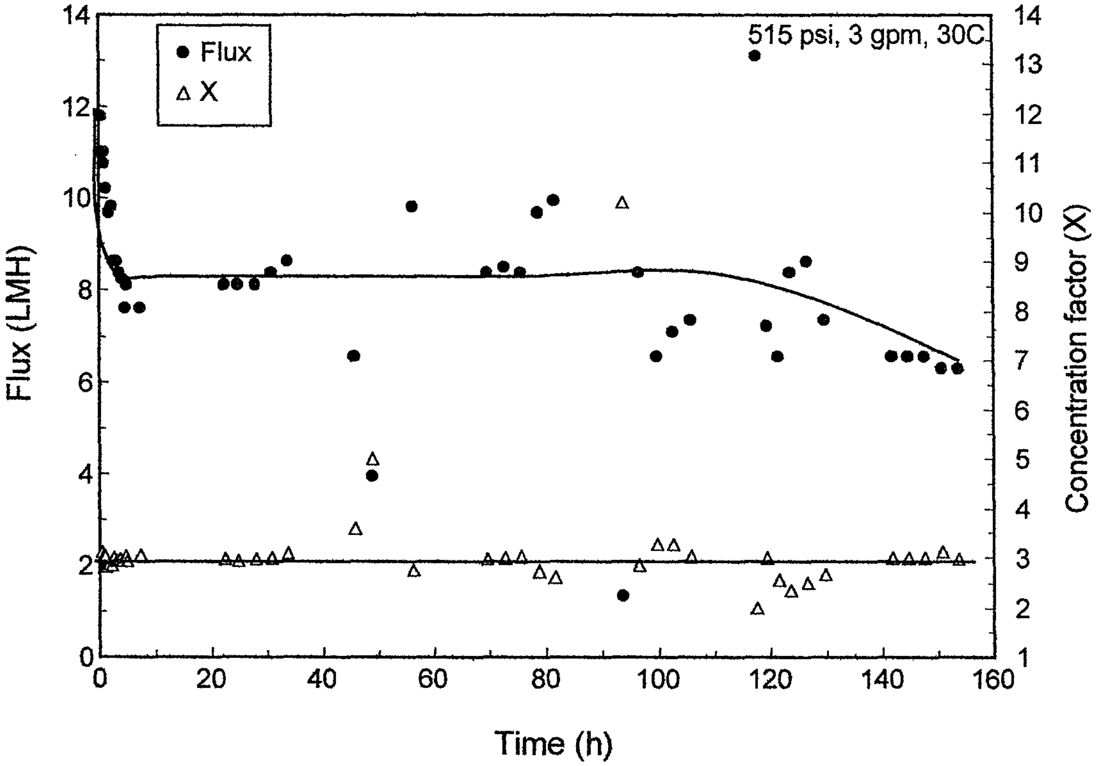


*Figure 89. Flux during continuous feed-and-bleed at 3X. Week 1 of on-site trial. (515 psi, 3 gpm, 30°C).*

the same total solids and pH as Run 531, was estimated as 76.7 psi. The retentate bleed valve was then opened and adjusted to get a X value of 3. Normally, the retentate and permeate would be discarded from the system, but to save on feed, they were instead recycled to the feed tank. At frequent intervals, the retentate and permeate would be removed from the system. When the volume in the feed tank had reached the minimum holdup, fresh feed would be pumped in and the cycle repeated.

Data for Run 604 is shown in Figures 82-84. However, when the retentate and permeate were recycled, the flux would drop rather steeply (even though all operating conditions were kept constant). When fresh feed was added, the flux would increase. This phenomenon is reflected in the spikes in the flux data shown in Figure 7.

Run 609 was operated in the true feed-and-bleed mode. Due to the logistics and difficulty of bringing 55-gallon drums of feed from the plant to our laboratory, a 24-hour run was planned with about 600 gallons of feed.

Figures 85-87 show data from this run. An average flux of 15 LMH was obtained and the average total solids in the retentate was 2.8%. No solids were observed in the permeate from this or any other run in this period. Interestingly, the pH of the permeate was lower than the retentate in this run (Figure 12) in contrast to the previous run (Figure 9).

Figure 88 is a comparison of six runs with the Guardian West feed. Although the general trends are consistent, there is no definite correlation between the total solids, pH and flux.

### 3.6. Guardian West electroplating waste streams: On-site trials

For the in-plant trials, the RO system had to be further modified to use the portable high-pressure 10 gpm Wanner pump, a new frame was needed to hold the module and heat exchanger, and several new valves and controls were needed for continuous 24/7 operation. A larger 100-gallon feed tank and a level controller was added in the feed tank to prevent overflowing and to ensure a steady feed to the RO system. To enable better control of the retentate bleed, a peristaltic pump was attached to the retentate valve instead of the manual valve that had been used thus far. Additional personnel were trained since the system was to be operated on a 24/7 basis.

The reverse osmosis pilot system was transported from our laboratory and installed at the Guardian West plant. An in-line prefilter fitted with a 25-micron stainless steel screen was also needed to remove suspended matter in the feed A 35-foot 1" diameter plastic hose was used to siphon the feed from the storage tank to the system feed tank. Photographs of the system as installed in the plant are shown in the Appendix (Figures A1-A8). With the cooperation of plant personnel, the system was installed and utility connections done within one week of arrival, except for a hot water connection. Without hot water to clean the membrane, the only way to clean the membrane was to remove the membrane and housing and take it back to our pilot plant at the university and install it in the other RO system in our laboratory. After cleaning the module, it was reinstalled in the system in Guardian West. This was done on a weekly basis.

The data are shown in Figures 89-103. Since the module was cleaned once a week, the

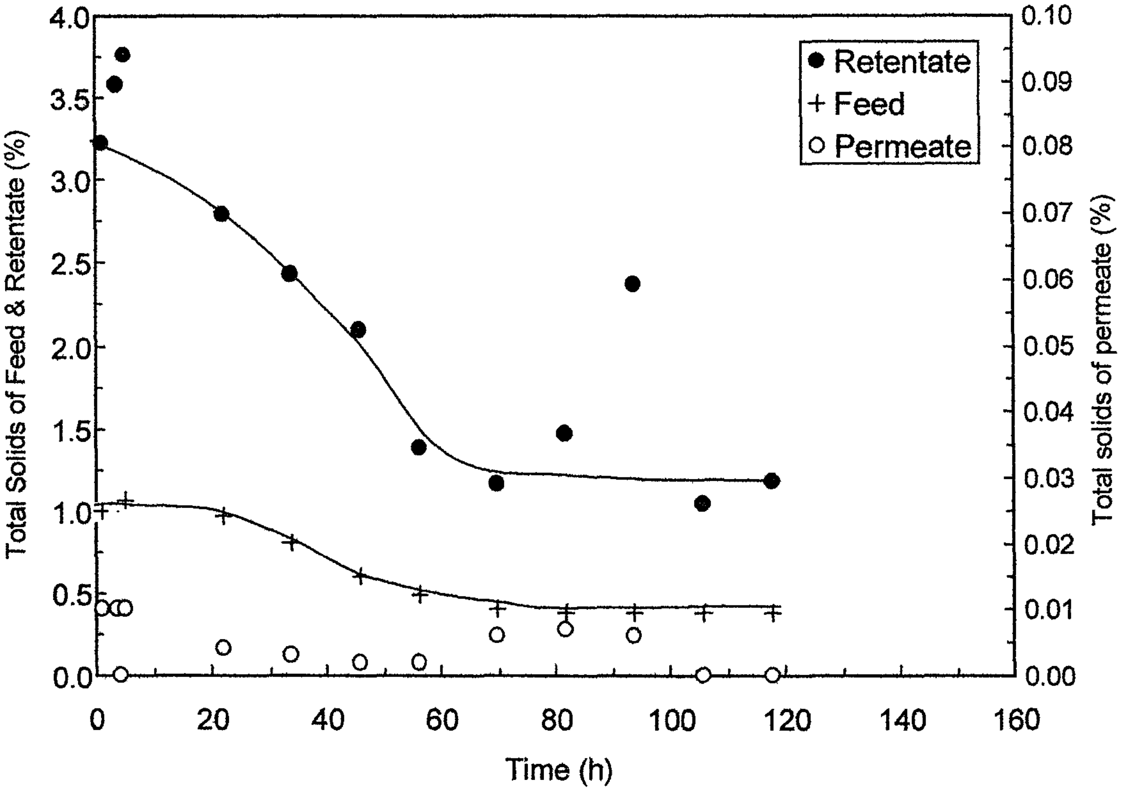


*Figure 90. Total solids during continuous feed-and-bleed at 3X. Week 1 of on-site trial. Same run as Figures 89 and 91.*

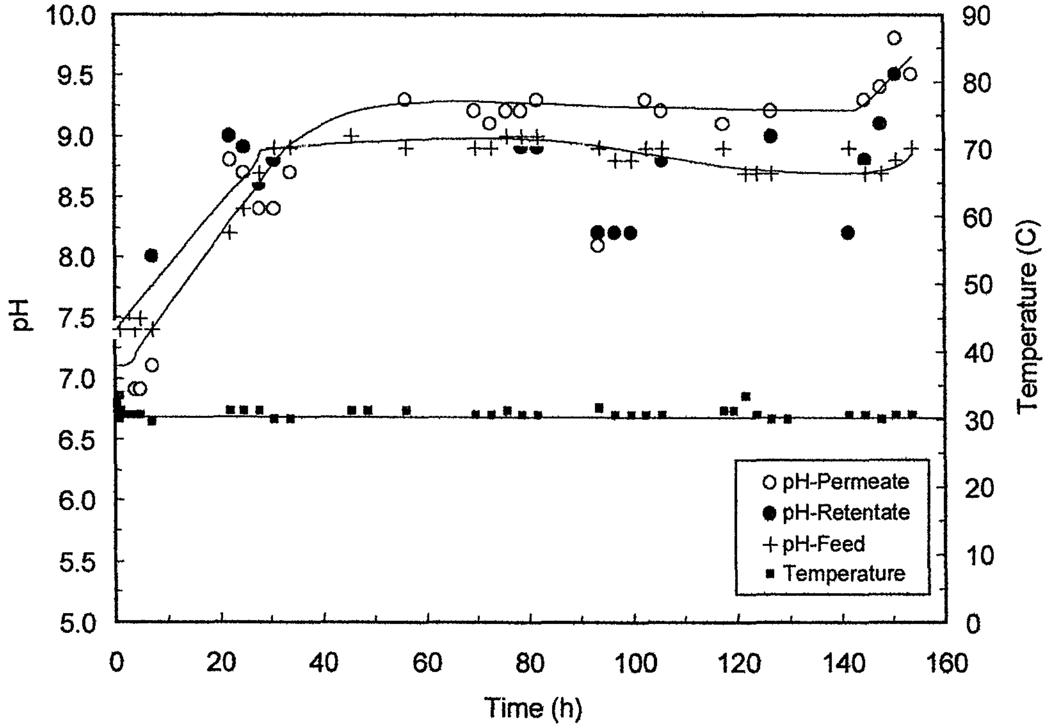


*Figure 91. pH and temperature during continuous feed-and-bleed at 3X. Week 1 of on-site trial. Same run as Figures 89 and 90.*

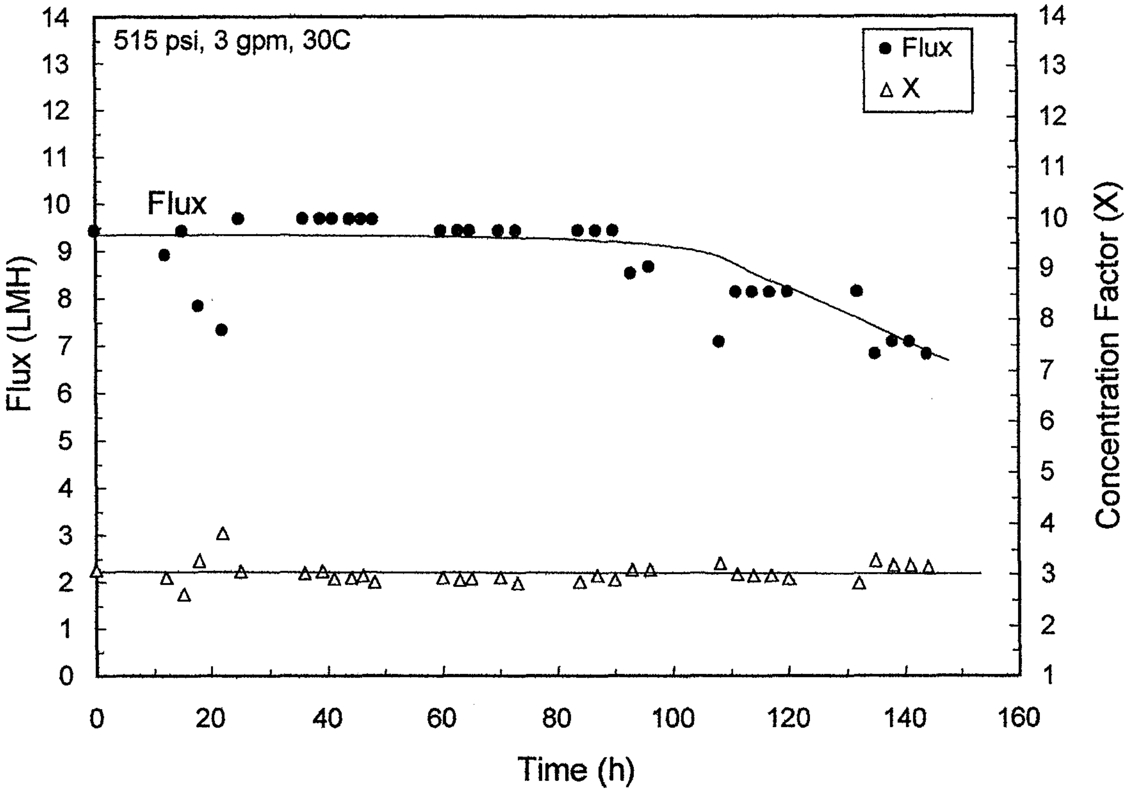


*Figure 92. Flux during continuous feed-and-bleed at 3X. Week 2 of on-site trial.*

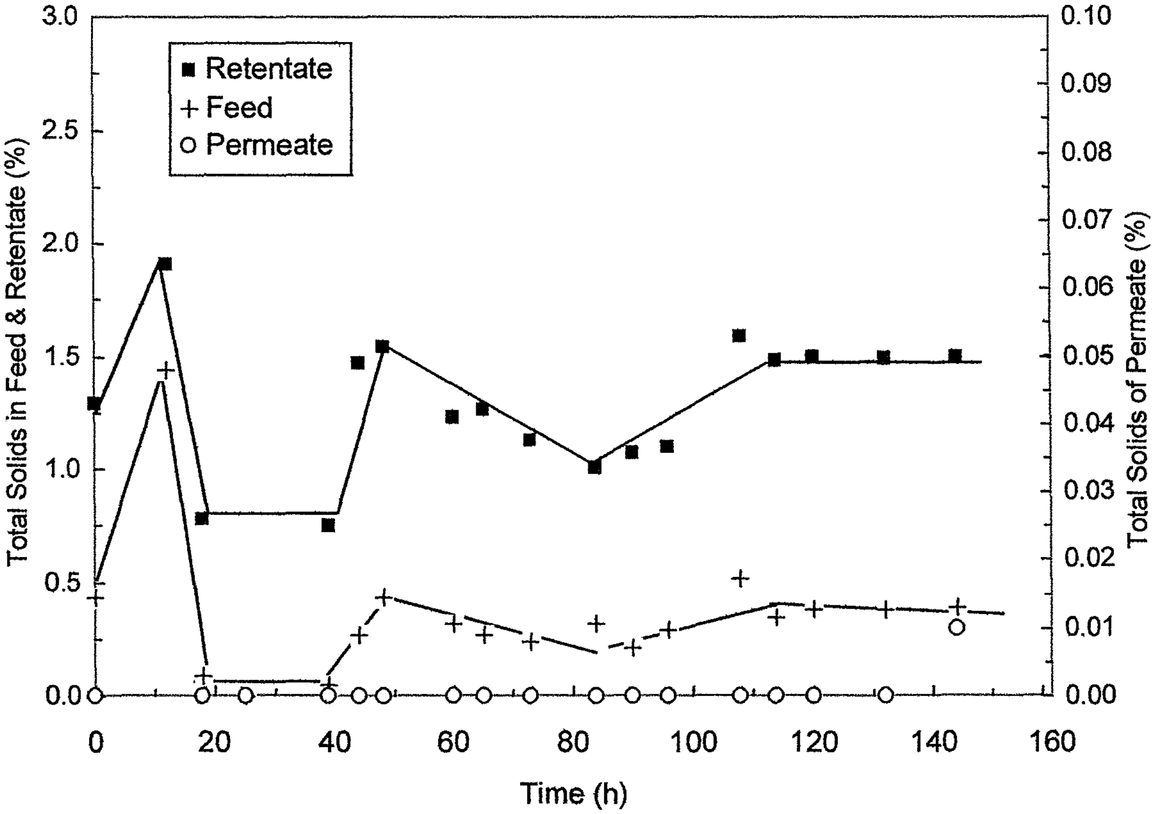


*Figure 93. Total solids during continuous feed-and-bleed at 3X. Week 2 of on-site trial. Same run as Figures 92 and 94.*

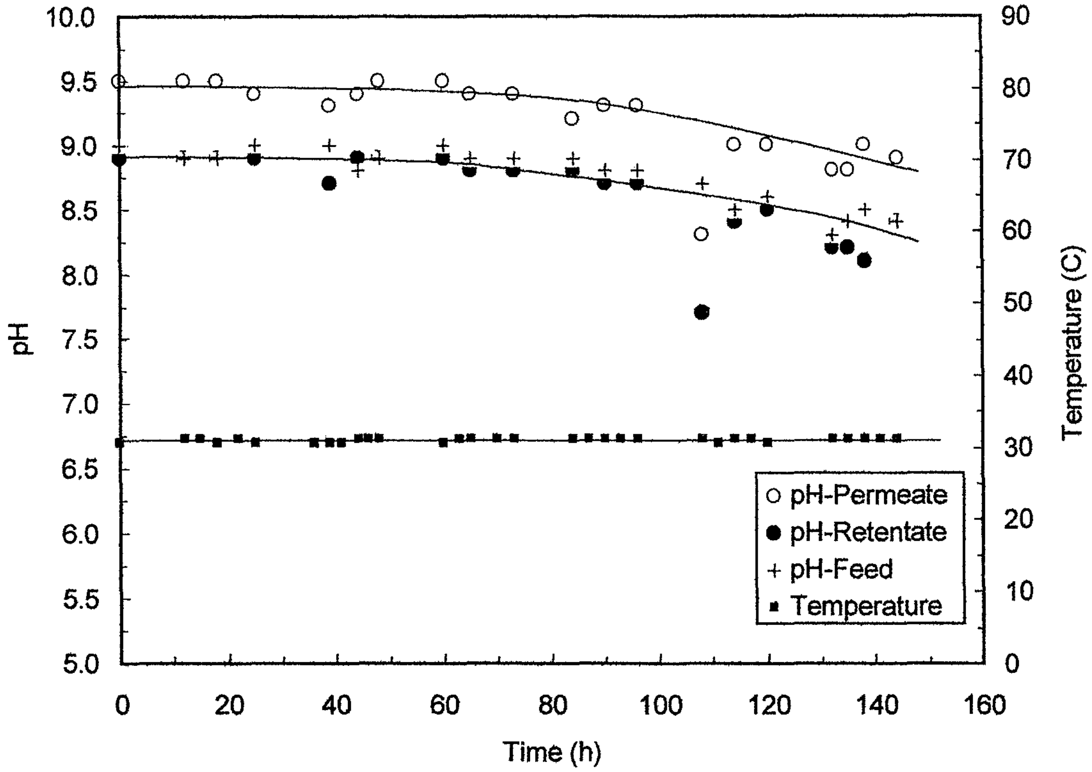


*Figure 94. pH and temperature during continuous feed-and-bleed at 3X. Week 2 of on-site trial. Same run as Figures 92 and 93.*

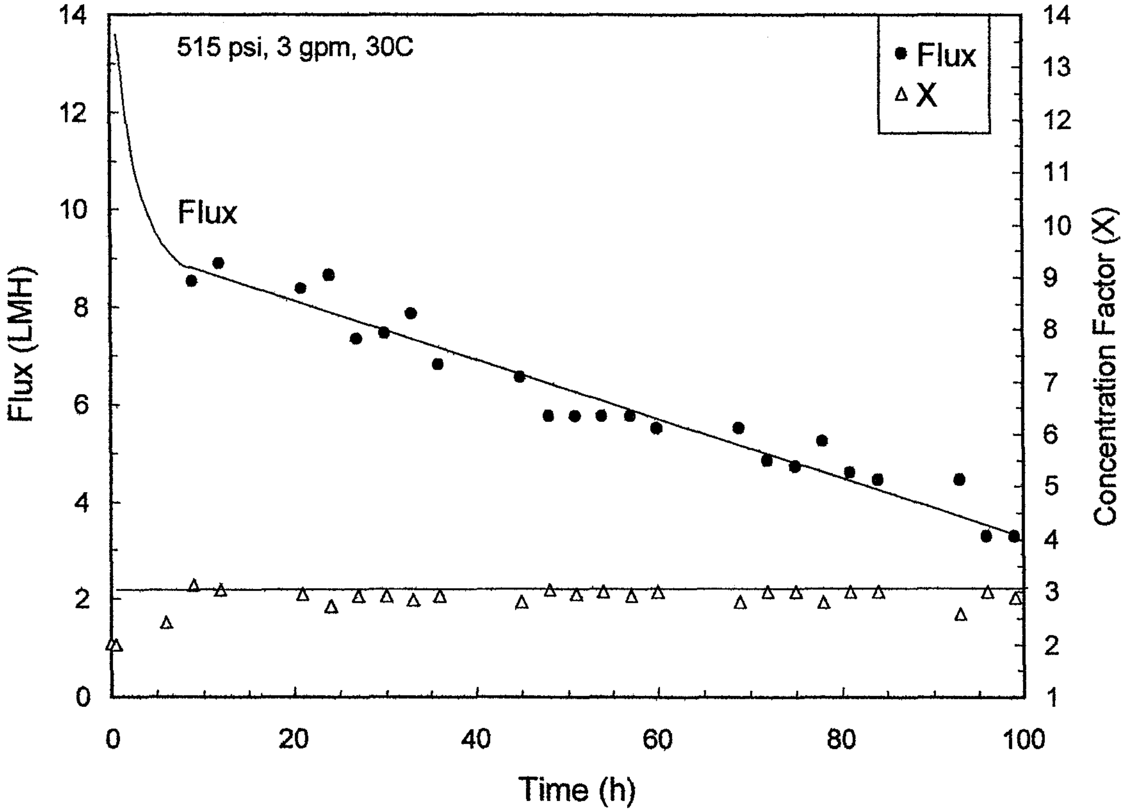


*Figure 95. Flux during continuous feed-and-bleed at 3X. Week 3 of on-site trial*

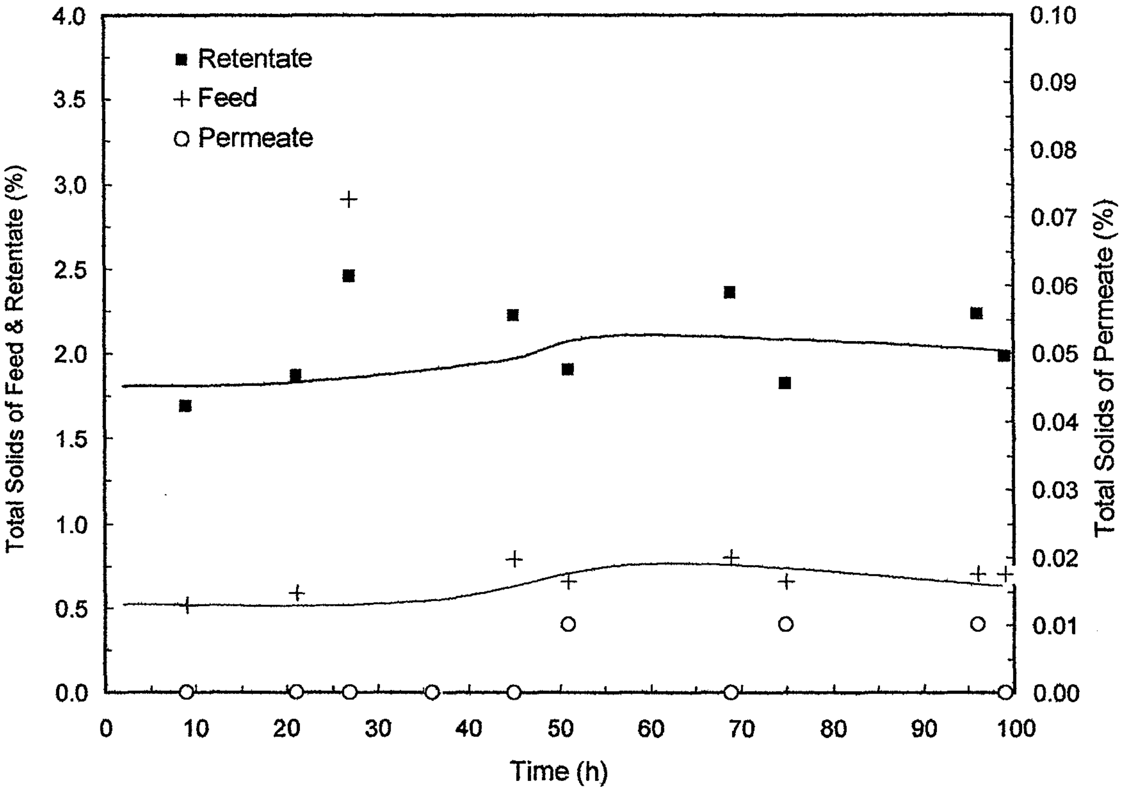


*Figure 96. Total solids during continuous feed-and-bleed at 3X. Week 3 of on-site trial. Same run as Figures 95 and 97.*

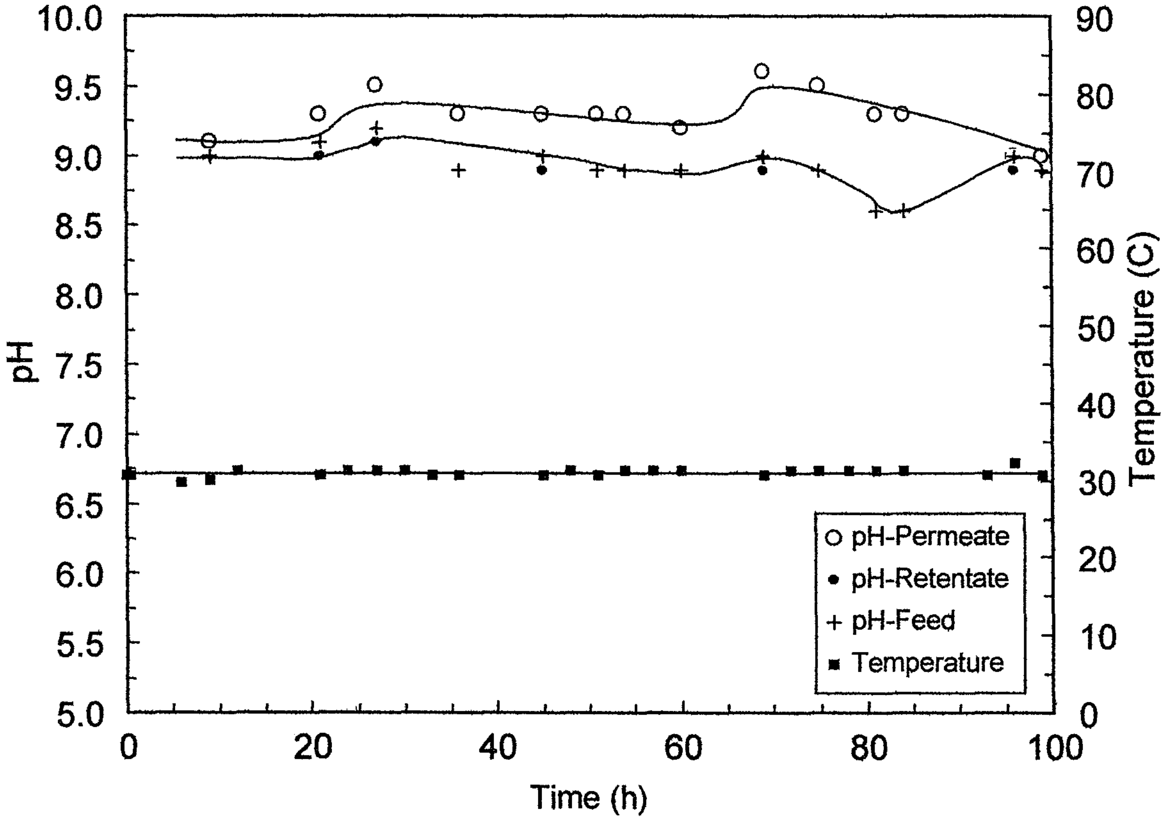


*Figure 97. pH and temperature during continuous feed-and-bleed at 3X. Week 3 of on-site trial. Same run as Figures 95 and 96.*

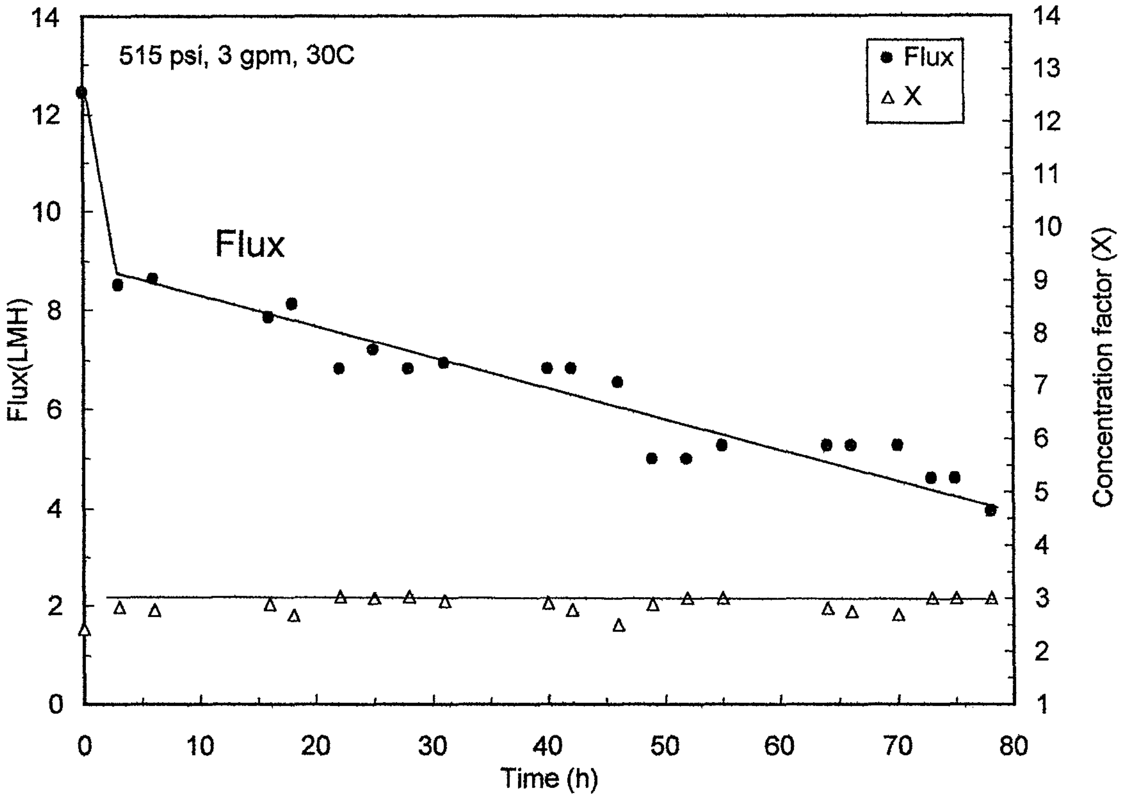


*Figure 98. Flux during continuous feed-and-bleed at 3X. Week 4 of on-site trial. (515 psi, 3 gpm, 30°C).*

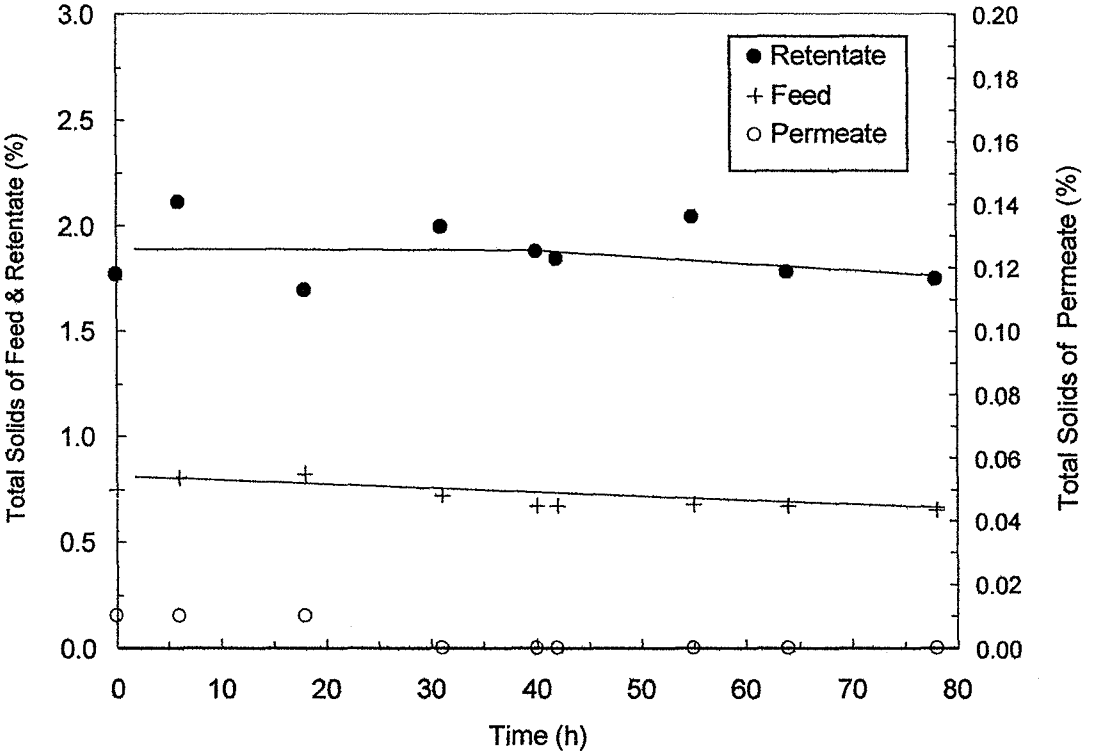


*Figure 99. Total solids and temperature during continuous feed-and-bleed at 3X. Week 4 of on-site trial. Same run as Figures 98 and 100.*

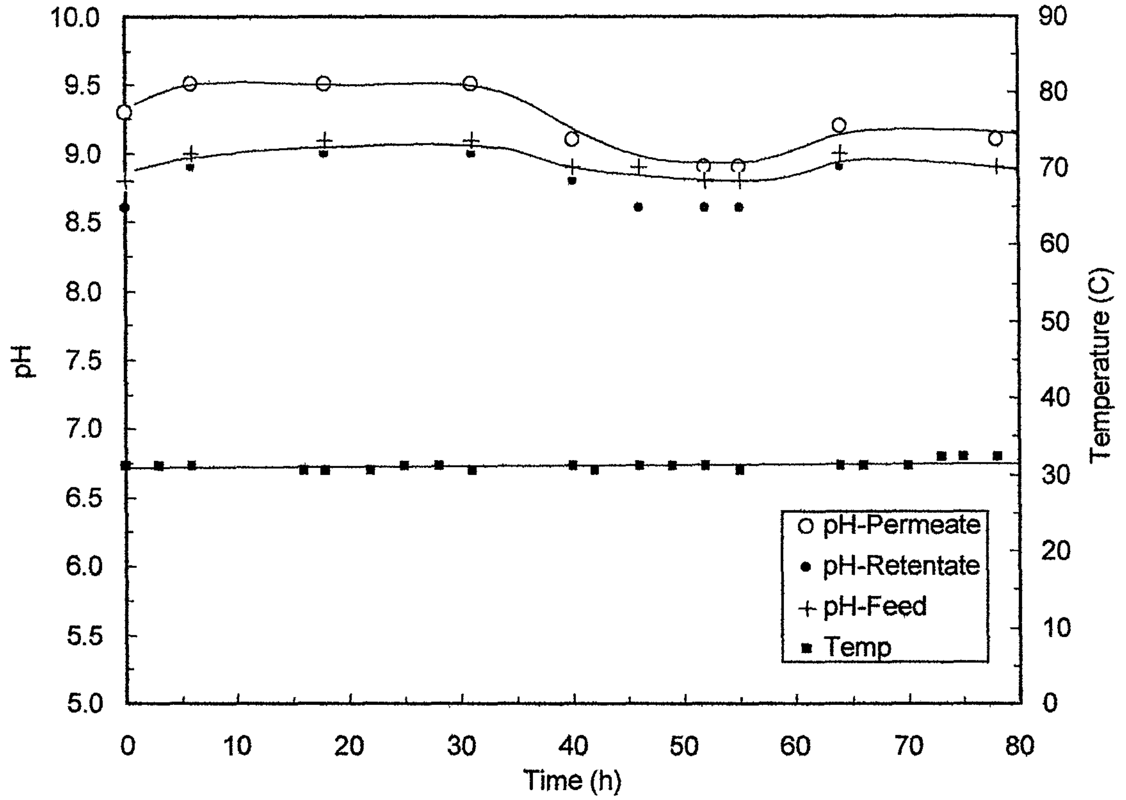


*Figure 100. pH during continuous feed-and-bleed at 3X. Week 4 of on-site trial. Same run as Figures 98 and 99.*

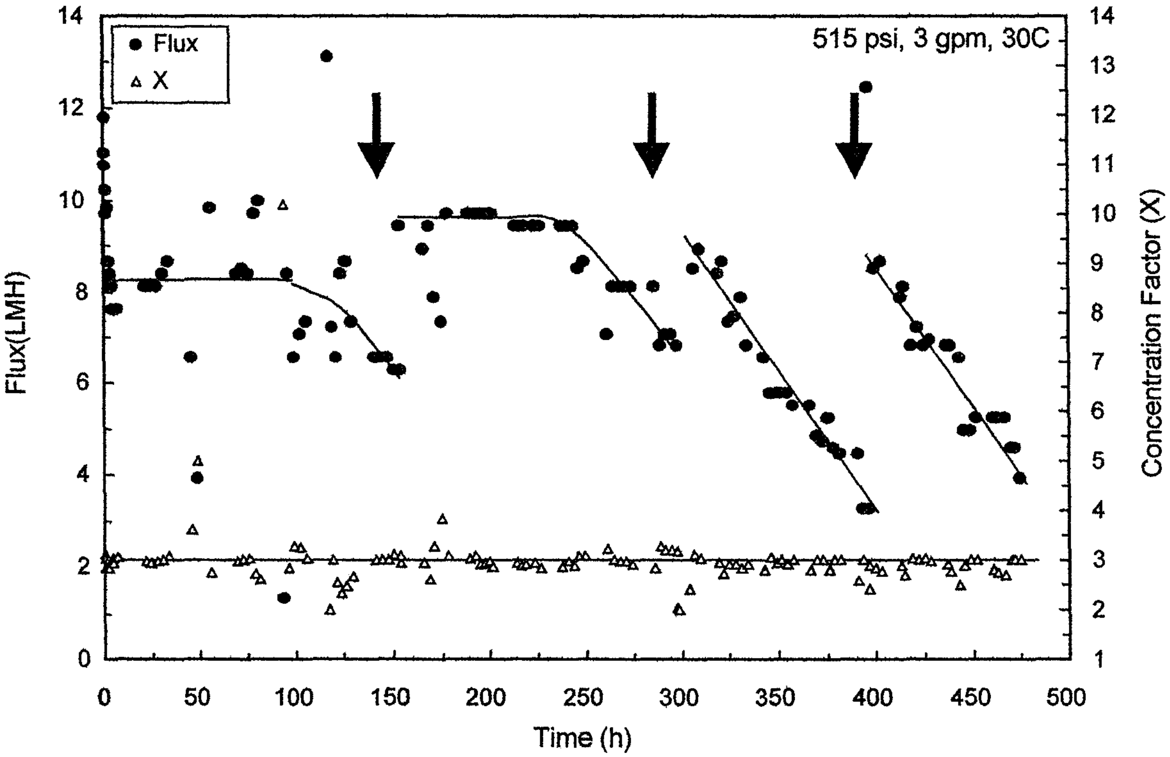


*Figure 101. Flux during continuous feed-and-bleed at 3X for all four weeks of on-site trial. (515 psi, 3 gpm, 30°C). Arrows show when the system was cleaned.*

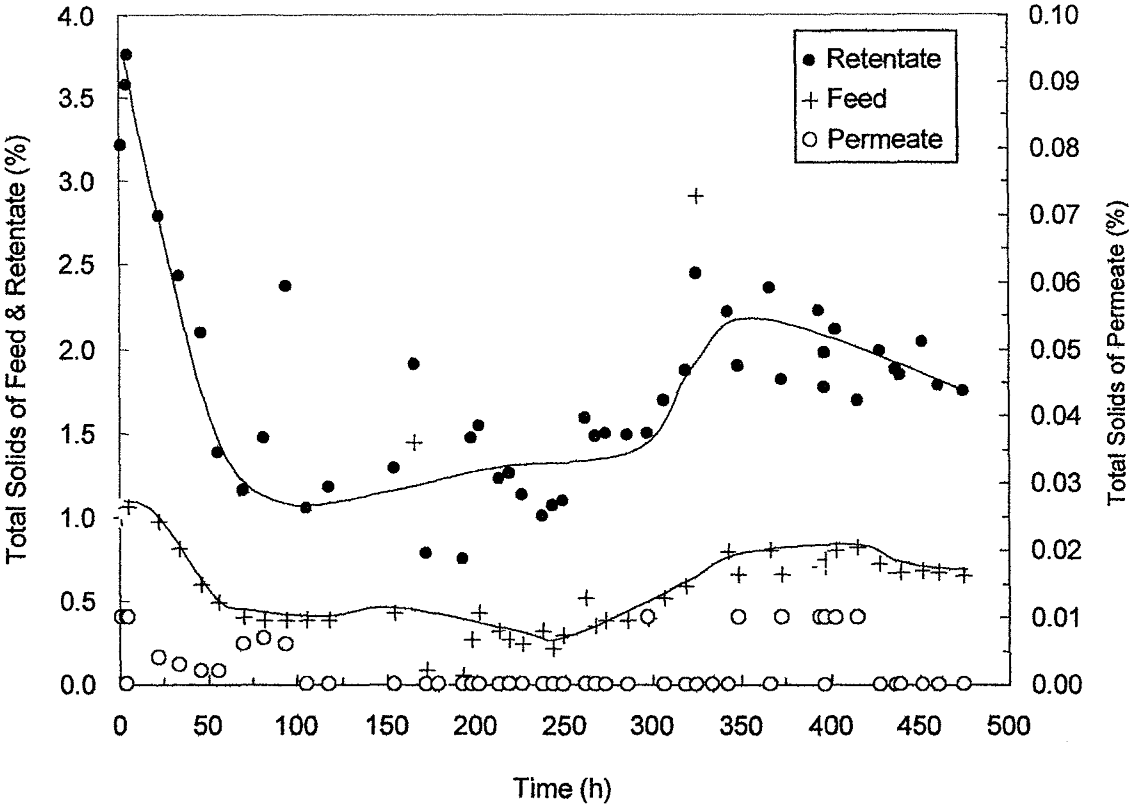


*Figure 102. Total solids of feed, retentate and permeate during continuous feed-and-bleed at 3X for all four weeks of on-site trial. Same runs as Figures 101 and 103.*

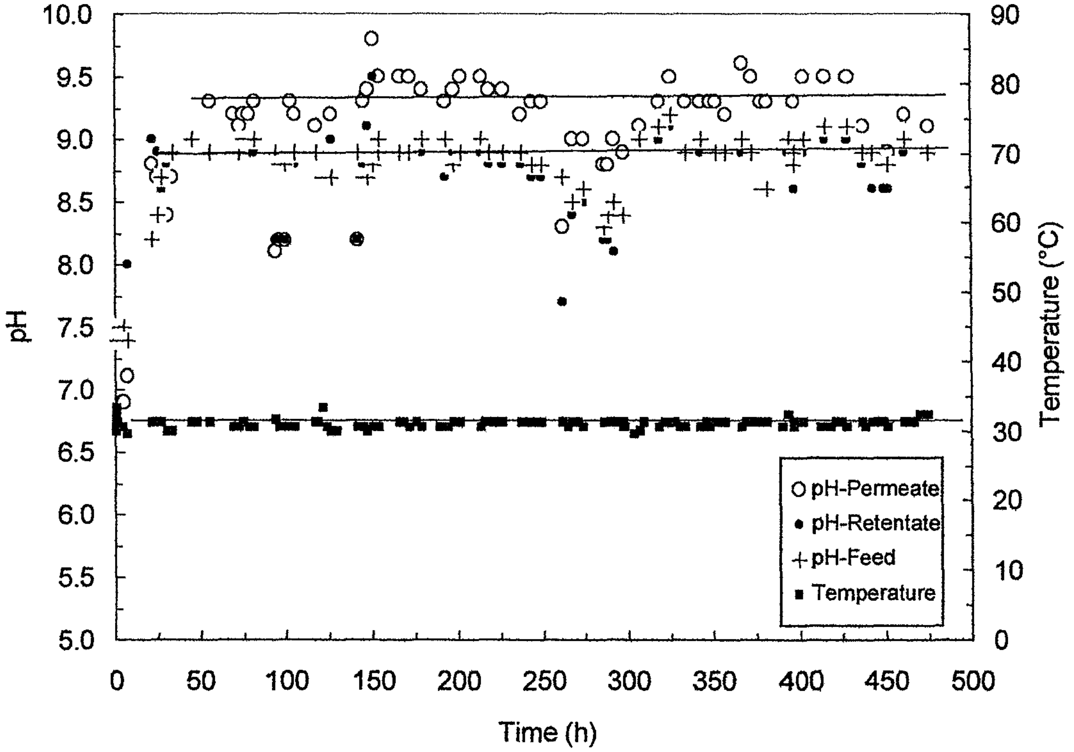


*Figure 103. pH and temperature during continuous feed-and-bleed at 3X for all four weeks of on-site trial. Same runs as Figures 101 and 102.*

data are reported on a weekly basis, e.g., Figures 89-92 show data from the first week. The system was operated in a continuous feed-and-bleed mode at a concentration factor of 3X. Since the feed properties (solids and pH) varied during the trial, the feed TS and pH are also shown in the graphs. Average flux was 3-10 LMH at 515 psi with feed concentrations of 0.5-1% TS. This flux seems to be low when compared to our results with the Guardian West feed done in our lab (as shown earlier in section 4.4), where we obtained 15 LMH with a feed concentration of 0.9% TS. However, the membrane module was cleaned daily in those trials, whereas the membrane was cleaned only once a week during the in-plant trials.

During the first week, the total solids in the permeate reached 0.01% (Figure 90). This is an unusually high level of permeate solids based on our past experience. Since this particular membrane module had been used for the past 18 months, we replaced it with a new module for weeks 2-4. As shown in Figures 93, 96 and 99, it improved the rejection of the solids although there were occasional spikes in the permeate solids (Figure 102).

The data for all four weeks is shown in Figures 101-103. There is considerable scatter in the data, which is partly a reflection of varying feed conditions. However, on average it appeared that the flux remained steady in the first week and in the first half of the second week, but showed fairly steep decreases in flux in the second half of the second week and in weeks 3 and 4. The steady portions could be because during those periods, the retentate and permeate were recycled for considerable periods of time due to difficulties in maintaining a good siphon from the main waste tank to our feed tank. Thus there was little fresh feed coming in during those periods. The feed siphon system was fixed in the second half of the second week, and from then there was no recycle of retentate and permeate. Thus the second half of the second week, the third and fourth weeks are more representative of a typical feed-and-bleed operation with fresh feed coming in to the system at all times. This brought in fresh fouling solids continuously which resulted in the continuous decline in flux observed in the later periods in Figure 101.

As shown in Figure 103, the pH of the permeate was higher than the retentate which is typical of what we had observed previously. The temperature remained reasonably steady at about 30°C despite the limited availability of hot and cold water.

The main problem was a lack of adequate amounts of fresh hot water to clean the membrane, as discussed earlier. This essentially meant an interruption of about one day per week while we cleaned the module. In commercial systems, a CIP (clean-in-place) system will be included that will obviate this problem. It was also observed that despite the 25-micron prefilter, some suspended particles were still being trapped at the inlet of the spiral membrane module. A tighter prefilter or larger spacer in the membrane module may have to be used.

Tables 16 and 17 show the analysis of selective anions and cations during the first week of the on-site trials and Table 18 shows the total organic carbon (TOC). The experiments were conducted at a concentration factor of approximately 3X, the retentate is approximately 3 times higher in concentration of the individual ions. The permeate quality is fairly good even though this membrane had been used for several months prior to this run. The ion and TOC levels are consistent with the data obtained earlier (Tables 4-7 and 11-13).

*Table 16: Cations in Guardian West electroplating waste stream during Week 1 of on-site trials.*

| Elapsed Time (hours) | Barium (mg/L) | | | Calcium (mg/L) | | | Chromium (mg/L) | | |
|---|---|---|---|---|---|---|---|---|---|
| | **Feed** | **Ret.** | **Perm.** | **Feed** | **Ret.** | **Perm.** | **Feed** | **Ret.** | **Perm.** |
| 2 | 0.035 | 0.09 | <0.01 | 51 | 150 | <1 | 3.1 | 11 | 0.054 |
| 6 | 0.035 | 0.10 | 0.026 | 48 | 180 | <1 | 3.5 | 12 | 0.08 |
| 23 | 0.022 | 0.05 | <0.01 | 18 | 33 | <1 | 2.1 | 8.1 | 0.04 |
| 35 | 0.020 | 0.07 | <0.01 | 14 | 36 | <1 | 1.4 | 4.2 | 0.01 |
| 47 | 0.055 | 0.17 | <0.01 | 17 | 58 | <1 | 1.0 | 2.9 | 0.05 |
| 57 | 0.069 | 0.18 | <0.01 | 15 | 42 | <1 | 0.94 | 2.2 | 0.05 |
| 71 | 0.085 | 0.24 | 0.01 | 17 | 49 | <1 | 0.39 | 1.2 | 0.15 |
| 93 | 0.092 | 0.35 | <0.01 | 19 | 70 | <1 | 0.11 | 0.43 | <0.01 |
| 105 | 0.086 | 0.39 | <0.01 | 18 | 99 | <1 | 0.24 | 0.61 | 0.027 |
| 117 | 0.100 | 0.11 | <0.01 | 18 | 34 | <1 | 0.29 | 0.33 | <0.01 |
| 129 | 0.088 | 0.07 | 0.034 | 18 | 27 | <1 | 0.16 | 0.43 | 0.04 |
| 141 | 0.085 | 0.08 | <0.01 | 18 | 23 | <1 | 0.24 | 4.4 | 0.03 |
| 153 | 0.081 | 0.08 | <0.01 | 18 | 23 | <1 | 0.12 | 6.7 | 0.04 |

*Table 16 (continued): Cations in Guardian West stream during Week 1 of on-site trials.*

| Elapsed Time (hours) | Copper (mg/L) | | | Magnesium (mg/L) | | | Nickel (mg/L) | | |
|---|---|---|---|---|---|---|---|---|---|
| | **Feed** | **Ret.** | **Perm.** | **Feed** | **Ret.** | **Perm.** | **Feed** | **Ret.** | **Perm.** |
| 2 | 0.065 | 0.21 | 0.01 | 5.6 | 16 | 0.18 | 0.41 | 1.4 | 0.033 |
| 6 | 0.065 | 0.18 | <0.01 | 5.0 | 17 | 0.17 | 0.50 | 1.7 | 0.056 |
| 23 | 0.040 | 0.13 | <0.01 | 8.1 | 18 | 0.18 | 0.45 | 1.7 | 0.026 |
| 35 | 0.035 | 0.090 | <0.01 | 6.3 | 21 | 0.23 | 0.48 | 1.3 | <0.01 |
| 47 | 0.022 | 0.063 | <0.01 | 14 | 45 | 0.16 | 0.89 | 1.9 | 0.046 |
| 57 | 0.020 | 0.041 | <0.01 | 12 | 34 | 0.30 | 0.92 | 1.3 | 0045 |
| 71 | 0.013 | 0.033 | <0.01 | 16 | 47 | 0.27 | 0.63 | 1.3 | 0.13 |
| 93 | 0.016 | 0.053 | <0.01 | 24 | 89 | 0.16 | 0.57 | 1.7 | 0.016 |
| 105 | 0.016 | 0.068 | 0.013 | 21 | 103 | 0.27 | 0.91 | 3.3 | 0.065 |
| 117 | 0.019 | 0.03 | <0.01 | 21 | 38 | 0.23 | 0.91 | 1.2 | <0.01 |
| 129 | 0.016 | 0.041 | <0.01 | 20 | 36 | 0.22 | 0.71 | 1.2 | 0.053 |
| 141 | 0.015 | 0.063 | <0.01 | 20 | 21 | 0.21 | 0.77 | 0.95 | 0.03 |
| 153 | 0.013 | 0.057 | <0.01 | 20 | 10 | 0.35 | 0.52 | 0.94 | 0.04 |

*.....Table 16 continued next page*

*Table 16 (continued): Cations in Guardian West stream during Week 1 of on-site trials.*

| **Elapsed Time (hours)** | **Sodium (mg/L)** | | | **Zinc (mg/L)** | | |
|---|---|---|---|---|---|---|
| | **Feed** | **Ret.** | **Perm.** | **Feed** | **Ret.** | **Perm.** |
| 2 | 2900 | 8700 | 36 | 0.12 | 0.45 | <0.05 |
| 6 | 2800 | 9600 | 37 | 0.11 | 0.48 | <0.05 |
| 23 | 2500 | 8100 | 21 | 0.10 | 0.41 | <0.05 |
| 35 | 2100 | 5900 | 15 | 0.09 | 0.29 | <0.05 |
| 47 | 1500 | 5200 | 16 | 0.10 | 0.25 | <0.05 |
| 57 | 1300 | 3700 | 11 | 0.07 | 0.17 | <0.05 |
| 71 | 1100 | 2800 | 11 | 0.06 | 0.16 | <0.05 |
| 93 | 940 | 3500 | 13 | 0.05 | 0.16 | <0.05 |
| 105 | 970 | 5400 | 25 | 0.06 | 0.24 | <0.05 |
| 117 | 870 | 2500 | 9 | 0.06 | 0.13 | <0.05 |
| 129 | 970 | 2900 | 9 | 0.065 | 0.14 | <0.05 |
| 141 | 990 | 4000 | 14 | 0.06 | 0.18 | <0.05 |
| 153 | 950 | 3800 | 15 | 0.05 | 0.21 | <0.05 |

*Table 17: Anions in Guardian West stream during Week 1 of on-site trials.*

| **Elapsed Time (hours)** | **Chloride (mg/L)** | | | **Nitrate (mg/L)** | | | **Sulfate (mg/L)** | | |
|---|---|---|---|---|---|---|---|---|---|
| | Feed | Ret. | Perm. | Feed | Ret. | Perm. | Feed | Ret. | Perm. |
| 2 | 500 | 1500 | 16 | <2 | <2 | <0.02 | 6200 | 20000 | 64 |
| 6 | 490 | 1700 | 17 | <2 | <2 | <0.02 | 6300 | 22000 | 63 |
| 23 | 330 | 940 | 15 | <2 | <2 | <0.02 | 5900 | 17000 | 23 |
| 35 | 250 | 710 | 5 | <2 | <2 | <0.02 | 4900 | 15000 | 23 |
| 47 | 180 | 580 | 4 | <2 | <2 | <0.02 | 3600 | 13000 | 22 |
| 57 | 170 | 450 | 5 | <2 | <2 | <0.02 | 2900 | 8100 | 13 |
| 71 | 150 | 150 | 3 | <2 | <2 | <0.02 | 2400 | 7100 | 11 |
| 93 | 130 | 510 | 3 | <2 | <2 | <0.02 | 2200 | 8700 | 13 |
| 105 | 130 | 810 | 9 | <2 | <2 | <0.02 | 2200 | 14000 | 35 |
| 117 | 130 | 400 | 3 | <2 | <2 | <0.02 | 2200 | 6100 | 9 |
| 129 | 130 | 470 | 4 | <2 | <2 | <0.02 | 2200 | 6700 | 11 |
| 141 | 140 | 570 | 5 | <2 | <2 | <0.02 | 2200 | 10000 | 17 |
| 153 | 140 | 530 | 5 | <2 | <2 | <0.02 | 2200 | 9000 | 21 |

*Table 18: Total organic carbon (TOC) in Guardian West stream during Week 1 of on-site trials.*

| **Elapsed Time (hours)** | **TOC (mg/L)** | | |
|---|---|---|---|
| | Feed | Ret. | Perm. |
| 2 | 130 | 370 | 27 |
| 6 | 120 | 410 | 18 |
| 23 | 140 | 380 | 18 |
| 35 | 100 | 330 | 12 |
| 47 | 82 | 280 | 15 |
| 57 | 99 | 250 | 18 |
| 71 | 85 | 230 | 6 |
| 93 | 77 | 320 | 8 |
| 105 | 71 | 430 | 9 |
| 117 | 78 | 180 | 6 |
| 129 | 78 | 220 | 8 |
| 141 | 81 | 240 | 14 |
| 153 | 81 | 220 | 6 |

## 4. SUMMARY AND CONCLUSIONS

This project has demonstrated the feasibility of treating electroplating waste by reverse osmosis (RO). Studies were conducted with model and real electroplating waste streams at bench-top and pilot scale. Sampling of the effluent at the company site over several weeks identified several anions and cations that could cause fouling of the RO membrane. During the course of the project, total dissolved solids ranged from 3600 to 18,000 ppm. The primary contributors were sodium and sulfate ions which varied from 950 to 7600 ppm and 2200 to 14,200 ppm respectively. The variation in feed composition has to be considered in the final process design. The high ion concentration indicated that the maximum practical recovery would be about 66-75% (a concentration factor of 3-4). pH of the feed varied from 8.0-9.3

Preliminary experiments showed little benefit of ultrafiltration as a pretreatment. Several RO membranes were screened and the Dow-FilmTec SW30 membrane was selected for further studies. Two pilot plant rigs were fabricated for trials in the laboratory and at the company site. The membrane was first characterized using model feed solutions of 1000-10,000 ppm sodium chloride and 10,000 ppm sodium sulfate. The effect of the major operating variables (transmembrane pressure, cross-flow rate, temperature and solids concentration in the feed) on flux and rejection were studied and modelled. Flux with 1% sodium sulfate was about 50 liters per square meter per hour (LMH) at a pressure of 500 psi and 30°C. It reduced to 20 LMH at 5% TS. Rejection was > 99.7%. The effect of added barium and calcium on performance of the membrane was negligible except at levels of 50 ppm added barium at which point the flux reduced slightly to 45 LMH.

Trials with the feed from the electroplating plant were conducted over a period of 8 months. Fouling (as manifested by a continuous decline in flux at constant operating conditions during total recycle of retentate and permeate) was minimal. Average flux was 20-30% lower than the model sodium sulfate solution at equivalent solids concentrations. Bacteria (added at a level of 0.0026% TS) had a slightly more deleterious effect on flux. Over a 100-hour operating period, flux decreased 17% with added bacteria compared to 11% decline without added bacteria. Permeate quality continued to be excellent.

On-site trials were conducted over a four-week period. The feed composition varied considerably over this period. Fluxes were initially 8-10 LMH at a concentration factor of 3X, 515 psi and 30°C. Flux declined over the one-week period (with no cleaning during the week) to ~3 LMH. Feed pH varied between 7.5 and 9.2, and the pH of the permeate was always higher (9.0-9.5) than the feed or retentate. The permeate quality was consistently good.

In conclusion, reverse osmosis can be successfully applied to the treatment of electroplating waste. If the TDS is 10,0000 ppm or less, a recovery of 66-75% can be obtained with less than 100 ppm solids in the permeate. This implies a reduction of 66-75% in the volume of waste water and simultaneous production of good quality water that could be re-used in the plant. The results of this project are expected to have broad applicability to a number of electroplating facilities that might want to consider the use of membranes for recycling effluent wastewater.

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# 6. APPENDIX

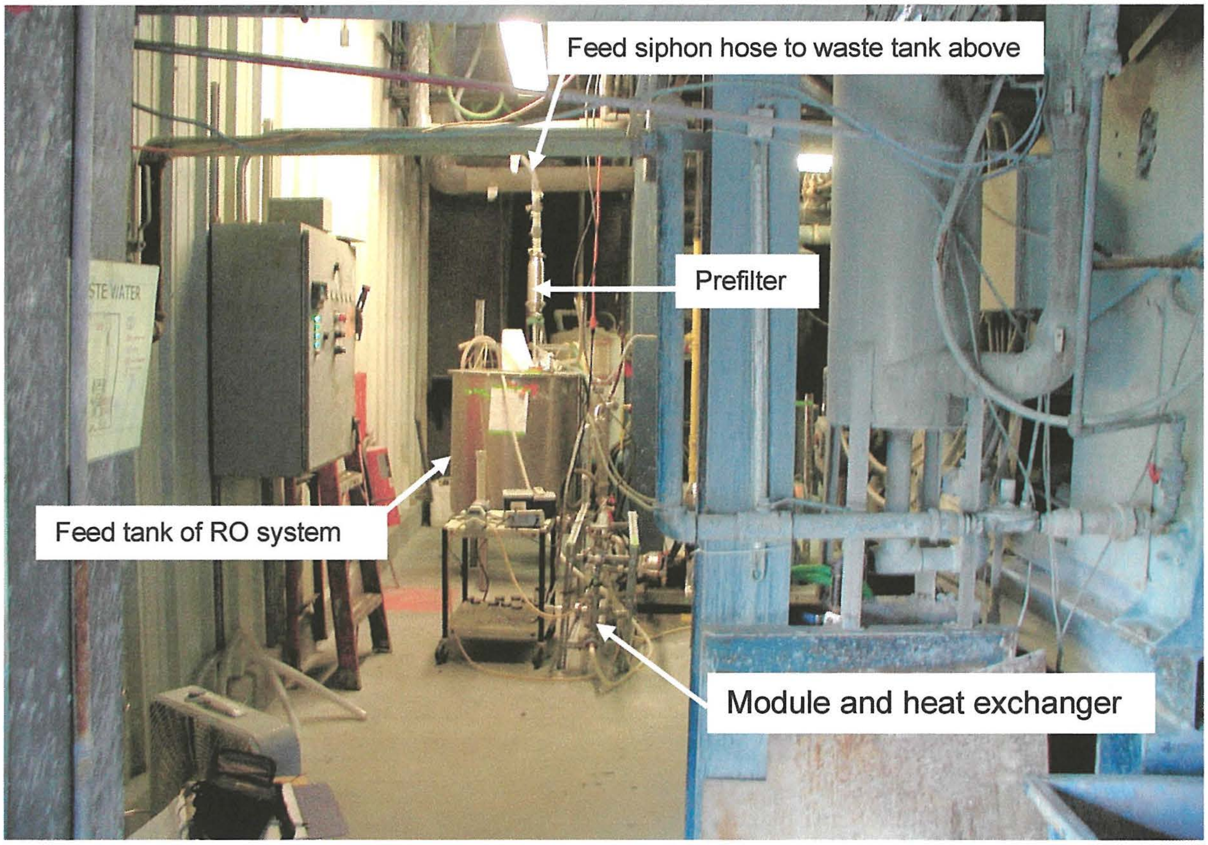

*Figure A1. Side view of pilot RO system installed in the Guardian West plant.*

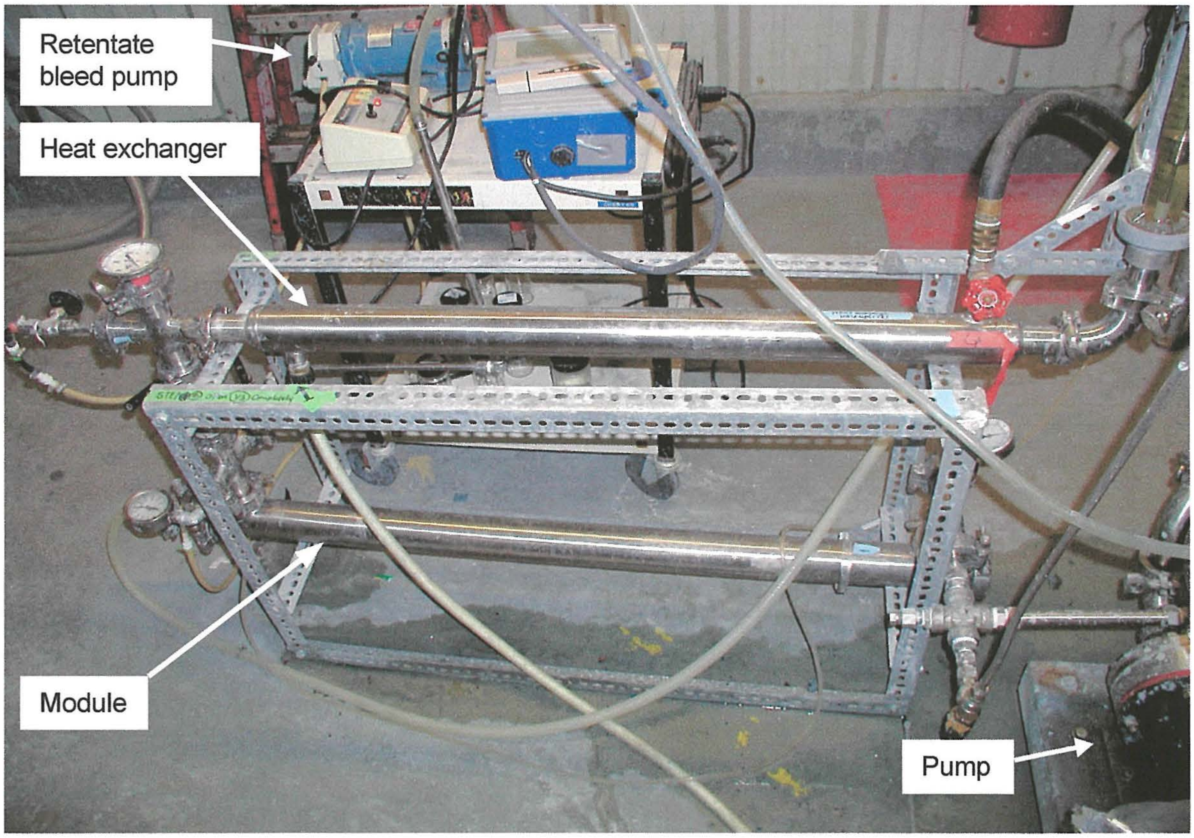

*Figure A2. Heat exchanger and RO module installed in the Guardian West plant.*

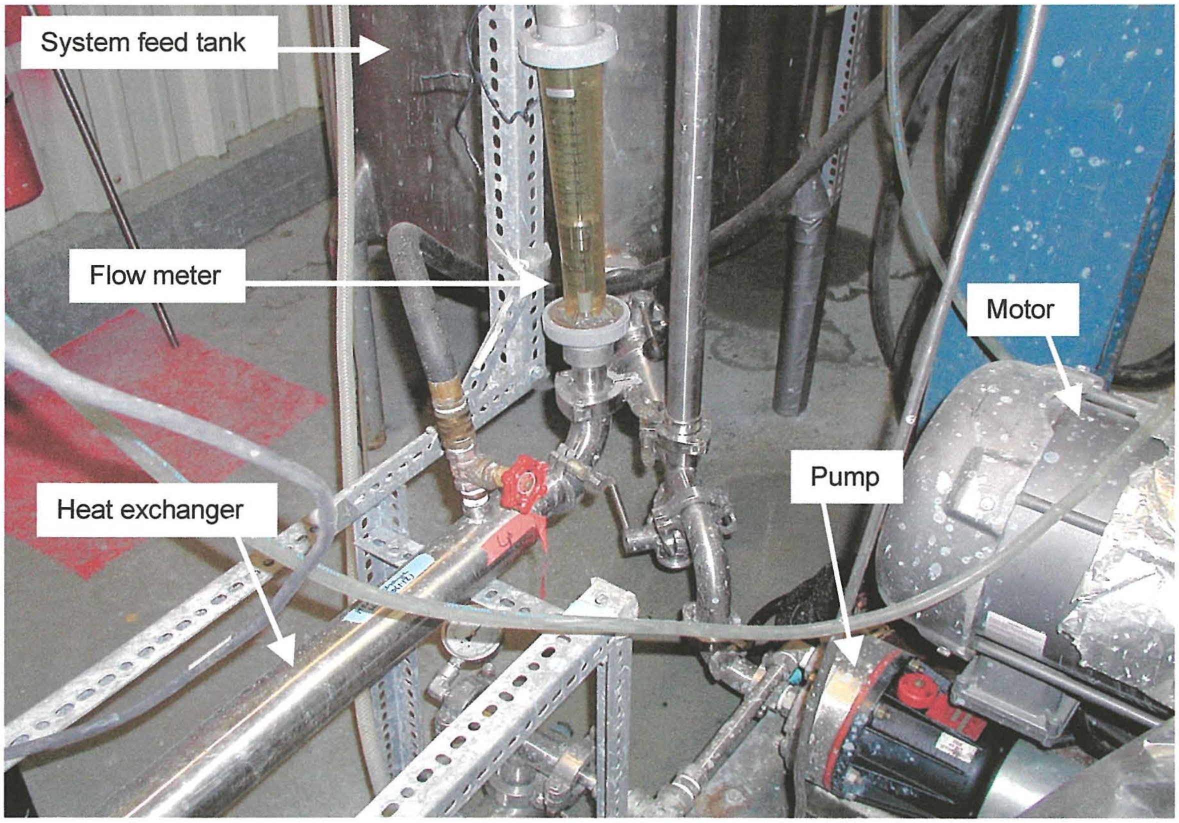

*Figure A3. Heat exchanger, retentate flow meter and high-pressure pump*

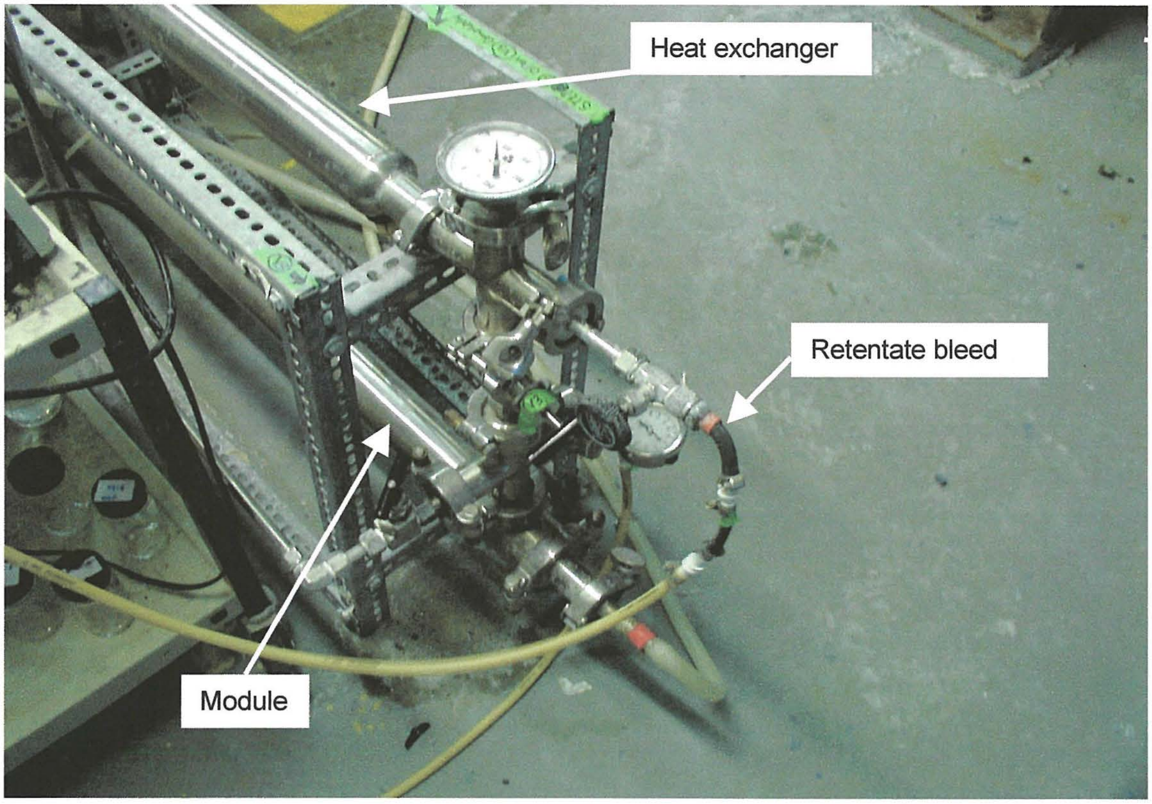

*Figure A4. Heat exchanger and module*

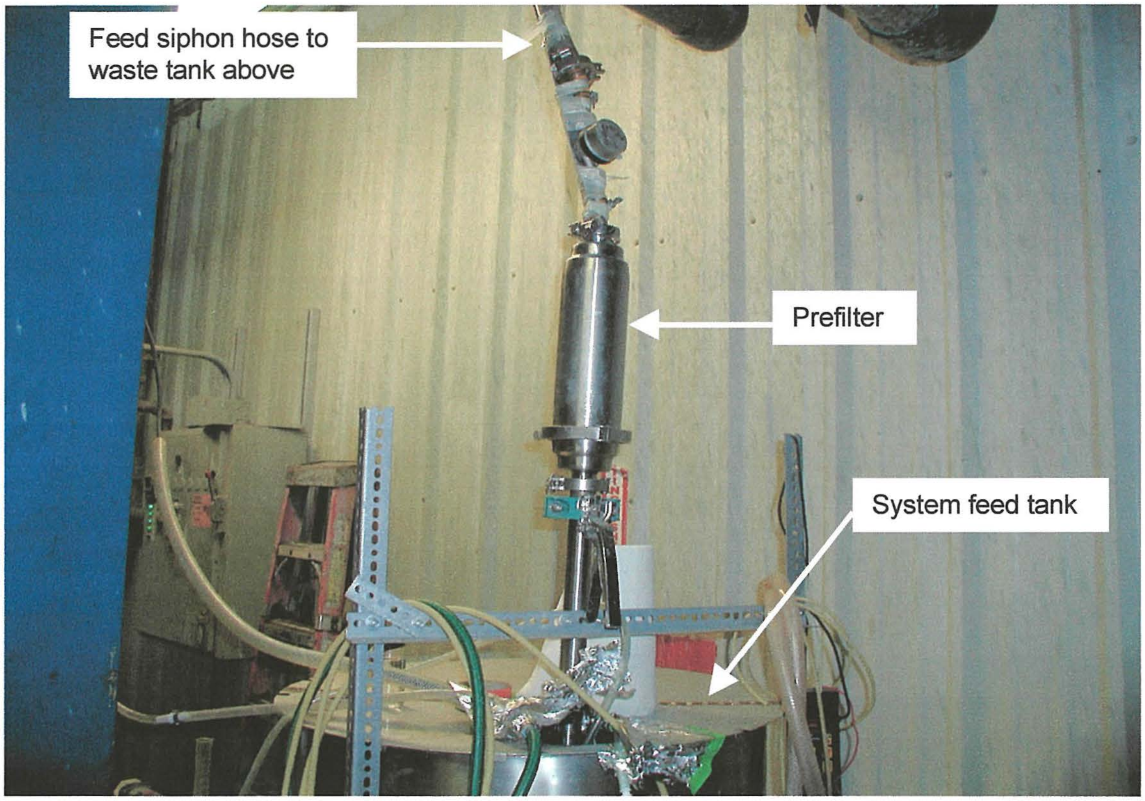

*Figure A7. Prefilter*

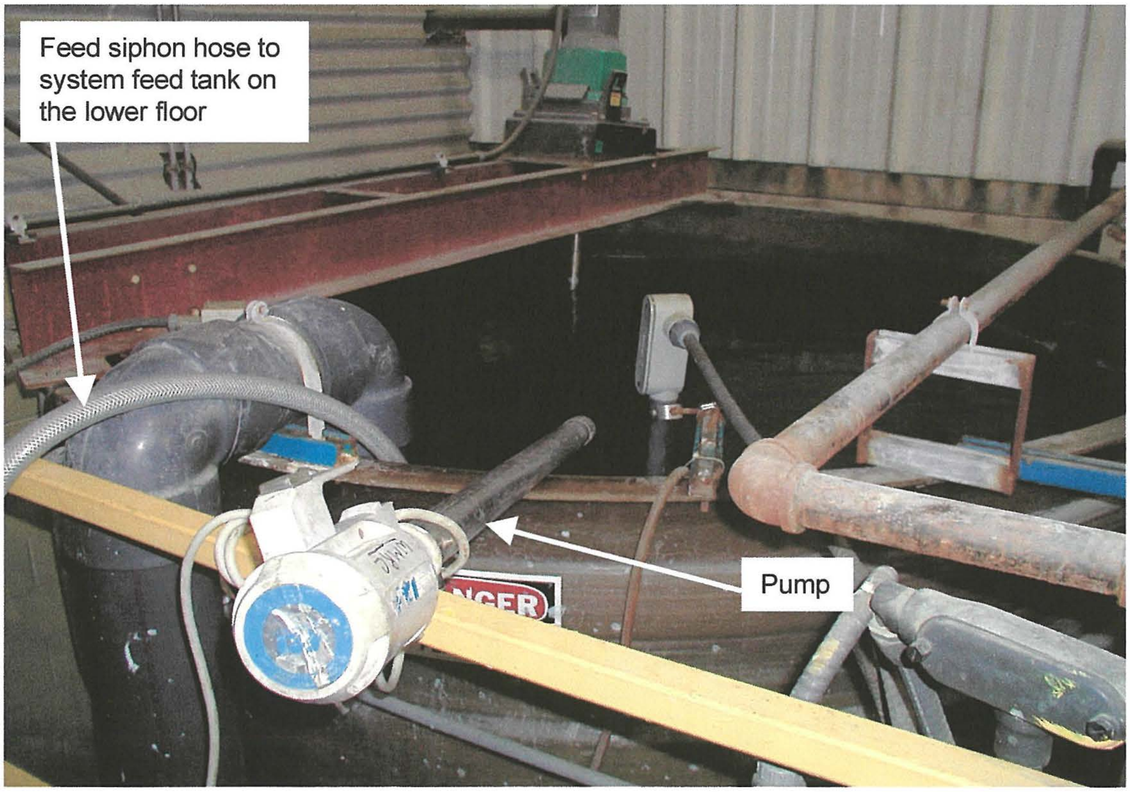

*Figure A8. Waste storage tank in the plant from which feed was taken. Pump was used to initially start the siphon.*