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MEMORANDUM

TO: J. R. WILEY, 773-A

FROM: D. G. PIPER, 676-8T DGP

F/H EFFLUENT TREATMENT FACILITY ECWPF PILOT TEST

INTRODUCTION

This report summarizes the results of tests that were performed on the ECWPF reverse osmosis (RO) unit in support of the F/H Effluent Treatment Facility (ETF). The objectives of the study were to: 1) confirm the results of single element RO testing which indicate the standard Filmtec SW30 membrane is susceptible to performance losses as a result of tributyl phosphate (TBP) fouling, and 2) determine if performance of the Filmtec SW30-HR high rejection membrane is comparable to that of the standard membrane. The study was also designed to test how the performance of the three-stage F/H ETF RO process would be affected by TBP fouling.

SUMMARY

1. Current plans to provide an organic removal step in front of the F/H ETF RO unit to prevent frequent fouling and cleaning cycles are fully justified by experimental data.

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2. Cleaning with sodium laurel sulfate created a discharge problem due to foaming, and may be unsuitable for use in the F/H ETF.

3. SRL will investigate further the use of acid cleaning solutions on the ECWPF unit after O-ring seal problems are corrected.

FACILITY DESCRIPTION

The results described in this report include five experimental runs on the ECWPF RO unit. The first experiment was a recirculation test; the last four were single-pass experiments. Recirculation tests consist of feeding the RO unit from the 1800 gallon ECWPF feed tank while recycling the concentrate and permeate back to the feed tank. The feed solution was not filtered during the run.

Single-pass experiments use two 10,000 gallon feed tanks (D and H) in the TNX tank farm. The 1800 gallon feed tank serves as a surge tank, allowing sufficient time (20 minutes) to switch between 10,000 gallon tanks. While feeding out of one tank, the other tank was drained, flushed, refilled with process water, and the next batch of chemicals added. In-tank agitators were run continuously after chemicals were added. Five-micron polypropylene Filterite cartridge filters were used upstream of the RO unit during single-pass runs to protect the membrane.

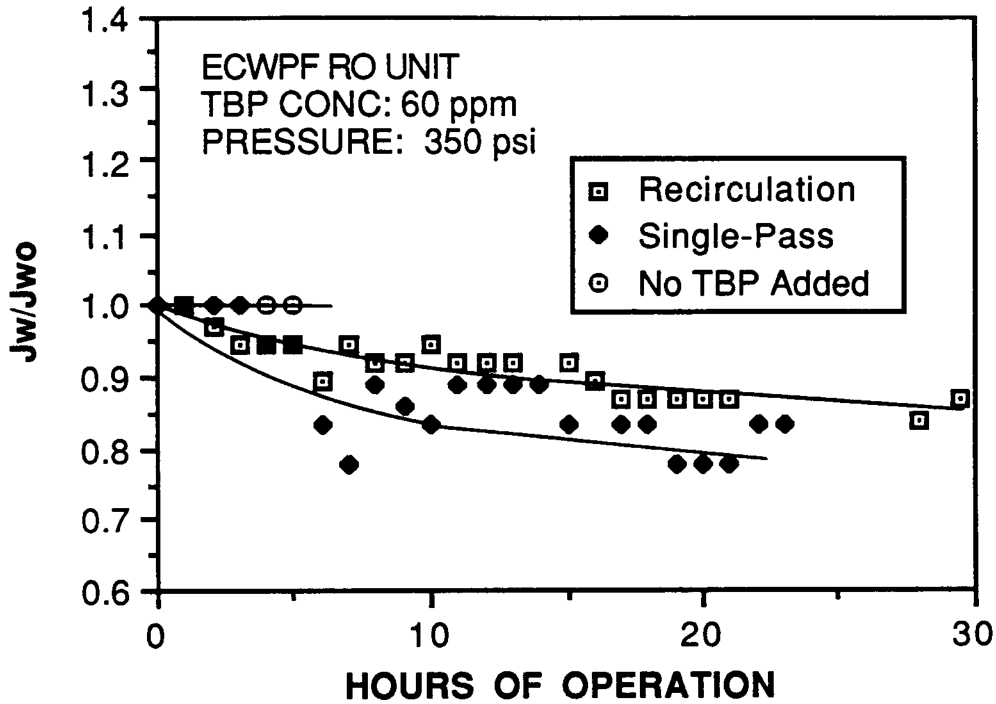
RESULTS AND DISCUSSION

Recirculation vs. Single-Pass Operation: Results from recirculation and single-pass operation are presented in Figure 1. The fluxes (J_w) are normalized by dividing them by the initial flux (J_{wo}) thereby forming a common starting point for the two operating lines making comparison of the curves easier. J_{wo} was 18.9 gfd (gal/ft^2/day) for the recirculation and 17.9 gfd for the single-pass experiments. Permeate fluxes reported in this study are experimentally measured fluxes, uncorrected for temperature and pressure. Standardized Permeate Flux (SPF) is not a reliable way to monitor performance on a staged RO system.

Recycling concentrate and permeate to the feed resulted in a consistently higher flux compared to single-pass operation. The flux drop plateaued after approximately five hours of recirculation but took approximately eleven hours to level off for single-pass operation. After 20 hours of recycle operation, the flux loss was only 13% compared with a flux drop of 19% after 20 hours of single-pass operation.

The results from the two experiments can be explained by a lower concentration of foulant material at the membrane surface when recycling feed. During recycle operation the concentration of foulant at the membrane surface is limited by the amount of foulant initially present in the feed. Single-pass operation, however, provides a continuous source of foulant such that a more concentrated boundary layer could be formed at the membrane surface, resulting in a lower flux, before steady-state is achieved.

FIGURE 1. COMPARISON OF RO UNIT OPERATING CONDITIONS



This difference in the two modes of operation was enhanced because additional trace inorganic (primarily Fe and Al and to a lesser extent Mn) was present in the single-pass experiments but not in the recycle experiment. For single-pass experiments the feed consisted of 60 ppm of TBP in a simulant with the salt concentrations given in Table I. The feed for the recycle experiment was a two g/l $NaNO_3$ solution with 60 ppm of TBP added. If the foulant mechanism is initiated by TBP complexing with metal ions, as believed, then the concentration of these compounds in the bulk feed and at the membrane surface would be higher in the single-pass experiments.

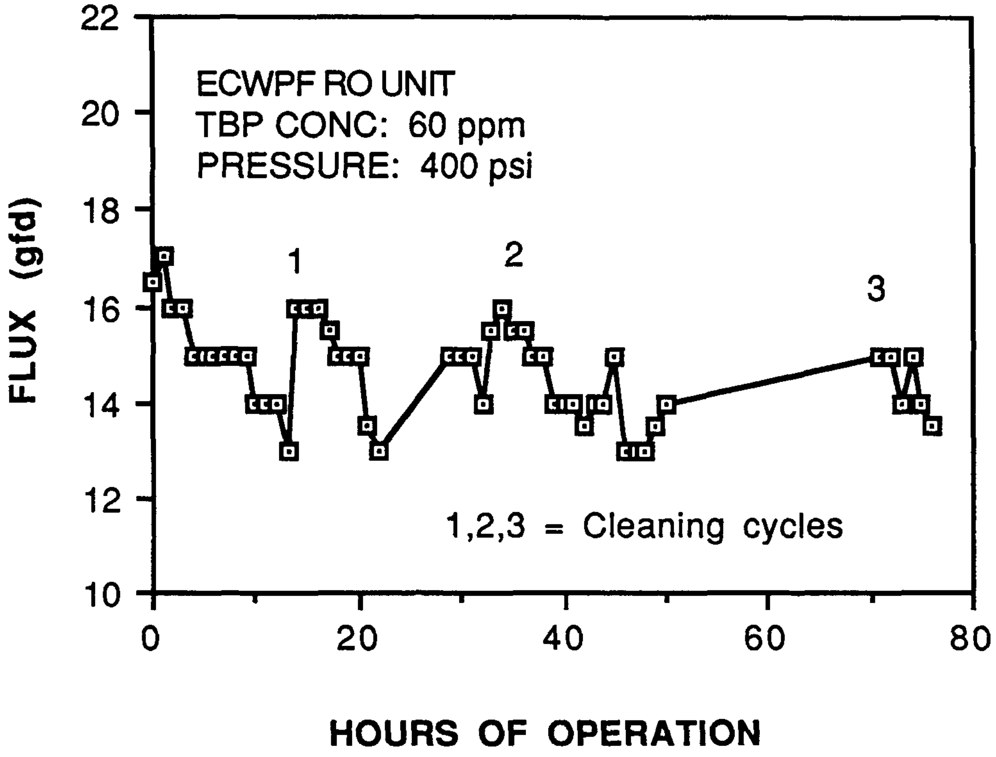
TABLE I. SIMULANT COMPOSITION

COMPOUND	CONCENTRATION (g/l)
$NaNO_3$	1.50
$NaHCO_3$	0.10
NH_4NO_3	0.10
Na_2SiO_3	0.026
$Al(NO_3)_3 \cdot 9H_2O$	0.017
$FeNO_3$	0.017
$CaCO_3$	0.011
Na_2SO_4	0.007
$NaNO_2$	0.002
$NaCl$	0.002
$MgCO_3$	0.001
NaF	0.001
$MnCl_2 \cdot 4H_2O$	0.001
$Zn(NO_3)_2$	0.001
$Ba(NO_3)_2$	0.00004

Water Cleaning Test: At the completion of the recirculation and single-pass tests described above, the RO unit was cleaned with water only. Water cleaning recovered 97% of water flux after the recirculation run and 93% following the single-pass experiment. In order to determine whether water cleaning alone could be used to consistently restore flux to the ECWPF RO unit, the ECWPF unit was operated continuously for 76 hours (beginning at 11:00 AM on June 23, 1987) during which time the RO membrane was fouled three times and cleaned each time with water. The fouling and cleaning cycles are shown in Figure 2.

The permeate flux was 16.6 gfd at the beginning of the 76 hours of operation. The flux dropped to 12.9 gfd, a 22% loss of flux, in 13 hours. At this point, as the result of an operational omission, a full 10,000 gallon tank of water only, without simulant, was processed through the RO unit. The flux increased to 15.9 gfd, a 96% recovery, after the impromptu water cleaning step. When simulant feed began to the RO unit once again the flux quickly dropped back to 12.9 gfd in only 6 hours.

FIGURE 2. EFFECT OF WATER CLEANING ON MEMBRANE PERFORMANCE



At this point RO unit operation was discontinued for cleaning. After a two and a half hour water cleaning cycle the flux was 14.9 gfd. This was a 90% flux recovery compared to the 16.6 gfd at the beginning of the 76 hour test run, but only a 79% recovery compared to the original 18.9 gfd before the first recirculation experiment.

The flux measured during the next 22 hours of operation was very erratic. At one point the flux increased to 15.9 gfd before declining to 12.9 gfd over a period of 12 hours and then increased slightly to 13.9 gfd at the end of the 22 hours.

The RO membrane was washed with water this time for a much longer period of time. A 15 hour water wash cycle returned the flux to 16.6 gfd, the same flux that was observed at the beginning of the 76 hours of operation. However, when the RO unit was started up, the flux dropped to 14.9 gfd in only five hours indicating that in spite of the flux recovery the unit was still fouled. Salt rejection measurements that were obtained throughout the experiment confirm that in spite of the flux improvement water cleaning had a marginal effect on removing foulant from the membrane.

Salt Rejection: A salt rejection problem was observed for the first time at 4:00 AM on June 24, 1987; this corresponds to the sixteenth hour of operation. The DF (based on permeate and concentrate conductivity) dropped from 42 to 10. Individual pressure vessel samples taken at this time showed that salt was breaking through in two of the four first stage pressure vessels. The water flux at this point was 15.4 gfd.

Water cleaning did not improve the DF. The DF was the same after the first water cleaning cycle as before. After approximately seven more hours the DF dropped to 5.5 and bottomed out at 4 after six more hours. Conductivity measurements at this time showed that, in addition to the two pressure vessels on the first stage, one of the two second stage pressure vessels was breaking through.

The fifteen hour water wash cycle had no effect on salt rejection. The DF remained at 4 until the unit was shut down. The third stage pressure vessel began to show salt break through after the final water washing.

Surfactant Cleaning: Surfactant cleaning with a basic solution of sodium laurel sulfate was used as a last resort in an attempt to clean the fouled membrane. An earlier ECWPF membrane surfactant cleaning test with 0.5 wt% sodium laurel sulfate caused a foaming problem.

No visible improvement in membrane performance, neither flux nor salt rejection, was observed following the surfactant cleaning. In light of the discharge problems caused by the foaming at TNX, surfactant cleaning is not recommended for the plant ETF process. Foaming in the ETF evaporator would be the likely result and would require injection of defoaming chemicals to prevent an evaporator upset.

Acid Cleaning: Prior to attempts to clean the ECWPF RO unit with acid solutions, a laboratory test was performed to determine the effectiveness of various acid cleaning agents. The acid cleaners were tested on some of the fouled polypropylene Filterite cartridge filters that had been used upstream of the RO unit. The foulant material on the cartridge filters were expected to be similar, and results from the filter tests were expected to translate to the RO unit.

Of the five acid cleaning agents tested, only oxalic acid was effective at removing foulant material. The foulant material was dissolved without leaving a residue or forming a precipitate at an oxalic concentration of 2 wt% (pH = 1). Four acid cleaners recommended by Filmtec; hydrochloric acid, phosphoric acid, sulfamic acid, and citric acid were minimally effective or had no effect at all. A photograph of the cartridge filters before and after cleaning are shown in Figure 3.

Clean-up of the ECWPF unit with oxalic acid, however, was unsuccessful. Salt rejection on the pressure vessels that were not previously breaking through was improved. The vessels that had appeared to be fouled were unchanged after the acid cleaning. This result indicates that the decline in salt rejection may be the result of leaking seals in the RO pressure vessels and may not be completely attributed to fouling. There is a possibility that TBP may have softened the rubber O-rings and caused them to fail.

CONCLUSIONS

Results from the recirculation runs with $NaNO_3$ and TBP show that TBP is a primary foulant. TBP was apparently able to complex with iron, readily available in the TNX process water, even when multivalent metal ions were not added in the simulant mixture. Operation of the F/H ETF process, continuously and at high utility, requires an organic removal step in front of the RO to prevent frequent fouling and cleaning cycles. Increasing the membrane area to compensate for the expected flux loss is not sufficient.

Results from the 76 hour continuous run show that membrane foulants can not be removed consistently by simple means such as water cleaning. Water cleaning may be used, however, as a temporary measure since partial flux recovery was observed. Continued application of water cleaning alone would ultimately lead to a decline in salt recovery.

Cleaning with a basic solution of sodium laurel sulfate appeared to clean the membrane the first time it was used. However, it can create discharge problems due to foaming, and may be unsuitable for use in the F/H ETF.

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FIGURE 3. Photograph of Acid Cleaned Cartridge Filters.