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Effect of forward osmosis (membrane) support layer fouling by organic matter in synthetic seawater solution

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ABSTRACT

Recently, the forward osmosis (FO) process has received considerable attention because it has lower fouling characteristics compared to the generally high-pressure process, reverse osmosis. Most studies concerning the FO process focus on the active layer, the upper side of the FO membrane. However, a study reporting the support layer has a negative influence on active layer fouling was insignificant. Therefore, our study investigates the effects of organic matter, such as humic acid (HA) and sodium alginate (SA) in draw solution, in conjunction with synthetic seawater containing divalent ions, like Ca^{2+} and Mg^{2+} on the support layer. Furthermore, we investigate the effects of the supplemented synthetic seawater on support layer fouling when the spacer is present on the draw solution side, contacting the support layer membrane surface. This assemblage mitigates the internal concentration polarization increasing an effective osmotic pressure. Using 500 ppm of organic matter in the draw solution, such as HA and SA, results in an increased water flux reduction rate, 43–45%, respectively, and further increased water flux reduction rate by 49.5 and 48%, respectively, with the spacer. The water flux reduction rate using HA is increased without a spacer compared to SA; however, in the presence of the spacer, the results are the reverse. As a result, excessively high concentrations of organic matter (500 ppm) influence membrane fouling. Placing a spacer in contact with the support layer and organic matter accelerates membrane surface fouling.

Keywords: Organic matter; Synthetic seawater; Support layer; Spacer; Fouling

1. Introduction

Since the 1960s, seawater and brackish water have been used to produce fresh water using membrane technology to overcome water shortage. In the past, the conventional high-pressure membrane process such as microfiltration, ultrafiltration (UF), nanofiltration, and reverse osmosis (RO) are used in desalination, wastewater treatment, and water purification [1–3]. Among the membranes, RO technology is widely used as one of the main processes in the membrane field. However, these membranes require hydraulic pressure as the

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driving force to transmit water through the membrane, which consumes a great deal of electrical energy [4]. Therefore, alternative solutions are needed to solve this energy problem [5].

Forward osmosis (FO) has high recovery rate, low energy consumption and weak membrane fouling characteristics compared to other membranes [6,7]. FO systems use natural osmotic pressure as the driving force to push water through the membrane [5,8]. In this system, the feed solution and draw solution are prepared to generate the proper amount of osmotic pressure. For example, a low concentration of deionized (DI) water or wastewater is selected as a feed solution and high concentration seawater or brackish water is usually used as draw solution. The two solutions with different concentrations form an osmotic pressure difference. Therefore, the draw solution is diluted by permeable water from the feed solution. Consequently, low hydraulic pressure is applied to this process compared to the RO system [9]. Because of these benefits, FO system applications have been applied in various fields, such as wastewater treatment [10,11], brackish water desalination [12], seawater desalination [13–15], power generation [16–18], food manufacturing [19,20], and fertilizer use [21–23].

The FO membrane is composed of active and support layers, which are necessary to support the active layer [24]. FO research is mainly focused on active membrane fouling and studies of the support layer are few, especially in regard to support layer fouling.

There are many kinds of contaminants in seawater, such as inorganic and organic materials. In case of the organic matters, humic acid (HA) is a major organic component of the seawater [25]. Similarly, another organic material, alginate, is detected when algal blooms release extracellular polymer substances into the seawater [26]. HA and alginate are considered important irreversible organic matter. Furthermore, when they are present at high ionic strengths, as in seawater, the fouling potential is increased [27]. Studies show calcium ions, which are abundantly present in seawater, are well known to generate compounds with carboxylic functional groups in organic matter and cause accelerated membrane fouling [4].

When we operate a membrane process that has an asymmetric structure, concentration polarization (CP) is unavoidable, especially the internal concentration polarization (ICP) that leads to reduced water flux. Thus, in previous studies, spacers were used to alleviate the CP phenomenon [28]. Although spacers cause flux recovery by easing the CP phenomenon, these results are limited only in the absence of foulants.

Thus, the object of this study is to evaluate the effects of synthetic seawater as a draw solution containing divalent ions, as well as HA and sodium alginate (SA), on support layer fouling. In addition, we studied whether the presence of the spacer located on the membrane surface increases the effective osmotic pressure, relieving the ICP, in the presence of organic matter. Because we wanted to ensure an effect, the results obtained in this study are measured with excessively high concentrations of organic materials in comparison with the real concentrations in the seawater.

2. Materials and methods

2.1. Forward osmosis (FO) membrane and spacer

Porifera manufactures the FO membrane used in this study. The chemical materials of the membrane are proprietary, however, we speculate that the flat sheet membrane is composed of a polyamide active layer and polysulfone support layer. Membrane size was adjusted to $77 \times 26\ \text{mm}^2$ (membrane effective area: $20.02\ \text{cm}^2$). Porifera also provided the spacer used in this study with the membrane, which is located in draw solution side in contact with the membrane support layer. The spacer was used to investigate effects of organic matter. The spacer size is the same as that of the membrane.

2.2. Feed and draw solution

Sodium chloride (NaCl, 99.0%, Samchun) was dissolved into DI water to obtain a 0.3-M NaCl feed solution. 0.3 M NaCl was used to simulate the water flux reduction by fouling during the experiment. Synthetic seawater was prepared as the draw solution during the experiments. The synthetic seawater stock solutions listed in Table 1 are based on the American Society for Testing and Materials (ASTM, D1141–98) and were stored in separate bottles at 4℃.

HA (HA, Sigma-aldrich) and SA (SA, Samchun) were used as model organic materials. The organic fouling from these organic materials is widely distributed over various components in water environment [1]. The stock solutions for the contaminants were prepared by dissolving them in DI water (10 g/L), separately. After making the stock solutions, the draw solution for the fouling experiments was prepared by mixing synthetic seawater stock solutions and foulants according to their concentrations, to make 100–500 mg/L of HA and SA, respectively.

2.3. Experimental setup

The membrane experiments were conducted in lab scale. The FO system consisted of two pumps (Longer

Table 1
The composition of the synthetic seawater

	Constituent	M.W (g/mole)	Dimension (g/L)
Stock 1	$MgCl_2 \cdot 6H_2O$	95.21	555.57
	CaCl2 (anhydrous)	110.98	57.94
	$SrCl_2 \cdot 6H_2O$	158.53	2.11
Stock 2	KCl	74.55	69.46
	$NaHCO_3$	47.01	20.10
	KBr	119.00	10.06
	H_3BO_3	61.83	2.71
	NaF	41.99	0.30

Pump WT3000–1FA), mass balance (AND GF-6000), chiller (CPT Inc.), and magnetic stirrer (MISUNG SCIENTIFIC CO., LTD MS-300HS). The concrete structure of the FO system is shown in Fig. 1. The cross-flow velocity was fixed at 8.5 cm/s using circulating pumps for both the feed and draw solutions. To measure the weight change of the draw solution, we used a mass balance to permeate the water flux with a computer program recording system every minute. All of the experiments were maintained at 25˚C using a chiller. A magnetic stirrer was used to mix the feed solution into a homogeneous solution.

The organic material draw solutions concentrations are provided in Table 2. The 0.3 M NaCl feed solution (2L) and a solution prepared by mixing artificial seawater and organic matter were used as a draw solution (1L) separately. To stabilize temperature, solutions circulated in tanks for one hour without FO cell connection, including the membrane and spacer. After this equilibration, we connected the FO cell. In addition, we did not adjust the pH, because the draw solution has high ionic strength similar to seawater [1]. Baseline experiments followed the procedure described above, except that no foulants were added to the draw solution. To determine the average water flux reduction rate, all experiments were repeated twice.

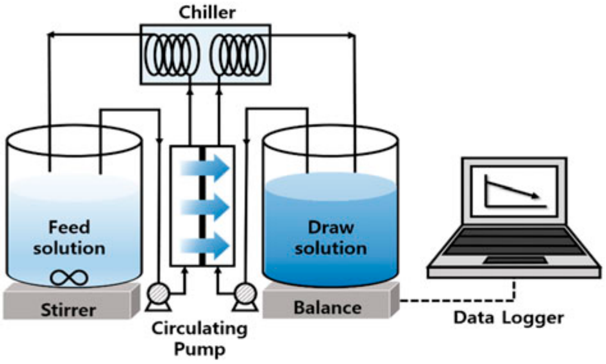

Fig. 1. Schematic diagram of the FO system.

2.4. Analytical methods

To identify the membrane surface characteristics, Field Emission Scanning Electron Microscopy/Energy Dispersive X-ray Spectrometer (FESEM/EDS, JMS-7600F, JEOL) analysis was performed. The EDS is installed with the FESEM to confirm which elements are present on the membrane. The voltage controlled for analysis has 15 kV (resolution: 1.0 nm), offering high spatial resolution. For the analysis, normal magnification was set to 100, 500, and 1,000 times and the working distance average was 8.4 nm. The membrane was placed at 25˚C in a 105-rpm shaking incubator for 24 h to extract the organic materials using 0.1 N Sodium Hydroxide (NaOH, DAEJUNG). After this process, a total organic carbon (TOC) analyzer (TOC-L, Shimadzu) measured dissolved organic carbon (DOC) to assess the amounts of HA and SA. Before measurement, the extraction solution was filtered by a 0.45 μm filter (NORM-JECT Syringe, Whatman™). During the lab-scale experiments, the permeability of the water flux value of the draw solution was recorded in a computer program (Win CT, AND). The water flux was calculated by the equation below [28]:

$$\text{Water flux (LMH)} = \frac{\Delta V}{A \Delta t} \tag{1}$$

where ΔV is the volume change of draw solution in the time interval Δt. A is the effective membrane area of 20.02 cm^2. The flux reduction rate is the evaluation index for these experiments.

Normalized water flux was calculated by the equation below:

$$\text{Normalized water flux (LMH)} = \frac{\text{Current flux}}{\text{Initial flux}} \tag{2}$$

Finally, the reduction rate was calculated by the equation below:

Table 2
Draw solution experimental conditions (Feed solution conditions: 0.3 M NaCl)

	Foulants	Spacer	Temperature	Operating time (h)
Concentrations	HA 100 ppm HA 500 ppm SA 100 ppm SA 500 ppm HA 500 ppm + SA 500 ppm	With/Without With/Without With/Without With/Without With/Without	25˚C	17

$$\text{Reduction rate of the water flux } (\%) = \frac{\text{the initial flux } - \text{the final flux}}{\text{the initial flux}} \times 100\% \qquad (3)$$

3. Results and discussion

3.1. Flux effects of foulants

Normalized water flux values, which were recorded every 60 min with 100 ppm concentrations of different organic materials in synthetic seawater are shown in Fig. 2. In the baseline experiments with the spacer, the water flux was expected to be enhanced due to promoting friction force in the boundary and decreased flow velocity generating momentum causing thickness loss. As a result, draw solutes accumulate between the membrane and spacer. Therefore, the draw solution concentration improves with an increased osmotic pressure difference [28].

We assume the solute at baseline does not hinder the water flux. Therefore, similar results of slightly alleviated water flux values compared to that in absence of the spacer were obtained in a previous study [28]. Also, in the case of organic matter in the feed solution, water flux drops, whether the spacer is under the same background condition or not [29]. Thus, we conducted experiments under the same conditions, but changing the feed solution to draw the organic matter solution, whether the results were similar or not. Therefore, when using a low foulant concentration in the draw solution, the water flux reduction rate was similar for both baseline and organic matter experiments.

In the current study, the results of a 20 ppm HA solution, the general concentration of HA in natural seawater [30] remain unreported, since there was little difference in the results from 100 ppm HA (5-fold higher concentration than the HA content in natural seawater). Therefore, we conducted experiments with a 500 ppm organic matter concentration, a 5-fold higher concentration than 100 ppm. Additionally, we conducted experimental observations without or with the spacer under the same conditions.

3.1.1. Effects of HA

To study the effects of HA in synthetic seawater, fouling experiments were performed with a

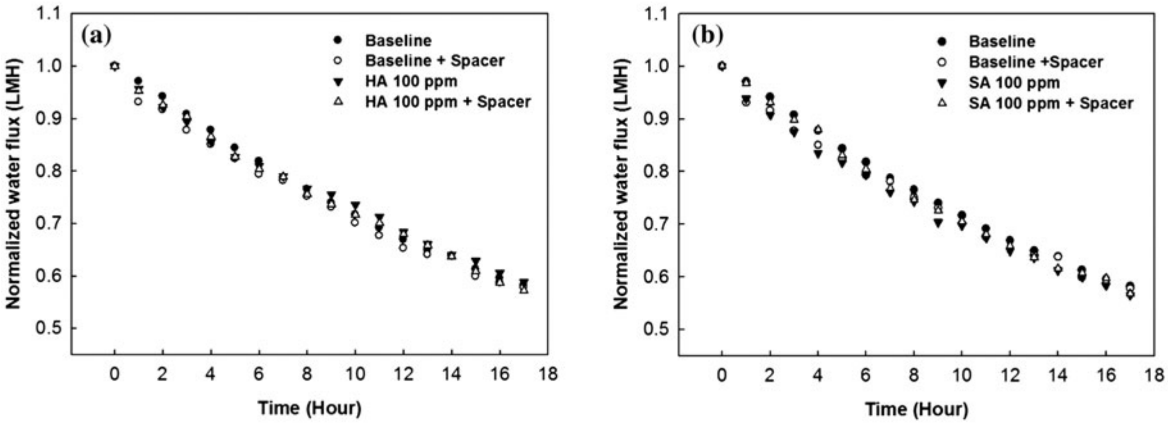

Fig. 2. The water flux decline curve for organic matter. Average values were taken to represent support layer fouling: (a) HA and (b) SA.

100–500 ppm HA draw solution, with or without spacer. Fig. 3 shows the normalized water flux reduction rate for HA and SA at different concentrations. As shown in Fig. 3(a), the difference between water flux reduction rates of HA at 500 ppm (43%) and 100 ppm (41.5%) was not noticeable. The reduction rate curve demonstrates that HA alone is not an effective factor in water flux reduction. This result is different from previous reports, in that divalent ions, like Ca^{2+}, in the feed solution interacts with HA, increasing adsorption; which is the interaction between membrane and accumulated materials due to reduced electrostatic repulsion [31]. However, we used the HA in a salt-rich draw solution and there is no strong driving force for organic matter to adhere to the support layer, which is the side that is diluted from the permeate water of the feed solution. As a result, HA in synthetic seawater decreases water flux, albeit not significantly.

To determine the effectiveness of the spacer when synthetic seawater with HA was used, we applied the spacer in contact with the support layer. Fig. 3(b) shows the effect of the spacer using HA in synthetic seawater. Compared to Fig. 3(a), as the concentration of HA increases, the water flux reduction rate increases. In case of the baseline spacer, a 42% water flux reduction rate occurred, while HA at 500 ppm with a spacer reduced water flux reduction rate by 49.5% (Fig. 3(b)).

In this study, solutes like HA in the support layer with the spacer, prevent water flux. HA, combined with divalent ions on the membrane surface and spacer, acts as an obstacle, and does not allow water to permeate from the feed to draw side. This means, under the spacer, HA accumulates between the membrane and spacer. Thus, in HA at 500 ppm with the spacer, HA disturbs the water flux more than HA at 100 ppm with the spacer. According to these results, we conclude that HA may affect the membrane surface when the spacer is present and concentration is 500 ppm on draw solution side of synthetic seawater.

3.1.2. Effects of SA

To verify the effect of SA during the fouling experiments, water flux reduction was measured as shown in Fig. 4. Similar to results shown above (Section 3.1.1), the water flux reduction rate was not remarkable. However, in case of the HA 500 ppm experiments, the water flux reduction rate increased by 1.5% (Fig. 3(a)). In contrast, the water flux reduction rate increased by 2.5% in SA 500 ppm experiments compared to baseline (Fig. 4(a)). In addition, the overall water flux reduction rate was higher when using the SA in draw solution. These results indicate that the SA, among organic foulants in synthetic seawater, has a negative effect on water flux reduction.

It was reported that in a forming gel layer, the average adhesion force (ASF) of SA in the presence of calcium ions is twice as strong as in the absence of the calcium ions. The ASF of HA is similar without calcium ions [4]. Our results demonstrate that formation of a cross-linked alginate gel layer on the support layer of the membrane surface in the presence of calcium ions causes stronger adhesion force than the reduced electrostatic repulsion by HA on the support layer surface.

On the other hand, when we use the spacer under SA 500 ppm conditions, the water flux reduction rate increased by 6% compared to that of the baseline with a spacer (Fig. 4(b)). This value showed less water flux reduction rate increase than HA, 7.5% (Fig. 3(b)).

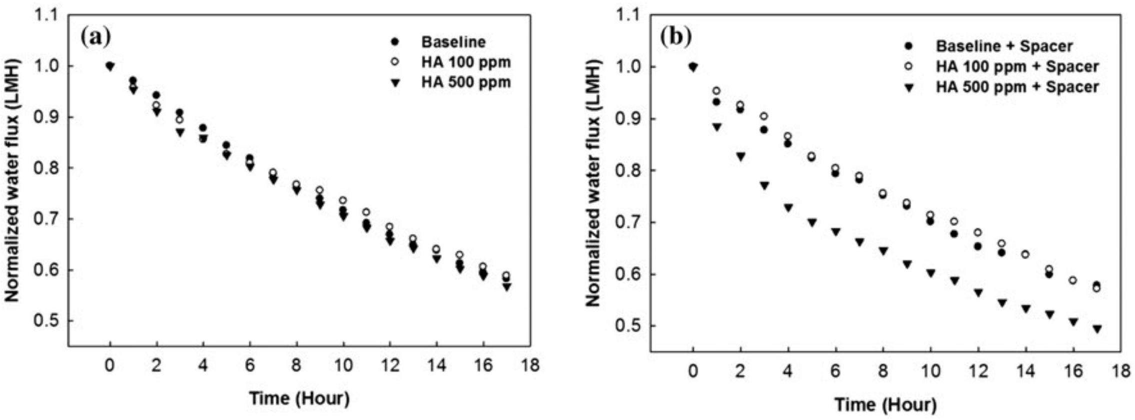

Fig. 3. Water flux decline curve for HA. Values were averaged to represent support layer fouling: (a) Without spacer and (b) With spacer.

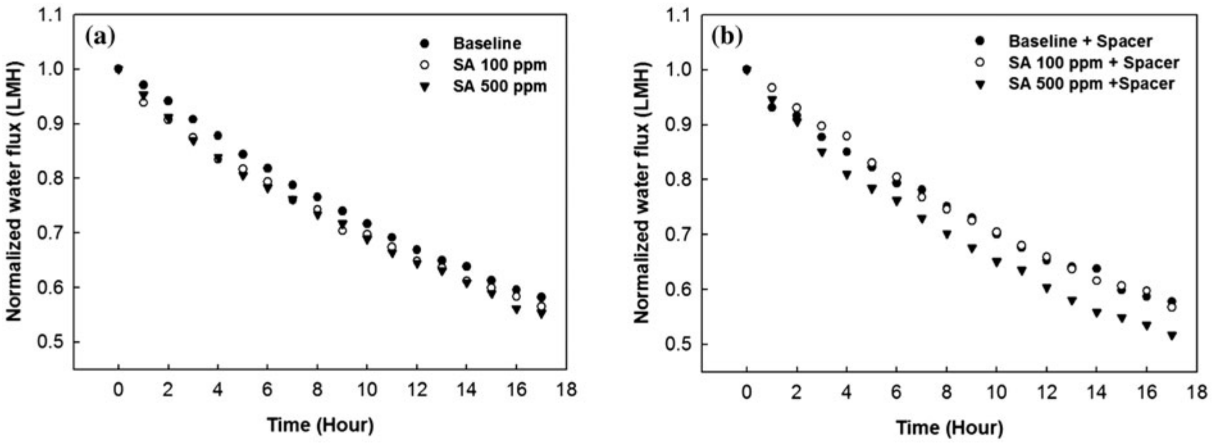

Fig. 4. Water flux decline curve for SA. Values were averaged to represent support layer fouling: (a) Without spacer and (b) With spacer.

Divalent ions like Ca^{2+} or Mg^{2+} play the significant role of a bridge with SA combining gel, which is deposited on the membrane surface [27]. Here, we infer that fouling in the SA experiments using a spacer is relatively reversible compared to the experiments with HA.

Membrane fouling by HA is caused by the adsorption inside the membrane pore, whereas SA fouling is due to internal pore constriction by the cake layer on the membrane surface [32]. Because HA in the presence of calcium tends to be more compacted by intermolecular electrostatic repulsion, HA caused fouling that is relatively more irreversible than that from SA [32,33]. Since the spacer generates turbulent flow on the membrane surface, fouling by SA is washed out compared to HA.

As discussed above, high concentrations, like 500 ppm of organic matter in synthetic seawater draw solution, affects the reduced permeate water flux. Specifically, SA's effect on the water flux reduction rate is more remarkable than that of HA alone. Moreover, in the FO process, spacers in contact with the support layer makes the water flux reduction decrease with foulants.

3.1.3. Effects of mixing solution

In these experiments, the mixed solution effect of HA 500 ppm and SA 500 ppm was studied. As shown in Fig. 5(a), the water flux reduction rate increased in the sequence of HA 500 ppm + SA 500 ppm (48.5%) > SA 500 ppm (45%) > HA 500 ppm (42.5%). A previous study of fouling behavior with a certain mixture in the UF membrane system shows that the aspect of water flux with mixing, is closer to that with polysaccharides alone. Polysaccharides are a component of SA [32]. Likewise, results provided in this study show that the water flux reduction rate of the mixing solution is closer to that of SA. As mentioned earlier, the fouling potential of SA is greater than that of HA without spacer. Therefore, our result supports the crucial negative effect of SA on water flux.

Fig. 5(b) is the same experiment as in Fig. 4(b) with the addition of a spacer. When the spacer was located on the draw solution side in contact with support layer, the water flux reduction rate increased in the sequence: HA 500 ppm + SA 500 ppm + spacer (52%) > HA 500 ppm + spacer (49.5%) > SA 500 ppm + spacer (48%). This result implies that the mixed solution has a higher water flux reduction rate than when only one contaminant is present.

Interestingly, HA results stood out in this experiment. They showed similar trends along the reduction curve of the mixed foulants. However, it was reported that fouling from HA is irreversible compared to SA, when divalent ions such as Ca^{2+} and Mg^{2+} are present [32]. In addition, a spacer increases momentum-loss thickness by reducing flow velocity and accumulates the solute on the membrane surface [28]. These data indicate that, with spacer, fouling from HA tends to predominate over SA in a mixed solution. Thus, both the mixed solution with spacer and HA solution have similar curves in this experiment. Therefore, we can once more verify the results in Sections 3.1.1 and 3.1.2.

3.1.4. Effects of spacer

As mentioned above, we observed the following results: the water flux reduction rate by SA was higher without spacer on the draw solution side, however,

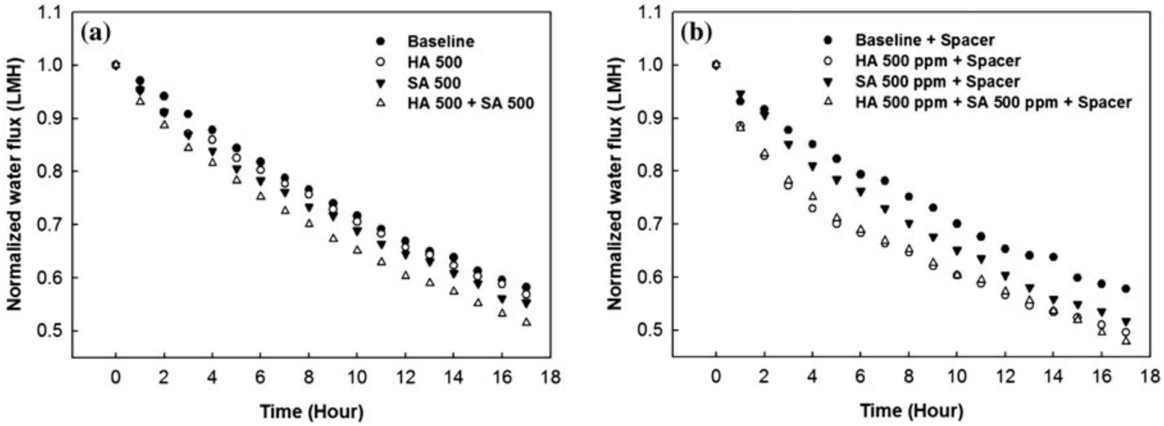

Fig. 5. The water flux decline curve for mixed solution. Values were averaged to represent support layer fouling. (a) Without spacer and (b) With spacer.

that of HA was higher with a spacer. Although the overall flux reduction rate appears different depending on the foulant, generally, a spacer yields a remarkable water flux difference (Fig. 6).

3.2. Analysis of DOC

Organic fouling on the membrane can be determined by DOC analysis by extraction using 0.1 N NaOH solution. The membrane was rinsed with DI water to eliminate unwanted materials deposited on the membrane surface before immersing into the NaOH solution. We defined the DOC values, which are stored in the membrane as foulant materials for these experiments.

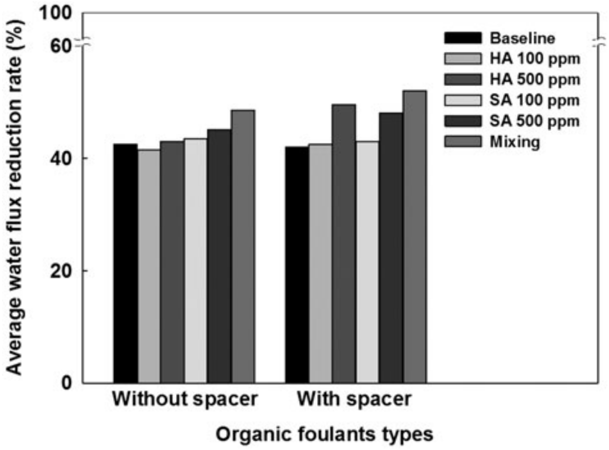

Fig. 6. Average water flux reduction rate comparison (%) without or with spacer.

The results show that foulants were not detected regardless of concentration change, spacer, foulant type, and mixing solution. We found that organic matter in the draw solution accumulates between membrane surface and spacer. It is implied that the driving force, which moves in the draw solution direction, disturbs the tendency of organic matter to adhere to the membrane side. Thus, organic matter is detected only on the membrane surface without affecting the inside of the membrane.

3.3. Analysis of SEM/EDS

Through SEM images, we confirmed that fouling matter covered the membrane surface (Fig. 7). Fig. 8 shows a 500× magnified image of a virgin membrane. The black part describes pores on support layer, pointed out in Fig. 9. On the other hand, as shown in Fig. 7, images of a fouled membrane by organic matter shows that they represent the gray area on the membrane, which proves there was accumulation of organic matter on the membrane. The brighter part is due to salt, because we omitted the washing procedure to identify the original fouled membrane surface after the experiments. As a result, the organic matter on the support layer is easily washed away by rinsing with DI water.

In addition, the EDS analyses data are displayed in Table 3. With regard to these data, we focused on the carbon value for the analysis of organic matter, which mainly consists of carbon. The support layer of membrane is manufactured by polysulfone, composed of carbon elements. Thus, many carbon elements are measured. The value of the carbon elements is not consistent with the concentration and organic foulants,

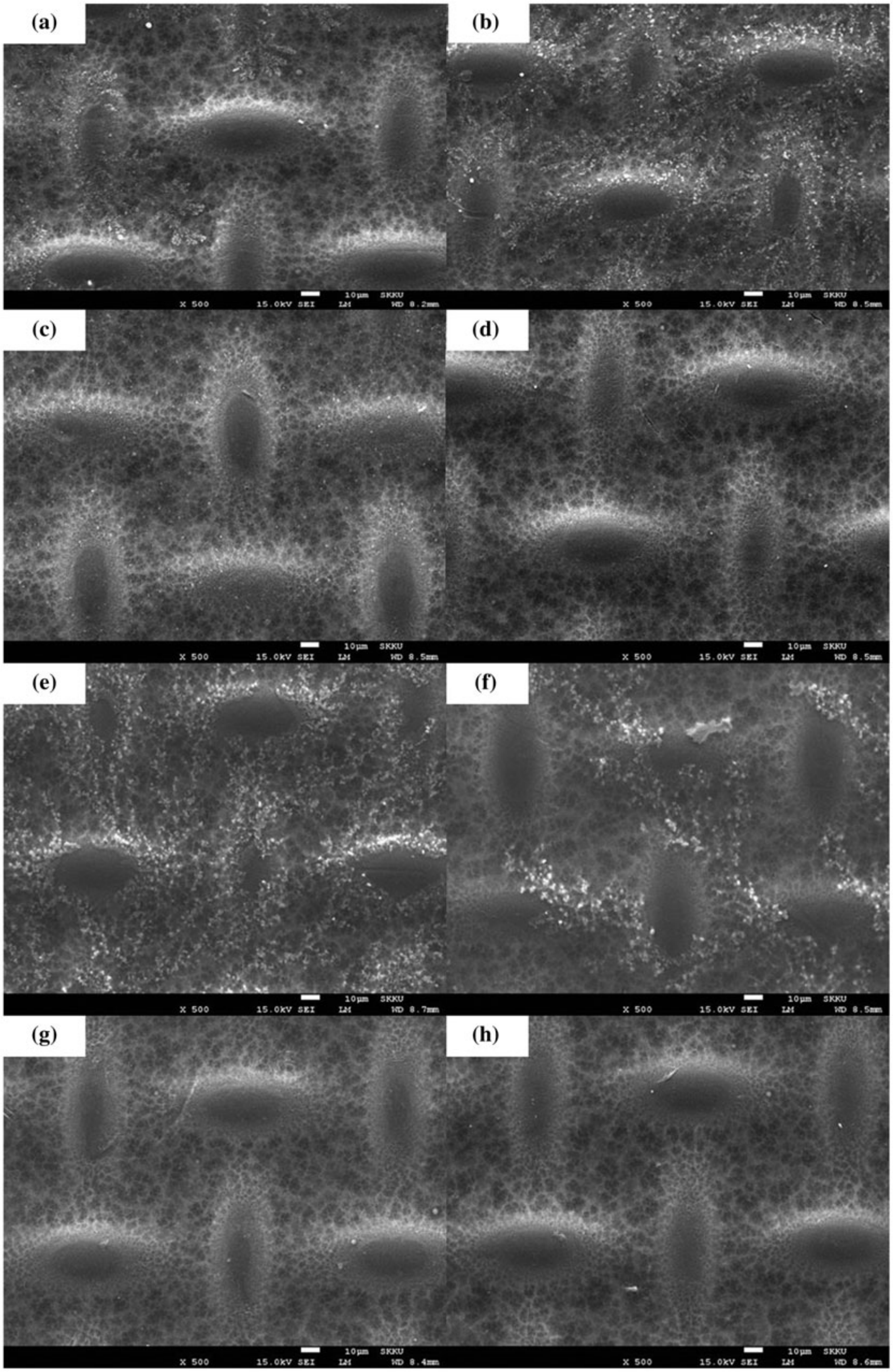

Fig. 7. SEM images on the fouled membrane by organic matter: (a) Baseline, (b) Baseline + spacer, (c) HA 500 ppm, (d) HA 500 ppm + spacer, (e) SA 500 ppm, (f) SA 500 ppm + spacer, (g) Mixing, and (h) Mixing + spacer.

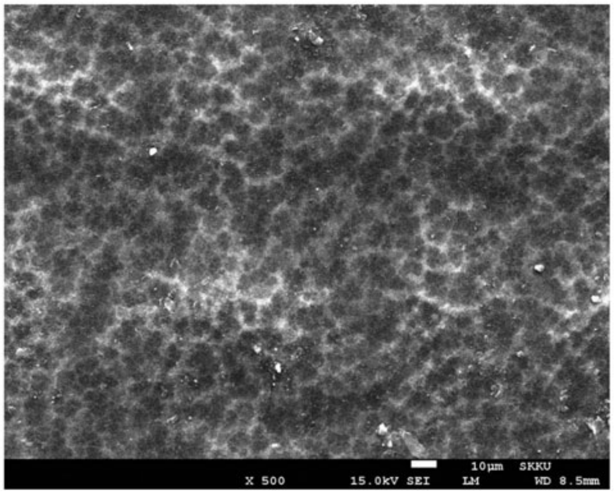

Fig. 8. SEM image of support layer of virgin membrane.

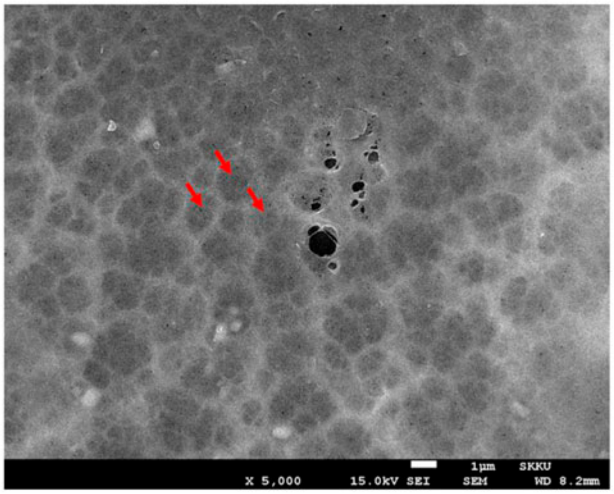

Fig. 9. SEM image of the support layer surface under HA 100 ppm condition.

Table 3
Atomic values (%) measured by EDS analysis on the fouled membrane

Contents/elements	C	Na	Cl
Virgin	73.59	–	–
Baseline	72.95	1.12	1.36
Baseline + spacer	67.02	2.36	2.35
HA 100 ppm	73.93	3.85	3.65
HA 100 ppm + spacer	71.94	2.80	2.93
HA 500 ppm	76.86	0.78	0.57
HA 500 ppm + spacer	78.70	0.25	0.17
SA 100 ppm	72.44	1.82	1.67
SA 100 ppm + spacer	73.44	3.60	4.35
SA 500 ppm	71.82	3.64	3.38
SA 500 ppm + spacer	63.50	1.07	1.03
Mixing	70.78	0.75	0.69
Mixing + spacer	70.07	0.12	0.09

since membrane structure has inhomogeneous characteristics. However, this shows a higher value in the cases where HA was applied. It indicates that there are many organic materials accumulating on the membrane surface. Salts are detected in synthetic seawater, Na and Cl.

3.4. Fouling mechanism of FO in the support layer

Until now, we investigated the fouling mechanism of the FO in support layer following the ICP equation below [24]:

$$\text{ICP modulus (FO mode)}: \frac{\pi' \text{ low}}{\pi' \text{ high}} = \exp(-J_w K) \qquad (4)$$

where J_w and K mean permeate flux (membrane property) and solute resistivity within porous support layer (solute resistance to diffusion), respectively.

Also equation on K is defined below:

$$K = \frac{t\tau}{D\varepsilon} \qquad (5)$$

where t, τ, ε, $t\tau$, and D point to support layer thickness, tortuosity, porosity, effective boundary layer, and diffusion coefficient of the solute, respectively. As shown in Fig. 10(a), the solute accumulated on the support membrane surface. Divalent ions play a significant role in generating an osmotic pressure difference. Thus, water flux is increased.

On the other hand, because organic matter accumulates on the membrane surface and the support layer is filled with a permeate water, it causes solution dilution within support layer, as a result, osmotic pressure difference is decreased. Eq. (5) describes Fig. 10(b). Values of t, τ, ε, and $t\tau$ are not changed, however, since support layer was diluted, the D value is decreased. The K value is hanged and increases slope, which means the distance difference between π' low and π' high widens. Thus, effective osmosis pressure is continuously decreased, since deteriorated ICP occurs by dilution in the support layer when the spacer is present in the support layer with organic matter.

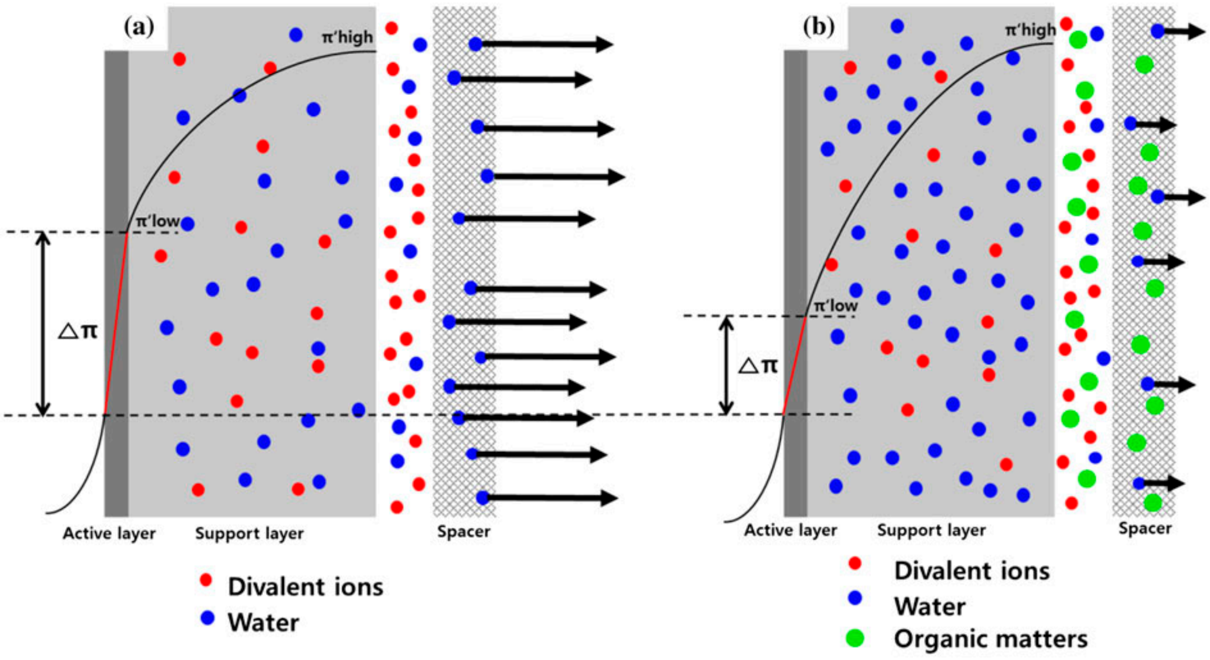

Fig. 10. A conceptual illustration of the permeate water flux on the draw solution side: (a) with spacer and (b) with spacer and organic matter.

4. Conclusions

Several experiments were conducted to clarify the effects of organic matter in synthetic seawater without or with a spacer. In this study, we found that organic matter on the draw solution side has a negative effect on water flux reduction rate when the concentration is as high as 500 ppm. Likewise, results revealed that a spacer added on the membrane in contact with support layer in a FO process might enhance the water flux reduction rate. Particularly, the water flux reduction rate on HA fouling appears increased by a spacer compared to SA. On the other hand, the effect of SA is much greater in the absence of the spacer than in HA. The DOC and FESEM/EDS results support these results.

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