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# Assessment of fouling of an RO process dedicated to indirect potable reuse

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## ABSTRACT

Autopsies of three elements of a three-stages reverse osmosis process (lead, central and tail elements of respectively Stage 1, 2 and 3) treating microfiltered wastewater effluent were performed to quantify the propensity of each stage to foul. As expected, results showed fouling on Stages 1 and 2 to be predominantly biofouling whilst Stage 3, the most heavily fouled, was subject to most pronounced scaling, predominantly by calcium phosphate. Chloramine dosing in the RO feed water appeared largely ineffective for membrane biofouling mitigation due to its low rejection by the RO membrane leading to a very low residual in the RO retentate stream. Five antiscalants (three commercialised and two under development) were tested at pilot scale and their efficiency for scaling mitigation compared. Results showed wide variations in effectiveness, with the novel reagents showing the most promising results. However, none of the antiscalants tested succeeded in completely suppressing scaling without the addition of sulphuric acid. A cost analysis showed the operating cost to be most sensitive to pH adjustment, with reduced acid dosing requirement providing a significant cost benefit.

*Keywords:* Membrane autopsy; Reverse osmosis; Antiscalant; Calcium phosphate; Wastewater reuse

## 1. Introduction

Increasing freshwater scarcity continues to further the technological progress and economic benefit of wastewater reuse, predominantly to preserve freshwater resources. The use of an integrated membrane system (IMS), the combination of micro/ultrafiltration (MF/UF) followed by reverse osmosis (RO) membranes, represents an important option for municipal wastewater reuse. Such schemes are currently used for advanced treatment of municipal effluents for reuse in industrial processes [1], environmental protection/restoration [2], irrigation [2,3] and indirect potable reuse (IPR) [4].

A major drawback of such systems is the fouling of RO membranes. Fouling leads to an increase in feed pressure of the system to maintain a constant flow, such that the energy demand also increases. Given that operation beyond some threshold pressure is not tenable, chemical cleaning or dosing for fouling amelioration becomes necessary. Both chemical cleaning and fouling appear to shorten the membrane life, leading to significantly increased operational costs due to membrane replacement [5,6]. It is therefore ultimately necessary to employ appropriate pretreatment to control and/or ameliorate fouling.

Four types of fouling arise on RO membranes: colloidal, biological, organic and inorganic [7]. Biofouling of RO membranes can be controlled through ensuring a chloramine residual in the influent [8]. Organic fouling

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can be minimised by applying an appropriately conservative flux [7]. Inorganic fouling, or scaling, by sparingly soluble salts such as calcium carbonate is suppressed by antiscalant (chemical) dosing, pH reduction and/or reduced recovery [9]. Colloidal fouling, as well as biofouling, is controlled by pre-treatment [7,10]. However, notwithstanding pre-treatment, fouling is always experienced to some extent. Autopsy of the RO membrane, whilst providing only a destructive examination, provides a means of assessing foulants and possible loss of membrane integrity, thereby informing appropriate pretreatment [5].

This paper provides results from a study of RO membrane autopsies relating to an IPR process, along with an appraisal of five antiscalant reagents.

## 2. Materials and methods

### 2.1. Pilot plant overview

The 600 $m^3\ d^{-1}$ IPR pilot plant has been described elsewhere [11]. Final effluent from a conventional activated sludge plant passes through a pre-filter (Bollfilter) before being filtered by a submerged microfiltration (MF) unit (Memcor). The MF permeate then passes through a RO system (Hydranautics) and on to an advanced oxidation process (AOP) and a degasser tower before undergoing pH correction. Chloramine dosing for biofouling control can take place at three different points in the process, pre pre-filter, pre MF and pre RO. Antiscalant and sulphuric acid are dosed pre-RO to control scaling. The plant is fully automated and data recorded on a supervisory control and data acquisition (SCADA) system. The RO process has three stages (Table 1) and is fed with tertiary MF effluent (Table 2). The RO process is fed from a balance tank by a feed pump at a constant flow rate of 8.2 $m^3\ h^{-1}$. 20 μm cartidge filters are used to protect the RO membranes. A high pressure pump is used to generate feed pressure.

Table 2
Average RO feed water quality

| Parameters | Values |
|---|---|
| Conductivity (μS $cm^{-1}$) | 1146 ± 38 |
| TOC (mg $l^{-1}$) | 8.0 ± 1.8 |
| pH | 7.3 ± 0.1 |
| Temperature (°C) | 13.7 ± 0.88 |
| Turbidity (NTU) | 0.04 ± 0.00 |
| Alkalinity (as $CaCO_3$) (mg $l^{-1}$) | 195 ± 15 |
| Phosphate (as $PO_4^-$) | 4.09 ± 0.75 |

### 2.2. Autopsies

Membrane autopsies were conducted on three RO elements: the lead element of the first stage, central element of the second stage, and the end element of the third stage, to examine fouling. This was performed after running the process at 85% recovery, a flux of 19 l $m^{-2}\ h^{-1}$ (LMH) and at a pH of 6.5. The antiscalant used was Antiscalant A at a dose of 2 mg $l^{-1}$ (Table 3). Autopsies comprised stereo and optical microscopic investigation, scanning electron microscopy coupled with energy dispersive X-ray (SEM-EDX), chemical analysis by inductively-coupled plasma optical emission spectrometer (ICP-OES), and total cell count determination using DAPI (4,6-diamidino-2-phenylindole) dye staining and fluorescence microscopy. Membrane analyses were performed by IWW Rheinisch-Westfälishes Istitut für Wasser Beratungs und Entwicklungsgesellshaft mgH (Germany).

### 2.3. Chemicals

Monochloramine, formed using sodium hypochlorite and ammonium sulphate in a 3:1 mass ratio (N:Cl), was dosed in-line upstream of the pre-filter at a constant dose of 1 mg $l^{-1}$ using a static mixer to control biofouling.

Table 1
RO process specifications

| | |
|---|---|
| Manufacturer | Hydranautics |
| Membrane type | ESPA2 (first stage) and ESPA2-4040 (second and third stage) |
| Materials | Composite polyamide |
| Area/element | 37.1 $m^2$ (ESPA2) and 7.9 $m^2$ (ESPA2-4040) (leading to an overall area of 365 $m^2$) |
| Configuration | Array 1:2:1 (six elements per vessel) (Total number of elements: 24) |
| Operating pH | 2–10.6 |
| Standard CIP | CIP 1: Recirculation of permeate water at pH 2.5 for 30 min followed by 1 h soaking<br>CIP 2 and 3: Recirculation of permeate water at pH 2.5 for 30 min on all stages flowed by 1 h soaking |

Table 3
Antiscalants properties

| Antiscalant | Commercial status | Target scalant | Type |
|---|---|---|---|
| A | Commercialised | Calcium carbonate | Phosphonic acid |
| B | Commercialised | Calcium carbonate<br>Calcium phosphate | Phosphonic acid |
| C | Commercialised | Calcium phosphate<br>Calcium carbonate | Phosphonate and carboxylic acid |
| D | Non-commercialised | Calcium carbonate<br>Calcium phosphate | Unknown |
| E | Non-commercialised | Calcium carbonate<br>Calcium phosphate | Unknown |

Antiscalant and acid for scaling inhibition were dosed in-line upstream of the RO process using another static mixer. Previous scoping trials determining optimum operating envelopes for scaling mitigation in the RO process using a single commercial antiscalant [11] established that both antiscalant and acid dosing were necessary to control scaling. With this antiscalant (reagent A, Table 3), the highest design recovery (85%) demanded adjustment to a pH below 6.25 (by dosing with sulphuric acid at ≈1.4 $l\ h^{-1}$) combined with an antiscalant dose of 2 $mg\ l^{-1}$.

To attempt to reduce sulphuric acid consumption, four different antiscalants (B, C, D and E, Table 3), all claimed by the suppliers to be effective against both the phosphate and carbonate salts of calcium, were assessed. Of these, two were commercialised and two were under development. For each antiscalant the appropriate dose, ranging from 2 to 4 $mg\ l^{-1}$, was determined based on feedwater quality and projections informed by the suppliers' own respective software.

All experiments were conducted under challenging conditions of a mean flux of 19 LMH and an overall recovery of 85%. Experiments were stopped once a 10% decrease in permeate flow for a single stage was observed, since this represents the point at which chemical cleaning is generally advised. Sulphuric acid was used to adjust the pH, which ranged from 6.35 to the natural pH of 7.2.

## 3. Results and discussion

### 3.1. Fouling determination and membrane integrity assessment

Fouling on first and second stage elements was observed as a brown, highly hydrated slimy deposit located at the intersections of the spacer material. This deposit was mainly composed of aggregated and suspended bacteria, with a few embedded inorganic particles. For the third stage, a brown-tainted particulate deposit was spread all over the membrane surface and was mainly crystalline inorganic particles, 1–40 μm in size. Bacterial aggregates and suspended cells were also observed (Table 4), and were more concentrated in Stages 2 and 3 than in Stage 1.

The inorganic component of the fouling layers of the three stages was analysed by ICP-AES (Table 4). The deposit analysed at Stages 1 and 2 was found to have a very high water content (≈97.5%) compared with that sampled at Stage 3 (78%). Data for mass loss on ignition at 550°C revealed the organic content of the deposits to be 87, 70 and 19% for Stages 1, 2 and 3 respectively. Carbonates, as detected by loss on ignition at 900°C, were only present in significant amounts for the Stage 3 deposit, providing 7.5% of the dry weight. The inorganic component of the deposits comprised mainly Ca and P; inorganic phosphates were detected in the deposits from all three RO modules. In addition, Na, Al, Cu, Fe, Mg, Pb, Zn and Si were found at low concentrations. In comparison, the amounts of Ca and P – most likely from calcium phosphate – were significantly higher on the membrane surface of Stage 3. Stage 2 showed slightly elevated Ca and P concentrations (about 500 times less than for Stage 3) whereas Stage 1 had the lowest Ca and P deposits (2000–3000 times less than Stage 3).

Fig. 1 compares the inorganic element concentration on Stages 1 and 3, and the expected concentration on Stage 3 if the deposition of inorganic particles is assumed proportional to the bulk retentate concentration, that is, a concentration factor of 100/15 between the lead elements of Stage 1 and the tail elements of Stage 3. As shown in Fig. 1, the concentrations of Ca and P are significantly greater than that based simply on retentate concentration, indicating calcium phosphate ($Ca_3(PO_4)_2$) formation. According to data in Table 4, a maximum Ca concentration of 40 $mg\ cm^{-2}$ is associated with phosphate. It must therefore be assumed that some calcium carbonate or other calcium salts (such as calcium salts of

Table 4
Elemental composition of fouling deposits on membrane surfaces determined by ICP-OES and total cell count determined by DAPI staining and fluorescence (< : undetected)

| Stage | Stage 1 | Stage 2 | Stage 3 |
|---|---|---|---|
| Wet weight (g m$^{-2}$) | 3.213 | 4.901 | 0.858 |
| Dry weight (g m$^{-2}$) | 0.077 | 0.127 | 0.190 |
| Dry weight (% of wet weight) | 2.4 | 2.6 | 22.1 |
| Loss on ignition 550°C (% of DW) | 86.6 | 70.1 | 19.4 |
| Loss on ignition 900°C (% of DW) | 87.3 | 71.9 | 26.9 |
| Element (ICP analysis) (mg m$^{-2}$) | | | |
| Al | 0.2256 | 0.6666 | 1.1234 |
| Ca | 1.4460 | 9.0671 | 46.1372 |
| Cd | 0.0001 | 0.0015 | 0.0063 |
| Co | 0.1031 | 0.0039 | 0.0266 |
| Cr | 0.0257 | 0.0294 | 0.0394 |
| Cu | 0.0386 | 0.1397 | 0.4262 |
| Fe | 0.2047 | 0.2588 | 0.4725 |
| K | 0.1973 | < | < |
| Mg | < | < | 0.9605 |
| Mn | 0.0039 | 0.0147 | 0.0926 |
| Na | 0.3856 | 1.4654 | 0.5994 |
| Ni | 0.0495 | 0.0093 | 0.0256 |
| Pb | 0.0109 | 0.9851 | 0.1852 |
| Total P | 1.4107 | 5.3913 | 29.1573 |
| Si | 0.3406 | < | 0.9004 |
| Zn | 0.0100 | 0.1245 | 0.5454 |
| Total cell count cm$^{-2}$ | $3.5 \times 10^6$ | $6.5 \times 10^6$ | $6.7 \times 10^6$ |

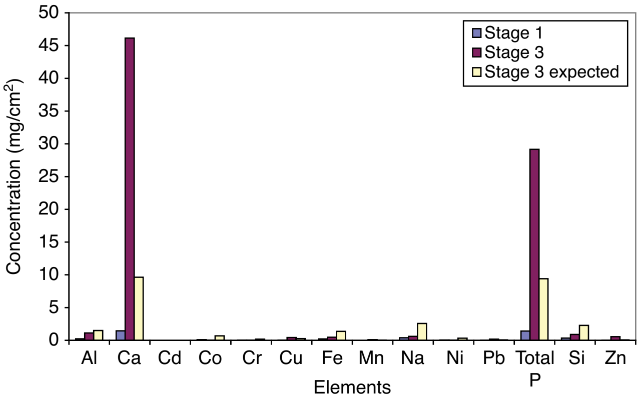

Fig. 1. Elements concentration (mg cm$^{-2}$) for Stage 1 and 3, and expected concentration of Stage 3 if inorganic fouling follows the concentration factor.

antiscalant) scale must also be present. The phosphorus may also have been associated with both biomass and the antiscalant itself, especially for Stage 1.

In general, membrane deposits in Stages 1 and 2 were dominated by organic matter (respectively 86.6% and 70.1%) with calcium phosphate making up most of the inorganic component. The Stage 3 deposit was conversely lower in organic content (19.4%) and higher in concentration of calcium phosphate with some metal carbonates in the inorganic fraction. A similar proportion of scale to organic matter (80%:20%, where most of the scaling was calcium phosphate) was observed by Ning and Troyer [12], and trends in organic/inorganic content over the three stages were largely in agreement with those reported by Xu et al. from their membrane autopsies of a two-stage pilot-scale RO process treating microfiltered municipal wastewater [8].

Fouling of the RO membrane was, as expected from normal practice, manifested as decreased permeate flow accompanied by decreased salt rejection and increased differential pressure at the third stage of the array. An acid clean at pH 2.5 completely recovered the flow on the third stage. This suggested that, whilst organic and/or biological fouling was evident, scaling was primarily responsible for reduced permeation.

### 3.2. *Scaling minimisation*

The choice of antiscalant A was originally made on the assumption of calcium carbonate being the primary scalant, contrary to the outcomes of the autopsy which suggested calcium phosphate scaling to predominate.

Table 5 shows that, as expected, the volume of water treated before a chemical cleaning is higher at lower pH. Antiscalant B appeared more efficient at low pH than antiscalant A. However at higher pH, antiscalant B was less efficient. Antiscalant C proved less efficient than antiscalant A, even though the former is designed for

Table 5
Volume of water treated (m$^3$) before a 10% decrease of the flow on the third stage for each antiscalant as a function of the pH for each tested antiscalant

| pH | Volume of water treated (m$^3$) before a 10% decrease of the flow on the third stage for each antiscalant | | | | |
|---|---|---|---|---|---|
| | A[1] | B | C | D | E |
| 6.35 | 1247 | 3865 | – | 12,841 | 5634 |
| 6.5 | 908 | – | 843 | 1114 | 5556 |
| 6.65 | 430 | – | – | – | 979 |
| 6.75 | 190 | 92 | – | – | – |
| 7.2 | 0.104 | 0.004 | 0.003 | – | – |

[1] Empirical model data based on pilot plant performance data [11].

calcium phosphate scaling suppression. It is possible that it was dosed at too high a concentration, causing clogging of the membrane channels. Antiscalants D and E, both of which are under development, provided better results than the commercialised reagents at sulphuric acid acidified pHs. It is likely that at pH values below 6.5, the limiting scalant had changed from calcium phosphate to some unidentified compound.

At the unadjusted pH of 7.2, the RO membranes immediately scaled for all of the commercialised antiscalants. This is contrary to the projection obtained from the antiscalants suppliers' software, which indicated that no pH adjustment was required for pH below 7.6 for the reagents to be effective. Although this was already known for antiscalant A [11], this insufficiently conservative projected performance was also noted by Xu et al., who reported significant amounts of calcium, aluminium and phosphorus scaling on the membrane whilst projections estimated that only barium sulphate would precipitate without antiscalant. Greenberg et al., who compared five different antiscalants, also reported all tested antiscalants as being ineffective against calcium phosphate scaling when treating secondary wastewater [13]. This may be due to calcium phosphate arising in colloidal form in wastewater effluent [12], such that it passes through the MF and blinds the RO membrane surface; antiscalants are not effective against suspended compounds since they act by suppressing precipitation. Ning and Troyer also suggested pH control to be critical, since phosphate nanoparticle concentration changes within the pH range of 5–7 [12].

### 3.3. Operating cost

The choice of antiscalant also impacts on the capital and operating costs of the RO process. From the results obtained in the current study, it is evident that the chemical cleaning interval and acid dose required depend on the choice of antiscalant. Cleaning-in-place (CIP) of the RO process can take up to 6 h depending on the extent of the scaling. At longer CIP intervals the percentage downtime decreases and the net flux increases commensurately, reducing the required number of membrane elements. Acid dosing can be reduced at lower recoveries. There is therefore a trade-off between various design and operating parameters and, according to the results, it appears that the operating envelop defined by Raffin et al. could be enlarged [11]. The reduction of acid dosing also impacts favourably on site health and safety issues relating to sulphuric acid storage.

Table 6
Ranges and prices of the different parameters

| | Parameters range | Reagent price (£ kg$^{-1}$) |
|---|---|---|
| Sulphuric acid (mg l$^{-1}$) | 0–185[1] | 0.17 |
| Antiscalant (mg l$^{-1}$) | 2–4 | 1.4–4 |

[1] Corresponding pH range: 6.25–7.2.

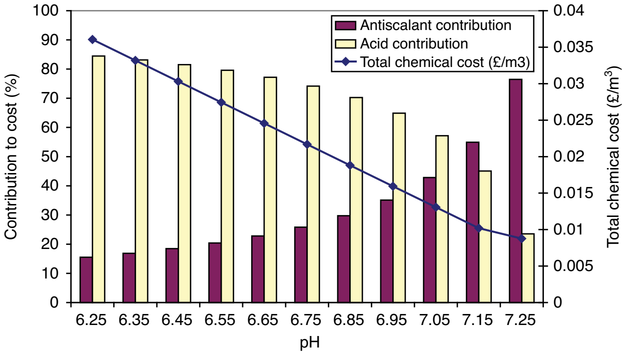

Fig. 2. Contribution of acid and antiscalant to cost and total chemical cost as a function of the adjusted pH (from a pH of 7.25 and an alkalinity of 195 mg l$^{-1}$ as $CaCO_3$) for Antiscalant *A* (concentration of 2 mg l$^{-1}$).

A sensitivity analysis was performed to assess the operating cost saving arising from chemical dosing (pH and antiscalants dose). Ranges and costs of the parameters used for the sensitivity analysis are summarised in Table 6. Fig. 2 represents the contribution of acid and antiscalant to chemical costs as a function of the adjusted pH. Similar trends are obtained with the other antiscalants. Costs of chemical dosing range from £0.008 m$^{-3}$ feed water for zero acid dosing and 2 mg l$^{-1}$ of antiscalant to £0.040 m$^{-3}$ feed water when dosing to a pH of 6.25 and antiscalant dose of 4 mg l$^{-1}$. On average, the operating cost incurred by chemical dosing is decreased by 7.8% for each 0.1 unit increase in the adjusted pH, which correspond to a decrease of £0.003 m$^{-3}$. An additional 0.55% (≈0.0002 £ m$^{-3}$) reduction arises with 0.1 mg l$^{-1}$ decrease in antiscalant dose. However, the each cost contribution from the acid and the antiscalant depends on their respective doses: the higher the dose of antiscalant the higher the pH that can be sustained and the greater the contribution of antiscalant cost to the overall operating cost (Fig. 2). Over the range of conditions studied, pH adjustment was found to have the greatest influence on operating cost, with possible operational cost reductions of 67–77% for zero acid dosing compared to adjustment to pH 6.25.

### 3.4. Biofouling minimisation

In the existing scheme biofouling is minimised by pre-treating the RO influent by MF, along with dosing to 1 mg l$^{-1}$ with chloramine. However, this pretreatment was found not to eliminate microbial activity in the RO feedwater, where a colony count at 22°C of ≈180 ml was recorded. As reported by Lazarova et al., low molecular weight dissolved organics passed through the MF membrane and provide nutrients for microorganisms immobilised in biofilms [3]. In this study, the DOC concentration reached up to 10 mg $l^{-1}$ in the RO feed water. Villacorte et al. showed that a biofilm may result from the deposition of transparent exopolymer particles arising from pre-treatment [14]. The concentration of phosphate in the RO feed water, especially when treating wastewater, may also contribute to biofouling. Vrouwenvelder et al. demonstrated that reduced phosphate concentrations can significantly constrain biomass accumulation, and it is well known that phosphonate-based antiscalants may promote RO biofouling by increasing phosphate concentration in the presence of an organic carbon substrate [15]. These authors advised limiting phosphate levels by implementing pre-treatment such as coagulation, and avoiding phosphonate-based antiscalants when treating wastewater effluents rich in organic substrate. Organic polymer-based antiscalants with highly assimilable organic compounds have been found to have a high biofouling potential, apparently from their nutrient content [16].

Volatile organic matter was found on membranes in all stages, portions of which are likely to have derived from biofilm. A higher bacteria count per square centimetre was recorded for the tail elements of the third stage (Table 4), corroborating the reported results of Xu et al. [8]. According to these authors, the chloramine residual decreased along the length of the module and between successive stages since its rejection by the RO membrane is low. In the current study the chloramine concentration in the RO permeate was found to be higher than that recorded in the feed water, with no residual chloramines in the retentate. Clearly, the impact of chloramines dosing on biofouling mitigation in the concentrate scheme would be expected to be negligible under such conditions.

## 4. Conclusions

Membrane autopsies have been conducted to assess fouling propensity of a RO membrane process treating wastewater effluent, along with pilot trials of a range of antiscalants which were compared with reference to scaling mitigation.

Autopsies showed the first and the second stages of an RO plant treating microfiltered secondary municipal wastewater to be subject to significantly less scaling than the third stage, respectively 15 and 4 times less than Stage 3. Scaling was mostly associated with calcium phosphate, although calcium carbonate was also present. Biofouling was observed on the three stages with higher concentrations at the third stage. This was explained by a lack of chloramine residual as its concentration decreases across the array.

Results show that antiscalant should be chosen carefully as their efficiency regarding scaling minimisation differs from one antiscalant to another. A future generation of antiscalants is being developed and showed some promising results. However, none of them appear capable of avoiding scaling without the addition of sulphuric acid. This might reflect by the relative inefficacy of the antiscalant against calcium phosphate colloidal fouling.

A simple analysis enabled different antiscalants to be appraised. A cost analysis quantified the benefit of employing a more effective antiscalant at more neutral pH levels: a small increase in adjusted pH can significantly reduce operational costs associated with acid consumption. An operating cost reduction of up to 77% can be obtained by increasing adjusted pH from 6.25 to 7.25 at wastewater alkalinity of 195 mg $l^{-1}$ as $CaCO_3$. Capital costs might also be reduced since slightly fewer membrane elements are required at longer chemical cleaning intervals and commensurately higher net fluxes.

From this study, it is clear that more attention is required regarding pre-treatment of the RO process to limit different types of fouling.

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