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# TUBULAR REVERSE OSMOSIS TREATMENT OF SECUNDA MINE WATER: A PILOT PLANT INVESTIGATION

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## ABSTRACT

Secunda Collieries experience a positive water balance. Because of the zero discharge principle applied by Sasol and because it is rather difficult to build storage dams, desalination using a tubular reverse osmosis pilot plant was investigated. The objectives were to prove the technical feasibility of the process and to obtain enough information :

(i) to design a full-scale plant and
(ii) to subsequently do economic evaluations regarding operating and capital costs.

With pretreatment consisting of flocculation/clarification and dual media filtration, excellent results were obtained. Average standard flux was 700 $\ell/m^2$.d at pressures of ca 4 MPa and at a water recovery of 70 % (which was the maximum that could be obtained even with the addition of scale inhibitor). The importance of operating the system at feed water temperatures of ca 25 °C was demonstrated. Salt rejection based on conductivity was very constant at ca 96 % resulting in permeate conductivity values of ca 20 mS/m. Fouling, which was mainly inorganic in nature, was reversible and could be removed by citric acid washes and regular replacement of sponge balls.

Treatment of mine water using tubular reverse osmosis is a technically feasible process. However, further research is needed to improve overall water recovery.

## KEY WORDS

Flux, salt rejection, water quality, cleaning actions, temperature.

## INTRODUCTION

As a result of mining activities, there is a positive water balance at the Secunda coal mines which are part of the Sasol operations. As a result of the high total dissolved solids of ca 4 000 mg/$\ell$ and the zero discharge principle which is being applied by Sasol, the water cannot be discharged to natural streams. Up to now dams which are expensive to construct and for which suitable sites are very scarce, had to be built to accommodate this positive water balance. In an attempt to find a permanent solution to this problem, desalination of the mine water using a tubular reverse osmosis pilot plant was investigated.

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The aim of the investigation was to prove the technical feasibility of the process and to obtain sufficient information to be able to :

(i) design a full-scale plant and
(ii) to subsequently do an economic evaluation regarding capital and operating costs for a full-scale plant.

## EXPERIMENTAL

A typical analysis of the mine water as well as general discharge standards are given in Table 1. It is clear that pretreatment was needed for suspended solids removal. A flow diagram of the pretreatment is given in Fig. 1.

Hypochlorite (5 mg/ℓ) was dosed to kill algae before flocculation using 15 mg/ℓ Fe as $FeCl_3$. Clarification was followed by dual media filtration (anthracite/sand).

A flow diagram of the tubular reverse osmosis pilot plant with full-scale size locally manufactured modules is given in Fig. 2. The pH was controlled at 5.5 to 6.5 to minimise hydrolysis of the cellulose acetate membranes. "Flocon" was used at a dose of 5 mg/ℓ as a scale inhibitor ($CaSO_4$ precipitation). An attempt was made to maintain a feed water temperature of 25 °C by using a heat exchanger. Feed flow rate was ca 1.7 $m^3$/h. A tapered configuration was used to maintain a linear flow velocity of 1 to 2 m/s through the modules in order to minimise fouling. Water recovery was controlled by continuous adjustment of the system operating pressure.

The pilot plant is fitted with a flow-reversal system and sponge ball cleaning facilities (one ball for every row of four modules).

From previous work and available computer programs it was predicted that 60 % water recovery could be expected without addition of scale inhibitor and without serious fouling problems. The pilot plant was initially operated at 60 % water recovery and later at 70 % water recovery, in both cases with addition of scale inhibitor.

**TABLE 1 Quality of mine water at different stages as well as river discharge standards**

| | | Raw mine water | Clarifier effluent | RO feed | RO concentrate | RO permeate | River discharge standard |
|---|---|---|---|---|---|---|---|
| pH | | 8.4 | 7.1 | 7.1 | 6.8 | 6.3 | 5.5 to 9.5 |
| Conductivity | mS/m | 450 | 460 | 465 | 975 | 16.7 | 75 |
| P-alk as $CaCO_3$ | mg/ℓ | 14 | N.A. | N.A. | N.A. | N.A. | N.S. |
| M-alk as $CaCO_3$ | mg/ℓ | 183 | N.A. | N.A. | N.A. | N.A. | N.S. |
| $Cl^-$ | mg/ℓ | 220 | 305 | 313 | 754 | 26 | N.S. |
| $SO_4^{2-}$ | mg/ℓ | 2 200 | 2 150 | 2 125 | >3 000 | 14 | N.S. |
| $F^-$ | mg/ℓ | 3.0 | 3.5 | 3.0 | 6.0 | 1.1 | 1 |
| $Na^+$ | mg/ℓ | 917 | 914 | 920 | 2 222 | 26 | 90 |
| $NH_4^+$ | mg/ℓ | <2 | N.A. | N.A. | N.A. | N.A. | N.S. |
| $K^+$ | mg/ℓ | 8 | N.A. | N.A. | N.A. | N.A. | N.S. |
| $SiO_2$ | mg/ℓ | 4.3 | N.A. | 4.3 | N.A. | N.A. | N.S. |
| $Ca^{2+}$ | mg/ℓ | 176 | 176 | 176 | 452 | 2.0 | N.S. |
| $Mg^{2+}$ | mg/ℓ | 80 | N.A. | N.A. | N.A. | N.A. | N.S. |
| $Fe^{2+}$ | mg/ℓ | 0.3 | 0.3 | 0.3 | 0.7 | 0.1 | N.S. |
| Suspended solids | mg/ℓ | 174 | 76 | 47 | 347 | <10 | 20 |
| COD | mg/ℓ | 70 | 45 | 40 | 900 | <2 | 75 |
| Turbidity | NTU | 9.0 | 6 | 0.9 to 1.5 | 9.0 | 0.9 | N.S. |
| TDS | mg/ℓ | 3 959 | 3 919 | 4 092 | 8 720 | 104 | N.S. |

N.A. = Not analysed; N.S. = Not specified.

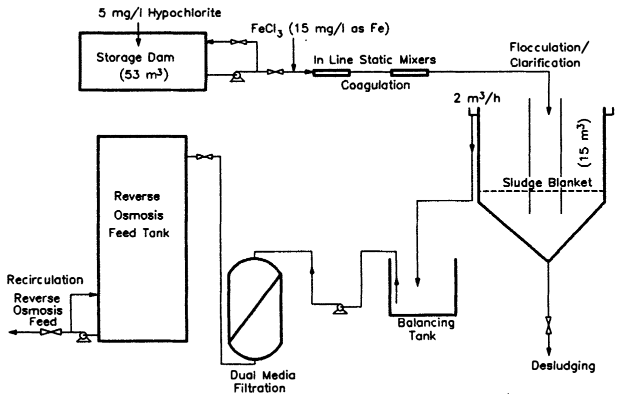


**Fig. 1. Pretreatment for reverse osmosis**

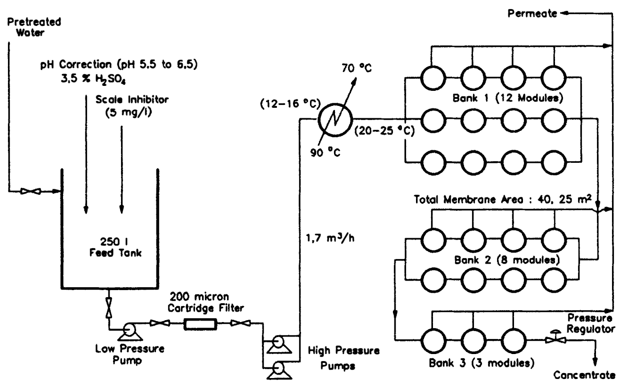


**Fig. 2. Schematic presentation of the tubular reverse osmosis pilot plant**

## RESULTS AND DISCUSSION

### Flux and the effect of pressure and temperature

Flux is the volume of product water flowing through a unit membrane area per unit time and is expressed as litre/$m^2$.day (*ℓ*md). Standard flux is the actual flux standardised to a pressure of 4 MPa and a temperature of 25 °C. Since the pilot plant was operated at constant water recovery and the latter is a function of feed and product flow rates, it can be expected that permeate flow rate (and actual flux) would be constant for a specific water recovery (feed flow rate was constant since a positive displacement pump was used). Actual fluxes were fairly constant (Fig. 3) for each of the two water recoveries with average values of 625 and 697 *ℓ*md for 60 and 70 % water recoveries respectively. Standard fluxes were normally higher than the actual fluxes and were also less constant as a result of pressure and temperature effects. Average standard flux was 700 *ℓ*md. The sharp decrease in standard flux during the first 40 h of operation is a result of membrane compaction which is normal for reverse osmosis systems

During the 1 800 h investigation, the standard flux was always within the design figures of 500 to 800 *ℓ*md for a standard 1 000 mg/*ℓ* NaCl solution.

There is an indirect relation between temperature and pressure i.e. for a fixed flux, pressure will increase with decreasing temperatures or in other words, if the temperature increases, the same flux can be maintained at lower operating pressures. The relation is very clear from Fig. 4, which compares pressures with temperatures, and Fig. 3 (flux).

The initial sharp increase in pressure (Fig. 4) is a result of membrane compaction. The following gradual increase in pressure is a result of the gradual decrease in temperature (Fig. 4). With the increase in temperature (after 859 to 1 234 h of operation), a decrease in pressure can be observed. The most important aspect here, was that the higher fluxes at 70 % water recovery could be obtained at lower operating pressures by only operating at the optimum temperature of 25 °C. The importance of operating at optimum temperatures is very clear.

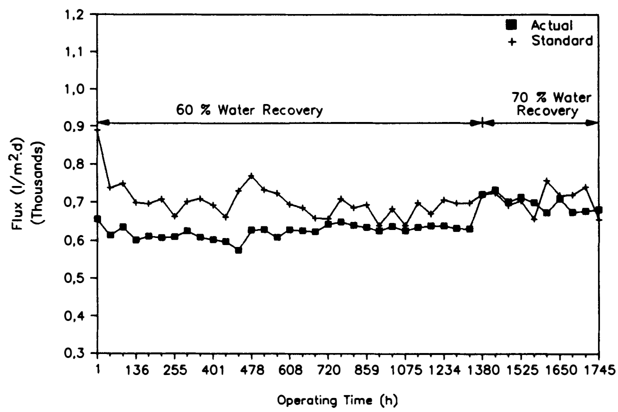


**Fig. 3. Flux versus operating time**

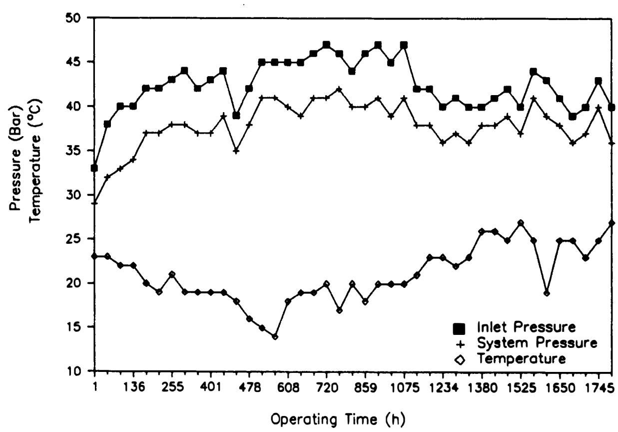


**Fig. 4. Relationship between pressure and temperature**

## Salt rejection and conductivity

Salt rejection was fairly constant throughout the test period of ca 1 800 h with an average of 96 % (Fig. 5). Design figures are 90 to 96 % with 1 000 mg/ℓ NaCl and 96 to 98 % with 1 000 mg/ℓ $Na_2SO_4$ solutions respectively. The results obtained indicated that the performance of the pilot plant was excellent.

The conductivities of the feed, concentrate and permeate were ca 460, 1 000 and 20 mS/m respectively (Fig. 6). Taken into consideration that the discharge standard was ca 75 mS/m, it is clear that a good quality permeate was obtained.

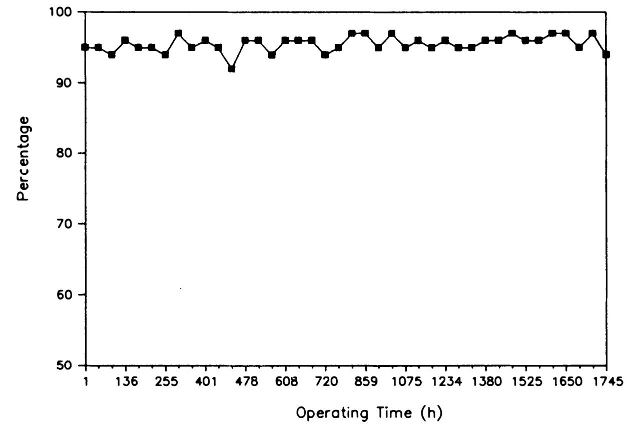


**Fig. 5. Salt rejection**

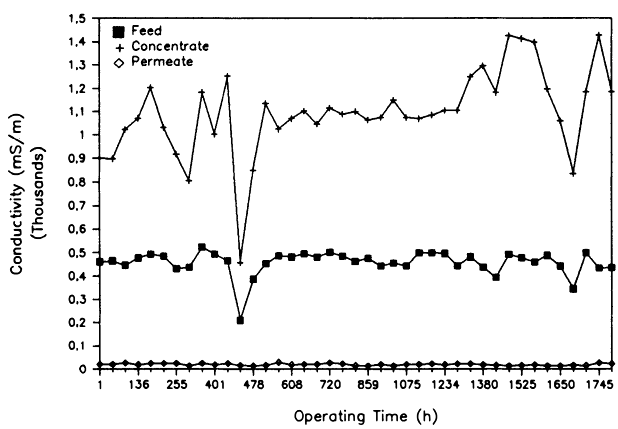


**Fig. 6. Conductivities of feed, permeate and concentration**

## Water quality

Silt density index (SDI) is normally used as criterion for reverse osmosis feed water quality. A SDI of 5 or less is preferred but could never be reached; not even in the laboratory on a very small scale and with available pretreatment. Values obtained during the test run with the pretreatment described in Fig. 1, varied between 5.6 and 6.2 with an average of 6.1. Despite this relatively high SDI the membranes performed well.

Average values of analyses obtained for the water stream at different stages are given in Table 1. Suspended solids decreased from 174 mg/ℓ to 76 mg/ℓ during flocculation and further to 47 mg/ℓ after dual media filtration. Turbidity decreased from 9 NTU to between 0.9 and 1.5 NTU after filtration which indicates that pretreatment was quite effective for suspended solids removal.

Removal of bivalent ions such as $SO_4^{2-}$ and $Ca^{2+}$ was 99 % whereas removal of monovalent ions such as $Na^+$ was 97 %. All concentrations of components in the permeate were well below the river discharge standard (Table 1).

The quality of the permeate is of such a nature that it can be used in the Sasol oil from coal factories. The quality of the permeate is better than that of the Vaal River with the exception of $Cl^-$ which is 10 mg/ℓ higher in the former compared to the latter.

## Cleaning of membranes

Monitoring and the need for cleaning : A decrease of 5 % in standard flux or a 10 % increase in operating pressure was an indication that membranes needed to be cleaned. A modular investigation, using a 1 g/ℓ NaCl solution, was used to determine the degree of fouling and also to identify individually fouled membranes. This investigation was done before and after each cleaning action in order to evaluate the effectivity. All the results obtained during the investigations are given in Table 2.

**TABLE 2 Modular survey using 1 g/ℓ NaCl solution**

| Hours | Situation | Standard flux ℓ/m².d | Salt Rejection, Percentage | | |
|---|---|---|---|---|---|
| | | | Bank 1 | Bank 2 | Bank 3 |
| 0 | Prior to investigation | 915 | 98 | 98 | 98 |
| 438 | Before Biotex wash | 832 | 96 | 95 | 95 |
| | After Biotex wash | 886 | 98 | 97 | 97 |
| | After citric acid wash | 907 | 97 | 97 | 96 |
| 720 | Before citric acid wash | 827 | 96 | 96 | 96 |
| | After citric acid wash | 929 | 96 | 96 | 96 |
| 1 075 | Before replacement of sponge ball | 841 | 95 | 94 | 94 |
| | After replacement of sponge ball | 911 | 96 | 95 | 94 |

Design (1 g/ℓ NaCl solution); Flux - 700 to 1 000 ℓmd; Salt rejection : NaCl - 90 to 96 %, $Na_2SO_4$ - 96 to 98 %.

*In situ* cleaning (CIP) : Three cleaning actions were compared i.e. biological wash, citric acid wash and regular replacement of sponge balls.

The biological wash was performed after 438 h of operation by circulating an enzyme active washing powder such as Biotex, through the system followed by rinsing with permeate. The effect of the Biotex wash was relatively small (Table 2) indicating that fouling was not organic in nature. The effect of shock chlorination in order to reduce the frequency for biological washes was not investigated.

The citric acid wash was performed after a further 282 h of operation by adjusting a 2 % citric acid solution to pH 4 using ammonium hydroxide and to circulate this solution through the reverse osmosis system. During the cleaning action, the citric acid solution turned yellowish indicating removal of iron from the membranes. This cleaning action was effective in restoring flux of the membranes (Table 2). It was also reflected in the lower pressures that had to be applied for the treatment of minewater after the cleaning action had been completed.

Following a recommendation from the suppliers of the membranes, the sponge balls were replaced with new ones when cleaning of the membranes was again due after a further 355 h of operation. The effect was quite remarkable as can be seen in Table 2. Flux could be fully restored without taking any further action. This is important taking into consideration the cost of chemical cleaning programs.

General : In general it was found that fouling took place throughout the system and not in certain module racks or individual modules. However, slightly higher fouling rates were observed in the last module rack which treated the most concentrated water. The fact that flux could be restored, was an indication that fouling was reversible.

## Permanent fouling

Permanent fouling which will eventually determine membrane life, is observed when, over a long period, there is a decrease in standard flux after the membranes had been cleaned properly and had been tested under standard conditions using a standard NaCl solution. During the investigation of 1 800 running hours, no permanent fouling could be observed (Fig. 7). However, a longer test period of 6 000 to 8 000 h is needed to make an accurate conclusion regarding membrane life.

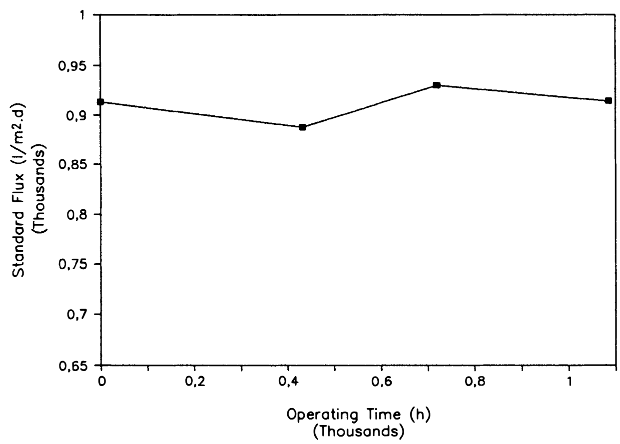


**Fig. 7. Standard flux using a 1 g/ℓ NaCl solution**

A slight decrease in salt rejection (from ca 98 to 96 %) using the standard NaCl solution, was observed (Fig. 8). This could be an indication that a very small degree of physical erosion could have taken place. However, salt rejection was always well within the specification when mine water was treated.

## Optimisation of the system

Since flocculation contributes towards operating costs and the presence of Fe in the feed water to the reverse osmosis system is not desirable, the effect of omitting flocculation from the pretreatment was investigated.

Without flocculation much higher fouling rates were experienced. The frequency for *in situ* cleaning was much higher. With flocculation, cleaning steps had to be performed every ca 350 h whereas cleaning was needed after only ca 28 h when flocculation was omitted (Fig. 9)

The cartridge filters also had to be replaced more frequently. It therefore seems as if pretreatment is essential.

Although the aspect was not fully investigated, available results indicated that a maximum water recovery of 74 % could be achieved with addition of scale inhibitor before scale formation would take place. This corresponds very well with the computer prediction of 69 %.

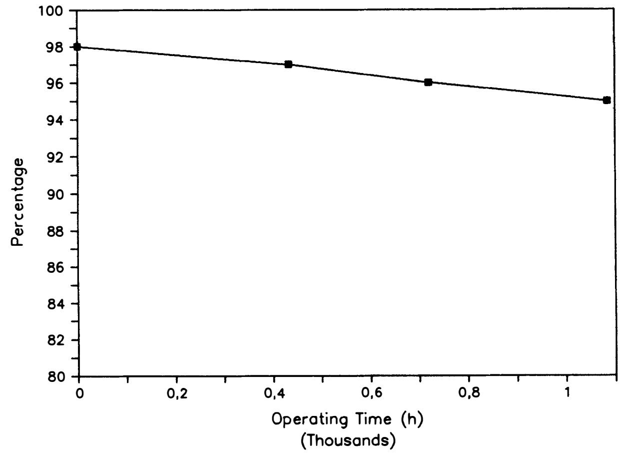


**Fig. 8. Salt rejection using a 1 g/ℓ NaCl solution**

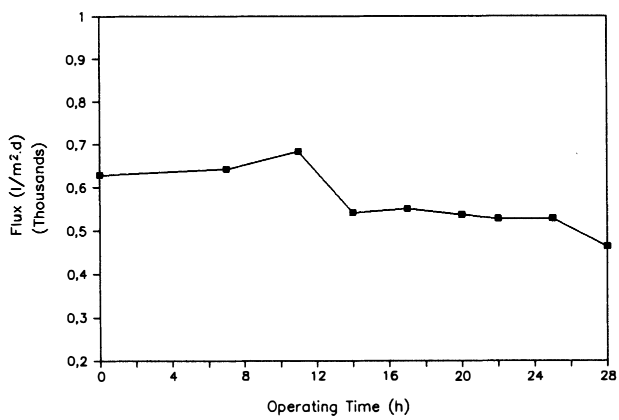


**Fig. 9. Flux versus time**
**(system operated without flocculation)**

## CONCLUSION

Reverse osmosis treatment of minewater at 70 % water recovery is a technically feasible process.

For optimal operation, the feed water temperature should be kept at 25 to 27 °C.

Optimal use of sponge balls for physical cleaning of the membranes (replacement every 3 weeks) will drastically reduce the need for expensive chemical cleaning programs.

Based on the results obtained, the suppliers have indicated that a membrane life of at least three years could be expected.

Further research is needed :

(i) to increase water recovery by application of, for instance, the SPARRO process on the reverse osmosis concentrate stream and
(ii) to determine membrane life more accurately.

From the data obtained it would be possible to do an economic evaluation regarding the application of tubular reverse osmosis treatment of Secunda mine water.

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