



Antiscaling effect of polyaspartic acid and its derivative for RO membranes used for saline wastewater and brackish water desalination

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HIGHLIGHTS

- PASP and its derivate were investigated as greener RO antiscalants.
- PASP derivate showed better antiscaling effect than PASP and a commercial product.
- PASP derivate led to larger crystals and a less dense scale layer on the RO membrane.
- PASP and its derivative appeared to be cost-effective.

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ABSTRACT

Polyaspartic acid (PASP) and its derivative (PASP-SEA-ASP) were tested as environmentally benign scale inhibitors at lab scale for a polyamide reverse osmosis (RO) membrane in the desalination of a synthetic brackish water containing various scale-forming ions. Their performance was compared with a commercially available RO antiscalant which contains phosphonic acid. The addition of the scaling inhibitors to feedwater led to increase in water recovery and removal efficiency of inorganic matter for the RO unit operated under constant transmembrane pressure, with PASP-SEA-ASP giving greater water recovery (90%) than PASP (87%) and the commercial antiscalant (85%). PASP-SEA-ASP was superior to PASP and the commercial antiscalant in scaling inhibitor/fouling reduction due to the reduced deposition of the scale forming ions on the membrane. Examination of the used membranes with scanning electron microscopy and X-ray diffraction suggested the different effect of the antiscalants was related to the resultant crystalline morphology on the membrane surface. The PASP and its derivative appeared to be more cost effective in maintaining permeate flux and inhibiting the formation of a dense scale layer. The present study demonstrated the great potential of these non-phosphorus containing agents for scale inhibition in domestic and industrial wastewater reclamation and brackish water desalination applications.

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1. Introduction

Membrane fouling caused by inorganic, organic and biological materials is a major problem limiting the efficiency of reverse osmosis (RO) based wastewater reclamation and brackish water desalination processes. Inorganic fouling, known as scaling, occurs after deposition of inorganic constituents on the RO membranes. The most common scale-forming constituents in membrane processes are CaSO_4 , CaCO_3 , BaSO_4 , calcium phosphates, silica, aluminum and ferric hydroxides [1,2]. Scaling can reduce permeate flow due to formation of a cake layer by surface blockage and/or bulk crystallisation with the inorganic deposits [3], leading to significantly increased operational cost for the membrane systems. Scaling may also physically damage the membranes because

of ineffective scale removal and occurrence of irreversible pore plugging during filtration. As such, scaling mitigation is a critical measure to ensure efficient RO operation for wastewater reclamation and brackish water desalination.

Several methods can be used to prevent the scaling of RO membranes, including (i) acidification of feedwater to reduce bicarbonate by converting it into carbon dioxide, (ii) scale inhibitor chemicals which change the physical and chemical nature of the depositor ions and hence the scale growth mechanisms, (iii) operating conditions of RO systems, and (iv) softening with lime or soda ash which react with bicarbonate in the feed to produce CaCO_3 sludge [4,5]. Antiscalants are used widely in RO processes due to their high effectiveness for preventing scaling. Antiscalants generally delay scale formation and allow higher water recovery by hindering scale growth under supersaturated conditions [6]. Reitz [7] investigated a number of commercial antiscalants for the control of scaling caused by SrSO_4 , CaSO_4 and

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CaCO₃ in RO systems and found that they were effective for inhibiting CaCO₃ precipitation. Another study by Lisitsin et al. [8] reported that zinc ion was promising to mitigate scaling for membrane processes. However, many commercial antiscalants can enhance biofouling of RO systems as they contain nutrients such as phosphorus [9,10,11]. Moreover, phosphorus-containing antiscalants remaining in the RO concentrate/brine can contribute to eutrophication of the receiving water bodies and consequent blooms of harmful algae. It has been suggested that most of the commercially available antiscalants are expensive, and could account for approximately 10% of the total treatment cost for the production of clean water [12]. As such, the development of low-environmental risk and cost-effective antiscalants is required for improving the sustainability of RO-based water purification processes.

Polyaspartic acid (PASP) is a polyamino acid which does not contain phosphorus and is biodegradable. It was reported as being an environmentally friendly antiscalant in the freshwater environment [13]. The functional groups of PASP have a scale inhibition effect as they have a side chain generated through the aminolysis reaction with amine-group [14]. Gao et al. [15,16] synthesised a PASP derivative (PASP-ASP) with the aim of enhancing the chelating performance of PASP through the introduction of carboxylic acid to its side chain, and the experimental study demonstrated the improved scale inhibition effect of the derivative in freshwater applications. It was suggested that PASP could ionise and form molecular chains of negative charge, leading to the formation of water-soluble complexes with Ca²⁺ and hence reduced deposition of the scale causing ions on membrane surface [13]. They also synthesised a PASP derivative (PASP-SEA-ASP) for enhancing both chelation and dispersion performance by introducing sulfonic acid and carboxylic acid to the side chain of PASP, with a view to inhibiting scale formation in a saline water environment such as seawater [17]. As there is a sulfonic acid group in the molecular structure of PASP-SEA-ASP, and when PASP-SEA-ASP is adsorbed onto the small crystal particles, a double layer can be formed and the charge state of the particle surface is changed. This can result in enhanced dispersion of the crystal particles in water. Although PASP and its derivative exhibited a promising antiscaling effect on carbon steel in freshwater and seawater for potential industrial applications such as scale inhibition for heat exchangers and water pipes, their effect on scale inhibition for polymeric RO membranes used for wastewater and brackish water desalination has not been investigated.

The present study was aimed at examining the antiscaling effect of PASP and its derivative PASP-SEA-ASP on polyamide RO membranes used for the desalination of a synthetic saline water containing various scale-forming ions, and to compare them with a phosphorus-containing commercial RO antiscalant. The antiscaling effect was assessed by determining the filtration performance in terms of water recovery within the test period and removal efficiency of inorganic matter, the quantity and chemical composition of the scale on the membranes, and examining the scale crystals formed on the membrane surface using scanning electron microscopy (SEM) and X-ray diffraction (XRD).

2. Materials and methods

2.1. RO feedwater

The chemical composition of the synthetic feedwater used for the RO tests is shown in Table 1. The chemicals were selected based on their wide occurrence in the scale on RO membranes. The chemical reagents used for the preparation of the RO feedwater were of analytical grade, supplied by Sigma-Aldrich.

2.2. Antiscalants

A commercial RO antiscalant that is widely used for wastewater desalination in many countries including Australia was tested in this study. According to the manufacturer, the antiscalant is based on

Table 1
Composition of the synthetic RO feedwater.

Composition/property	Unit	Concentration
NaCl	mg/L	15,000
CaCl ₂	mg/L	8000
MgSO ₄	mg/L	2000
SiO ₂	mg/L	1000
pH	–	7.9
Conductivity	μs/cm	52,800

phosphonic acid and polycarboxylic acid. The PASP and PASP-SEA-ASP were synthesised in our lab with the methods described in Section 2.3. The purity of PASP and PASP-SEA-ASP was determined as 99% and 98.5%, respectively. The antiscalant dosage of 1 mg/L (1 ppm) was used for all RO antiscaling experiments.

2.3. Synthesis of antiscalants

2.3.1. PASP

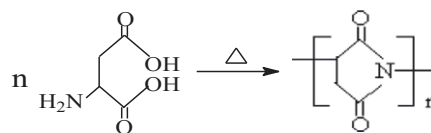
PASP was synthesised through two steps. The first step was the synthesis of polysuccinimide, which was carried out in an electric thermostatic drying oven through pyro-condensation of L-aspartic acid (Industrial grade, Sigma Aldrich) monomer at 240 °C for 4 h. The second step was the synthesis of PASP. Firstly, polysuccinimide was suspended in three times its weight of water. The aqueous polysuccinimide suspension was stirred at 12 °C, and then NaOH solution (15%, industrial grade) was added. The reaction was for 4 h at 12 °C and pH 8–9. Solid PASP was obtained through the separation process involving the addition of double volume of absolute alcohol, washing of the resultant precipitates with deionised water, and then drying at 80 °C for 48 h. PASP stock solution (36%) was prepared by dissolving the solid PASP in deionised water. The relevant synthetic reactions are expressed in Schemes 1 and 2.

2.3.2. PASP-SEA-ASP

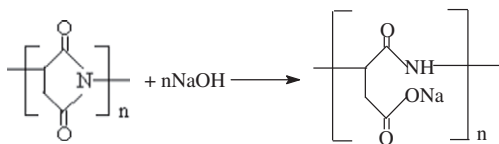
Synthesis of PASP-SEA-ASP was through two steps. In the first step, polysuccinimide was synthesised as described in Section 2.3.1. The second step was the synthesis of PASP-SEA-ASP, in which polysuccinimide was first suspended in three times its weight of water. The polysuccinimide suspension was stirred at 25 °C, and then aspartic acid and 2-aminoethanesulfonic (Industrial grade, Sigma Aldrich) which were dissolved in NaOH solutions (15%) were added. The reaction was for 24 h at 25 °C and pH 8–9. The pH of the mixture was then dropped to 5.2 by adding 2 N HCl to remove the unreacted 2-aminoethane sulfonic acid through filtration. Unreacted aspartic acid was removed from the solution by further dropping the pH to 2.8 and then filtration. Solid PASP-SEA-ASP was obtained through a separation process in which a double volume of absolute alcohol was added to the filtrate and the resultant precipitate was rinsed using deionised water and dried at 80 °C for 48 h. The stock solution of PASP-SEA-ASP (30%) was made through dissolving the obtained solid in deionised water. The relevant synthetic reaction is expressed in Scheme 3.

2.4. RO tests

RO tests were performed using a flat-sheet membrane (Polyamide RO AG membrane, GE Osmonics) with a bench-scale cross flow RO rig (SEPA CF, GE Osmonics). Before the filtration tests, RO membranes



Scheme 1. Synthesis route of polysuccinimide.



Scheme 2. Synthesis route of polyaspartic acid.

were soaked in Milli-Q water at 4 °C overnight after which deionised water was passed through the membranes for removing chemical preservatives and membrane compaction. During RO operation, a constant transmembrane pressure of 10 bar and cross-flow rate of 0.1 m/s was maintained. A refrigerated circulating bath (Neslab RTE 7, USA) was used to maintain the temperature of the feedwater at 22 ± 2 °C. Each test was performed in duplicate and the average values of the results were reported, a new membrane was used for each run. RO concentrate and permeate samples were collected for analyses of water quality and determining the water recovery over the testing period. The schematic diagram of the RO testing system is presented in Fig. 1.

The following equation was used to calculate the water recovery:

$$WR = \frac{V_p}{V_f} \times 100\%$$

where *WR* is water recovery (%); *V_f* is the total volume of feedwater (L); *V_p* is the total volume of permeate (L).

2.5. Analytical methods

The substances forming the scale on the RO membranes were extracted with acid solution (0.1 N H₂SO₄). The fouled membrane was soaked in 2 L of acid solution at room temperature for 24 h and then manually agitated using forceps for 5 min to extract the scale. The concentration of the metal ions of interest was determined by atomic absorption spectroscopy (AA-6501F, Shimadzu, Japan). Prior to the analysis, all samples were pre-filtered using 0.45 μm membrane filters and then acidified using 40% nitric acid to stabilise the metal content. Stock solution for each metal species was prepared for obtaining a standard curve to determine the concentration of the metal ion.

The dry weight of the unused and used membrane was measured to calculate the amount of scale attached to the membrane for each antiscalant. The membrane was dried for 12 h in a desiccator at room temperature, and then weighed with an electronic balance (PA 114C, Ohaus, China).

A scanning electron microscope (SEM) (FEI Quanta 200, Nova) was employed to examine the scaling layer on the membrane surface. The used membranes were dried at room temperature overnight prior to the SEM examination. A small piece of the membrane was cut out and coated with gold, and then held within the SEM chamber with

conductive and double-sided carbon tape. The accelerating voltage of 30 kV was maintained and images were acquired at 4000× magnification.

X-ray diffraction (XRD) analysis was undertaken for virgin and used membranes using a Bruker AXS D4 Endeavor X-ray diffractometer equipped with Lynxeye linear strip detector and copper anode. The analysis was conducted using Cu-Kα radiation at 40 kV and 40 mA beam intensity. The data was collected between 5° and 55° 2-theta ranges with a step size of 0.02° 2-theta and the counting time per step was 1 s. Prior to the analysis, the used membranes were dried overnight at room temperature.

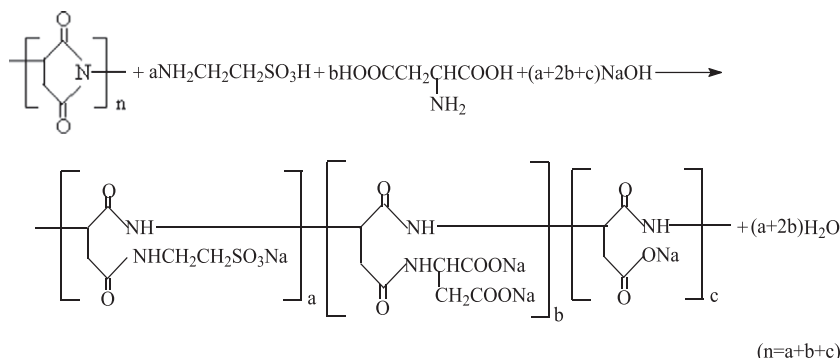
3. Results and discussion

3.1. Impact of scale inhibitors on RO permeate water quality

The concentration of the metal ions in feedwater, permeate and concentrate was examined to indicate the scale formation conditions. The removal efficiency of the ions by the membrane was increased by adding antiscalant to the feedwater, with PASP-SEA-ASP giving greater ion removals than PASP which in turn performed better than the commercial antiscalant (Table 2). The greater concentration of the metal ions in the concentrate stream for the PASP-SEA-ASP and PASP also suggested less salt attachment to the membrane surface compared with the commercial antiscalant. Analyses and quantification of the deposits extracted from the membrane surface confirmed that the antiscalants led to reduced scale formation, with antiscaling effect in the order of PASP-SEA-ASP > PASP > commercial antiscalant (Table 2 and Fig. 2).

In a previous study of the antiscaling effect of PASP-ABSA copolymers [18], it was suggested that the reduced calcium scale formation was due to the chelation of calcium ions and the subsequent formation of stabilised and soluble chelates, which was attributed to the presence of sulfonic groups and carboxylic ion in the scale inhibitors. In the present study, it was likely that the antiscalants led to the formation of chelated structures with metal ions [19,20], resulting in less passage of salt through the RO membrane and hence lower ion concentration in the permeate. The higher ion concentration in the RO concentrate stream would be a result of good solubility of the chelates [18]. However, antiscalants cannot infinitely increase the solubility of inorganic compounds, after the solubility reaches a certain value, carbonate scale will be produced. Since PASP-SEA-ASP possesses both carboxylic acid groups and sulfonic acid groups, whereas PASP has only carboxylic acid groups, greater inhibition of the deposition of the scale forming ions on the membrane was expected for PASP-SEA-ASP compared with PASP. Further work to examine the function of chelation of the antiscalants and examine the solubility of calcium carbonate as a function of the antiscalants may be required to confirm the suggested mechanism.

The water recovery was determined as 83% for the RO test without using antiscalant, 85% for the commercial antiscalant, 87% for PASP



Scheme 3. Synthesis route of PASP-SEA-ASP.

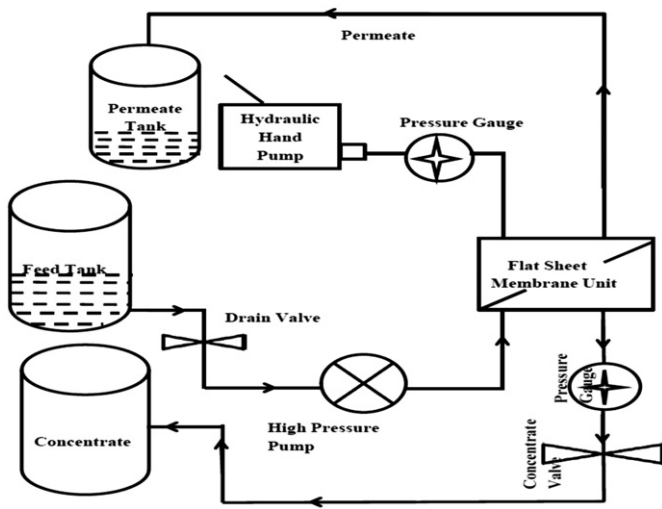


Fig. 1. A schematic diagram of the RO system.

and 90% for PASP-SEA-ASP. These results were consistent with the expectation that the antiscalant exhibiting greater scale inhibiting effect would result in less membrane inorganic fouling and hence higher water productivity.

3.2. Characterisation of the scale formed on membrane surface

3.2.1. SEM images of scale crystals

SEM analysis of the used and unused membranes was carried out to examine the scaling of the RO membranes in the presence of the various antiscalants (Fig. 3). SEM image of the virgin membrane was also obtained for comparison (Fig. 3a). In general, scale formation occurs via several stages including ion pairing, formation of micro-aggregates, micro-crystals and then macro-crystals, and eventually formation of scale [21]. When no antiscalant was added to the feedwater, the scale deposits on the RO membrane appeared to be present over the entire membrane area sampled and formed a dense scale layer, mainly consisted of distorted calcite crystals which appeared to have rough rounded edges. Changes in crystal morphology were shown, which was most likely due to antiscalant addition to the feedwater. The crystals resulting from the antiscalants were significantly larger in size (Fig. 3c, d, and e). This was most probably because the antiscalants increased the solubility of the inorganic compounds and delayed the formation of the deposits, and when the concentration of solute increased to a certain value, deposit formation began [19,20]. The resultant

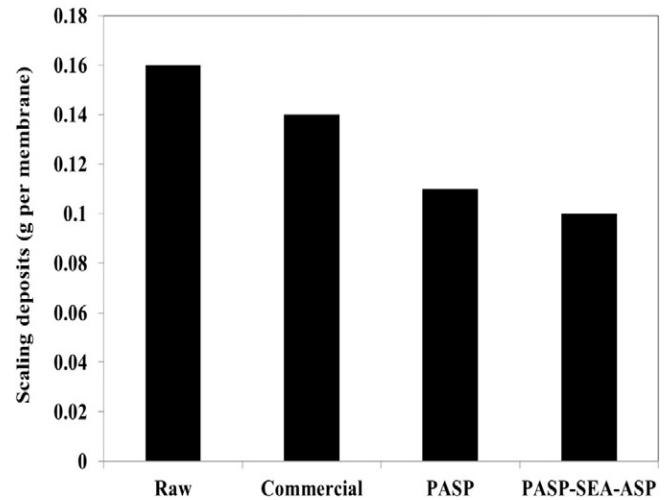


Fig. 2. Scale deposits per membrane surface area for RO tests without and with addition of antiscalant.

crystals were significantly larger in size because the formation of the carbonate scale was influenced by other ions such as Mg^{2+} , Na^+ , Si^{4+} and Ca^{2+} . With advancing outgrowth of the crystals, adsorbed scale inhibitor molecules were eventually overgrown [22]. This was in line with a previous study in which the crystals were larger in size due to the influence of other ions on carbonate scale in a groundwater environment [23]. The build-up of larger crystallites was the result of the formation of loose and irregular crystallites. However, some researchers found that scale inhibition was due to the formation of smaller and more dispersed carbonate crystallites when there was only Ca^{2+} and HCO_3^- in the synthetic feedwater [15,24,25].

PASP and PASP-SEA-ASP appeared to lead to the formation of the crystals with irregular shapes and loose structures. For PASP, the crystals lost the sharp edges, and their morphology was changed from smaller fragments to extended stick forms. The resultant crystals from the addition of PASP-SEA-ASP were markedly larger in size and were spindle shaped. A similar observation was made by Weijnen and Rosmalen [22] who suggested that the larger and spindle shaped crystals were the result of the increased adsorption of the polyelectrolyte inhibitor (based on either polyacrylic acid or polymaleic acid) onto the gypsum crystal surface. The addition of the PASP-SEA-ASP to the feedwater could affect the scaling process through (i) threshold inhibition, as lattice formation of regular crystals was inhibited via adsorption on the active growth sites of crystals; (ii) crystal dispersion, whereby micro-crystal growth and agglomeration was prevented via the increased anionic charge of the membrane surface [26]; and (iii) crystal modification, whereby the build-up of a regular crystalline lattice was prevented via modification of the micro-crystal morphology/shape [21].

Overall, PASP-SEA-ASP led to the formation of larger sized crystals, which were more sparsely distributed on the membrane surface compared with PASP and the commercial antiscalant. This could result in a partial coverage of the membrane by the loose structures, leading to significantly improved mass transfer, which was consistent with the water recovery results (Section 3.1). A similar observation was made by Rahardianto et al. [27] who noted that the fractional flux reduction was marked compared with the fractional surface area covered with scale.

3.2.2. XRD analysis

The XRD spectra of the RO membranes were to help identify the various compounds such as calcite, aragonite, and vaterite that are responsible for membrane scaling. The XRD results showed the presence of vaterite at 32° 2-theta and 76° 2-theta, calcite at 46° 2-theta and 57° 2-theta, and aragonite at 66° 2-theta, and 85° 2-theta (Table 3, and

Table 2
Concentration of ions in RO feedwater, permeate, concentrate and the solutions containing deposits extracted from the used membranes.

		Without antiscalant	Commercial antiscalant	PASP	PASP-SEA-ASP
Feed (mg/L)	Na^+	5842			
	Mg^{2+}	184			
	Si^{4+}	624			
	Ca^{2+}	2338			
Permeate (mg/L)	Na^+	176	148	132	112
	Mg^{2+}	16	12	6	10
	Si^{4+}	46	38	26	28
	Ca^{2+}	94	82	68	62
Concentrate (mg/L)	Na^+	17,540	18,960	20,448	23,376
	Mg^{2+}	388	456	524	676
	Si^{4+}	1376	1584	1640	1842
	Ca^{2+}	4560	5176	5872	6284
Solution containing the extract of membrane scaling layer (mg/L)	Na^+	3640	2942	1492	856
	Mg^{2+}	98	112	66	28
	Si^{4+}	396	342	312	186
	Ca^{2+}	1536	1448	1224	972

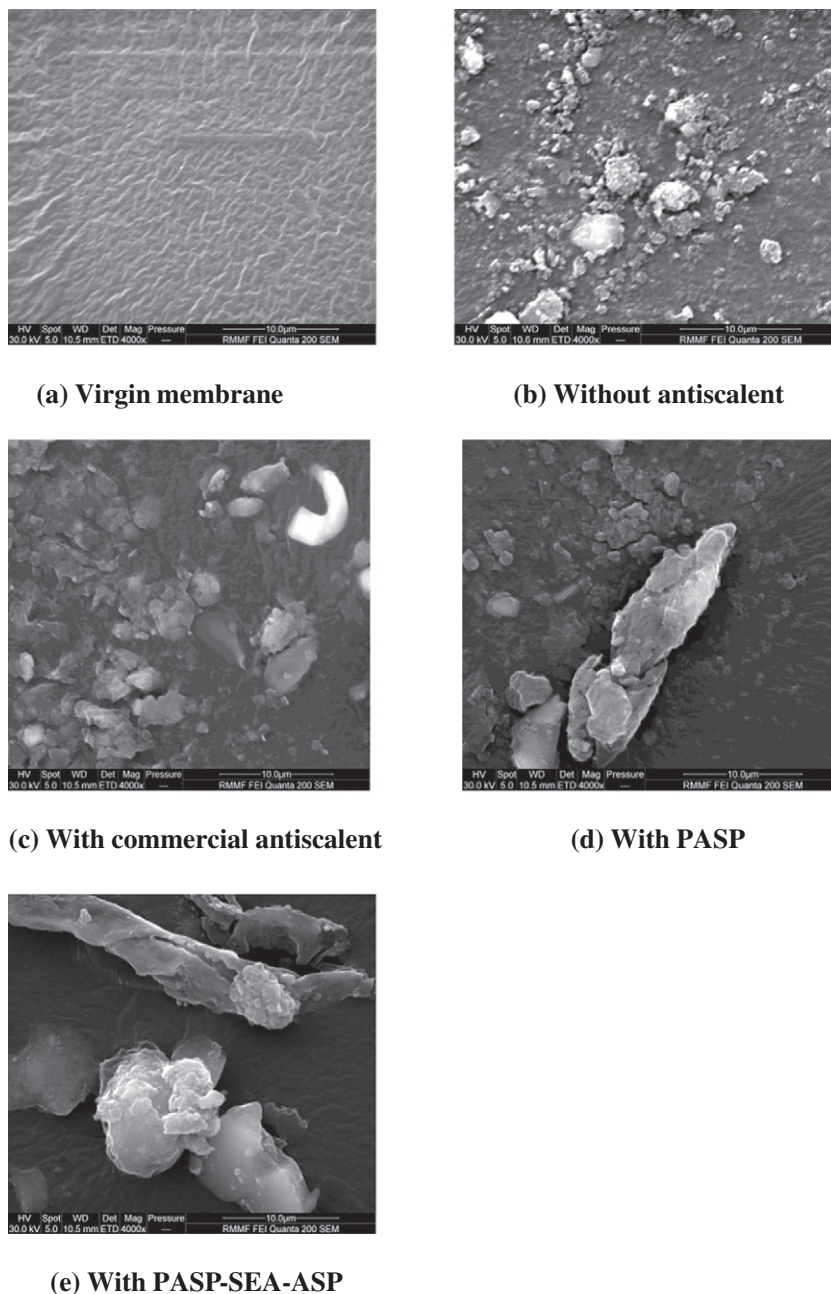


Fig. 3. SEM images of the virgin membrane (a), the membranes after filtration of the synthetic water without antiscalant (b), with commercial antiscalant (c), PASP (d) and PASP-SEA-ASP (e).

Fig. S1 in Supplementary Materials). A similar pattern was reported by Tang et al. [28] who found that different forms of compounds such as calcite and vaterite were formed at 46° 2-theta, 32° 2-theta,

Table 3
Intensity data of the various compounds on the different membrane samples.

Compounds	2-theta	Intensity for different membrane samples			
		Without antiscalant	Commercial	PASP	PASP-SEA-ASP
Calcite	46°	1852	2763	3139	3787
	57°	1073	1268	1291	1421
Vaterite	32°	4138	8051	10,832	10,214
	76°	815	892	901	929
Aragonite	66°	741	843	832	900
	85°	605	618	622	629

respectively. These results indicated that the scale formed had a significantly different crystal composition. The addition of antiscalant apparently led to the changes in the morphology of the crystal precipitates, resulting in displacement and enhancement of the diffraction peaks.

At the same degree, the peaks exhibited different intensities for the different antiscalants. The vaterite peak at 32° 2-theta was the highest peak for all samples, indicating vaterite was a major component in the scale crystals on the membranes. There was a higher peak intensity at 32° 2-theta for PASP than PASP-SEA-ASP. However, overall, the diffraction peaks for PASP-SEA-ASP were stronger than for the commercial antiscalant or PASP (Table 3), indicating that the morphological changes for the crystals resulted from the presence of PASP-SEA-ASP in the RO feedwater. It should be noted the intensity of the diffraction peaks for PASP-SEA-ASP is related not only to the composition, but also to the crystallinity and the size of scale crystals [29]. PASP had stronger

peaks than the commercial antiscalant. These changes in the peaks illustrated that the crystal structure was significantly different with the addition of various scale inhibitors. These changes likely resulted in the loose structure of the crystals, and hence a less dense scale layer.

4. Conclusions

PASP and its derivative PASP-SEA-ASP were tested as potential environmentally friendly antiscalants for mitigating scaling of a RO membrane in the filtration of a synthetic saline feedwater containing various scale forming ions, and compared with a commercially available antiscalant. All scale inhibitors were capable of improving RO performance in terms of water recovery and rejection of inorganic matter, with PASP-SEA-ASP performing better compared with PASP which in turn was better than the commercial antiscalant. SEM analysis confirmed the formation of larger crystals and less dense scale layer on the membrane surface for the feedwater with added PASP-SEA-ASP compared with the other antiscalants, which suggested the effective chelation of scale-forming ions.

Overall, this study exhibited the potential of polyaspartic acid and its derivative as scale inhibitors for their applications in wastewater and brackish water desalination. As the cost of chemical per unit of water treated was $\$4.5 \times 10^{-6}/\text{m}^3$ for commercial antiscalant, $\$2.1 \times 10^{-6}/\text{m}^3$ for PASP-SEA-ASP and $\$1.5 \times 10^{-6}/\text{m}^3$ for PASP, PASP and its derivative appeared to possess an advantage on cost-effectiveness. It is recommended that further investigations into the impact of process variables such as pH, inorganic species, the presence of organic matter in feedwater, and membrane materials on their scale inhibition effect be conducted at lab and pilot scales before being employed for full scale applications.

Supplementary data to this article can be found online at <http://dx.doi.org/10.1016/j.desal.2016.11.019>.

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