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Comparative evaluation of physical cleaning method for organic and inorganic foulants mitigation in pressure retarded osmosis (PRO)

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ABSTRACT

Membrane fouling is one of the major problems to be solved to sustain stable pressure-retarded osmosis (PRO) performance. This phenomenon caused diminished water flux productivity. So an effective cleaning method for fouled membrane is necessary. This study was carried out to compare applicable cleaning methods to mitigate organic and inorganic foulants on the PRO membrane. Fouling experiments were divided into two types: organic fouling and inorganic–organic fouling. Humic substance and calcium carbonate were used as model compounds. The fouled PRO membranes were cleaned by seven types of cleaning methods namely: (i) physical flushing, (ii) pressure-assisted physical flushing at feed side, (iii) pressure-assisted physical flushing at draw side, (iv) osmotic backwashing, (v) reverse osmosis flushing, (vi) pressure-assisted osmotic backwashing with low pressure (PAOB-I), (vii) pressure-assisted osmotic backwashing with high pressure. PAOB-I was a more effective method to mitigate foulants on the PRO membrane compared with the other cleaning methods in this study.

Keywords: Membrane fouling; Cleaning method; Pressure-retarded osmosis; Osmotic backwashing

1. Introduction

The world needs more energy due to growing population. So, the world has tried to find sustainable energy. Osmotically driven (OM) membrane process of renewable energy has drawn interest of many researchers [1]. Pressure-retarded osmosis (PRO) is a promising technique of the new renewable energy techniques (wind, solar, and tidal generation, etc.) [2]. However, the net energy PRO productivity is not enough to apply [3]. PRO is used as a supporting energy source to reverse osmosis (RO) system. RO–PRO hybrid system has been studied. RO is connected by PRO. In the RO–PRO system, PRO uses brine coming out of the RO system, as a draw solution (DS), wastewater as a feed solution (FS). The RO–PRO system had a lot of benefits compared with stand-alone RO system. RO energy consumption can be reduced. The generated energy from PRO can support a total operational energy of RO.

In previous study of RO–PRO system using a cellulose triacetate (CTA) the minimum net energy consumption was 1.2 kWh/m^3. Considering 2.0 kWh/m^3 of RO energy consumption, the RO–PRO system can theoretically reduce the energy consumption up to 40% [4]. However, there are some problems to run PRO, one of the major problems is membrane fouling due to the organic and inorganic foulants in the solution. Membrane fouling is an unavoidable phenomenon during PRO operation, because FS uses wastewater. The inorganic scaling is occurred when the sparingly soluble salts exist in the feed water such as barium sulfate, calcium carbonate, and calcium sulfate. These organic foulants convert into bulk crystallization. In surface crystallization, a growth of the scale deposited on the membrane surface hinders the membrane performance. In bulk

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crystallization, a sediment of crystals is formed on the membrane surface. These combined phenomena lead to adverse effect; it could decline membrane lifespan and increase operation cost. So, proper cleaning method is necessary to keep stable PRO process [5–7]. Concentration polarization (CP) is a major factor to affect pressure-driven membrane desalination processes. Its presence hinders permeate due to changed osmotic pressure. In osmotic processes, CP occurs on both sides of the PRO membrane. The membrane has asymmetric layers – active and porous support layer. The porous support layer of the membrane protects from the shear and turbulence related with cross flow during operation. Fig. 1 shows the schematic diagram of concentration polarization (CP) on the membrane. [8]. Two types of CP take place in the membrane – external concentration polarization (ECP) and internal concentration polarization (ICP). When the solute accumulates within porous support layer and depletes solute in outside of membrane, CP occurs. As a result of lower OM force, water flux is also declined proportionately. CP affects strongly water flux [9]. To minimize CP, if pressure is applied during cleaning, the permeate water flux of cleaning could be increased. The advantage of pressure-assisted cleaning can get higher permeate water flux (J_w) even with less concentrated draw solution. The hydraulic pressure has impact on the membrane properties and overall

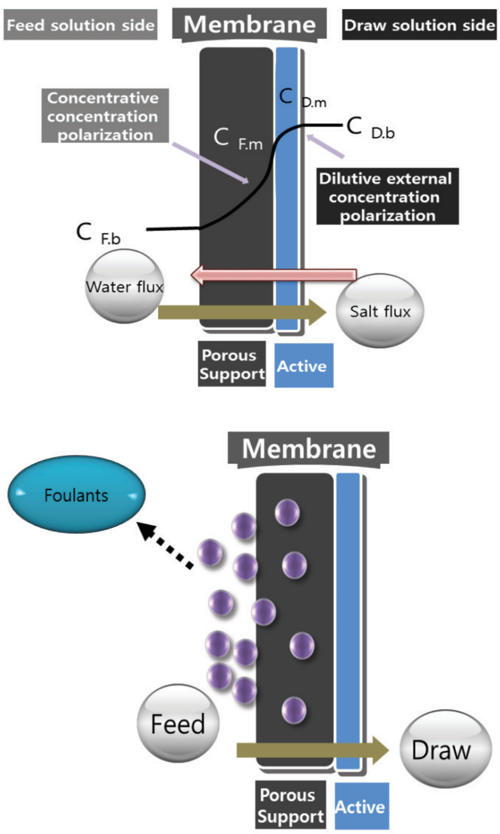


Fig. 1. Asymmetric membrane with the active layer facing the draw solution (AL-DS), internal concentration and external concentration polarization (ICP, ECP). (C = concentration polarization, F,b = feed boundary, D,b = draw boundary, F,m = feed membrane, D,m = draw membrane, ICP = internal concentration polarization, ECP = external concentration polarization, RSD reverse salt diffusion.)

performance. It induces membrane deformation [10]. First, this study investigated the seven types of cleaning methods for PRO membrane to mitigate the organic fouling with humic acid 100 mg/L. The fouled PRO membranes were cleaned and compared by cleaning methods: (i) physical flushing (PF), (ii) pressure-assisted physical flushing at feed side (PAPF-F), (iii) pressure-assisted physical flushing at draw side (PAPF-D), (iv) osmotic backwashing (OB), (v) reverse osmosis flushing (ROF), (vi) pressure-assisted osmotic backwashing with low pressure (PAOB-I), (vii) pressure-assisted osmotic backwashing with high pressure (PAOB-II). Second, in the organic and inorganic fouling – $CaCO_3$ solution of 1,000 mg/L with humic acid 100 mg/L, three types of cleaning methods were chosen to compare the effect on membrane performance as follows: (i) cleaning without OM cleaning – PF, (ii) cleaning with OM cleaning – ROF, and (iii) cleaning with OM cleaning – PAOB-I. Field emission scanning electron microscope (FESEM) and excitation emission matrix (EEM) were used to prove the phenomena of the results. According to the recovery rate, the effectiveness of the cleaning methods was evaluated. Declining water flux (J_w) was the performance parameter studied to find out influence of the cleaning methods on the membrane.

2. Materials and methods

2.1. Experimental setup

In this study, the schematic diagram of the PRO setup is shown in Fig. 2. It is worthwhile to note that several types of cleaning methods were conducted in the same solution for the purpose of finding the optimum method for the stable PRO operation. The membrane orientation, 'active layer facing feed solution', was investigated. The cross flow and stable hydraulic pressure were applied to maintain the beginning of the PRO operation. The lab-scale PRO cell is made of SUS (Steel Use Stainless) for flat-sheet membrane. It has an effective area of membrane, that is, 0.064 m^2 (0.08 m length × 0.08 m width). The 3-version PRO commercial membrane was obtained from Toray (Toray Chemical Co., Korea). The applied hydraulic pressure in the DS was monitored as an electronic pressure gauge (GR200 graphic recorder, Hanyoung Nux Co., Korea). An electronic scale (Ranger 7000, Ohaus Co., USA) was used to record the declining water weight to calculate permeate flux. It is placed underneath the FS container. Each fouling

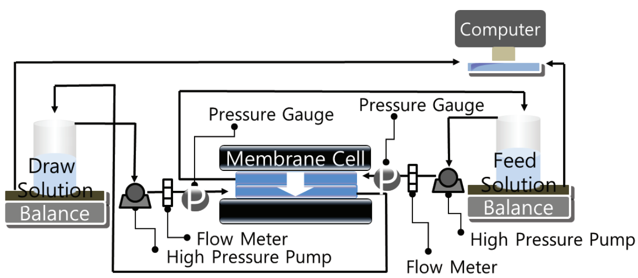


Fig. 2. The schematic diagram of pressure-retarded osmosis (PRO) in lab scale. Feed solution: low concentrated solution, Draw solution: high concentrated solution. The PRO membrane is placed between the membrane cell. Water permeates the membrane due to osmotic gradient. Decreasing weight of the both solution is automatically recorded at the computer.

experiment kept stability of the temperature (DS, FS = 20°C). A chiller (RW-0525G, Jeiotech Co., Korea) was used to control the temperature. A booster pump (Hyosung Co., Korea) was used to maintain the stable hydraulic pressure (P = 15 bar) for the PRO process. DS concentration was made from 70,000 mg/L NaCl (sodium chloride, Samchun Co., Korea). DS concentration was kept constantly by dosing a higher concentrated stock solution into the DS. DS and the brine of the RO system have same concentration – 70,000 mg/L NaCl. The FS and DS volume (2 L) were maintained constantly. FS and DS flow rate (1 LPM) were fixed. Each fouling experiment lasted for 5 h before cleaning.

Table 1 shows the testing conditions applied for all experiments.

The optimum applied pressure (ΔP) for the PRO operation was a little different depending on the condition of device, as described in Eq. (1):

$$J_w = A(\Delta\pi - \Delta P) \quad (1)$$

where A = water permeation coefficient of the membrane, $\Delta\pi$ = osmotic pressure difference, ΔP = hydraulic pressure difference in PRO membrane, as described in Eq. (2):

$$\text{Differential pressure (DP)} = P_f - P_c \quad (2)$$

P_f = feed pressure, P_c = concentrate pressure.

2.2. Condition of the feed solution

In this study was conducted: (i) organic fouling – seven types of cleaning methods, (ii) organic and inorganic fouling – three types of cleaning methods.

First, organic solution was composed of distilled water (DW) with humic acid – 100 mg/L (humic acid sodium salt, Sigma-Aldrich Co., Germany). Humic acid was selected as model organic foulant because wastewater effluent and natural water include humic acid. It is reported that humic substances have been found in 2nd and 3rd treated wastewater and it has been extensively used for study of membrane fouling [11–14].

Second, inorganic solutes – $CaCO_3$ (calcium carbonate, Showa Co., Japan) and $CaSO_4$ (calcium sulfate, Shimakyu's Pure Chemicals Co., Japan) were used as a model scalant. All three types of feed solutions were used in this study. Table 2 shows the experimental conditions for foulants. Especially, $CaCO_3$ solution of 1,000 mg/L with humic acid 100 mg/L was used as a model solution in the inorganic and organic fouling. Before using it, all stock solutions were stored at 4°C.

Table 1
Testing conditions applied for all experiments

Items	Conditions
Membrane	3-version PRO membrane (Toray Co.)
Effective membrane area	0.0064 m^2 (0.08 m × 0.08 m)
Pressure	15 bar
Membrane orientation	AL-DS (PRO mode)
Temperature	20°C
Flow rate	Both (feed, draw) 1 LPM
Fouling time	5 h

2.3. Cleaning methods

The fouled PRO membranes were cleaned by seven types of cleaning methods:

(i) PF was performed using draw and feed side – DW. (ii) PAPF-F was performed as PF with 5 bar hydraulic pressure in the FS. (iii) PAPF-D was performed as PF with 5 bar hydraulic pressure in the DS. (iv) OB using draw side – DW, feed side – 0.6 M NaCl was performed by switching the solution position in terms of the membrane orientation. (v) ROF was performed using draw side – 0.6 M NaCl, feed side – DW with 40 bar hydraulic pressure in the DS. (vi) PAOB-I using draw side – DW, feed side – 0.6 M NaCl was performed as OB with 5 bar hydraulic pressure in the DS. (vii) PAOB-II using draw side – DW, feed side – 1.2 M NaCl was performed as OB with 5 bar hydraulic pressure in the DS. Table 3 shows the cleaning conditions for all experiments.

Table 2
Experimental conditions for solution

Items	Conditions		
Feed solution	Humic acid 100 mg/L	Humic acid 100 mg/L + $CaSO_4$ 1,000 mg/L	Humic acid 100 mg/L + $CaCO_3$ 1,000 mg/L
Draw solution	NaCl 1.2 M		

Table 3
Cleaning methods for PRO membrane

Items	Conditions		
Solution	(1) DW	(2) 0.6 M NaCl	(3) 1.2 M NaCl
Cleaning time	30 min		
Flow rate	FS:DS = 1:1 LPM		
Cleaning methods	(i) Physical flushing (PF) (ii) Pressure-assisted physical flushing at feed side (PAPF-F) (iii) Pressure-assisted physical flushing at draw side (PAPF-D) (iv) Osmotic backwashing (OB) (v) Reverse osmosis flushing (ROF) (vi) Pressure-assisted osmotic backwashing-I (PAOB-I) (vii) Pressure-assisted osmotic backwashing-II (PAOB-II)		
Cleaning conditions	(i) FS:DS = (1):(1) (ii) FS:DS = (1):(1) + 5 bar in the FS (iii) FS:DS = (1):(1) + 5 bar in the DS (iv) FS:DS= (2):(1) (v) FS:DS = (1):(2) + 40 bar in the DS (vi) FS:DS = (2):(1) + 5 bar in the DS (vii) FS:DS = (3):(1) + 5 bar in the DS		

2.4. Morphology analysis of membrane by FESEM

In this study, to find the difference, all fouled and cleaned membranes were analyzed by FESEM. The samples were cut by cross section polisher (CP, IB-19510CP, JEOL Ltd., Japan) to place the analysis device. The cross section of the membrane can maintain state stability by using CP. Distinctions between membrane surface were examined by using field emission scanning electron microscope (FESEM, JEOL-7800F, JEOL Ltd., Japan).

2.5. F-EEM analysis of cleaning solution by time

In this study, the cleaning solution of the feed side was circulated as close-type circulation during cleaning. Because membrane fouling phenomenon more occurred in the support membrane – feed side membrane layer, the cleaning solution was analyzed over time. To find the amount of the detached foulants from the fouled membrane depending on the different types of cleaning methods, while the fouled membrane had been cleaned, cleaning solutions were taken over time (0, 5, 15, 30 min). The fluorescence spectroscopy (AQUALOG, Horiba Instruments Inc., Japan) was used for the EEM analysis. The samples were analyzed after being filtered with 0.45 μm filter. Fluorescence EEMs (F-EEM) and absorbance spectra were analyzed using from 250 to 550 nm using 2 nm for excitation intervals and 2.33 nm for emission using a medium gain and 0.5 s integration for emission detection.

3. Results and discussion

3.1. Comparison according to cleaning methods

Figs. 3 and 4 represent the effectiveness of the all cleaning methods on organic fouling with humic acid 100 mg/L. In the initial stage, ROF had a highest recovery performance. All cleaning methods could work as cleaning except PAPF-F. PAPF-F was not working as a cleaning method. When an initial recovery rate was measured after fouling and cleaning, the recovery rates were PF: 81%, PAPF-F: 0%, PAPF-D: 87%, OB: 90%, ROF: 100%, PAOB-I: 92%, PAOB-II: 91%. ROF had the best efficiency among the methods, however, as time passed, PAOB-I started showing the fine effect.

3.1.1. Impact of hydraulic pressure orientation on fouled membrane during cleaning

Cleaning efficiency was evaluated by using seven types of cleaning methods. All cleaning test had recovery ability except the PAPF-F. All cleaning types were operated for 30 min. The hydraulic pressure was used as a support factor to increase permeate flux. However, adverse result can occur depending on the orientation of the hydraulic pressure. In this study, there were a lot of organic foulants in the FS. So, these foulants were accumulated on the feed-side surface of the membrane and in the porous support layer of the membrane.

Cross flow cleaning and permeate flux were used to detach the foulants from the membrane. The pressure factor can accelerate the permeability to clean inside of the membrane (orientation from DS to FS) even without a higher draw

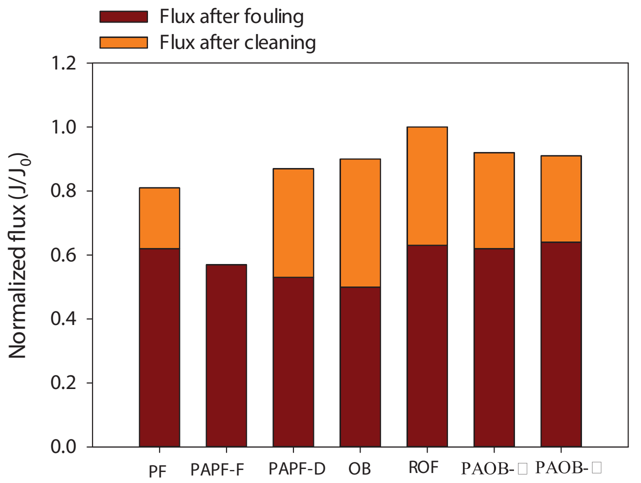


Fig. 3. Comparison between recovery rate (recovery rate PF: 81%, PAPF-F: 0%, PAPF-D: 87%, OB: 90%, ROF: 100%, PAOB-I: 92%, PAOB-II: 91%).

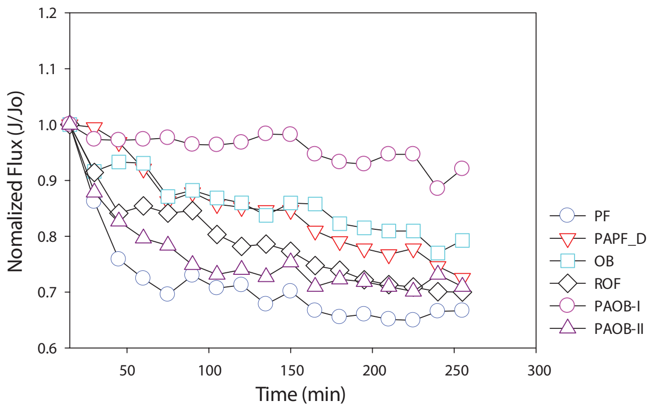


Fig. 4. Comparison after cleaning methods in the organic fouling.

solution as a driving force. This phenomenon can be inferred from the results (OB and PAOB-I) in Fig. 3. OB and PAOB-I had same experimental condition without pressure. Due to the pressure, PAOB-I had better recovery than OB. However, in PAPF-F, when the pressure was applied in the FS (orientation form FS to DS), it assumed the fact that it could press down the foulant and make the compact cake layer on the feed-side surface of the membrane. It could cover the surface of the membrane and hinder the permeability. PAOB-I had 0% recovery rate among the other types of cleaning with same experimental conditions. From this result, the fact that the membrane pore was blocked from foulants was assumed.

3.1.2. Comparison between effect of the cleaning methods

Fig. 4 shows the effectiveness of the cleaning methods. The PAOB-I was more effective than the other kind of cleaning methods. The PRO membrane has asymmetric shape – active layer and porous support layer. In the PRO system, pressure factor is necessary to generate the energy. So, the porous support layer plays an important role against the applied pressure. The porous support layer is thicker than active layer to withstand the applied pressure. The water transport across the membrane is affected by membrane structure and ICP in the porous support layer. The foulants are accumulated inside the porous support layer during PRO operation. The deposited foulants substantially affects water transfer across the membrane. The accumulated foulants in the porous support layer induce ICP [9]. PF can rinse well the foulants on the surface of the membrane. The cleaning methods based on osmotically driven (OM), such as ROF, OB, PAOB-I, and PAOB-II, can use permeate water created by osmotic pressure to clean inside of the membrane during cleaning. To compare the effect of the cleaning methods, (i) the pristine membranes were forced to get fouling, (ii) the fouled membranes were applied different cleaning methods, and then (iii) these membranes were forced to get fouling again. The membrane filter function could work, but as time passed , the PAOB -I recovery performance started showing the fine effect. The fouled membrane applied PAOB-I had lesser declining slope of flux compared with others. From this phenomenon, it assumed the fact that rest of the fouling and membrane deformation caused by the applied higher pressure during the cleaning could affect the PRO membrane performance. Deformed membrane shape can affect performance in different ways [10].

3.2. Cleaning for inorganic and organic fouling

In the organic fouling – humic acid 100 mg/L cleaning methods, PAOB-I had higher recovery performance compared with the others. (i) Cleaning without OM cleaning – PF, (ii) cleaning with OM cleaning – ROF, and (iii) cleaning with OM cleaning – PAOB-I of the seven types of the cleaning methods were applied to the inorganic and organic fouling for reproducibility to investigate this phenomenon.

3.2.1. Fouling tendency according to the inorganic and organic component

Fig. 5 shows the results of the declining flux of three types of solution (humic acid, humic acid + $CaSO_4$, humic acid + $CaCO_3$). Declining rate was different according to the solute in the solution. All fouling experiment had been run for 5 h. A severe decline was observed about 1 h. Organic–inorganic component (humic acid + $CaCO_3$) had big impact on the membrane performance compared with the others. $CaCO_3$ could make scaling in the membrane. Humic acid + $CaCO_3$ used as a model foulant in the organic–inorganic fouling experiment. Three types of cleaning methods were applied to identify for reproducibility of the previous result of organic fouling study: PF, ROF, and PAOB-I.

3.2.2. Comparison between PF, ROF, and PAOB-I

Fig. 6 shows a distinct difference between PF, ROF and PAOB-I. The PAOB-I was more effective than the others and had the similar result as previous result in organic fouling study. In the initial stage of the declining flux, the fouled membrane filtering function could work after cleaning. However, similar to previous result, as time passed, PAOB-I was showing better performance. PF and ROF were sharply declined than PAOB-I. The result of the membrane treated PAOB-I had showed the lesser declining flux. The fact that rest of the accumulated foulants in the membrane and deformed membrane structure after applying three kinds of cleaning (PF, ROF, and PAOB-I) could affect the performance of the membrane, was assumed.

3.3. Morphology analysis of membrane by FESEM

Fig. 7 shows the distinction according to cleaning methods. To compare the changed structure in terms of the before and after shape, the cross section of the pure membrane is shown in Fig. 7(a). Fig. 7(b) shows the cross section of the fouled membrane. As can be seen, the inorganic and organic foulants were accumulated in the membrane. The foulants stuck and covered the membrane pore. This phenomenon can affect the permeability related to the flux. The two types of the membrane by ROF and PAOB-I were chosen. The cross section of the membrane after ROF is shown in Fig. 7(c). The cross section of the membrane after PAOB-I is shown in Fig. 7(d). The cross section of the membrane was flushed by a permeate water flux of ROF and PAOB-I. Both cleaning methods can clean the foulants in

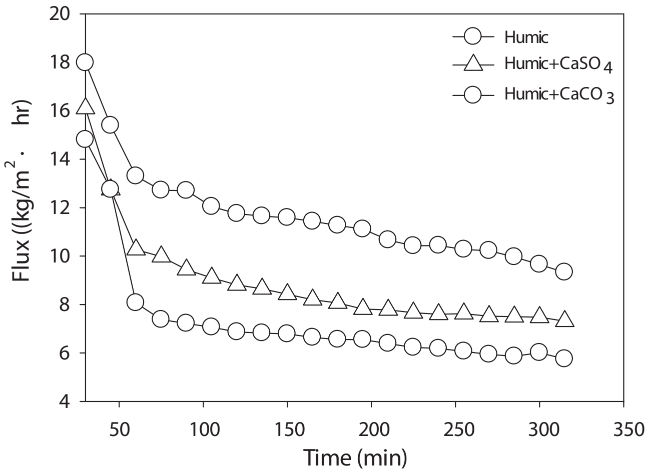


Fig. 5. Flux decline according to the inorganic and organic foulant.

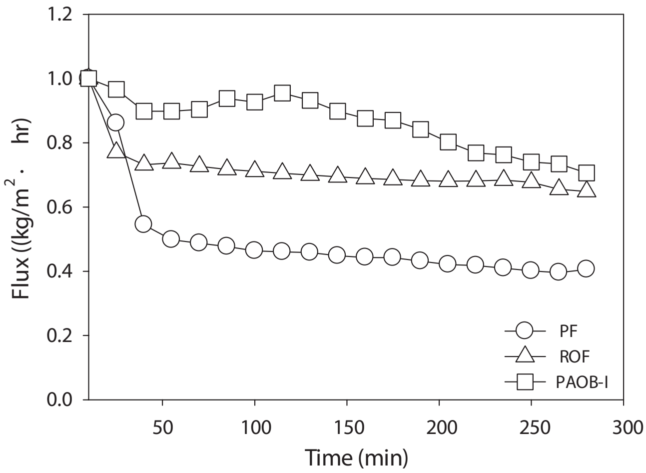
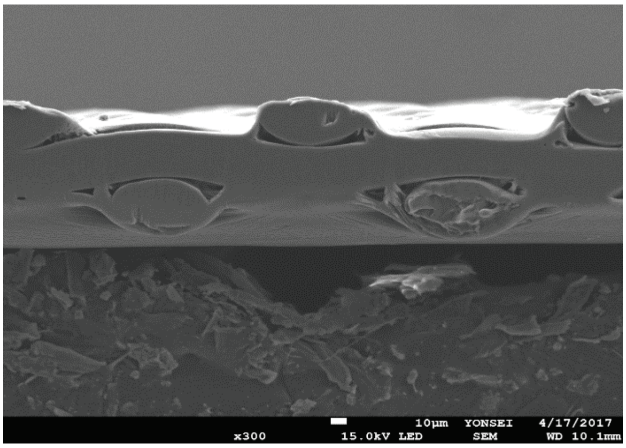
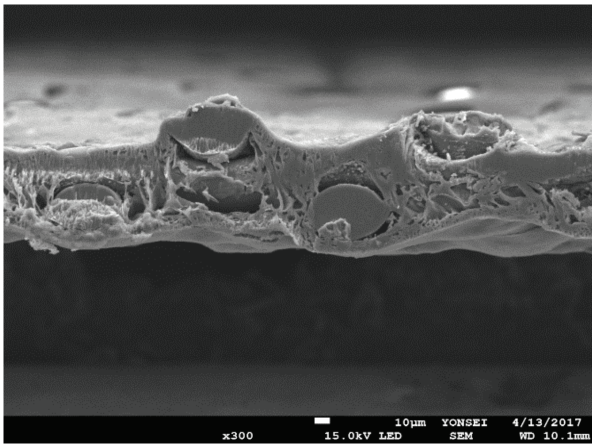


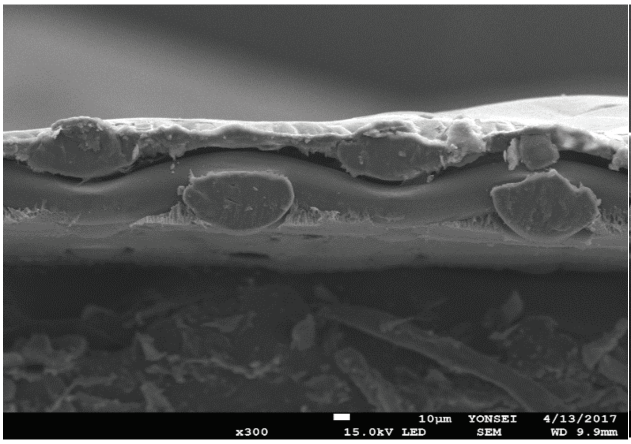
Fig. 6. Comparison of the flux decline after different cleanings (PF, ROF, and PAOB-I).



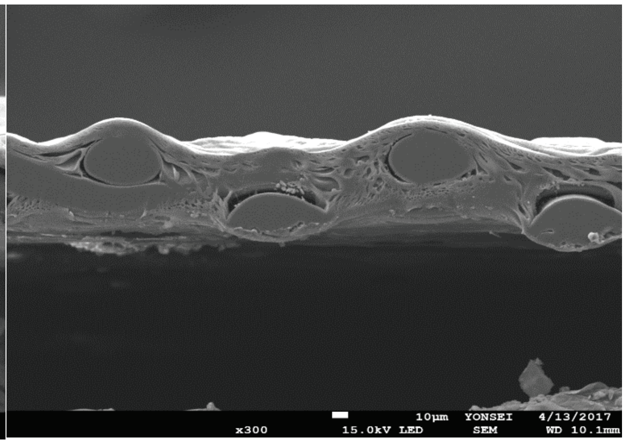
(a)Cross section of the pure membrane
(Magnification: X300)



(b)Cross section of the fouled membrane
(Magnification: X100)



(c)Cross section of the membrane after ROF
(Magnification: X300)



(d)Cross section of the membrane after
PAOB-I(Magnification: X300)

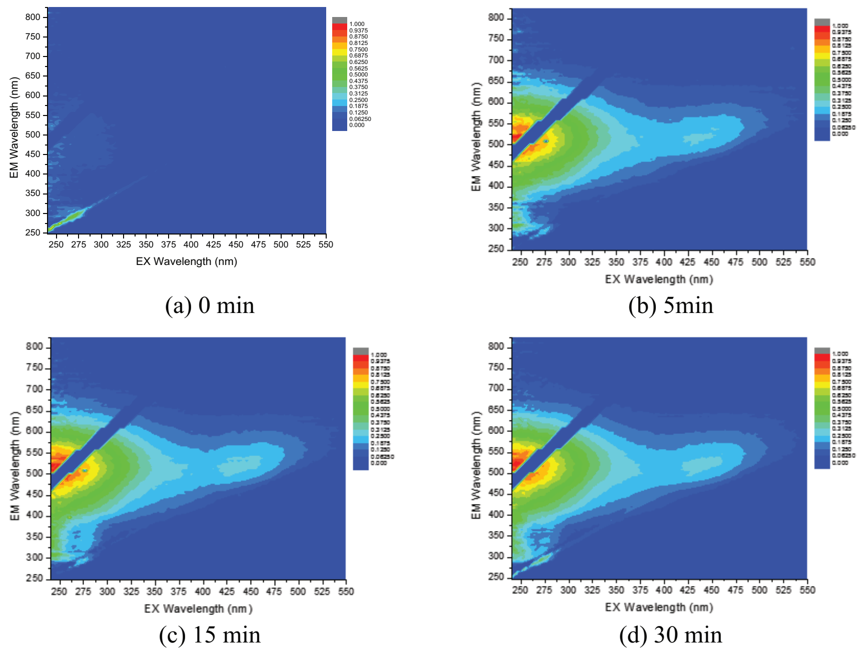
Fig. 7. FESEM images of the cross section of the membranes. (a) Cross section of the pure membrane (magnification: X300). (b) Cross section of the fouled membrane (magnification: X100). (c) Cross section of the membrane after applying ROF (magnification: X300). (d) Cross section of the membrane after applying PAOB-I(magnification:X300).

the membrane. However, the big distinct feature is shown in Figs. 7(c) and (d). Both ROF and PAOB had pressure factor. Due to the pressure, both cleaning methods can induce membrane deformation during cleaning. As shown in Fig. 7(c), the active layer was detached from the porous support layer and changed the shape. Because ROF used higher pressure – 40 bar than PAOB-I – 5 bar, deformed membrane could result in adverse effect. Reverse solute diffusion (RSD) plays an important role in the PRO membrane performance. The ICP can be increased by the RSD. The drop of the membrane performance is attributed to the limiting factor of RSD – the decreased selectivity of the membrane cause greater passage of draw solute inside the porous support layer, thereby reducing the osmotic power as a driving force [15,16]. A higher applied pressure can induce severe membrane deformation. The extent of membrane deformation is proportionally caused by the applied pressure. Membrane deformation can cause more RSD into the feed solution and inside of the membrane. Its phenomenon can enhance the ICP and induce adverse effect [17,18]. Membrane deformation after ROF was more severe than PAOB-I in Figs. 7(c) and (d). It shows the fact that the higher pressure can lead to membrane deformation. Due to the deformed structure of the membrane, even though the ROF had the best recovery in the initial stage after cleaning, the adverse effect started showing over time.

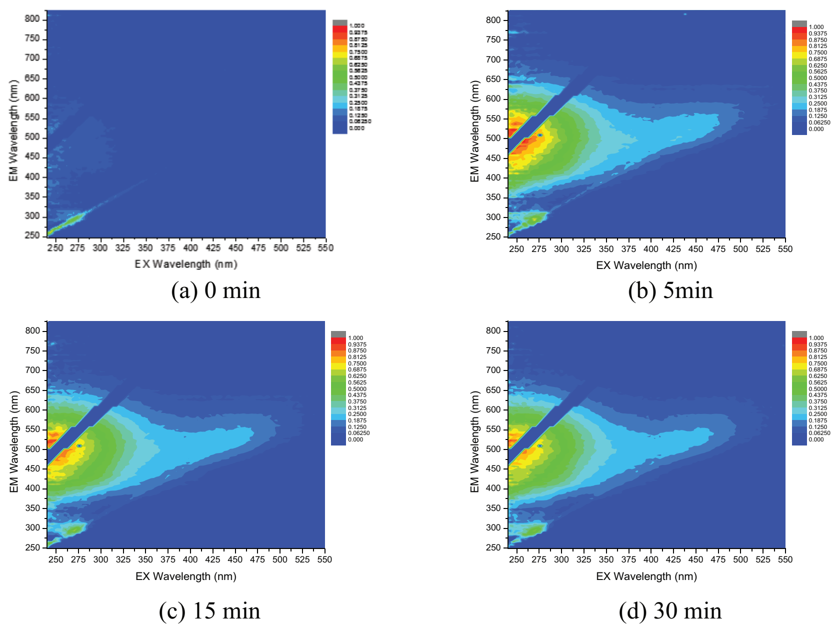
In the organic fouling study, even though, PAOB-II used 1.2 M NaCl draw solution to get a higher driving force than PAOB-I, PAOB-I had been more effective than PAOB-II. This result assumed that a rest of the solute of the PAOB-II cleaning solution in the membrane could increase RSD and ICP and affect the performance [17,18].

3.4. Analysis of the cleaning solution by EEM

Fig. 8 shows, as a sensitive analytic method, EEM can find the foulants in the cleaning solution flowed from fouled membrane during cleaning for 30 min. Because cleaning solution in the feed side was circulated as a close circulation type, the circulated cleaning solution was taken over time (0, 5, 15, 30 min), and EEM was used to observe the detached organic matter from the fouled membrane as time passed. Each EEM gave spectra information about the humic composition of cleaning samples. EEM is an appropriate method for characterizing the foulants extracted from the fouled membrane depending on the cleaning methods. The optimum cleaning method can be identified by EEM.



(A) Cleaning organic fouled membrane with ROF



(B) Cleaning organic fouled membrane with PAOB-I

Fig. 8. Analysis of the EEM in ROF and PAOB-I over time. (A) Cleaning organic fouled membrane with ROF, (a) 0 min, (b) 5 min, (c) 15 min, (d) 30 min. (B) Cleaning organic fouled membrane with PAOB-I, (a) 0 min, (b) 5 min, (c) 15 min, (d) 30 min.

4. Conclusions

This study was conducted to find the optimum cleaning method for the PRO membrane. Fouling experiments were divided into two types: organic fouling and inorganic–organic fouling. The seven types of cleaning methods were applied to the fouled membrane in organic fouling experiment and three types of the cleaning methods were chosen (PF, ROF, and PAOB-I) to apply in inorganic–organic fouling for reproducibility.

- The result can be changed depending on the pressure orientation. When the cleaning uses assisted pressure to the fouled membrane, the applied pressure can increase the permeate water into the membrane even without a higher driving force. The applied pressure orientation from DS to FS can work effectively, however, in the opposite orientation, from FS to DS, it could compress the cake on the feed-side membrane surface. The pressed cake can block the pore of the membrane and hinder the permeability during PRO operation.
- A higher applied pressure can induce severe membrane deformation. The membrane deformation can cause more RSD into the feed solution and inside of the membrane. Its phenomenon can enhance the ICP. ROF, 40 bar, used higher pressure than PAOB-I, 5 bar, during the cleaning. Even though ROF had the best recovery rate, the adverse effect of the deformed membrane started showing over time.
- Even though, PAOB-II used 1.2 M NaCl draw solution to get a higher driving force than PAOB-I, PAOB-I had been more effective than PAOB-II. This result assumed that a rest of the solute of the PAOB-II cleaning solution in the membrane could increase RSD and ICP and affect the performance.
- EEM is an appropriate method for characterizing the foulants extracted from the fouled membrane depending on the cleaning methods. The optimum cleaning method can be identified by EEM.
- PAOB-I was a more effective method to clean the fouled membrane compared with the other cleaning methods in this study.

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