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The influence of feed pH on the performance of a reverse osmosis membrane during alginate fouling

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ABSTRACT

The performance of a brackish water reverse osmosis membrane in the presence of a model polysaccharide foulant, sodium alginate, is investigated. The concentration of foulant, the feed pH and the presence of calcium are all shown to impact upon this performance. The flux decline is greatest at low feed pH, high calcium concentrations and high foulant concentrations. Sodium and chloride rejection falls as foulant concentration increases, due to cake-enhanced osmotic pressure. Effects are again greatest at low feed pH and high calcium concentrations. In part, this reflects a shift of the isoelectric point of the membrane from 4.3 to below 3.0 as alginate is added, reflecting the lower pK_a of the alginate foulant. Conversely, calcium rejection improves in the presence of even small quantities of alginate foulant at all pH values. These increases in rejection are believed to relate to a direct interaction between the dissolved ions and the alginate which act to sequester the salt.

Keywords: Reverse osmosis; Polysaccharide; Fouling; Rejection

1. Introduction

Reverse osmosis (RO) is a widely adopted technology for both waste water reclamation and seawater desalination. The technology has advanced significantly during the past decade but membrane fouling remains one issue that limits performance [1–10]. Many of the recent studies on RO membrane fouling have used protein and humic acids as foulants [11–13], while less work has focused on polysaccharides. Polysaccharides are the main constituents of extra polymeric substances which have been reported as major foulants in membrane bioreactors [14–16] and RO systems [17–19]. Indeed, recent work has shown that such polysaccharides have a dominant impact upon the fouling cake structure in mixtures of beyond 25% with humic acids [20].

A fundamental understanding of membrane fouling by polysaccharides and their influence on membrane performance is necessary for the efficient operation of desalination plants. Many authors have shown that fouling of RO membranes causes a flux decline, which can be attributed to foulant adsorption, cake layer formation or cake-enhanced osmotic pressure. This latter phenomenon is caused by a build up of salt within the fouling cake that in turn increases the osmotic pressure at the membrane surface and so limits the available transmembrane pressure [21,22]. Salt rejection can either increase or decrease as fouling occurs [23–25]. When the solutes are rejected better by the membrane than by the fouling layer, they accumulate within this region, leading to cake-enhanced osmotic pressure and declining rejection. This is the case most commonly observed [25]. Conversely, when the solutes are rejected better by the fouling layer than

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by the membrane, rejection can improve. In particular, foulants can accumulate in membrane defects or "hot spots", blocking low resistance pathways through the membrane, and thus increasing rejection and reducing flux [11,26]. Alternatively, rejection may increase due to additional electrostatic interactions between the dissolved ions and the fouling layer. Such behaviour has been noted previously by Tang et al. [11,27].

Fouling behaviour is influenced by solute properties such as the molecular weight, membrane properties such as the pore size and hydrophobicity and hydrodynamic factors such as the crossflow velocity and the membrane flux [28]. Of particular relevance to the present work are the charge properties of the membrane, the solute and the foulant. Electrostatic interactions between foulant molecules themselves directly influence their intermolecular adhesion and thus the structure of the fouling cake. Similar interactions between these molecules and the membrane surface influence the adhesion of the fouling cake to the membrane. Finally, electrostatic interactions between the solute and both the membrane and the foulant will influence whether rejection increases or declines as fouling continues. Fouling can also be influenced by Van Der Waals forces, which are largely insensitive to such charge interactions [29].

Lee and Elimelech [30] found that at pH values greater than 5, the carboxylic acid groups on an alginate polysaccharide were completely deprotonated, leading to strong electrostatic repulsion between the negatively charged molecules and a decrease of intermolecular adhesion; which they measured using atomic force microscopy. Protonation occurred between pH 5 and pH 3, consistent with the recorded pK_a of alginic acid being 3.0–3.1 [31,32]. Thus at pH 3.0, the carboxylic groups were completely protonated leading to an increase in intermolecular adhesion and a rapid flux decline during fouling experiments. These authors showed that the presence of calcium in the feed solution also significantly increased intermolecular adhesion and fouling rates, due to complexation of these divalent cations to the carboxylic groups. The impact of calcium was significantly greater than that of magnesium, which they attributed to the formation of intermolecular bridges between the alginate molecules formed by the calcium species, commonly referred to as the "eggbox" model [33,34]. However, this work did not consider the influence of membrane charge and also did not report the effect upon membrane rejection.

This paper investigates the impact of fouling with polysaccharides (alginates) on the performance of brackish water RO membranes, with a focus on both membrane charge and salt rejection, as this has not been the focus of previous work.

2. Materials and methods

2.1. Materials

The RO membrane used for this study was a brackish water AK membrane from GE Osmonics. Salt solutions were prepared from AR grade crystalline NaCl and $CaCl_2 \cdot 2H_2O$ as supplied by Chem Supply (South Australia). All solutions were prepared to a total ionic strength of 0.0342 M, either as 2 g/L of NaCl or as 0.17 g/L $CaCl_2 \cdot 2H_2O$ and 1.80 g/L NaCl. Deionized water with resistivity of 18.2 MΩ cm was used exclusively in all experiments (MilliQ, Millipore). The pH was adjusted with AR grade HCl, NaOH and $Ca(OH)_2$ corresponding to the co-ions present in the feed solution. Alginic acid was supplied as the sodium salt (Kimitsu Chemical Corporation, Japan).

2.2. Equipment

Membranes were tested in a rig consisting of three parallel Sterlitech CF042 membrane cells. Each cell had a membrane area of 42 cm^2 and a channel depth of 2.3 mm. No spacer was used. Further details of the flat sheet membrane cross flow rig are provided in our earlier paper [35]. Membranes were washed and soaked in deionized water overnight before use. They were then installed in the rig and compacted at 1,600 kPa for 2 h. Water was run for two hours through the unit at the experimental pressure, then replaced by salt water. The entire permeate from each membrane unit and a small amount of retentate was collected every 30 min over a 3-h period and stored for analysis. After completion of the experiment, fresh water was run to wash and clean the membranes. Unless otherwise stated, the experimental pressure was 800 kPa and the retentate flowrate was 4.2 L/min across each cell, giving a crossflow velocity of 0.67 m/s. While this velocity is high relative to full-scale RO operations, it was chosen to reduce the impact of concentration polarization, as the cells did not accommodate spacers. The processing temperature was 30 ± 1℃.

Fouling experiments were carried out following the same procedure with the feed solution containing sodium alginate at 5 or 20 mg/L. The effect of divalent ions was investigated by adding calcium chloride and reducing sodium chloride content in a manner such that the solution ionic strength was unchanged. The fouled membranes were cleaned by washing with salt water (2,000 mg/L), with sodium hydroxide solution (pH = 11.0) and finally deionized water until permeate flux was restored to the original values (±2%).

2.3. Analysis

A Varian 720-ES ICP-Optical Emission Spectrophotometer was used for determination of sodium and calcium concentrations. Chloride concentration was determined by potentiometry using a Metrohm autotitrator Titrando Dosino with Ag–Pt electrodes and 0.02 N silver nitrate solution. pH and conductivity were measured using an Orion 720A$^+$ pH-meter and an Orion 3 star conductivity meter. The apparent rejection (R_{app}) was obtained from the following equation:

$$R_{\mathrm{app}} = (1 - C_{\mathrm{p}}/C_{\mathrm{r}}) \times 100 \tag{1}$$

where C_{p} is the average value of the element concentration of three permeate solutions collected and C_{r} is the retentate concentration at the same period.

The normalized flux was calculated from:

$$J = J_{\mathrm{salt}}/J_{\mathrm{water}} \tag{2}$$

where J_{salt} is the water flux during salt water circulation and J_{water} is the corresponding flux during water circulation (39.5 ± 5 L/m^2 h).

The streaming potential was measured with a commercial electrokinetic analyser EKA from Anton Paar GmbH, Austria at a total ionic strength of 2 mM. The relationship between the measurable streaming potential and the zeta potential is given by Helmholtz-Smoluchowski equation [36]:

$$\zeta = \frac{\mathrm{d}U}{\mathrm{d}p} \cdot \frac{\eta}{\varepsilon \cdot \varepsilon_0} \cdot \frac{L}{Q \cdot R} \tag{3}$$

where ζ is the zeta potential, $\mathrm{d}U/\mathrm{d}p$ is the slope of a plot of streaming potential vs. pressure, η is solution viscosity, ε_0 is the permittivity, ε is the dielectric constant, L and Q are the length and the cross-sectional area of the capillary system, and R is the AC resistance of the cell.

3. Results and discussion

3.1. Adsorption of foulant on RO membrane

The adsorption of polysaccharide to the membrane surface can be demonstrated by the change of the zeta potential of the membrane surface. Fig. 1 illustrates the surface zeta potential of an AK membrane as a function of pH in the absence and presence of alginic acid. In a standard NaCl electrolyte, zeta potential generally decreases to a plateau at higher pH, indicative of a typical polyamide surface [35,37]. With the addition of 1 mg/L sodium alginate, the zeta potential becomes more negative and the isoelectric point (IEP) falls from 4.3 to a value outside the measurement range. As more alginate is added, the zeta potential becomes even more negative. Extrapolation of the curves gives an IEP of around 2.6, which coincides with the IEP for alginate fouling reported by Negaresh et al. [38] further confirming the polysaccharide had adsorbed to the membrane surface and is dominating the surface charge.

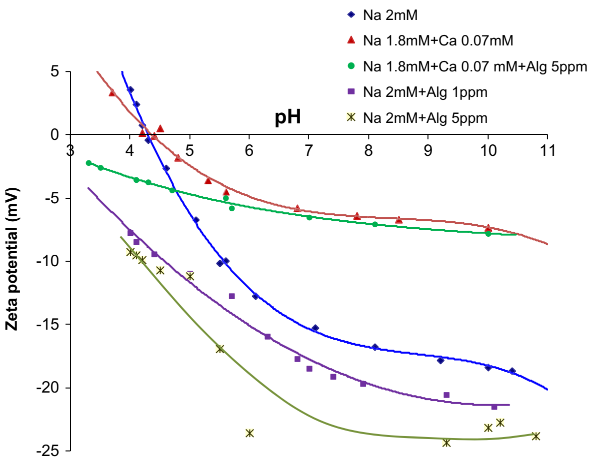

Fig. 1. The zeta potential of an AK membrane in sodium chloride and mixed solutions as a function of pH. The ionic strength of all solutions was 0.002 M.

When calcium chloride is added, the zeta potential of the membrane becomes less negative due to the adsorption of divalent cations to the membrane surface [37]. Calcium ions will also interact with the alginate and neutralize its negative charge [39]. However, it is interesting to notice that the extrapolated IEP in this case is also equal to the IEP of the fouled membrane in a calcium-free solution.

3.2. Effect of pH and Alginate Concentration on membrane flux

As reported in our earlier work [35], in a solution purely of sodium chloride, the normalized flux through the membrane varies little with pH (Fig. 2). There is some evidence of an increase around the IEP of 4.3, but this is within experimental error bounds.

Consistent with the work by Lee and Elimelech [30] this flux changes marginally with the addition of sodium alginate at high pH (Fig. 2 and Table 1). Carboxylic groups are the dominant functional groups

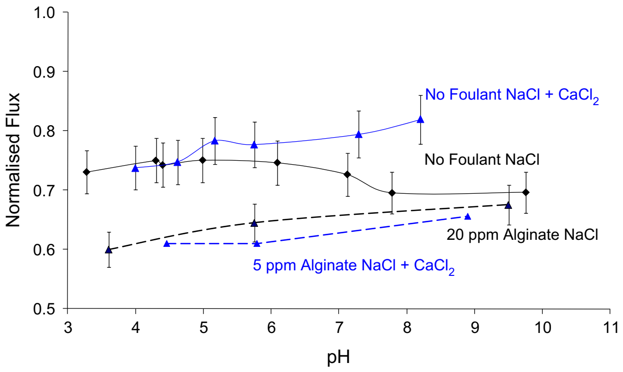

Fig. 2. Variation in normalized flux as a function of pH in a solution of total ionic strength 0.034 M (either as 2 g/L NaCl or as 1.80 g/L NaCl and 0.17 g/L $CaCl_2 \cdot 2H_2O$) and 5–20 ppm alginic acid added. Crossflow velocity 0.67 m/s, transmembrane pressure 800 kPa. Error bars are ±5%, based on replicate measurements. Absolute flux of pure water was 39 ± 5 L/m^2 h.

of alginic acid and above pH 5.0 they are completely deprotonated. This leads to an increase in the electrostatic repulsion between the negatively charged alginate molecules and thus a decrease of intermolecular adhesion among the foulants. At high pH, the membrane surface is also negatively charged (Fig. 1) causing electrostatic repulsion between the two materials. As a result, the foulant is not attracted to the membrane and the fouling effect is relatively small. However, as pH reduces, more alginate molecules are protonated, leading to an increase in their intermolecular adhesion. When the pH falls below the membrane IEP (pH = 4.3) the foulant and the membrane material have opposite charge, encouraging more foulants to be adsorbed onto the membrane surface. Under these conditions, even a very small quantity of polysaccharide has a significant effect on membrane fouling.

3.3. Salt rejection

As shown in our prior work [35], in a pure sodium chloride solution, a high sodium rejection is achieved at high and low pH while low rejection is seen at the IEP of the membrane (Fig. 3(a)). High chloride rejection is also obtained at high pH but falls continuously with decreasing pH (Fig. 3(b)). The negative charge of the membrane at high pH repels chloride ions resulting in a high chloride rejection. The sodium rejection is also high to maintain the charge balance of the components in the solution. As the IEP is approached, rejection of both species falls, as these charge effects are diminished. At pH values below this point, the positive charge of the membrane repels the sodium ions, giving it a high rejection. Chloride permeation continues to increase, with the negative charge balanced by the increasing permeation of the smaller hydronium ions [35].

Upon the addition of alginate, the sodium and chloride rejections both decrease for all pH values (Fig. 3(a) and (b)). This decrease can be related to cake-enhanced osmotic pressure. At pH 6.0 and 9.5, the fall in rejection is quite small, as at these conditions, both the fouling cake and the membrane charge are comparable and hence both have a similar effect upon the dissolved ions (Fig. 1). However, at pH 3.5, the rejection falls quite significantly. Under these conditions, the fouling cake is uncharged, whereas the membrane is positively charged. As discussed by Ng and Elimelech [25] when solutes are rejected better by the membrane than by the fouling layer, they accumulate within this region, leading to cake enhanced osmotic pressure. The minimum originally observed in the sodium rejection at the IEP has also disappeared. This reflects the move of the IEP to around 2.6, as shown in Fig. 1.

3.4. Effect of calcium ions

The addition of calcium to a non-fouling salt mixture causes the flux at high pH to increase (Fig. 2), as reported in our earlier work [35]. This is believed to arise from an expansion of the membrane matrix due to repulsion between like-charged dissociated functional groups on the pore walls [40]. The calcium also acts to reduce the negative charge on the membrane, which in turn will reduce the repulsion between the membrane surface and the alginate foulant. However,

Table 1
The effect of sodium alginate on membrane flux as a function of feed pH and foulant concentration

Sodium alginate concentration (mg/L)	Decline in flux relative to no foulant case (%)			
	pH 3.5	pH 4.3 (IEP)	pH 6	pH 9.5
5	14	3.5	0.4	0.3
20	17	15	12	1.6
180	25	23	18	3.8

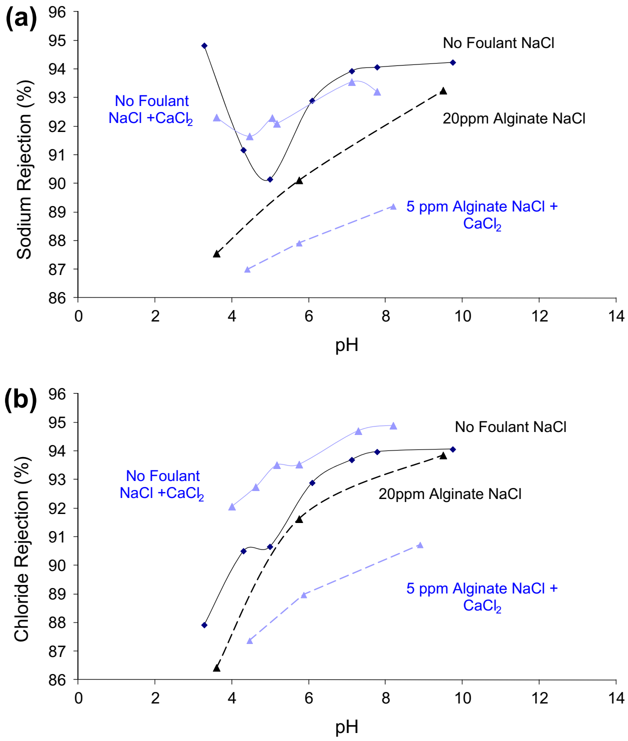

Fig. 3. The effect of pH on the apparent rejection of ionic species in salt solutions with total ionic strength of 0.034 M (either as 2 g/L NaCl or as 1.80 g/L NaCl and 0.17 g/L $CaCl_2 \cdot 2H_2O$) and 5–20 ppm alginic acid added. Crossflow velocity 0.67 m/s, transmembrane pressure 800 kPa. (a) Sodium rejection and (b) chloride rejection.

in the presence of even 5 mg/L of sodium alginate, the normalized flux reduces significantly regardless of the feed pH (Fig. 2). Since the total ionic strength was kept constant, the effect of charge screening or double layer compression should be comparable to the pure NaCl solution. The greater fouling in this case may be attributed to the complexation of calcium ions with the carboxylic groups of the alginate polysaccharide. Bridges between neighbouring alginic molecules are formed, resulting in an egg-box-shaped gel network [33,34] which results in a thicker and more compact fouling layer [4,5,13].

As previously reported [35], the replacement of some sodium with calcium in a non-fouling mixture causes chloride rejection to increase, reflecting strong calcium rejection (Fig. 3(b)). Sodium rejection is relatively unchanged, although the rejection minimum at the IEP is less prominent. However, as above, the addition of even 5 mg/L of alginate causes a dramatic decline in rejection of both sodium and chloride. Conversely, these small additions of alginate cause the calcium rejection to increase from 96–97 to 99–99.5% across the full pH range (data not shown). These results may again be explained by calcium ions forming complexes with the carboxylic groups of the alginate polysaccharide. As the alginate is itself completely rejected, this acts to increase calcium rejection. Further, the calcium complexes act to neutralize the negative charge of the foulant layer. This enhances the build-up of sodium and chloride within this fouling cake, enhancing the osmotic pressure in this region and hence further reducing sodium and chloride rejection.

4. Conclusions

The present study has confirmed that the performance of an RO membrane is influenced by foulant concentration, feed pH and the presence of calcium ions in the solution. The flux decline that occurs after the addition of alginate as a model polysaccharide is greatest for higher concentrations and at low pH. The presence of calcium ions promotes the fouling process dramatically due to their capacity to form bridges between the alginic molecules and the membrane surface. As a result, the normalized flux declines rapidly.

The sodium and chloride rejection falls upon the addition of alginate across all pH values. However, the fall is most dramatic at low pH values, where the membrane is positively charged, but the foulant is neutral. This can be readily related to cake-enhanced osmotic pressure. Calcium rejection also increases uniformly at all pH values and all foulant concentrations.

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