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Separation of antiscalants from reverse osmosis concentrates using nanofiltration

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ABSTRACT

One of the serious problems found in desalination using reverse osmosis (RO) is concentrate or brine management. This concentrate can actually be used as a raw material for salt production. However, antiscalants added during RO process should be removed before salt crystallization. This paper presents the separation of antiscalants from RO concentrate solution using nanofiltration (NF) membrane. Sodium hexametaphosphate (SHMP) and disodium ethylenediaminetetraacetate (Na_2EDTA) were used as models of antiscalants. The effect of antiscalants concentration and addition of natural organic matters on both flux behavior and rejection were investigated. The results showed that the permeate flux behavior was influenced by the sodium chloride solution itself as a solution background, antiscalant type and antiscalant concentration. Osmotic pressure, concentration polarization and fouling or scaling contributed to flux decline during nanofiltration of antiscalants. NF membrane demonstrated very high rejection for both SHMP and Na_2EDTA.

1. Introduction

Reverse osmosis (RO) as today's leading process for producing fresh water from seawater and brackish water has replaced the conventional desalination process. This technology has successfully been utilized to solve desalination problems due to its ability to produce superior and stable quality of water in a relatively little energy demand. However, this process results in concentrated saline water, also known as RO concentrate waste, which contains high concentration of dissolved salts [1,2]. This RO brine waste usually contains concentrations of total dissolved solids (TDS) 68,130 mg/L, Ca^{2+} 961 mg/L, Mg^{2+} 2940 mg/L, Cl^{-} 42,500 mg/L, Na^{+} 17,000 mg/L, HCO^{3-} 267 mg/L, SO_4^{2-} 6420 mg/L and chemical residues from pre-treatment process of RO [3]. The management of the concentrated waste stream is one of the remaining obstacles for the implementation of desalination using RO membranes, since the concentrate is usually unusable and has to be discharged or further treated. Normally, brines resulted from desalination plants in coastal area are directly discharged to the sea, posing adverse environmental effects on the receiving marine environment [4]. In addition, treating concentrate of RO is very costly.

Currently, brine utilization is getting more and more attention from both researchers and industries. In principle, brine utilization is directed to either increasing water or obtaining valuable components from seawater [5–11]. Among them, salt production by evaporating ~~of~~ RO concentrate is very interesting from practical point of view. Membrane distillation and membrane crystallizer have been proposed to increase water recovery and to obtain salt crystals [6,8,11]. However, these processes did not separate antiscalants from the salt crystals. It is important to note that antiscalants are present in RO brine as a consequence of control of scaling formation on RO membrane surface during desalination [12]. Therefore, separation of antiscalants from RO brine is very important when salt production will be performed.

Common antiscalants used for desalination using RO are polyacrylic acid, carboxylic acids, polyphosphates, phosphonate (threshold agents), anion polymer, sodium hexametaphosphate (SHMP), trisodium phosphate, crystal modifiers, sequestering agent (disodium ethylenediaminetetraacetate/Na_2EDTA), and dispersant [13–15]. Among several types of antiscalants, SHMP and Na_2EDTA are the most frequently used due to their effective ways to prevent scaling in the desalination process [16–18]. The existence of antiscalants in the RO concentrates waste has negative impact on the environment and health. SHMP is toxic, causing heat to the water body, damaging the reef, eutrophication and heavy metal accumulation. In addition, SHMP causes irritation of the mucous membranes if inhaled, intestinal damage if ingested, gastrointestinal irritation, nausea, diarrhea, affect the nervous system in high doses, heart disorders, and decreased blood pressure. Na_2EDTA leads to eutrophication and if it enters into the human body in excessive amounts causes body deficiencies of Ca and other minerals. This is because of

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Na_2EDTA has an effective binding metal ions such as calcium ions [19,20]. Therefore, the proper method is needed to eliminate antiscalant from reverse osmosis concentrate.

Several techniques have been proposed for separation of antiscalants. Boels et al. [21] and McCool et al. [13] reported that iron coated waste filtration sand and intermediate concentrate demineralization can eliminate antiscalant from the RO concentrate. However, this method requires chemicals addition, generates sludge, low permeate quality, high operating costs, and the antiscalant in the RO concentrate is still not completely separated. In this study, separation of antiscalant from waste concentrates RO using nanofiltration membranes was investigated. Nanofiltration was selected because it requires lower operating pressures and temperatures, high rejection of organic molecules, low operation and maintenance costs [22]. SHMP and Na_2EDTA were used as models of antiscalants dissolved in RO concentrate waste. Furthermore, sodium alginate was used as a model of organic matters in the waste concentrates. It should be noted that concentrate wastes, as byproducts of desalination using RO membrane, contain organic substances [23].

2. Materials and methods

2.1. Materials

In this study, SHMP and Na_2EDTA were used as models of antiscalants, while sodium alginate (SA) was used as a model of natural organic matters. SHMP and SA were purchased from local company, Semarang, Indonesia. Na_2EDTA was purchased from Merck, Germany. NaCl (technical grade) was purchased from PT. Unichem Candi Industri, Indonesia. KH_2PO_4, $(NH_4)_6Mo_7O_{24}\cdot 4H_2O$, NH_4VO_3, HCl, H_2SO_4 and NaOH were purchased from Merck, Germany. NaCl (p.a. grade) was purchased from Sigma Aldrich Germany. Nanofiltration membrane (NF 270) was obtained from Dow Filmtec Membranes, USA.

2.2. Methods

The filtration experiments were performed by using a home-made laboratory scale for cross flow filtration [24].The set-up consisted of a feed tank (3 L volume), a pump, a pressure indicator connected to feed side of membrane to determine the trans-membrane pressure and a flat-sheet membrane cell. Fig. 1 shows the simplified diagram of experimental set-up. A new circular membrane disk was used in each experiment. The membrane was firstly compacted by filtering pure water for at least 0.5 h at a pressure of 600 kPa. The volume of feed was much larger than the volume taken as a sample for the analysis. In addition, the retentate and permeate were returned to the feed tank. All experiments were performed at room temperature (28 ± 2 °C) and at a constant trans-membrane pressure (500 kPa).

Synthetically prepared RO concentrate solution was used in this study. The RO concentrate solution was prepared by dissolving sodium chloride (technical grade) in pure water to obtain 60,000 ppm solution. A certain concentration of either SHMP or Na_2EDTA with and without addition of sodium alginate was dissolved in this synthetic concentrate solution. The concentration of SHMP was varied, i.e. 2, 4, and 6 ppm, while the concentration of Na_2EDTA was varied, i.e. 2, 3, and 4 mM. The selection of antiscalants concentration was based on the concentration when they are used in RO process.

The flux profile was expressed in term of normalized flux (J/J_o). The initial flux (J_o) was measured by passing pure water after the compaction was conducted, while the permeate flux (J) was gravimetrically measured every 10 min during the filtration was performed. The concentrations of SHMP and Na_2EDTA were analyzed using spectrophotometer UV–Vis (Genesys 10S UV–Vis, USA) at wavelength of 400 nm and 263 nm, respectively. The membrane surface was visualized by using a Scanning Electron Microscope (JEOL JSM-6510LA SEM, Japan). The outer surface of the sample was coated with gold/palladium and sputtered for 0.5 min before analysis.

3. Results and discussion

3.1. Flux behavior during nanofiltration of antiscalants

The nanofiltration behavior of antiscalant was investigated by filtering the feed solutions containing different concentration of SHMP and Na_2EDTA. In addition, the feed solution containing NaCl only (0 ppm of antiscalant) was also filtered. The experiments were performed at constant trans-membrane pressure. The results are expressed in term of flux (normalized flux) as a function of filtration time and presented in Figs. 2 and 3.

It is shown that both antiscalants solution displayed rapid flux decline in the early stage of filtration. Thereafter, relatively constant permeate after 30 min of filtration was demonstrated by SHMP, whereas gradual decrease was shown by Na_2EDTA. The flux decline during nanofiltration can basically be attributed to concentration polarization, osmotic pressure and fouling or scaling. The presence of concentration polarization on the membrane surface can increase the level of flux decline due to osmotic pressure. Furthermore, concentration polarization can also facilitate fouling by altering interactions

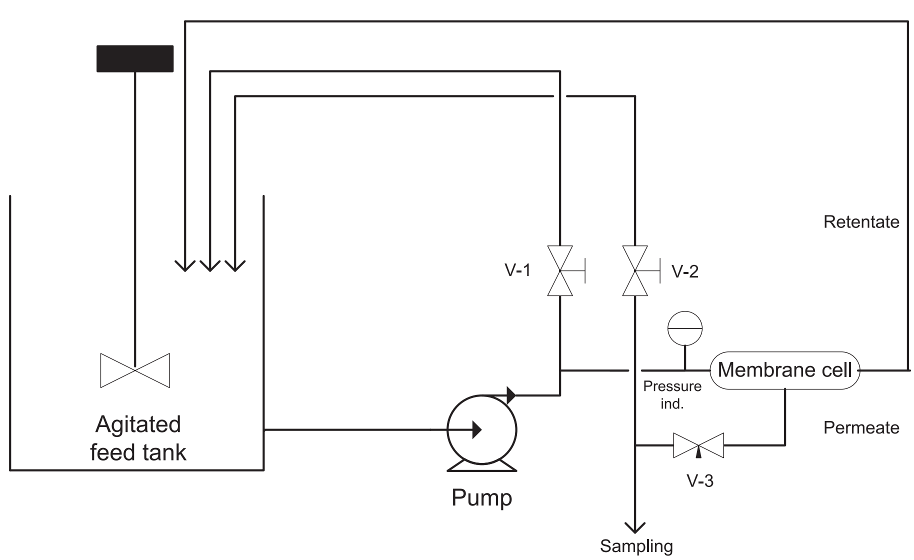

Fig. 1. The simplified diagram of nanofiltration experimental set-up [24].

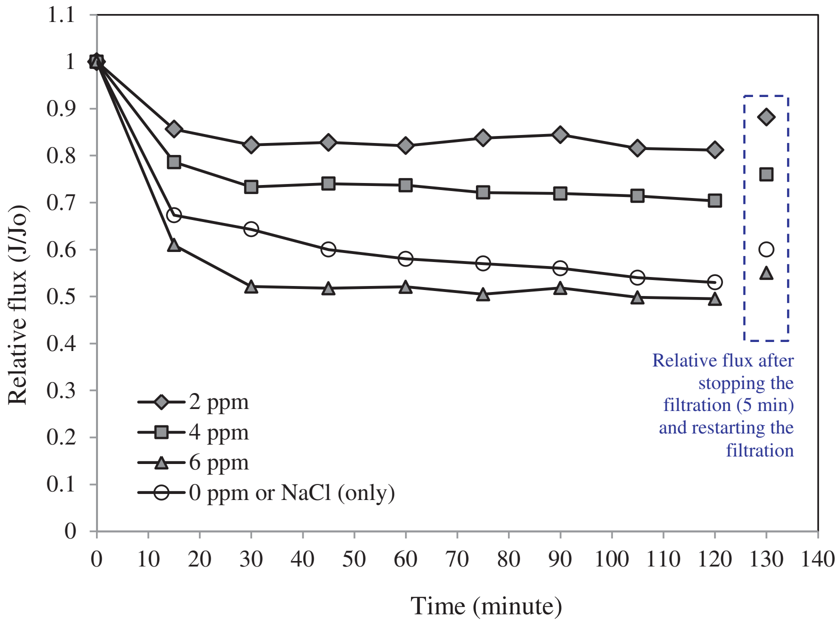

Fig. 2. Flux behavior during nanofiltration of SHMP solution (SHMP was dissolved in 60,000 ppm of NaCl solution (technical grade)). The NaCl (only) solution was prepared using NaCl p.a. grade. The trans-membrane pressure was 500 kPa.

between membrane and feed components.

The contribution of concentration polarization on the rapid flux decline at the beginning of filtration was investigated by stopping the filtration just after 5 min filtration for at least 10 s. The filtration was then started again. The results showed that the flux was lower than the initial value but was higher than the flux at 5th min filtration before stopping the filtration (data not shown). The contribution of concentration polarization was also investigated by stopping the filtration at the end of filtration for 5 min and restarting again the filtration. The fluxes demonstrated higher values compared to the fluxes before stopping the filtration but lower than the initial water flux (Jo). These two examination experiments of concentration polarization suggest that concentration polarization contributed to the rapid flux decline in the beginning of filtration as well as the flux decline for the whole filtration but it was not only the reason for flux decline. The observation in more detail shows that the increases in fluxes after stopping the filtration were similar for all concentrations of antiscalants, i.e. 6–7% for SHMP and 7–8% for Na_2EDTA. These phenomena indicate that the concentration polarization effect was mainly derived from NaCl rather than from antiscalants (note that the concentration of NaCl was very high, i.e. 60,000 ppm).

The effect of osmotic pressure was investigated by measuring the pure water permeability of the membranes after used. The results are presented in Table 1. The results showed that the permeability of all membranes after used were lower than the virgin membrane but higher than the permeability flux measured using feed solution (converted from fluxes in Figs. 2 and 3). The results suggest that the contribution of osmotic pressure to flux decline was confirmed. The contribution of osmotic pressure to flux decline during nanofiltration was also reported in previous publication [25].

Surprisingly, the permeate fluxes of antiscalants solution except for the highest concentration (6 ppm for SHMP and 4 mM for Na2EDTA) were higher than the permeate fluxes obtained using NaCl (only) solution as the feed. The presence of antiscalants with a certain

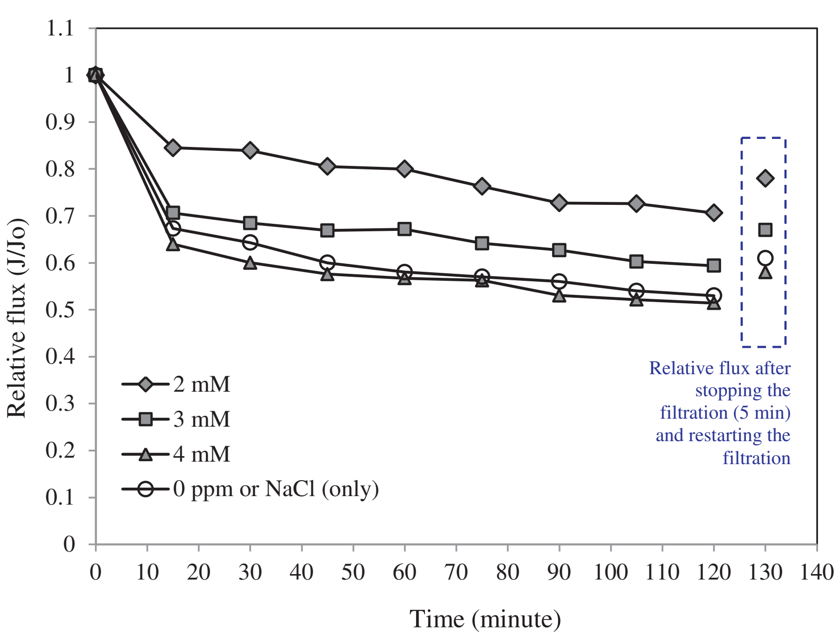

Fig. 3. Flux behavior during nanofiltration of Na_2EDTA solution (Na_2EDTA was dissolved in 60,000 ppm of NaCl solution (technical grade)). The NaCl (only) solution was prepared using NaCl p.a. grade. The trans-membrane pressure was 500 kPa.

Table 1
Pure water permeability and normalized water permeability of various membranes.

No	Membrane	Permeability (L/m^2 h bar)	Lp/Lp_o
1	Virgin membrane	16.92	–
2	Membrane used for filtering NaCl solution (only)	15.90	0.94
3	Membrane used for filtering SHMP 2 ppm	15.40	0.91
4	Membrane used for filtering SHMP 4 ppm	14.55	0.86
5	Membrane used for filtering SHMP 6 ppm	14.04	0.83
6	Membrane used for filtering Na_2EDTA 2 mM	14.72	0.87
7	Membrane used for filtering Na_2EDTA 3 mM	12.86	0.76
8	Membrane used for filtering Na_2EDTA 4 mM	11.34	0.67
9	Membrane used for filtering SHMP 2 ppm + ALG (25 ppm)	12.86	0.76
10	Membrane used for filtering Na_2EDTA 2 mM + ALG (25 ppm)	12.01	0.71

Note: Lp_o was permeability of virgin membrane.

concentration increased the permeate fluxes. It seemed that the function of antiscalants to prevent precipitation on the membrane surface as reported by Andrade et al. [26] occurred in this nanofiltration. Nevertheless, if the concentration was increased the antiscalants could give a negative impact on permeate flux, i.e. decreasing the permeate fluxes.

Previous explanation demonstrates that concentration polarization and osmotic pressure contributed to flux decline of nanofiltration of antiscalant solution. However, fouling still has a portion as the reason for the decreasing permeate flux. Comparing the flux decline in filtering SHMP and Na_2EDTA solutions showed that the flux decline during filtration of Na_2EDTA solution was larger than the flux decline during filtration of SHMP solution. These results indicate that the fouling mechanism during nanofiltration of SHMP and Na_2EDTA solution was different. It is believed that the gel layer formation is not the possible mechanism for both antiscalants filtrations. Thus, the possible fouling mechanism during nanofiltration of SHMP solution was complete blocking as indicated by rapid flux decline at the beginning of filtration followed by steady state permeate flux. The possible fouling mechanism during nanofiltration of Na_2EDTA solution was intermediate blocking as indicated rapid flux decline followed by gradual flux decline. The difference in fouling mechanism was due to the difference in interaction between SHMP − NaCl and Na_2EDTA − NaCl.

In addition to flux behavior, the rejections of salt and antiscalants were observed. The results are presented in Table 2. It is seen that the rejection of NaCl was relatively low for all conditions (19–25%). These results are in a good agreement with the study by Su et al. [27]. Considering the low rejection of NaCl, the membrane used in this study (NF 270) should be considered to be a loose nanofiltration membrane. This explanation is supported by previous publications [26,28]. The rejection of Na_2EDTA was slightly higher than the rejection of SHMP. As the concentration was increased the rejection of antiscalants did not change significantly. Because electrostatic interaction between membrane and feed should be dominated by NaCl-membrane the electrostatic interaction should be not the reason for this slight difference in rejection. Thus, steric hindrance should be the reason. It is important to inform that our membrane surface charge measurement by streaming potential [29] showed that the isoelectric point of the membrane was 4.3. All feed solutions had pHs above the isoelectric point of the membrane.

The effect natural organic matter on permeate flux behavior was investigated by addition of sodium alginate (ALG) into the feed containing antiscalant. The concentration of ALG was 25 ppm. The results are presented in Fig. 4. First of all, the presence of alginate decreased the effect of NaCl on flux decline as found in filtration without ALG. The presence of ALG increased the flux ratio from 0.55 to ~0.9. The possible reason for this phenomenon was that the presence of ALG decreased the osmotic pressure and concentration polarization caused by NaCl ions. Further, it was clearly observed that the addition of ALG increased the flux decline significantly. The increase in flux decline was more significant for Na_2EDTA. The presence of ALG at high ionic strength caused aggregation leading to the formation of organic layer on the membrane surface. The ability of EDTA to form aggregate was higher than SHMP. Similar phenomenon was observed by Resosudarmo et al. [30] and Ang et al. [18]. It should be noted that the presence of multivalent ions is possible (note that in this study technical grade of NaCl was used for all experiments except for the solution NaCl only/without antiscalants). The addition of ALG increased the rejection of phosphate, EDTA and NaCl (Table 2).

3.2. Membrane visualization

The membrane surface morphology was visualized by using SEM. Fig. 5 shows the SEM results of fresh membrane and the membrane after used for the filtration of feed containing ALG, SHMP, SHMP and ALG, Na_2EDTA, and Na_2EDTA and ALG. The SEM results confirmed the deposition of antiscalants, ALG and salt crystals on the membrane

Table 2
Rejection of phosphate, EDTA and NaCl at different concentrations of antiscalant.

SHMP concentration (ppm)	Phosphate rejection (%)		NACl rejection (%)	
	Without ALG	With ALG	Without ALG	With ALG
2	96.9	98.1	19.5	21.2
4	97.8	97.2	19.7	23.1
6	98.3	96.9	20.1	22.8

Na_2EDTA concentration (mM)	EDTA rejection (%)		NACl rejection (%)	
	Without ALG	With ALG	Without ALG	With ALG
2	96.5	99.4	24.5	30.9
3	96.6	98.8	23.7	31.6
4	98.9	97.6	24.9	31.2

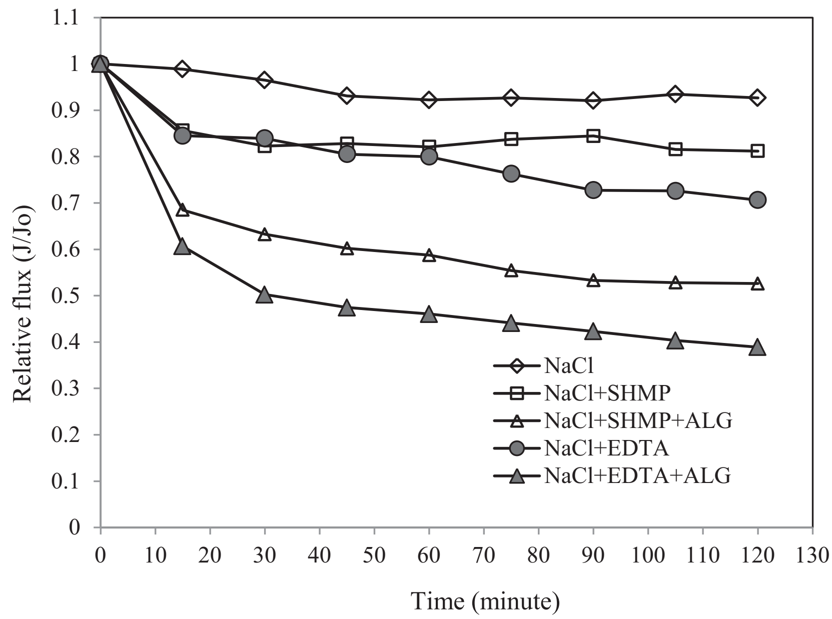

Fig. 4. The effect of ALG (25 ppm) addition on flux behavior during nanofiltration of SHMP (2 ppm) and Na_2EDTA (2 mM) solution (dissolved in 60,000 ppm NaCl solution). The NaCl (only) solution was prepared using NaCl p.a. grade. The trans-membrane pressure was 500 kPa.

surface.

It is seen from Fig. 5 that there is no particle on the surface of fresh membrane was found, while the presence of some salt crystals was observed on the membrane surface after used for filtration. Surprisingly, the presence of alginate layer on the membrane surface was not clearly observed. The very high concentration of NaCl in the feed will interact with sodium alginate added causing the remaining alginate that could be deposited on the membrane surface was very

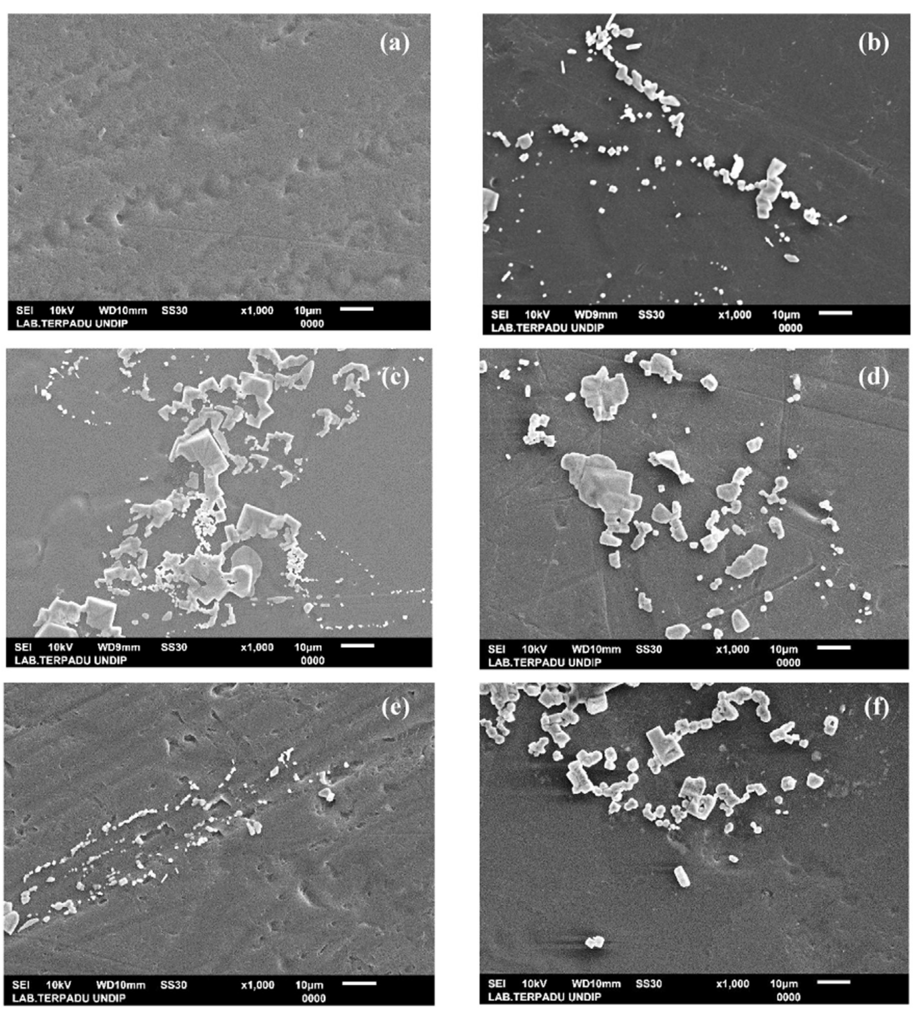

Fig. 5. Scanning electron micrographs of membrane surface of NF membranes: (a) fresh membrane before used (b) after used for filtering ALG solution, (c) after used for filtering SHMP solution, (d) after used for filtering SHMP + ALG solution, (e) after used for filtering Na_2EDTA solution, and (f) after used for filtering Na_2EDTA + ALG solution.

low. Nevertheless, FTIR spectra confirmed the presence of alginate on the membrane surface. New significant peaks within the wave length 3120–3600 cm^{-1} indicating OH stretching and at ~1600 cm^{-1} and at ~1415 cm^{-1} indicating antisymmetric and asymmetric stretch of carboxylate group were observed (the IR spectra are not shown). Overall the SEM results support the previous explanation that the flux decline during nanofiltration of antiscalant solution (dissolved in very high NaCl concentration) was dominated by the interaction of NaCl solution with the membrane (scaling). Because scaling by salt crystallization has taken place on the membrane surface, cleaning and membrane reuse will be important issues in practical implementation.

4. Conclusion

Separation of SHMP and Na_2EDTA antiscalants using nanofiltration for the possible treatment of RO concentrate has been investigated. The results showed that the permeate flux behavior was influenced by the NaCl solution as the background of antiscalant solutions, antiscalant type, antiscalant concentration and the presence of organic matter. The contributions of osmotic pressure, concentration polarization and fouling or scaling on flux decline were confirmed. The presence of antiscalant with a certain concentration increased the permeate flux when very high concentration of NaCl was used as the feed background solution. Different fouling mechanism was observed during filtration of SHMP and Na_2EDTA. The presence of ALG as natural organic matter increased the occurring fouling. NF membrane showed very high rejection of antiscalants, i.e. higher than 96% for both antiscalants used. The presence of ALG slightly increased the antiscalant and NaCl rejection. Overall, this high rejection of antiscalants suggests the opportunity of using NF for further processing of RO concentrate for the raw material of salt crystal production.

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