

Investigation of organic fouling during ultrafiltration dedicated to reverse osmosis pretreatment

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ABSTRACT

Ultrafiltration (UF) membrane has been widely proposed as a pretreatment unit to reduce fouling in RO membrane. However, less attention has been paid for UF fouling study using high saline water such as brackish water or seawater. This paper presents a study of ultrafiltration fouling of organic matters dissolved in high saline water. Alginate, humic acid and protein were used as models of organic matters present in seawater. The fouling behavior was investigated by observing the adsorptive fouling (membrane-solutes interactions) and ultrafiltration fouling (membrane-solutes-solutes interactions). The results showed that organic fouling behavior of UF membrane in high saline water was different with in fresh water. The presence of salt ions in a feed containing organic matter increased adsorptive fouling (for alginate and humic acid) but it decreased adsorptive fouling for BSA. The UF fouling behavior of an organic matter in NaCl solution was the most similar with the fouling behavior demonstrated by organic matters dissolved in real seawater. Further, the fouling behavior was influenced by salt concentration, salt type and organic foulant type.

Keywords: Ultrafiltration; Fouling; RO pretreatment; Organic fouling; Natural organic matter

1. Introduction

Desalination using reverse osmosis (RO) membrane is a promising technology to replace thermal-based desalination processes. Unfortunately, fouling and scaling are usually found as two main problems of this technology. While scaling can be controlled by using an antiscalant, fouling is still a big issue in application of RO. Fouling, deposition of organic matters on the membrane surface, reduces flux, increases frequency of membrane washing and cleaning, decreases membrane lifetime and lower product quality [1]. Therefore, control of fouling is a key performance-limiting factor for desalination using RO membrane.

Methods for control of fouling are dependent on the behavior and mechanism of fouling occurred. One

of the methods for control of fouling for RO membrane is pretreatment. Several pretreatment processes have been proposed including chlorination, clarification, dissolved air floatation (DAF), ozonation, coagulation-flocculation, inhibitors, ultrafiltration, microfiltration, and nanofiltration [2–6]. Among them, ultrafiltration (UF) is frequently chosen due to more consistent in product quality, smaller foot print, high removal of fine suspended solids, microbes and macromolecules [6]. With UF as a pretreatment, the performance and life time of a reverse osmosis membrane can be improved significantly [6–8]. The performance of UF membrane will finally determine the performance of a reverse osmosis membrane process. Unfortunately, problem of fouling occurs not only in RO membrane but also in UF membrane, even in some cases fouling in UF is worse than in RO. Therefore, study on fouling in UF as a pretreatment

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of RO is very important. Understanding the causes and consequences of fouling is paramount important for developing fouling control.

Many studies on UF fouling have been conducted including mechanism [9–13], characterization [14,15], and control [16–18]. However, most studies of UF fouling focused on processing of fresh water or feed containing very low concentration of salt ions. Less attention has been paid for UF fouling using high saline water such as brackish water or seawater, whereas UF as a pretreatment of RO deals with high saline water. Study on seawater pretreatment for RO has been conducted by using submerged UF and MF membranes. Nevertheless, this process was conducted for the removal of relatively large undesirable component microalgae [19]. Di Profio et al. [20] focused on the optimization of operating condition of submerged UF membrane as RO pretreatment rather than investigation of fouling behavior. They used real seawater without initial pretreatment and no source of organic carbon was added. Resosudarmo et al. [21] reported UF fouling study dedicated to pretreatment of RO membrane. They used real seawater and mixed organic foulants and therefore the responsible ions and organic compound causing dominant fouling have not been well understood. Moreover, they focused on the effects of calcium ions and backwash. This paper presents an investigation of organic fouling on UF membrane used for filtering an organic matter in high saline water. The effect of different types of salts on UF fouling was investigated by adsorption fouling and ultrafiltration fouling. Humic acid, alginate, and bovine serum albumin were used as models of natural organic matters present in seawater. These NOMs have been considered to be the main foulant present in water and wastewater [22].

2. Materials and methods

2.1. Materials

Humic acid (HA), alginic acid sodium salt from brown algae (ALG), and bovine serum albumin (BSA) as models of organic substances were purchased from Sigma-Aldrich Chemie GmbH (Steinheim, Germany), Sigma-Aldrich Co. (St Louis, USA), and Agdia Inc. (Elkhart, USA), respectively. NaCl, CaCl₂, CaSO₄, and MgCl₂ were purchased from Merck, Germany. Polyethersulfon (PES) UF membrane was from Microdyn-Nadir, Germany. The pore size of this PES UF membrane was 50 kDa and was expected to be a moderate pore size for UF. Real sea water obtained from Semarang coast, Java Sea, Indonesia was also used. The characteristic of this real seawater is presented in Table 1.

2.2. Methods

2.2.1. Adsorptive fouling experiments

The method used for adsorptive fouling study followed our previous publication [10]. Briefly, the experiments were carried out using a dead end filtration system with Amicon 8010 as a membrane cell. Adsorptive fouling was per-

Table 1
Characteristics of real sea water used

No	Component	Concentration (mg/L)
1	Total dissolved solids (TDS)	34,354
2	Cl	18,845
3	Na	10,234
4	K ⁺	365
5	Mg ²⁺	1239
6	Ca ²⁺	398
7	SO ₄ ²⁺	2638

formed by contacting the membrane surface with a feed solution containing salt and a model of organic substances. A certain concentration of organic substance (either BSA, ALG or HA) with concentration of 1 g/L was dissolved in a salt solution. It should be noted that the adsorption fouling is influenced by foulant concentration, thus relatively high concentration of NOM models was used in this experiment. The salt solution used to dissolve NOM included NaCl (5000 and 30000 mg/L as model for brackish and sea water), CaCl₂ (500 mg/L), CaSO₄ (500 mg/L) and MgCl₂ (1000 mg/L). The concentrations of NaCl, CaCl₂, CaSO₄ and MgCl₂ were determined by considering their concentration in sea water and/or brackish water.

First, the membrane was compacted by filtering pure water at a pressure of 450 kPa for 0.5 h. The pressure of 450 kPa was selected by considering the operation condition and the capability of the equipment used. The pressure was then lowered to the desired pressure and pure water flux was measured (J_o). The filtration was stopped and an organic substance solution was added to the cell and the outer membrane surface was exposed to that organic substance solution for 3 h. The organic substance solution was removed and the membrane was rinsed. The pure water flux was again measured (J_o). The adsorptive fouling was expressed in term of relative flux reduction (RFR) according to:

$$RFR(\%) = \frac{J_o - J_a}{J_o} \times 100 \quad (1)$$

2.2.2. Ultrafiltration experiments

The experiments were performed by using a cross flow filtration mode (Fig. 1), which is similar with our previous study [15]. 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 450 kPa. An organic solution model was added to the feed tank and was pumped into membrane cell. The concentration of organic foulant was 25 mg/L, which was dissolved in different salt solutions (NaCl: 5000 and 30000 mg/L, CaCl₂: 500 mg/L, CaSO₄: 500 mg/L and MgCl₂: 1000 mg/L). The selection of organic model concentration was expected approaching their concentration in sea water. In order to maintain constant feed concentration, the volume of feed was designed much larger than the volume taken as sample for the analysis. In addition, the retentate and permeate were returned to the feed tank. All experiments were performed at room

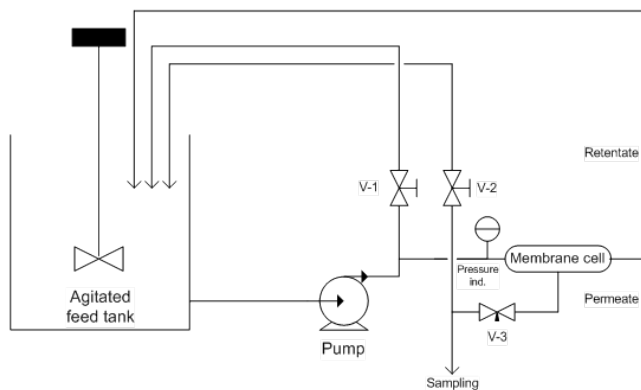


Fig. 1. Schematic of ultrafiltration experiment set up.

temperature ($28 \pm 2^\circ\text{C}$) and at a constant trans-membrane pressure (100 kPa). The flux profile over time was gravimetrically measured.

Besides the flux profile, the solute rejection was also observed and was determined using Eq. (2). The feed concentrations at the beginning and at the end of the experiment were averaged to obtain representative values of concentration in the feed side.

$$R (\%) = \left(1 - \frac{C_p}{C_f} \right) \times 100 \quad (2)$$

where C_p and C_f are the concentration of solute in permeate stream and in feed sides of the membrane, respectively.

The concentrations of HA and BSA feed were analyzed using a UV spectrophotometer (Shimadzu UV mini 1240 UV-vis Spectrophotometer). HA and BSA concentrations were determined by measuring UV absorbance at 255 nm and 280 nm, respectively. Analysis of alginate concentration followed the colorimetric method developed by Dubois using phenol-sulfuric acid method (cf. [23]). The samples were firstly reacted with phenol in acid medium forming an orange-yellow color. Their absorbances were then measured at 480 nm using UV-spectroscopy.

3. Results and discussion

3.1. Adsorptive fouling

The effect of monovalent ion on adsorptive fouling was studied by using sodium chloride. Alginate (ALG), humic acid (HA) or bovine serum albumin (BSA) with concentration of 1 g/L was dissolved in different concentration of NaCl solution. The extent of adsorptive fouling was expressed in term of relative flux reduction (RFR). The results are presented in Fig. 2.

The presence of NaCl increased RFR for both ALG and HA organic foulants indicating higher adsorptive fouling than in the absence of NaCl. The presence of 30000 mg/L NaCl in alginate solution increased RFR from 14 to 23%. Nevertheless, when the NaCl concentration was lowered to 5000 mg/L the resulting RFR was similar with RFR of NaCl-free condition. The use of NaCl solution 30,000 mg/L and 5000 mg/L as the back-

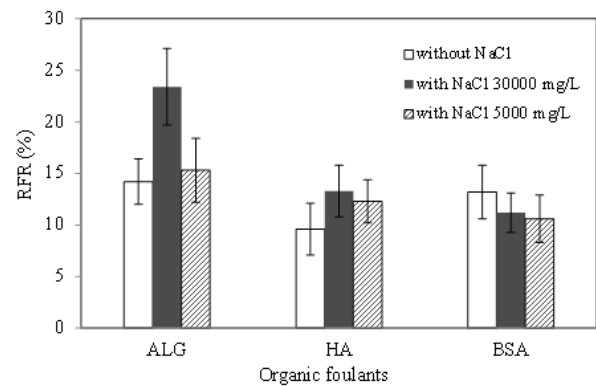


Fig. 2. RFR after static adsorption (3 h) of various organic foulant solutions. The concentration of organic foulant in NaCl solution was 1 g/L.

ground solutions of HA increased RFR from 9.6% to 13% and 12%, respectively.

As the ionic strength was increased the RFR increased indicating more adsorptive fouling occurred. The presence of monovalent ions increased ionic strength and then decreased the electrostatic repulsion between negatively charge of ALG molecules and membrane leading to higher adsorption on the membrane surface. It is important to mention that our zeta potential measurement at pH 10 to pH 4 showed that the zeta potential of membrane was -28.3 – -13.6 mV (the zeta potential 0 mV was observed at pH ~ 4.7). The explanation for alginate adsorption on membrane surface seems to be valid also for HA. The presence of monovalent ions increased ionic strength and would decrease the electrostatic repulsion between negatively charge of HA molecules and membrane resulting in HA deposit on membrane surface. The adsorption of humic acid was more significant in the presence of salt ion. Liu et al. reported that the charge of HA changed from -43.9 to -23.7 mV and from -31.5 to -20.3 mV by increasing ionic strength (by addition of NaCl) from 0 to 40 mM and calcium ions from 0 to 2.0 mM, respectively [11]. In general, the results obtained in this study are in agreement with the results reported in previous publications [18,21].

Surprisingly, the addition of NaCl slightly decreased the adsorptive fouling by BSA (from 13 to 11%). No difference in RFR was observed when NaCl was increased from 5000 to 30000 mg/L. The possible reason behind this result would be increasing solubility of BSA or conformation changes of BSA at high ionic strength. It is important to note that in this experiment very high ionic strength was applied. Such observation was also found in previous publication by She et al. [24].

Beside monovalent ion, the effect of divalent ions on adsorptive fouling was also investigated. CaSO_4 , CaCl_2 , and MgCl_2 were added into the feed containing organic foulants. The results are presented in Fig. 3.

As also observed in monovalent ion, the presence of divalent ions increased RFR by both ALG and HA but slightly decreased RFR by BSA. ALG and HA showed similar behavior, where CaCl_2 demonstrated the highest impact on RFR followed by CaSO_4 and MgCl_2 , respectively. The ALG, HA and BSA should have negative charges in water.

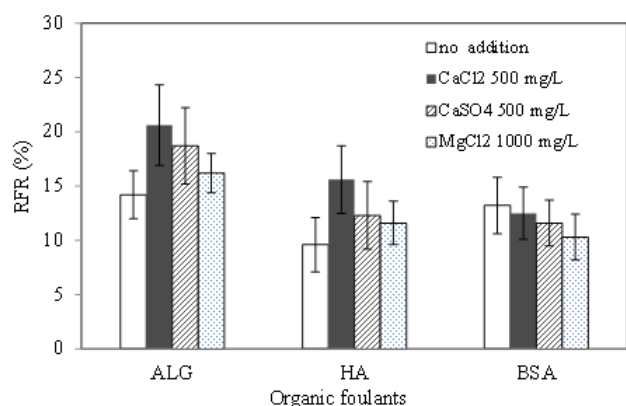


Fig. 3. RFR after static adsorption (3 h) of various organic foulant solutions. The concentration of organic foulant was 1 g/L.

This means they should have electrostatic repulsion with the membrane. Nevertheless, the presence of divalent ions promoted the formation of bridge among foulant molecules (here ALG and HA) leading to agglomeration of organic foulant on the membrane surface.

Regardless the block structure of alginate (the ratio of its constituent monomer), the presence of calcium and magnesium ions causes the formation of cross-linked structure supporting aggregation. Each calcium or magnesium ion can attach two of the polymer strands. The binding of metal ion (calcium or magnesium) with carboxylic acid group of HA forms HA-metal complexes. Fig. 3 suggests that the strength to form foulant aggregate was larger for calcium than for magnesium. Furthermore, besides metal ions, the anion (see CaCl₂ and CaSO₄) also influenced the extent of adsorptive fouling. She et al. reported that anion played an important role in fouling behavior [25].

Different with ALG and HA, BSA does not have significant amount of carboxyl group and therefore the formation complexes between BSA and metal ions would not be formed. The reason used for monovalent can also be used to explain the slight decrease in RFR by addition of divalent ions into the feed containing BSA. In addition, the presence of anion (Cl⁻ and SO₄²⁻) might increase the negative charge of the membrane surface leading to higher electrostatic repulsion.

3.2. Ultrafiltration fouling

UF fouling behavior was investigated by ultrafiltration of ALG, HA and BSA solutions (25 mg/L in different salt solutions). The experiments were performed with constant trans-membrane pressure (100 kPa). The results are expressed in term of flux (normalized flux) as a function of filtration time as shown in Figs. 4–6.

Different flux behavior for different organic foulant solution was observed during ultrafiltration experiments. This means different fouling behavior has occurred. Rapid flux decline at the early stage of filtration followed by relatively constant permeate flux after 1 h filtration was shown by ultrafiltration of alginate solution. At the beginning of filtration or short period of filtration, concentration polarization played a major role for the flux decline besides foul-

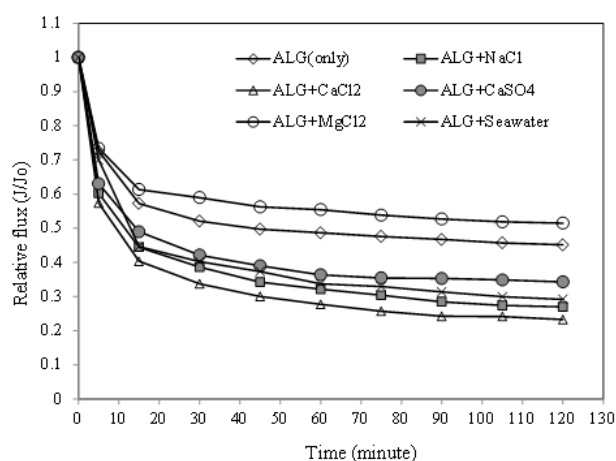


Fig. 4. Flux behavior during ultrafiltration of ALG solution (25 mg of ALG was dissolved in 1 L of different salt solutions). The concentrations of NaCl, CaCl₂, CaSO₄ and MgCl₂ were 30,000 mg/L, 500 mg/L, 500 mg/L and 1000 mg/L, respectively.

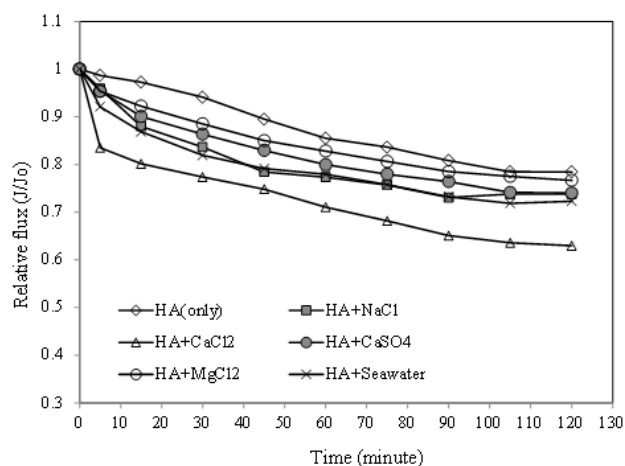


Fig. 5. Flux behavior during ultrafiltration of HA solution (25 mg of HA was dissolved in 1 L of different salt solutions). The concentrations of NaCl, CaCl₂, CaSO₄ and MgCl₂ were 30,000 mg/L, 500 mg/L, 500 mg/L and 1000 mg/L, respectively.

ing. Fouling by pore blocking is believed to be the dominant reason for the rapid flux decline followed by relatively constant permeate flux. The adsorption fouling should not be the reason for the flux decline at the early stage of filtration because it requires a certain time to occur [14,15]. Thereafter, cake formation caused by adsorption and foulant deposition should take place. Because relatively constant permeate flux was observed, porous cake layers should be formed.

Ultrafiltration of HA solution showed moderate flux decline at the early stage of filtration followed by a gradual decrease in permeate flux. Besides concentration polarization, fouling by pore constriction should be the reason for this moderate decline at the beginning of filtration. Thereafter, cake formation by adsorption and foulant deposition on the membrane surface was taken

place. In this case the formation of cake layers affected the permeate flux as indicated by their gradual decreases. The fluxes behavior at ultrafiltration using NaCl and sea water as the background solution is similar with our previous publication [26].

For the case of ultrafiltration of BSA solution, the permeate flux behavior showed the characteristic in between the permeate fluxes of ultrafiltration of ALG and HA solutions. Thus the fouling mechanism should be the combination of pore blocking and pore narrowing followed by cake layer formation.

Overall, the presence of ions increased the extent of fouling except $MgCl_2$ in ultrafiltration of ALG and BSA solutions as well as $CaSO_4$ in ultrafiltration of BSA solution. Similar results were observed by Resosudarmo et al. [21] where the presence of Red Sea salt (33 g/L) increased membrane fouling rate by HA and alginate. Even though the effect of calcium ions on organic fouling (HA and alginate), i.e. whether increase or decrease, are under debate by many authors in reported publications (e.g., [15–17,27]), this study confirmed that the calcium ions increased the extent of membrane fouling except for ultrafiltration of BSA with

addition of $CaSO_4$. The presence of SO_4 as divalent anion should contribute to this behavior.

The high concentration of salt ions in the feed solution would reduce electrostatic repulsion between organic foulant and membrane surface. Consequently, the extent of fouling increased. Divalent ions promote the formation of bridge among alginate molecules resulting a rigid lattice alginate deposit on membrane surface. This result is similar with previous publication by Jermann et al. [28]. It was reported by Meng et al. [29] that in the absence of calcium ion, alginate had filament structure whereas much bigger and more compact structure was formed in the presence of calcium ions. Further, the zeta potential of alginate tended to decrease after the addition of calcium ions forming less stable structure and provoking aggregate formation [29]. These conditions increased the formation of fouling layer. These explanations are supported by rejection data, where the highest rejection of ALG and HA was achieved when $CaCl_2$ was added (Table 2). The reason behind the increase in permeate flux during ultrafiltration of BSA solution after the addition of $CaSO_4$ and $MgCl_2$ on the flux behavior has not been understood until now.

Unlike in adsorption experiments, the addition of salt ions (NaCl and $CaCl_2$) increased the extent of fouling by BSA. The presence of ions in the feed containing protein decreased the flux because screening of the charges of the protein increased. This condition would decrease the electrostatic repulsion leading to more serious membrane fouling.

Comparing the flux behavior, it was observed that the flux decline caused by ALG was greater than caused by HA and BSA. This behavior indicates that under high salinity, ALG caused more severe fouling than HA and BSA. Ultrafiltration experiments showed that the fouling behavior shown by organic solution with addition of NaCl (30,000 mg/L) was the most similar with the fouling behavior shown by organic macromolecules dissolved in real seawater.

Irrespective the lower concentration of foulant model in feed stream, the ultrafiltration fouling was more significant than adsorptive fouling as indicated by their higher RFR for all foulant models at all conditions (see Table 2). The foulant model, which showed high RFR after adsorptive

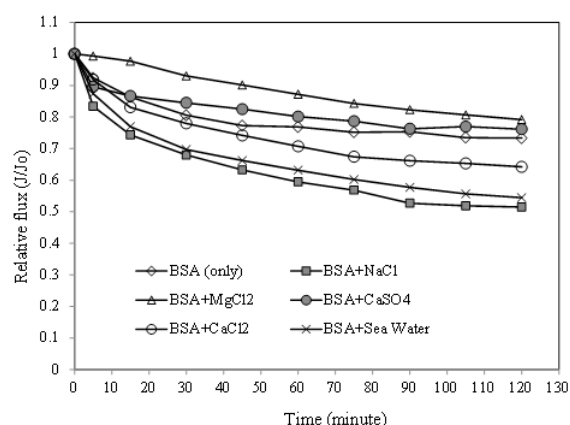


Fig. 6. Flux behavior during ultrafiltration of BSA solution (25 mg of BSA was dissolved in 1 L of different salt solutions). The concentrations of NaCl, $CaCl_2$, $CaSO_4$ and $MgCl_2$ were 30,000 mg/L, 500 mg/L, 500 mg/L and 1000 mg/L, respectively.

Table 2
Flux reduction and rejection of different organic substances at different feed conditions

Solution background	Alginate			Humic acid			BSA		
	RFRads ¹ (%)	RFRuf ² (%)	Rejection (%)	RFRads (%)	RFRuf (%)	Rejection (%)	RFRads (%)	RFRuf (%)	Rejection (%)
Fresh water	14.2	54.9	78.3	9.6	21.6	59.7	13.2	26.7	87.2
NaCl 30000 mg/L	23.4	73.1	83.2	13.3	26.2	63.1	11.2	48.5	85.3
$CaCl_2$ 500 mg/L	20.6	76.8	90.8	15.6	37.1	74.5	12.5	35.8	86.1
$CaSO_4$ 500 mg/L	18.7	65.8	88.8	12.3	26.1	71.4	11.6	23.9	84.5
$MgCl_2$ 1000 mg/L	16.2	48.5	85.3	11.6	23.3	69.3	10.3	20.9	85.5
Seawater	22.8	70.8	83.9	14.1	27.7	65.5	11.9	45.6	86.7

Note: ¹RFRads: relative flux reduction after adsorptive fouling
²RFRuf: relative flux reduction during ultrafiltration fouling

fouling, it also showed high RFR after ultrafiltration. These phenomena suggest that besides membrane-solute-solute interactions, ultrafiltration fouling was influenced by membrane-solute interactions (adsorptive fouling). Furthermore adsorptive fouling is very useful for the evaluation of the potential fouling by certain foulants.

4. Conclusion

Study on adsorptive and ultrafiltration fouling using alginate, humic acid and bovine serum albumin dissolved in high saline water as the feed has been conducted. It is important to note that these three foulants used in this study are the potential foulants that are usually found in surface water including sea water. The experimental results showed that both adsorptive and UF fouling were clearly observed. The high saline water even increased the extent of both adsorptive and ultrafiltration fouling (ALG and HA). Consequently, care should be taken for the control of UF fouling during application of UF for pretreatment of RO membrane.

A good correlation between adsorptive and ultrafiltration fouling was observed, where foulant model that showed high RFR after adsorptive fouling, it also showed high RFR after ultrafiltration. Furthermore, adsorptive fouling is very useful for the evaluation of the potential fouling in ultrafiltration for the pretreatment of RO application. Ultrafiltration experiments showed that the fouling behavior exhibited by an organic solution with addition of 30000 mg/L NaCl was the most similar with the fouling behavior demonstrated by organic substance dissolved in real seawater.

The results of fouling mechanism study suggest that even though control of fouling by fluid management or hydrodynamic method is believed to be one of the methods that should be applied for controlling of fouling but the availability of low fouling membrane is also important (note that adsorptive fouling was significant).

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