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REMEDIATION OF LAUNDRY WASTEWATER WITH A LOW-PRESSURE AROMATIC POLYAMIDE THIN-FILM COMPOSITE REVERSE OSMOSIS MEMBRANE FOR MEMBRANE FOULING MINIMISATION AND REUSE APPLICATION

In areas with limited freshwater resources, affordable technologies can remediate greywater for reuse applications and increase the water supply. These wastewaters contain various chemicals, which make them challenging to treat. Reverse osmosis (RO) membrane systems could be the solution to removing these harmful chemicals. Membrane fouling has been investigated and using a commercial antiscalant to treat laundry wastewater effluent with a polyamide (PA) thin-film composite (TFC) reverse osmosis membrane. Attenuated total reflectance Fourier transform infrared spectroscopy (ATR-FTIR), energy-dispersive X-ray spectroscopy (EDX), and scanning electron microscopy (SEM) were used to assess the antiscalant effects. The anionic surfactant rejection was above 99.8% for experimental tests conducted. The average COD removal rate was in the range of 91–96%, irrespective of the antiscalant dosing. However, the presence of antiscalant at the dose of 8 mg/dm^3 significantly reduced fouling intensity. The flux decline ratio amounted to 56 and 72% for the RO process with antiscalant and no antiscalant dosing, respectively.

1. INTRODUCTION

Water intake is increasing at double the rate of the world's population, according to Guppy and Anderson [1]. Unless steps are taken to limit water use or eliminate contaminants from wastewater for reuse, the existing water shortage can only worsen [2, 3]. Domestic [4], industrial [5], hospital and laundry wastewater are examples of wastewater

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sources reclaimed [6]. Laundry wastewaters carry a mixture of organic (soaps, fat, detergents, oil, chlorinated and aromatic solvents, and microbial organisms) and inorganic substances (sand and soil dust, heavy metals, metal ions, and particles) [7]. Laundry detergents have long been considered significant sources of pollution in households, workplaces, and industries, particularly in large cities [8]. Since so many species depend on surface water, pollution can cause substantial shifts in biota. Anionic surfactants can bind to biological molecules. Protein and peptide binding changes the polypeptide chain, altering the surface charge. This change in biological function can damage habitats and modify biodiversity [8]. Physical water treatment methods, such as microfiltration and ultrafiltration, can effectively minimise turbidity and pathogens. However, they fail to strip organics, resulting in less microbiologically safe product water. As a result, using a tertiary method such as reverse osmosis (RO) to help remove toxic contaminants from greywater can be beneficial [9].

Many studies have identified scaling as a significant challenge in wastewater treatment [10, 11]. Scaling occurs as minerals in feed water precipitate on the membrane surface after reaching a point of saturation [12, 13]. Different categories of membrane fouling are known: the accumulation of organic macromolecules influences organic fouling, inorganic fouling is induced by the precipitation of inorganic salts, colloidal/particulate fouling is caused by the aggregation of colloidal/particulate particles, and microorganisms cause biofouling [14].

Scale formation is a complicated process involving crystallization and distribution mechanisms. Crystallization and precipitation occur as the saturation limit crosses and the solution becomes supersaturated. The two mechanisms through which scale occurs are surface crystallization and bulk crystallization. The membrane's surface composition and the system's working conditions influence scaling [15].

Fouling cannot be eliminated but it can be reduced. Minimization and mitigation measures methods are two techniques for coping with fouling. Fouling may be minimized by implementing an appropriate pre-treatment measure, whilst mitigation is more concerned with repeated chemical washing [16]. Scale formation resulting from fouling has become a downside in RO activity as it induces a decrease in permeate water flux, membrane deterioration, output loss, and increased operational expenses. Antiscalant is thus required to maintain RO process efficiency while making the operation more cost-effective by lessening membrane cleaning and substitution [15].

In this study, a lab-scale RO system was used to analyze the treatment of industrial laundry wastewater. Impacts of laundry detergent and antiscalant concentrations on membrane efficacy were also investigated. Using attenuated reflective Fourier transform infrared spectroscopy (ATR-FTIR) and scanning electron microscopy (SEM) fitted with energy-dispersive X-ray spectroscopy (EDX), the surface properties of membranes were investigated to determine the degree of scaling. This study brings awareness to maintaining a minimum of organic and inorganic fouling on RO membranes in laundry wastewater treatment.

2. MATERIALS AND METHODS

Reagents and antiscalant. The chemicals used were of analytical quality and were not purified further. Sodium phosphate (Na_2HPO_4), sodium carbonate ($NaHCO_3$), and sodium sulfate (Na_2SO_4) were purchased from Sigma-Aldrich. The liquid softener, linear alkylbenzene sulfonate (LAS), and detergent were obtained from a local store. Avista Technologies provided the antiscalant phosphonic acid ($H_2O_3P^+$) (Vitec 3000).

Membrane performance and scaling tests. A thin film composite aromatic polyamide (PA-TFC) RO membrane (XLE-4040) manufactured by Dow Filmtec was used. Experimental tests were conducted using an experimental laboratory membrane filtration system (Sepa CF Cell, GE Osmonics, USA). Before use in the RO cell, the membranes were cut to a size of 14.5×9.5 cm and placed in deionized water for 24 h. Deionized water (conductivity not higher than 7 µS/cm) was used to flush-rinse the RO cell system before experimental testing with the wastewater. The feed water was prepared according to the Public Sanitation Foundation/NSF/ANSI (American National Standards Institute) Standard 350 [17] to mimic laundry wastewater (10 g of test dust per 100 dm^3 of water, 1.33 g of Na_2HPO_4, 1.33 g of Na $_2SO_4$, 13.2 cm^3 of liquid detergent, 7 cm^3 liquid fabric softener, 0.667 g of $NaHCO_3$) as suggested by Ward et al. [18]. The cell system was operated at a feed pressure of 1 MPa, 5 m/s cross-flow velocity speed, and a 75% initial water recovery rate. The RO system experimental tests were conducted as shown in Fig. 1 for durations of 19 h and 104 h for short experimental and long experimental tests, respectively. All tests were carried out in a batch mode with the permeate recirculated to the feed.
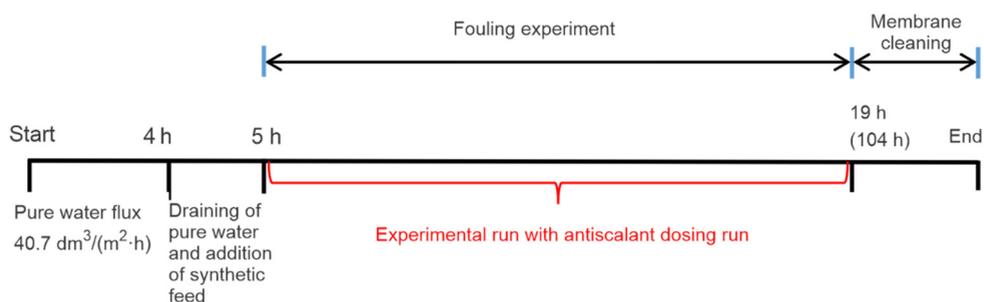

Fig. 1. Scheme of fouling tests using the RO system

The effects of surfactants and COD concentrations on the RO system and antiscalant performance were investigated at different concentration levels, as shown in Table 1. The flow rates of streams (feed, permeate, and brine), as well as the physical parameters (temperature, conductivity, total dissolved solids (TDS)), were measured recurrently after a constant interval of time for all experimental tests.

The antiscalant at different concentrations (4 and 8 mg/dm^3) was added to the feed solution during the experimental test. A 5 cm^3 drop of the antiscalant mentioned above was added every 45 min for the 19-h experimental test, and every 5-h extended experimental test. The salt rejection, water permeation, surfactant, and COD removals were evaluated to assess membrane system performance accurately [19].

<div align="right">T a b l e 1</div>

Parameters of experimental tests of the laundry wastewater model used in the study

Test No.	Detergent concentration [cm^3/100 dm^3 H$_2$O]	Surfactant concentration [mg/dm^3]	COD [mg O$_2$/dm^3]	EC [µS/cm]	pH
1	13.4	83	460	235	
2	19.8	103.5	629	281	9.3–10
3	26.4	128	825	284	

The water flux J_v and salt rejection (R_{EC}) were calculated using the following equations [11]

$$J_v = \frac{Q}{A} \tag{1}$$

$$R_{EC} = \left(1 - \frac{C_{pEC}}{C_{fEC}}\right) \times 100\% \tag{2}$$

J_v is the permeate/water flux (dm^3/(m^2 h)), J_f is the permeate fractional flux (dimensionless), A is the active area of the membrane (m^2), and Q is the volumetric flow rate of permeate (dm^3/h). C_{fEC} (µS/cm), C_p (µS/cm), and R_{EC} (%) are the feed conductivity, the permeate conductivity, and salt rejection, respectively. The percentage removal of COD (R_{COD}) and surfactant (R_S) were determined from

$$R_{COD} = \left(1 - \frac{C_{pCOD}}{C_{fCOD}}\right) \times 100\% \tag{3}$$

where C_p and C_f are the COD values in the permeate and the feed (mg O$_2$/dm^3), respectively.

$$R_s = \left(1 - \frac{C_{ps}}{C_{fs}}\right) \times 100\% \tag{4}$$

where C_{ps} and C_{fs} are the surfactant concentrations (mg/dm^3) in the permeate and the feed, respectively.

The flux decline ratio (FDR) and the fractional flux (J_f) were recorded to evaluate the severity of fouling. The FDR and J_f were calculated using the initial flux of permeate (J_i) and time-dependent flux of permeate (J_t) (all in $dm^3/(m^2 \cdot h)$)

$$FDR = \frac{J_i - J_t}{J_i} \times 100\% \tag{5}$$

$$J_f = \frac{J_t}{J_i} \tag{6}$$

Analytical method. According to the protocol, the COD was analyzed using the HI83224-spectrophotometer (Hanna Company). The surfactant concentration (mg/dm^3) was determined according to ISO7875-1 standards as Methylene Blue active substances (MBAS). Membrane morphology changes after experimental tests and the elemental and quantitative composition of the membrane surface were observed using SEM and EDX analysis. Samples of top view images were scanned at magnifications of 10 000× and 40 000×, with a 5.0 kV landing electron. The ATR-FTIR study was carried out using Nicolet iS10 FTIR apparatus with OMNIC software to detect the alterations on the membrane after experimental tests with and without the antiscalant. The ATR-FTIR spectra were recorded at an 8 cm^{-1} resolution; 64 scans; wavenumbers ranging from 4000 to 400 cm^{-1}; nominal incident angle of 45°. The conductivity of the RO system streams (feed, brine, and permeate) from which the salt rejection percentage was calculated was measured using a conductivity meter (EC 300 meter).

3. RESULTS AND DISCUSSION

3.1. SCALING ANALYSIS: ATR-FTIR AND SEM-EDX

The impact of antiscalant on the surface of the membrane before and after fouling was analyzed using ATR-FTIR analysis. The analysis resulted in a structure for distinguishing various chemical groups on the membrane surface. To extract those functionalities in both virgins and fouled membrane samples, the spectra were zoomed in to a range of 2000–600 cm^{-1}.

Figure 2 shows a characteristic broadband tipping point at 3300 cm^{-1} for the virgin XLE polyamide (PA) thin-film composite (TFC) membrane. This band is intricate because of the stretching movements of N–H and carboxylic groups, which overlap the active layer of PA. The peaks at 3000 and 2900 cm^{-1} are consistent with aliphatic C–H bond stretching and aromatic =C–H bond stretching, respectively [20]. The peaks at 1240, 1480, 1510, and 1590 cm^{-1} identify the RO membrane's polysulfone support layer [14, 21], while the peak frequency at 1240 cm^{-1} indicates a C–O–C asymmetric stretching vibration in the support layer.
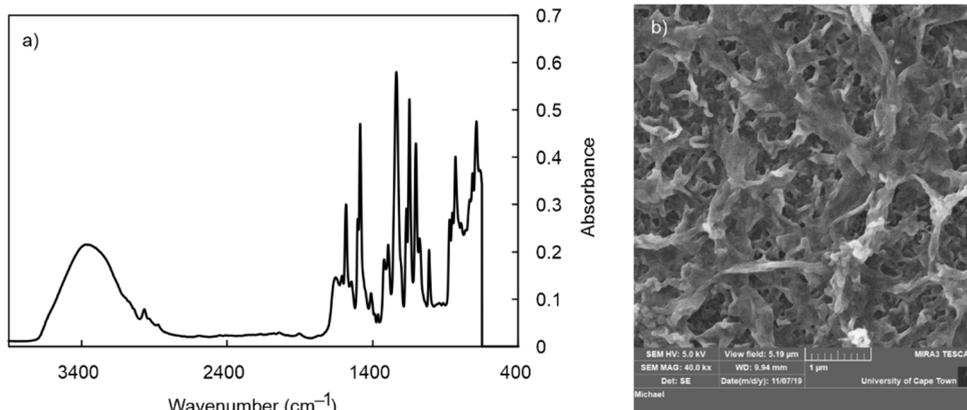
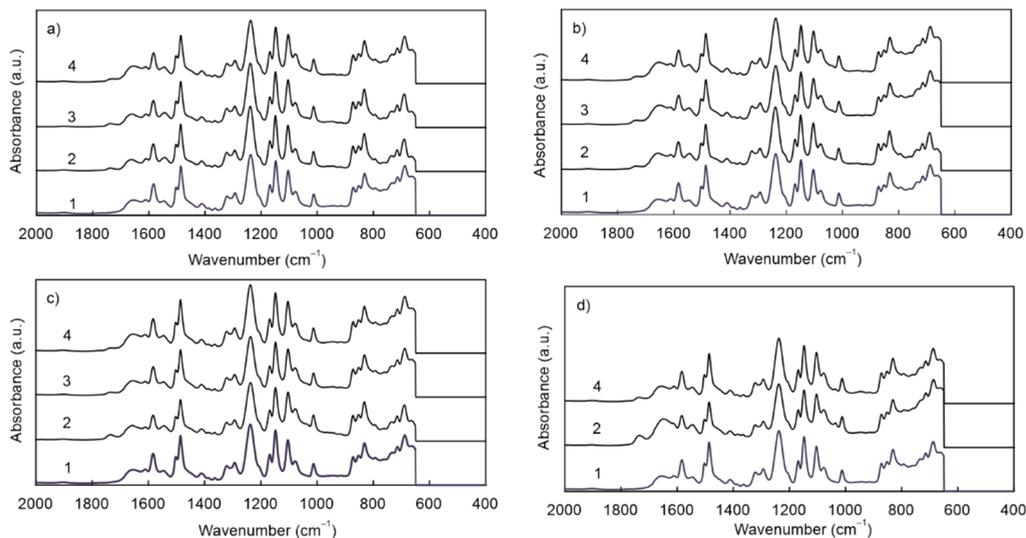Fig. 2. Virgin membrane: a) ATR-FTIR spectra, b) SEM top view image

Fig. 3. ATR-FTIR spectra of the membranes at various surfactant concentrations:
a) 83 mg/dm^3, b)103.5 mg/dm^3, c) 128 mg/dm^3, d) long-term tests 1 (antiscalant 0 mg/dm^3,
surfactant 128 mg/dm^3, 105 h) and long-term 105-h tests, 128 mg/dm^3,
antiscalant doses: 1 – virgin sample, 2 – 0 mg/dm^3, 3 – 4 mg/dm^3, 4 – 8 mg/dm^3

ATR-FTIR spectra of the zoomed membranes in the range of 2000–600 cm^{-1} for a better comparison of the virgin membrane and membranes after scaling are shown in Fig. 3. Peaks at 1600 cm^{-1}, 1485 cm^{-1}, and 685 cm^{-1} are far less prolonged in the experimental tests without antiscalant dosing than in those with antiscalant due to the layer which protects the carbonyl group in the PA layer. Observing the wavelength peaks of

antiscalant-dosed membranes is close in amplitude to their virgin states, suggesting less scaling than in experimental tests without antiscalant.
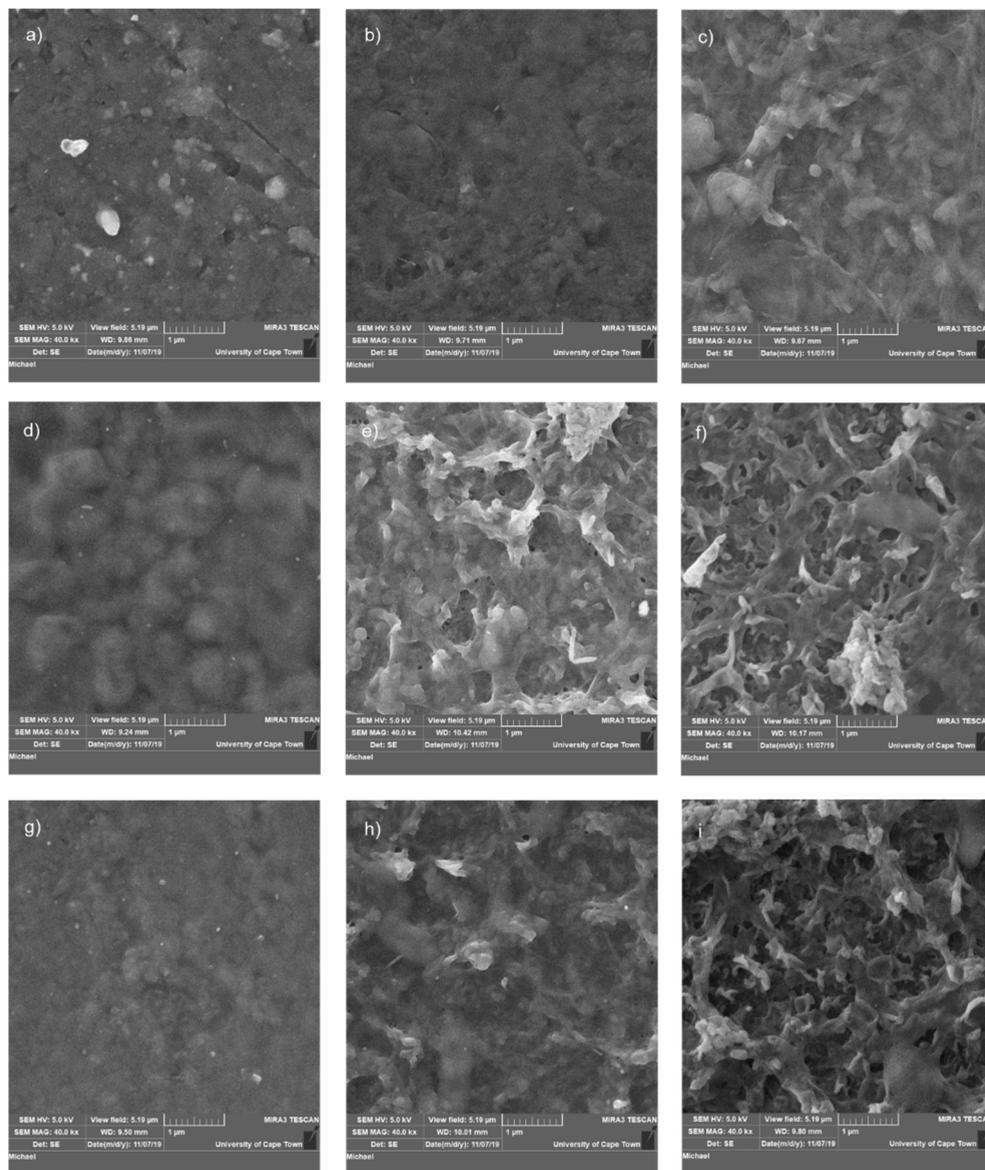

Fig. 4. SEM images of the membrane surfaces (top-view) after tests with various concentrations of surfactant and antiscalant; surfactant doses: a)–c) 83 mg/dm^3, d)–e) 103.5 mgdm^3, f)–h) 128 mg/dm^3; antiscalant doses: a), d), g) 0 mg/dm^3, b), e), h) 4 mg/dm^3, c), f), i) 8 mg/dm^3

According to the study, the higher the antiscalant dosage, the higher similar the membrane spectra became to their virgin states. This observation suggests that with a higher dose, less antiscalant accumulates on the membrane's surface, aligned with the SEM-EDX findings. A similar pattern occurs when looking at the extended experimental test (Fig. 3d). The membrane surface in the long-term experimental test without antiscalant shows peaks with lower intensity (1600 cm^{-1}, 1420 cm^{-1}, and 1000 cm^{-1}), indicating scaling strength. More clear peaks can be seen on the long-term laboratory test membrane (with 8 mg/dm^3 of antiscalant). The peaks' magnitude around the spectrum is similar to the virgin membrane, indicating reduced fouling.

Figures 4a–4i show SEM representations of the top surface layer. Foulant aggregation on RO membranes is determined mainly by foulant-surface contact [20]. In the RO process, the feed pressure is an essential factor in the deposition and adhesion of foulants onto the surface of the membrane [21]. The SEM performed an energy dispersive X-ray (EDX) analysis on the specimens. The EDX findings of membranes after scaling experimental tests with different surfactant and antiscalant concentrations are shown in Table 2.

T a b l e 2

EDX results of the virgin RO membrane and membranes following laboratory testing
of various laundry detergent concentrations and antiscalant dosages

Chemical element	Virgin membrane	Surfactant dosage [mg/dm^3]								
		83	103.5	128	83	103.5	128	83	103.5	128
		Antiscalant dosage [mg/dm^3]								
		0			4			8		
Carbon, wt. %	77	77	77	76	76	76	77	75	76	78
Oxygen, wt. %	17	17	17	18	18	19	16	19	17	17
Sulfur, wt. %	6	6	6	6	6	6	7	6	6	6

When comparing the chemical elemental composition identified on the surface at different experimental conditions during short-term scaling tests, no significant differences were observed. This demonstrates that only a thin layer of a scalant accumulated on the membrane surface during short experimental runs [10]. For experimental tests without antiscalant, the amount of carbon declined as laundry detergent concentration increased, caused by the foulant layer masking the carbonyl group existing in the PA layer. The finding is consistent with the previously mentioned ATR-FTIR spectra (Fig. 3). The EDX findings revealed that the presence of sulfonate groups in the surfactant molecule could be a reason for the increase in the membrane surface layer's total oxygen and sulfur composition.

3.2. MEMBRANE PERFORMANCE DURING SCALING TESTS

The membranes' pure water flux ($EC \leq 5$ µS/cm) before scaling experimental tests was 40.7 dm^3/(m^2·h) at an applied feed pressure of 1 MPa. The permeate flux of the

membranes during the laundry wastewater treatment and antiscalant addition is shown in Fig. 5, where the initial laundry wastewater flux was in a range of 37.5 to 45.4 dm^3/m^{2}h depending on the experimental conditions. Both experimental tests showed a gradual decrease of the initial permeate flux with time, indicating the existence of fouling, as reported by Aziz and Kasongo [11]. The fouling seems to be minimal in the first few minutes of the process. The flux decrease became more apparent over time, and the flux gap between the membranes with antiscalant expanded. A more significant flux reduction was observed as the laundry detergent dosage increased.
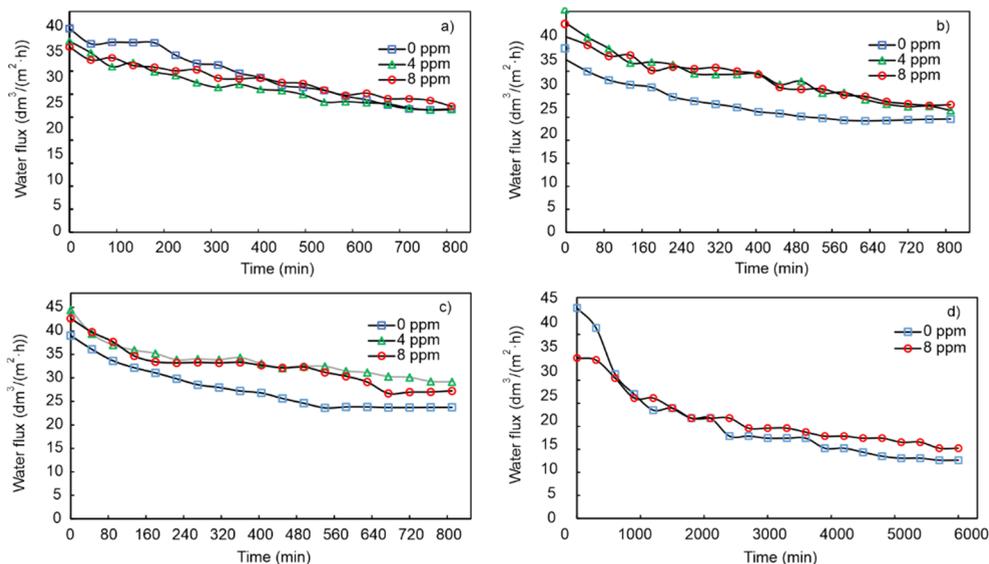

Fig. 5. Average permeate fluxes of membranes at various surfactant concentrations:
a) 83 mg/dm^3, b) 103.5, c) 128 mg/dm^3, d) 128 mg/dm^3; antiscalant concentrations given in the figures

The permeate flux at 83 mg/dm^3 laundry detergent (surfactant) becomes constant after 800 minutes, while the flux at 128 mg/dm^3 laundry detergent reaches a steady state after 450 minutes. Although it seems that a steady decline in flux could not be prevented (Fig. 5), the deposition of fouling substances on the antiscalant-added membrane was considerably lower than that on the membrane without antiscalant. The reduction in flux became less noticeable as the antiscalant concentration was increased. Figure 5d shows that the permeate flux was greater than that of the membranes without antiscalant after 105 h of the experimental test (6300 minutes). Thus, indicating that the antiscalant has reduced fouling behavior in the wastewater by electrostatic interaction with foulants [11].

The membranes' flux decline ratio (FDR) is shown in Fig. 6 at various antiscalant concentrations. The FDR is also regarded as an indicator of membrane surface fouling. The smaller the FDR, the lower the flux decline is, since fouling is reduced on the surface of the membrane [20].
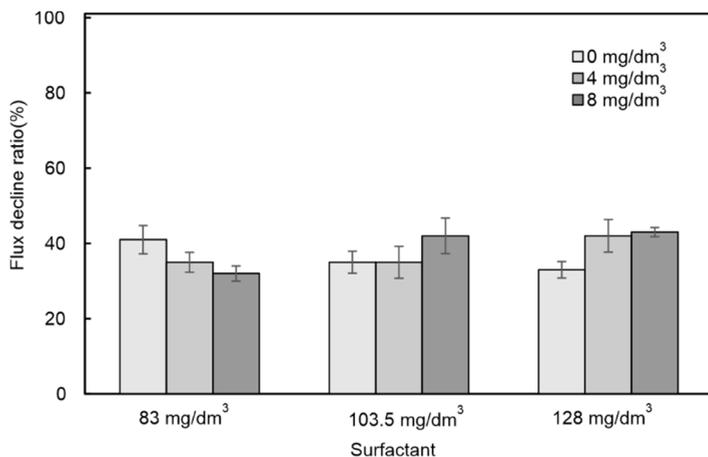
Fig. 6. Flux decline ratio at the end of the experiment

With a surfactant content of 83 mg/dm^3, the FDR values decrease as the antiscalant dosage increases from 0 to 8 mg/dm^3. It indicates the effectiveness of the antiscalant dosage to minimize membrane scaling. However, this trend is not observed with surfactant contents of 103.5 and 128 mg/dm^{3,} where the values of the FDR are observed to be the same when comparing antiscalant dosages of 0–4 mg/dm^3 and 4–8 mg/dm^3. It may be explained by the increase in surfactant level, which renders the action of the antiscalant less effective. The results suggest that a continuous review of the optimum antiscalant dosage is essential to ensure the effectiveness of the antiscalant.
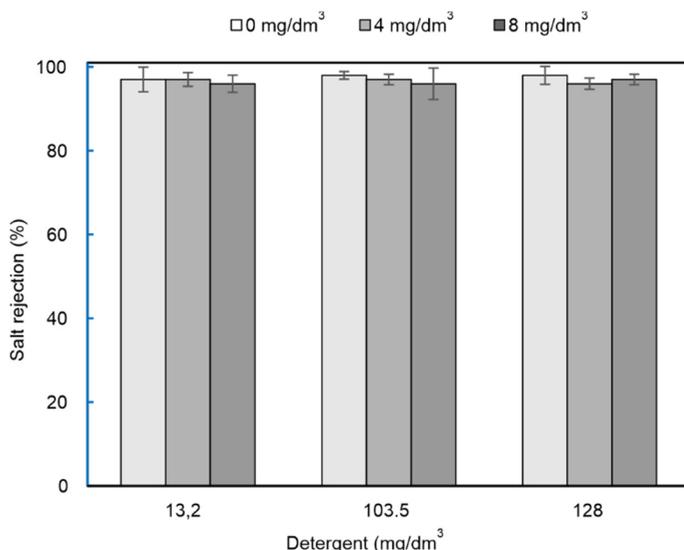

Fig. 7. Salt rejection of membranes at various detergent and antiscalant concentrations

An antiscalant must not degrade the efficiency of membrane salt rejection output over time and preserve the original flux. Figure 7 indicates the overall salt rejection in various conditions. In each condition, salt rejection is relatively high. A closer investigation reveals that the membranes without antiscalant dosing performed marginally better anti-scaling properties in terms of salt rejection. The antiscalant reacted with ions in the wastewater to create a steady, water-soluble complex molecule. It delayed the formation of precipitates and dispersed ions susceptible to precipitation formation, resulting in a lower flux decline [11, 15].

Assessing COD and surfactant removals as a measure of membrane system efficiency were necessary. Surfactant removal surpassed 99.8% in nearly all experimental tests, as seen in Fig. 8. Two factors accounted for the significant rejection of organic molecules: size exclusion and electrostatic repulsion [2]. The successful surfactant removal can be explained by the fact that the adsorption of surfactant to the surface makes the membrane surface hydrophobic, creating effective surfactant elimination. As a result, it causes fouling matter to rise to the surface of the developing bubble and float onto the surface [22]. A related pattern was found when comparing the COD rejection (Fig. 8) to average salt rejection (Fig. 7). The dynamics of size exclusion, charge, and electrostatic forces with the surface of the membrane are believed to be the cause of the removal efficiency [23].
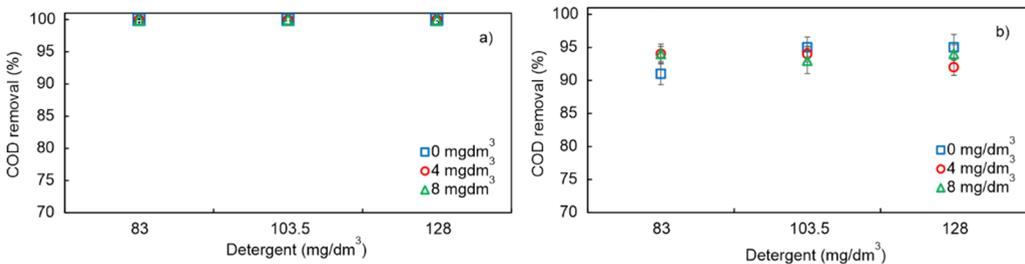

Fig. 8. Surfactant and COD percentage removal of membranes
t different detergent and antiscalant concentrations

Table 3

EDX results of membranes after 105-hour experimental test
without and with 8 mg/dm^3 antiscalant [wt. %]

Chemical element	Virgin membrane	Antiscalant addition [mg/dm^3]	
		0	8
Carbom	77	72	75
Oxygen	17	23	18
Sulfur	16	5	6

Long-term experiments were performed with a concentration of 26.4 cm^3/100 dm^3 H$_2$O laundry detergent (128 mg/dm^3 surfactants and 825 mg O$_2$/dm^3 COD synthetic feed);

8 mg/dm³ antiscalant firstly, and after that without antiscalant. There were significantly different relative ratios of the elements, as shown in Table 3. The effectiveness of the antiscalant was demonstrated when the 8 mg/dm³ dosage was used. The relative ratios of elements were close to those of virgin membranes.

Membranes SEM photos of experimental tests both with and without antiscalant are shown in Fig. 9. An inconsistent structure deposit covers almost entirely the membrane surface in Fig. 9a, while Fig. 9b partially demonstrates the ridge and valley membrane structure. Partly spread scalant shows the antiscalant uses' efficacy to minimize membrane fouling.
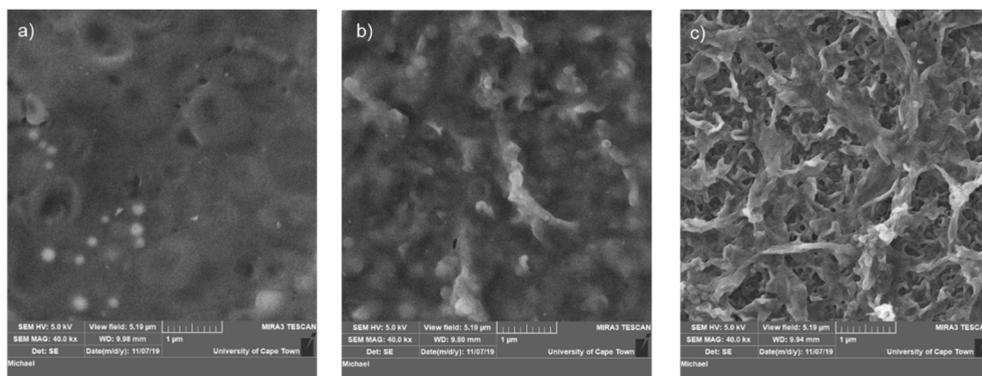

Fig. 9. SEM photographs of membrane surfaces after long experimental tests of 105 h
depending on antiscalant concentrations: a) 0 mg/dm³, b) 8 mg/dm³ c) virgin membrane

The surfactant performance removals were over 99.8% for both experiments with no anti-scaling agent and 8 mg/dm³ anti-scaling agent. The salt rejection and COD percentage removals were identical during the long experimental tests, as seen in Fig. 10.
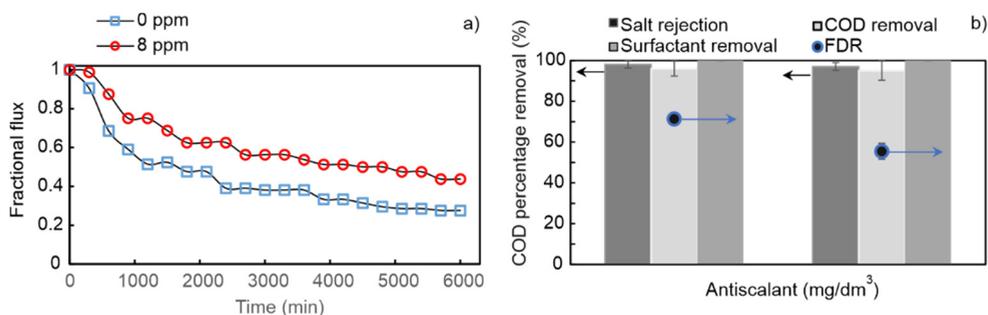

Fig. 10. Permeate fractional flux (a), COD and surfactant removal percentages (b)
of long-term experimental tests at 0 and 8 mg/dm³ antiscalant concentrations

A significant difference in flux reduction (expressed as fractional flux J_f) can be seen between the tests without antiscalant and with 8 mg/dm³ of antiscalant (Fig. 10).

The flux decline was expressed in terms of J_f to allow a fair comparison of experimental conditions with different initial flux values. It is evident here that hydrophobic and electrostatic implications between the surface of the membrane and surfactants, which are the reason for the adsorption of the surfactants on the surface [24], were reduced by the activities of an antiscalant that help avoid membrane fouling through interaction with the elements of concern [11]. This again illustrates the high COD removal caused by the mechanism of the electrostatic interaction [25]. The average FDR percentage for each condition was 72% (without antiscalant) and 56% (8 mg/dm^3 antiscalant).

4. CONCLUSIONS

In the study, scaling reduction on RO membranes has been assessed in removing laundry water using an antiscalant. More foulants accumulated onto the membrane surface with reduced or no antiscalant dosage. SEM analysis revealed a morphological shift in the membrane's surface. It was observed that using the correct amount of antiscalant inhibited and decreased the surfactant deposition as it disrupted scaling activity on the membrane surface with a surfactant level of 83 mg/dm^3. It resulted in a lower flux decline as the antiscalant concentration increased. The change in the percentage of carbon, oxygen, and sulfur on the membrane surface was not significant with the short-term experimental tests. However, a change was significant during the long-term experimental test. They were observed to be lower on the membrane surface with antiscalant dosage decreased as the laundry detergent dosage increased according to the EDX analysis. Surfactant removal was higher than 99.8% in nearly all experimental tests conducted at various surfactant concentrations in the feed. The *EC* and COD removal rates on the RO bench-scale system were 97–98% and 91–96%, respectively. The COD reduction was marginally higher with membranes without antiscalant addition than with antiscalant addition. The surfactant and COD concentrations were consistent with municipal disposal and future reuse requirements. The research results showed that the low-pressure RO membrane could be applied in real terms to treat laundry wastewater, given that a pilot-scale process analysis and various water recovery rates have been assessed.

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