Operating experiences of reverse osmosis plants treating Rand Water Board water: Pitfalls and problems*

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Abstract

Results of a survey of the operating experience of reverse osmosis users treating Rand Water Board water are presented. Common problems and pitfalls are identified and solutions offered. A major cause of poor reverse osmosis performance is membrane fouling. Laboratory investigations into the nature of membrane fouling by Rand Water Board water are described.

Introduction

The mineral quality of water supplied by the Rand Water Board (RWB) to the Pretoria-Witwatersrand-Vereeniging-Sasolburg (PWVS) region is steadily deteriorating due to greater reuse and contamination by mining, industrial and domestic effluents. Increasingly desalination will be required to produce demineralized water for applications ranging from laboratory use to feed water for high-pressure boilers. If reasonable membrane life can be achieved, the reverse osmosis (RO) process will be established as the desalination process of choice because of its low energy and chemical requirements and simplicity of operation.

The life of RO membranes, their flux and salt rejection performance is mainly dependent on two factors, viz. fouling, which results in decreased permeate flux, and, depending on the type and nature of the fouling, either an increase or decrease in salt rejection (Belfort and Altena, 1983); and chemical or biological degradation of the active membrane layer, which is characterized by an increase in permeate flux and a decrease in salt rejection.

More than 150 small to medium sized RO units have been installed in the PWVS area during the past ten years. However, reports have from time to time reached the National Institute for Water Research (NIWR) of poor RO performance, which was attributed to membrane fouling and it was decided to investigate the situation at first hand by means of a survey in order to:

- establish the extent of the problem;
- assess the performance of RO plants operating with RWB water as feed;
- identify problems and pitfalls of plant design, operation and maintenance;
- recommend action to maintain and improve performance; and
- define future research requirements.

Survey of RO plants

A survey questionnaire was prepared and the person(s) responsible for the operation of RO plants at 15 centres were interviewed. Most of the plants were small (< 1 500 ℓ/h) and were used in medical, pharmaceutical and laboratory applications where bacteria and pyrogen-free demineralized water is required.

It was difficult to assess plant performance because with only one exception regular and adequate records were not maintained. Generally, therefore, membrane life was the main measure of plant performance that could be quantified. The survey findings are summarized in Tables 1 and 2.

Membrane life

Two measures of membrane life were adopted, viz., average rate of membrane replacement in terms of calendar months, and average productive life (i.e. actual operating hours). This figure was derived from operator estimates of plant utilization.

Judging by the findings of our survey the performance of local plants has been very disappointing, particularly when one considers that some overseas plants have achieved membrane lifetimes of three years and more. The longest production life recorded was 7 600 h, which is only equivalent to 10,5 months of continuous operation.

Permeate quality (i.e. conductivity) was the criterion used at all plants to determine the need for membrane replacement. The survey highlighted aspects of pretreatment, plant operation and design that probably contributed to the poor membrane life.

Maintenance of adequate records

Only one plant had maintained operating records on a regular basis. At two plants records were kept for a limited period. However, they had been discontinued because the operators were satisfied with performance at the time and believed that the records were unnecessary. That this was not the case is illustrated in Figure 1.

At a further two plants operators started in 1983 to keep records after experiencing various operational problems. At most plants some measurements or analyses had been performed but these were inadequate to evaluate plant performance.

Reasons for the lack of records included: inadequate instrumentation to measure all the necessary parameters; unreliable instrumentation and no means of calibration; and inadequate training and information regarding the importance of record keeping. Even when performance records were kept they were never normalized and presented graphically to facilitate interpretation.

The lack of adequate operating records and the neglect of translating records into a suitable graphical form are serious

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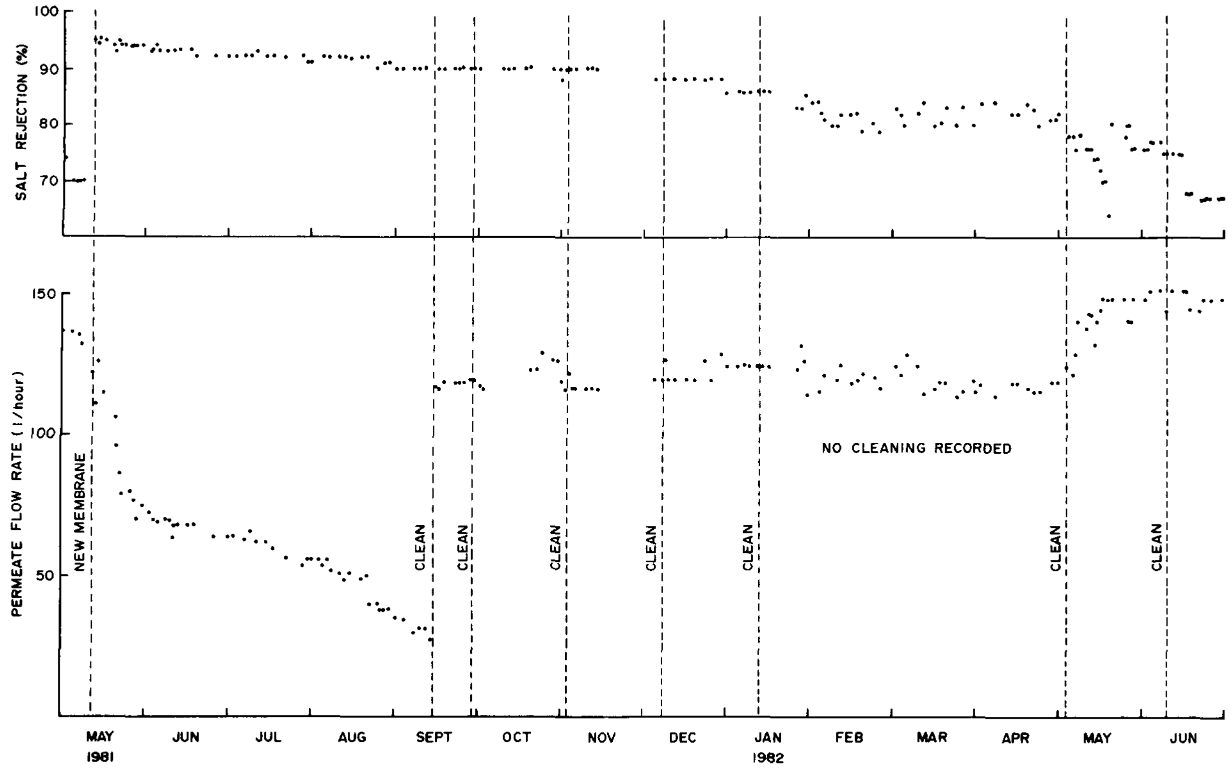

Figure 1
Salt rejection and permeate output data (normalized for pressure and temperature) for one of the RO plants surveyed.

TABLE 1
DETAILS OF APPLICATIONS, PLANT CAPACITIES, MEMBRANE LIFE, DISINFECTION, CLEANING AND PLANT UTILIZATION

Plant No.	Application	Capacity(a) (ℓ/h)	Criteria for membrane replacement(b)	Average replacement rate(a) (months)	Average productive life(a) (h)	Operating records	Disinfection		Membrane cleaning		Utilization (%)	Comments
							Agent	Frequency	Agent	Frequency		
1	Pharmaceutical	1 500	PC	10,5	1 800	No	$Na_2S_2O_5$	Weekly	Alkaline detergent or acid	Monthly	25	
2	Medical	5 x 50	PC	None at June 1983	–	No	$Na_2S_2O_5$ Formalin	Weekly Monthly	No	–	32	Units installed Nov. 1982
3	Industrial	180	PC>1mS/m Flux	9	3 300	No	H_2O_2	6 weeks	Citric acid	Irregular	50	
4	Pharmaceutical	80	PC	12	200	No	H_2O_2	6-8 weeks	No	–	2	
5	Medical	2 x 200 (2 x 36)	PC	7	550	No	Formalin and H_2O_2	Weekly	No	–	11 (20)	
6	Pharmaceutical	30 (60)	PC	None at June 1983 (9)	– (200)	No	Formalin	Weekly	Citric Acid	Irregular	36 (3)	New unit installed May 1983
7	Medical	7 x 60	PC	15	3 250	No	$Na_2S_2O_5$ Formalin	Daily Weekly	No	–	30	
8	Pharmaceutical	285	PC	12	1 000	No	No	–	No	–	12	
9	Medical	5 x 35	PC	No records	–	No	Formalin	Monthly	Proprietary formulation	Monthly	32	
10	Pharmaceutical	2 x 250	PC>8mS/m	4	2 200 (1 set lasted 7 600)	Partial	H_2O_2	Weekly	EDTA and citric acid	Weekly	75-85	
11	Industrial	720	PC	24	4 200	Kept for a period	Formalin	Irregular	No	–	24	
12	Laboratory	140	PC	No records	1 membrane lasted ~ 3 100	Kept for a period	No	–	Proprietary formulation	Irregular	30-40	
13	Industrial	2 x 14 000	PC	RO1 ≡ 36 RO2 < 12	RO1 ≡ 5 300 RO2 < 1 200	Yes	No	–	EDTA, TRITON X100, STPP(c)	3-6 months	14	
14	Medical	10 x 60	PC	11,5	1 100	No	Formalin	2 weeks	No	–	13	
15	Laboratory	60	PC	6	260	No	No	–	No	–	6	

(a) Figures in parentheses refer to plants previously installed at that centre.
(b) PC-Permeate conductivity (c) STPP-Sodium tripolyphosphate

TABLE 2
TYPES OF MEMBRANES USED AND PRETREATMENT SYSTEMS.

Plant No.	Membrane Type(a)	Pre-treatment						
		pH Control		Filtration			Pre-softening	Other
		Agent	Range	Primary	Cartridge Nominal pore size (μm)	Carbon		
1	(CA) FT30	1983 only Citric acid	6	–	3	–	–	$Na_2S_2O_5$ dechlorination
2	FT30	–	–	Diatomaceous earth	5	Yes	–	
3	(CA) FT30	CO_2 gas	6,5-7,0	–	3	Yes	–	
4	(CA) FT30	–	–	–	10 and 1-2	–	Ion exchange	
5	(CA) CA + FT30	–	–	–	10	–	–	
6	(CA) FT30	1983 only Citric acid	6	–	10	Yes	–	
7	CA	–	–	Diatomaceous earth	10	–	Yes	Ceramic candle filters
8	PA-Hollow fine fibre	–	–	–	10	Yes	Yes	
9	CA	H_2SO_4	5-7	–	3	–	–	
10	(CA + PA) CA	(H_2SO_4) Citric acid	5,5-7	–	10 and 5	–	–	
11	CA	H_2SO_4	6-6,5	–	10 and 5	–	–	
12	(CA) FT30	–	–	–	15 and 5	–	–	
13	CTA	H_2SO_4	4,5	Sand	3	–	–	Chlorine and SHMP dosing (b) and decarbonation
14	(CA) CA + FT30	–	–	Diatomaceous earth	10	Yes	Yes	STERACIL candle filters
15	(CA) FT30	–	–	–	Yes, but pore size unknown	–	Yes	

(a) Membrane types in parentheses are those previously used and replaced
CA – Cellulose acetate
CTA – Cellulose triacetate
PA – Polyamide
FT30 – FilmTec FT-30, a composite polyamide membrane
(b) SHMP – Sodium hexametaphosphate

shortcomings because users do not become aware of plant deterioration until it is serious or too late, and different types of membrane deterioration are not identified and thus appropriate steps cannot be taken to overcome specific problems.

Some records, which have been normalized and plotted in Figure 1, illustrate these points. The salt rejection performance

$$\left(\frac{\text{conductivity of feed} - \text{conductivity of permeate}}{\text{conductivity of feed}} \times 100\right)$$

of the plant decreased steadily during the first nine months of operation and was not improved by cleaning, even though flux was restored. This is indicative of a gradual chemical degradation of the membranes because if the decrease in salt rejection had been brought about by the build-up of a fouling layer, it would have been restored when the layer was removed by cleaning. From about January 1982, the salt rejection decreased more rapidly and the membranes had to be replaced at the end of June.

One may speculate as to the reason for the failure of the cellulose acetate (CA) membranes and it is likely that it was caused by hydrolysis of the membranes because pH control of the feed water was not applied. However, lack of interpretation of the records and follow up action resulted in complete deterioration by May 1982, after a mere 3 100 h of operation.

Pretreatment

The purpose of pretreatment is to alter the chemical and physical properties of the feed water so as to obtain optimum plant performance. The main objectives are:

- to remove particulate and colloidal substances, which may foul the membranes and reduce plant output;
- to remove, modify or inhibit sparingly soluble substances, which may precipitate and foul the membrane surface;
- to adjust the pH so as to prevent deterioration of some membrane types and reduce carbonate scaling;
- to remove or modify substances, such as chlorine and other oxidants or solvents, which may chemically destroy some membrane types; and

- to prevent biological growth and consequent membrane fouling or degradation.

The survey indicated that some of the following pretreatment operations were occasionally employed:

pH control

pH control was used at only seven plants and in two cases this facility was installed in 1983 (Table 2).

Since on average RWB water has a pH > 8 (RWB Annual Reports, 1980-1982), adjustment to between 5 and 7 is required when CA membranes are used to reduce the rate of membrane hydrolysis. This does not apply to polyamide (PA) based membranes since they have a wider pH tolerance.

Three of the seven plants presently using CA membranes have no pH control. In the past pH control was not used at a further five plants with CA membranes.

Calcium carbonate and magnesium hydroxide scale formation is also controlled by reducing the pH. Instead of pH control, five plants used softeners to reduce the calcium and magnesium concentrations and thus minimize the risk of scale formation.

Scale inhibitors

Sodium hexametaphosphate was used as a scale inhibitor at plant no. 13, which operated at a recovery of approximately 75 %. Inhibitors are required when plants operate at high recoveries to prevent calcium carbonate and calcium sulphate from precipitating on the membrane surface. However, most of the plants that were visited operated at low recoveries (25-35 %).

Dechlorination

When free chlorine is present in a feed water, PA and some other composite membranes require dechlorination (e.g. reduction by sodium metabisulphite addition or by activated carbon) to prevent halogenation of the membrane. However, the RWB adds ammonia after chlorination to produce monochloramine. This is a more stable compound than chlorine but a weaker oxidant.

Of the ten plants using PA membranes at the time of the survey, nine used FILMTEC FT-30 membranes. These membranes are resistant to monochloramine although they are damaged by free chlorine (Petersen *et al.*, 1982).

Membrane cleaning

Membrane cleaning is carried out at seven plants only. In the remaining plants, membranes have been replaced without any attempts at cleaning. This is most unfortunate because cleaning can significantly improve performance, as the following example shows: In plant no. 10, membranes were replaced seven times in the seventeen months from start up. The establishment of a weekly cleaning schedule (EDTA and citric acid) appears to be the major reason why the eighth membrane set lasted 14 calendar months (7 600 productive hours).

Some membrane manufacturers recommend that membranes should be cleaned when the flux has decreased by 10 % or the salt passage has increased by 25 %.

Plant design

The average utilization figures indicate that most plants have capacities far greater than required. Only one plant was operated for more than 50 % of the time, while eight had utilizations of less than 25 %.

The overdesign of the plants may have been intentional in order to provide spare production for future requirements. However, if it was done to compensate for flux decline due to fouling, then this overcapacity has been provided on false assumptions:

- When fouling occurs, permeate quality generally deteriorates as well. This is because intrinsic salt rejection is related to permeate flux (Johnson *et al.*, 1966) and foulant deposits on the membrane surface cause increased concentration polarization, which in turn results in an increase in solute passage (Sheppard and Thomas, 1971). Since all of the operators are specifically concerned about permeate quality (Table 1), only a limited amount of fouling is acceptable. Once this point is reached, it matters little that the plant has spare capacity, as none of the permeate will meet requirements. The operating pressure can be increased, which will improve the permeate quality somewhat. However, this in turn exacerbates the fouling problem.
- Chemical degradation of membranes continues whether the plant is in operation or not. Unlike fouling, this deterioration is a function of time, not permeate production. Moreover, when the plant is off-line, membranes tend to deteriorate more quickly due to microbiological growth, particularly when they are not preserved (by e.g. formaldehyde).
- Foulant deposits become more difficult to remove with time. Over-capacity means that plants initially do not require regular cleaning since production requirements are met despite fouling. However, when cleaning is implemented, it will probably not be as effective in restoring productivity due to the 'aging' of foulant deposits.

At seven installations a number of small plants were used in parallel. Most of these plants are poorly utilized (< 33 %). Apart from the increased capital costs, running costs escalate since these arrangements multiply plant monitoring and maintenance and increase the average membrane replacement rate.

The installation of suitable storage tanks and instrumentation to allow for semi-continuous unattended operation would cost a fraction of the present investment in delicate membranes and would probably result in a better quality water overall at a lower cost. Existing installations should be modified so that fewer units may be operated at more than say, 80 % utilization, while spare machines are 'mothballed'.

Note that plants should always be flushed before shutdown (whether shutdowns are automatic or manual) to avoid the precipitation of salts concentrated at the membrane surface.

Nature of membrane foulant deposits

Investigations into the causes of membrane fouling and into the nature and composition of foulants are in progress at the NIWR. Some preliminary results relevant to this survey are reported here.

Electron microscope studies

The foulant deposits formed when passing RWB water through microporous membranes under simulated RO operating conditions were examined by electron microscopy. Figure 2 is an inclin-

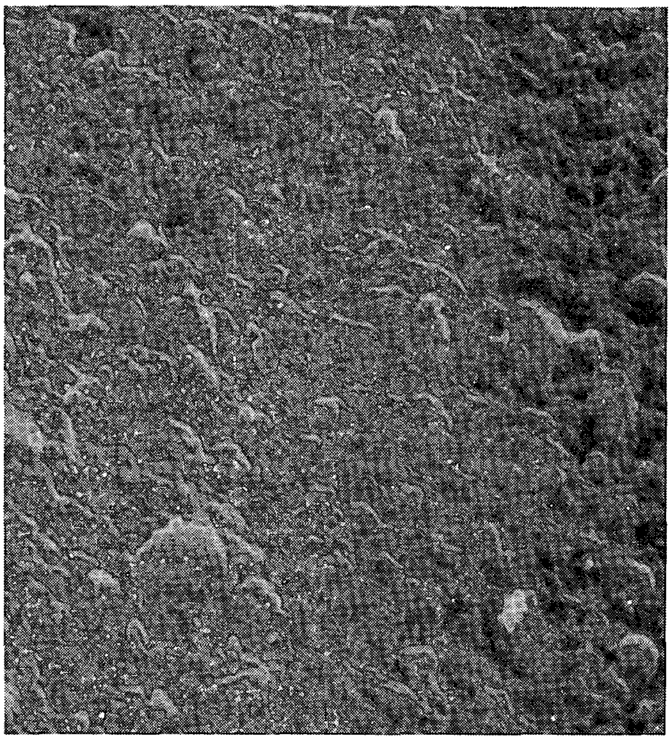

Figure 2
Fouled surface of a microporous membrane (CELGARD 5510) after treating RWB (CSIR) water. Note the very fine particle size of the deposits (scanning electron photomicrograph, 10 000x)

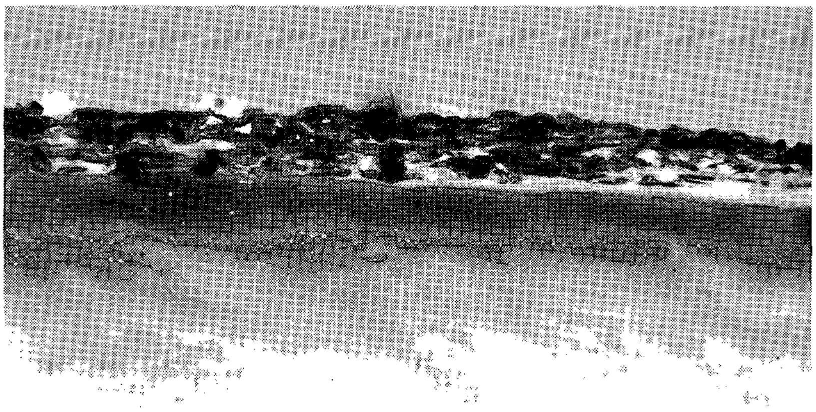

Figure 3
Cross-section of a foulant deposit formed on a microporous membrane (CELGARD 2500) (transmission electron photomicrograph, 12 600x).

ed top view of the foulant deposit formed on the membrane surface. It consists mainly of submicron particulates; the largest particle in the picture (near the bottom left) is only 1,2 μm in diameter. Similar deposits are formed on RO membranes.

In Figure 3 a photograph of the cross-section of the foulant deposit shows that the deposit was largely composed of layers of plate-like materials, probably clay colloids, orientated parallel to the membrane surface. The thickness of the plates varies between 0,01 and 0,08 μm. This picture of the microstructure of the foulant deposit – ultrathin plates stacked one on top of each other – explains why the permeate flux is so seriously reduced and why the salt rejection deteriorates due to increased concentration polarization. Improved pretreatment methods are required because the filters that are currently used do not remove these finely divided particulates.

Identification of RO membrane foulants

Samples of foulant deposits in a number of membrane modules were analysed in an attempt to identify the substances responsible for fouling. A knowledge of the nature of the foulants will, it is believed, enable one to prescribe pretreatment methods to remove the substances and prevent fouling; and determine the most suitable cleaning methods to remove foulants and restore the membrane flux and salt rejection performance.

Methods used for identification of foulants

Deposits were scraped from the membrane surfaces of six modules from four plants that treated RWB water. The following techniques were used to characterize and identify the constituents of the deposits:

- X-ray fluorescence on samples B-1, C-1 and D to obtain qualitative analyses.
- Combustion followed by gas chromatography to determine C, H and N concentrations.
- Atomic absorption spectroscopy (AA) and inductive coupled plasma – optical emission spectroscopy (ICP-OES) to obtain quantitative analyses of samples of the deposits that had been brought into aqueous solution after fusion with lithium metaborate at 900 °C (method described by Pradhon, 1976)
- X-ray powder diffraction (XRD) for mineral identification.

Composition of membrane foulant deposits.

The quantitative analyses are presented in Table 3 in terms of the overall percentage composition of the dried samples (105 °C overnight).

The sum of the constituents for each deposit does not add to a total of 100 % because no account is taken of combined oxygen.

At plant A mainly calcium carbonate scale formed (confirmed by XRD). The large amount of scale present in the module meant that it was difficult to determine the significance of other types of fouling that may also have occurred. The scaling was probably not a result of the failure of the pre-softener (scaling has not been noted in similar low recovery systems which operate without pre-softeners), but due to a malfunction (e.g. pump wear or a leaking O-ring) which led to a low linear velocity in the module and a high recovery. Note that if calcium carbonate deposits form, high pH's may prevail at the membrane surface and cause hydrolysis of the membrane, especially when the unit is not operating.

The high proportions of aluminium and silicon in the deposits of plants B, C and D indicate that fouling may have been caused by either clay colloids and particulates, or by precipitation of aluminium hydroxide, aluminium silicate and colloidal silica, or combinations of these. Some colloidal and particulate fouling (anticipated by the microscopic studies described in the previous section) was confirmed by XRD.

Iron and zinc were significant components of the deposits at plants B, C and D, and B and C respectively. Since the concentrations of iron and zinc are generally low in RWB water (maxima in 1982/83 were 185 µg/ℓ Fe and 38 µg/ℓ Zn; RWB, 1982b), these

TABLE 3
OVERALL PERCENTAGE COMPOSITIONS OF MEMBRANE FOULANT DEPOSITS SCRAPED FROM SIX MODULES USED AT FOUR PLANTS (A, B, C AND D) WHICH TREATED RWB WATER

Pretreatment	Softener, diatomaceous earth, ceramic candles and 10 µm cartridges	Activated carbon, softener, diatomaceous earth, ceramic candles and 10 and 5 µm cartridges		5 µm cartridge only		Sand filtration, Cl_2, acid and sodium hexametaphosphate dosing, 5 µm cartridges	Analytical methods
	A	B-1	B-2	C-1	C-2	D	
H	0,4	2,7	3,1	—	3,3	1,8	Combustion + GC
C (Total)	12,0	10,0	15,6	—	17,3	7,8	Combustion + GC
N	<0,3	1,0	1,4	—	1,6	2,1	Combustion + GC
Na	2,8	4,2	5,2	2,9	1,3	1,3	ICP-OES
K	1,0	0,05	1,3	1,4	1,1	2,3	ICP-OES
Mg	1,3	0,3	0,6	0,9	0,7	0,7	ICP-OES
Ca	44	0,9	1,5	2,5	3,1	0,6	ICP-OES
Sr	0,16	0,006	<0,008	0,011	0,0057	0,0055	AA (N_2O-Acet)
Ba	0,12	<0,025	0,058	<0,05	<0,026	<0,025	AA (N_2O-Acet)
Al	0,35	4,8	6,0	7,1	9,3	8,9	AA (N_2O-Acet)
Si	1,1	10,7	18,3	16,7	19,7	18,9	AA (N_2O-Acet)
P	0,2	0,23	0,33	0,33	0,25	0,27	Colourimetric
S	<0,3	<0,3	<0,3	—	—	<0,3	
Ti	0,007	0,29	0,55	0,17	0,12	0,22	ICP-OES
Cr	<0,0025	0,037	0,032	0,031	0,049	0,024	AA (N_2-Acet)
Mn	0,015	0,12	0,080	0,21	0,16	0,023	AA (Air-Acet)
Fe	0,21	2,9	3,9	4,6	4,6	5,4	AA (Air-Acet)
Co	0,045	0,015	0,058	<0,003	<0,001	<0,001	ICP-OES
Ni	0,011	0,013	0,025	0,012	0,014	0,09	AA (Air-Acet)
Cu	0,012	0,017	0,045	0,056	0,054	0,014	AA (Air-Acet)
Zn	0,27	1,3	2,1	0,79	0,78	0,027	AA (Air-Acet)
Cd	<0,001						AA (Air-Acet)
Pb	0,009	0,055	0,11	0,040	0,022	0,008	AA (Air-Acet)

GC – Gas chromatography
ICP-OES – Induction coupled plasma – optical emission spectroscopy
AA – Atomic absorption spectroscopy
Acet – Acetylene

foulants probably result from the galvanized iron pipes used for water reticulation. Iron fouling may be removed by cleaning with chelating agents like citric acid and EDTA. A slightly acidic environment (pH = 5) keeps zinc ions (as well as copper, lead, cadmium and nickel) in solution. pH control at plant D is probably the reason why there was a negligible amount of zinc in this deposit.

Some organic fouling took place at plants B, C and D, while at plant A most of the carbon was present as calcium carbonate scale. Studies are presently underway to establish the nature of the organic foulants. However, the organic composition (total H, C and N was between 13,7 and 22 % for plants B, C and D) appears to be low when compared to similar studies in the literature; for example Peterson *et al.* (1980) found that the foulant deposits, formed when operating on surface waters pretreated with ferric chloride or alum, contained between 49 and 55 % carbon and 5 to 9 % nitrogen.

These preliminary results have indicated the complexity of the fouling problem and the need for further research into pretreatment techniques and cleaning formulations.

Cost benefit analysis of improved operation

It was anticipated that substantial savings would result if the membrane life of a typical plant could be extended to a more reasonable three years, such as is obtained under similar conditions overseas. To this end the following analysis was carried out using plant no. 7 as an example.

The potential savings were estimated by comparing the past annual membrane replacement costs calculated from the survey data; and the projected costs if improved pretreatment and cleaning extended membrane life to 24 000 h.

In the calculations the following assumptions were made:

- The total volume of feed water required will be 3 700 m^3/a. (At present 7 machines with a production capacity of 60 ℓ/h operate at 30 % utilization and 30 % recovery.)
- Utilization will be increased from 30 to 70 % by using 3 machines instead of 7.
- Cost of a 60 ℓ/h membrane module = R850 (recent quote by a supplier).
- Improved pretreatment will consist of pH control with hydrochloric acid to reduce the rate of membrane hydrolysis and will cost 2,5 c/m^3 (75 g of 33 % HCl at 30 c/kg required to reduce 1 m^3 of RWB to pH = 7); in-line coagulation with ferric chloride at 0,11 c/m^3 (10 mg/ℓ $FeCl_3$ at 11 c/kg); and direct filtration by a depth media filter to remove coagulated particulates and colloids at 2 c/m^3.
- membranes will be cleaned once per month and will require two hours of labour at a marginal cost of R5/h; and 150 ℓ (3 membrane modules at 50 ℓ each) of a 0,5 % proprietary cleaning formulation (at R2/kg) gives a chemical cost of R1,50 per clean.

The cost comparison is presented in Table 4. Although these are only 'ball-park' estimates, it is clear that if the measures are successful then the reduction in membrane replacement costs (R4 845-R655 = R4 190) far outweigh the extra pretreatment and cleaning costs (R309).

Conclusions

Although poor experiences in some of the early RO plants were attributed to fouling, the impression is that they resulted from a lack of experience and insufficient attention to pretreatment requirements and essential cleaning of the membranes. The membrane life that has subsequently been achieved at some plants, although still unsatisfactory by international standards, is promising and shows that fouling and membrane deterioration can be substantially reduced.

From the observations made, it is concluded that better plant performance can be achieved if attention is paid to operating records and their interpretation; pretreatment; membrane cleaning; and plant design and utilization.

TABLE 4
COMPARISON OF ESTIMATED COSTS FOR PLANT NO. 7 AT TIME OF SURVEY AND AFTER PROJECTED IMPROVEMENTS (MEMBRANE LIFE INCREASED BY PRETREATMENT AND REGULAR CLEANING).

	At time of survey	After projected improvements
Average productive life of membrane element, h	3 250	24 000
Number of membrane elements replaced per annum	5,7	0,77
Membrane replacement costs, R	4 845	655
Pretreatment costs, per annum		
● pH control at 2,5 c/m^3	–	93
● $FeCl_3$ at 0,11 c/m^3	–	4
● Diatomaceous earth filtration at 2 c/m^3	–	74
Cleaning costs, per annum		
● Labour at R5/h	–	120
● Cleaning chemicals	–	18
Total pretreatment and cleaning costs, per annum	–	309
Total membrane replacement, pretreatment and cleaning costs	R4 845	R964

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