Desalination and Water Treatment
www.deswater.com

doi: 10.1080/19443994.2016.1189703

57 (2016) 26621–26628
November

Relating membrane surface properties and flux recovery during the chemical cleaning of forward osmosis membrane

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Received 18 December 2015; Accepted 24 December 2015

ABSTRACT

In this study, we evaluated the efficiencies of chemical cleaning agents for the removal of foulant and related the changes of membrane surface properties to the flux recovery in forward osmosis (FO) membrane process. Commercially available chemical cleaning agents were added and processed according to the protocols of reverse osmosis (RO) membrane processes into two steps—acid treatment, then base treatment, and the flux recovery and membrane surface properties were tested in FO mode. The flux of the fouled membrane was only 23% of initial water flux tested in FO mode, and the addition of cleaning chemicals could improve the water flux to be 65 and 83% of initial water flux after each step, respectively. The reverse flux selectivity of fouled membrane was increased up to 61 and 86% after each cleaning steps due to the decrease in the concentration polarization inside the layer. The removal of biofouling layer during each cleaning step was demonstrated by scanning electron microscopy (SEM) and FT-IR, while incomplete removal of colloidal matters on membrane surfaces was observed. The total membrane surface free energy was increased after the cleaning steps due to the increase in electron-donating and electron-accepting character. Overall, chemical cleaning agents designed for RO could remove various membrane foulants as well as recover the hydrophilicity of membrane surfaces and consequently be applied for the cleaning of membranes operated in FO process, but additional cleaning protocol should be developed for the removal of colloidal matters from membrane surfaces.

Keywords: Forward osmosis; Membrane fouling; Chemical cleaning; Membrane surface properties

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Presented at the 8th International Desalination Workshop (IDW) 2015, November 18–21, 2015, Jeju Island, Korea

1. Introduction

Reverse osmosis (RO), pressure-driven membrane process, has been widely used in the field of wastewater reclamation [1] and desalination [2,3] due to the higher salt rejection with the simple operation. RO uses hydraulic pressure as the driving force for water transport through the membrane, thus finally leading to the long-standing problem of fouling [4]. To restore the membrane performance, chemical cleaning process is generally adopted. Chemical cleaning agents typically used in RO cleaning process to remove the foulants deposited on membrane surfaces can be categorized as acidic (e.g. HCl and citric acid) and caustic (e.g. NaOH and KOH) solution with some additives such as surfactants dependent on the nature of fouling layer. Several researches on RO chemical cleaning have been focused on the finding of superior chemical cleaning agents and optimum conditions for acidic and caustic chemical agents. For example, Madaeni and Samieirad [5] reported that caustic solution such as NaOH has the higher cleaning efficiency, compared to acid solutions due to the enhancing of negative charges on organic molecules, leading to the stronger repulsion between organic matter and membrane surface, resulting in the loose fouling layer. Moreover, Ang et al. [6] suggested that the chemical cleaning by dual or combined cleaning agents is more effective than membrane cleaning by individual cleaning agents. From their experiments, the cleaning efficiency of NaOH was approximately 60%, while that of EDTA followed by NaOH was approximately 90%. In general, the acidic solution was effective in the removal inorganic precipitates, while the alkaline agent was effective in the removal of organic fouling including biological matters [7].

Recently, forward osmosis (FO), an osmotic pressure-driven process, has gained wider attention recently in many applications such as desalination [8,9], wastewater treatment [10,11], and liquid food processing [12,13], where RO processes have been applied [14]. The apparent advantages of FO process emanate from its operation without applied hydraulic pressure, which has the potential for lower energy consumption and fouling propensity [15]. In early study, Kim et al. [16] demonstrated that the absence of hydraulic pressure in the FO was the main reason for better fouling reversibility. Although organic and particulated foulants deposited on membrane surface which operated in FO mode could be easily removed by simple physical cleaning such as the increase in cross-flow velocity [17] and air scouring [18], and chemical cleaning would be required in FO process when the feed solution contains high and complex foulants such as effluents from wastewater treatment processes [19]. However, little information is available for the applicability of chemical cleaning in FO processes nor the mechanism of membrane flux recovery.

It has been reported that the changes of membrane surface properties such as hydrophilicity and surface roughness significantly affect the membrane permeability [20,21]. The hydrophilicity of membrane surface can be measured by water contact angle, and more sophisticatedly, by the calculation of surface free energy [22]. For quantifying the surface free energy between foulants and membrane surface, Derjaguin–Landau–Verwey–Overbeek (DLVO) theory has been frequently used as the sum of Lifshitz–van der Waals (LW) and electrostatic (EL) interactions in water. However, DLVO theory has been failed to explain the fouling behavior of membrane processes due to the polar properties of membrane surface and foulants [23]. The extended DLVO (XDLVO) approach is proposed to take into account the impact of surface hydrophilicity by adding polar or Lewis acid–base (AB) interactions to the classical DLVO theory [20]. There are several studies on the application of XDLVO theories to explain the adhesion of foulants on the membrane surface, while there is no study in our knowledge on the application of XDLVO theory on the changes of membrane surface properties before and after the chemical cleaning.

Consequently, we tested the applicability of acidic and caustic chemical cleaning agents designed for RO to FO process and evaluated the changes of membrane surface properties to investigate the mechanism of flux recovery in FO process in this study. Water flux and reverse salt flux were investigated after the addition of acidic and caustic cleaning agents in FO mode, and membrane surface properties including roughness, surface free energy, FT-IR, and EDX were measured to understand the recovery mechanism of the membrane performance.

2. Materials and methods

2.1. Membrane

A thin film composite brackish RO membrane (ESPA2-LD, Hydronautics) was fouled with media-filtered brackish water from Asan reservoir until the flux became 20% of the initial flux in FO mode. Prepared fouled membrane pieces were then chemically cleaned with two steps including steps A and B as recommended by the RO cleaning chemical supplier (Primetech, Korea). Fouled membrane was soaked with 1.5% of chemical cleaning agent A at 35℃ and pH of 2.7 for 12 h, called Step A. For Step B, membrane sample was prepared by soaking membrane from Step A with 1.5% of chemical cleaning agent B at 35°C and pH of 11 for 12 h. All membrane samples were stored and soaked in deionized (DI) water before using.

2.2. FO flux recovery test

All experiments were conducted in a laboratory scale FO unit [24]. Each membrane sample (virgin, fouled, Step A and Step B) was placed with the active layer facing the feed solution in membrane cell, which was 7.75 cm in length, 2.6 cm in width with a channel height of 0.30 cm (the total effective membrane area was 20.15 cm^2), for the examination of flux changes in FO mode. DI water and 2 M NaCl were used as feed solution and draw solution, respectively. The flow in both sides was controlled by two variable speed pump (LongerPump WT3000-1FA, China). Each experiment was performed based on the bench-scale setup for 3 h in FO mode at a cross-flow velocity of 15 cm/s. The water flux was determined by measuring the weight changes of the draw solution every 5 min with a digital weight scale (AND GF-4000, USA) which was connected to a computer during the entire experiment. The feed conductivity was also determined by conductivity device (Vernier LabPro, USA). All experiments were carried out at 20 ± 1°C using custom-made temperature controller.

2.3. Membrane surface analysis

Contact angles of each membrane surfaces were measured by contact angle analyzer (Phoenix, SEO Korea) using three liquids including distilled water (W), ethylene glycol (E), and hexadecane (H). The surface free energy and interfacial interaction free energy were then calculated by Lifshitz–van der Waals/Lewis acid–base (LW/AB) method as suggested in the following section [20]. The foulants remaining on membrane surface after each step were collected and measured as total solids by silicon knife. In addition, membrane morphologies were analyzed by scanning electron microscope with energy-dispersive X-ray spectroscopy (SEM/EDX) (Leica, Germany) and the functional group of membrane samples was determined by Fourier transform infrared spectroscopy (FT-IR, Perkin–Elmer, USA).

2.4. Calculation of surface free energy

The membrane surface properties were examined by the surface free energy and the interfacial interaction free energy which explained the hydrophilicity/hydrophobicity property and the interfacial interaction free energy on membrane surface. Surface free energy and interfacial interaction free energy were described by the extended Derjaguin–Landau–Verwey–Overbeek (XDLVO) theory. The XDLVO theory describes the total interaction energy between water molecules and a membrane surface as the sum of Lifshitz–van der Waals (LW), Lewis acid–base (AB), and electrostatic (EL) interactions, expressed in Eq. (1) [25]:

$$\Delta G_{\mathrm{XDLVO}}^{\mathrm{Total}} = \Delta G^{\mathrm{LW}} + \Delta G^{\mathrm{AB}} + \Delta G^{\mathrm{EL}} \tag{1}$$

where $\Delta G_{\mathrm{XDLVO}}^{\mathrm{Total}}$ is the total interaction energy between a membrane surface and water molecules, ΔG^{LW} is the Lifshitz–van der Waals interaction term, ΔG^{EL} is the electrostatic interaction term, and ΔG^{AB} is the acid–base interaction term. Subramani and Hoek [26] suggested that the effect of electrostatic interaction is not a key factor influencing the total interfacial interaction free energy. Therefore, the interfacial interaction free energy was calculated as the sum of Lifshitz–van der Waals and Lewis acid–base (LW/AB) only.

In this work, hexadecane (H) was used as an apolar liquid for the calculation of van der Waals component (γ^{LW}). The surface tensions of two polar liquids (water (W) and ethylene glycol (G)) could be used to determine the electron donor (γ^{-}) and electron acceptor (γ^{+}) components or acid–base component. These parameters included the van der Waals, electron donor, and electron acceptor surface free energy components which are then used to derive the interfacial interaction free energy.

The interaction between water and membrane surface was described in terms of interfacial interaction free energy between membrane surface and water molecules. The interfacial interaction free energy between membrane surface (component 1) and water (component W) ($\Delta G_{1\mathrm{W}}$) is decomposed into the Lifshitz–van der Waals or hydrophobic part ($\Delta G_{1\mathrm{W}}^{\mathrm{LW}}$) which indicated the hydrophobic attraction force between water and membrane surface and Lewis acid–base or polar part ($\Delta G_{1\mathrm{W}}^{\mathrm{AB}}$) which indicated the polarity and the adsorption of membrane and water molecules [27]. The interfacial interaction free energy could be expressed as Eq. (2):

$$\begin{aligned}\Delta G_{1\mathrm{W}} &= \Delta G_{1\mathrm{W}}^{\mathrm{LW}} + \Delta G_{1\mathrm{W}}^{\mathrm{AB}}\\ &= -2\left(\sqrt{\gamma_1^{\mathrm{LW}}\gamma_{\mathrm{W}}^{\mathrm{LW}}} + \sqrt{\gamma_1^{+}\gamma_{\mathrm{W}}^{-}} + \sqrt{\gamma_1^{-}\gamma_{\mathrm{W}}^{+}}\right)\end{aligned} \tag{2}$$

where γ^{LW}, γ^{+}, and γ^{-} are the surface free energy component, expressed in mJ/m^2.

In this approach, surface free energy (γ) is decomposed into Lifshitz–van der Waals component (γ^{LW}) and Lewis acid–base component (γ^{AB}) which is split into a Lewis acid component (γ^{+}) and a Lewis base component (γ^{-}). The LW/AB method could be expressed as Eq. (3):

$$\left(\gamma_L^{LW} + 2\sqrt{\gamma_L^{+}\gamma_L^{-}}\right)(1+\cos\theta) = 2\left(\sqrt{\gamma_S^{LW}\gamma_L^{LW}} + \sqrt{\gamma_S^{+}\gamma_L^{-}} + \sqrt{\gamma_S^{-}\gamma_L^{+}}\right) \quad (3)$$

where θ is the contact angle in the solid/liquid/gas system, γ_S^{LW}, γ_S^{+}, γ_S^{-} are the surface free energy components of the solid and γ_L^{LW}, γ_L^{+}, γ_L^{-} are the surface free energy components of the testing liquids.

3. Results and discussion

3.1. Effect of chemical cleaning agents on membrane performance

The changes of normalized flux (J_W/J_{W0}), reverse salt flux (J_S), and reverse flux selectivity (J_W/J_S) are shown in Fig. 1. After the fouling, flux was significantly reduced up to 23% of initial flux due to the deposition of foulants and buildup of biofouling layer on the membrane surface. After each cleaning step, the normalized flux was recovered to 65 and 83% of initial flux of virgin membrane after cleaning steps with Step A and Step B, respectively (Fig. 1(a)). It implied that the fouling layer was successfully removed from the membrane surfaces; thus, the chemical cleaning agents for RO processes could be effective to FO processes. For the investigation of membrane selectivity changes due to the fouling layer, the reverse flux selectivity (J_W/J_S) was calculated and is shown in Fig. 1(b). Interestingly, the reverse flux selectivity of each membrane was correlated well with the normalized flux at the tested condition. Fig. 1(b) shows that the reverse flux selectivity of virgin membrane was 0.64 L/mmol, while that of fouled membrane was only 0.12 L/mmol, or 19% of virgin membrane, due to the decrease in permeate flux. The reverse flux selectivity was increased to 61% (0.39 L/mmol) and 86% (0.55 L/mmol) of virgin membrane after cleaning with Step A and Step B, respectively. The value of the reverse flux selectivity is independent of the structure of the membrane support layer and is solely determined by the selectivity of the membrane active layer [28]. Consequently, the increase in the reverse flux selectivity after the chemical cleaning is due to the recovery of membrane selectivity possibly by the removal of fouling layers and recovery of surface physicochemical properties.

3.2. Effect of membrane cleaning on membrane surface properties

It is well known that membrane surface hydrophilicity significantly affects the permeability and fouling propensity of semipermeable membranes such as RO and FO [29]. In this work, the changes of membrane surface properties such as hydrophilicity/hydrophobicity and surface free energy of membrane surfaces were investigated. The measured contact angles of the membrane surfaces as well as their calculated surface free energy and interfacial interaction free energy are listed in Table 1.

Contact angle measurement is a well-established method for measuring the hydrophilicity of surface

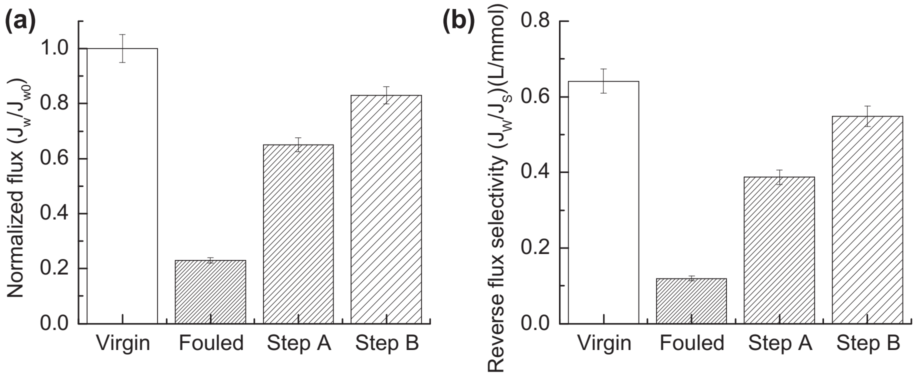

Fig. 1. The normalized flux (a) and reverse flux selectivity (b) of membrane samples. Note that the initial flux was about 6.2 LMH at DI as feed solution and 2 M NaCl solution as draw solution.

Table 1
Contact angle and surface energy parameters (mJ/m^2) of membrane samples

	Contact angle (°)						
Samples	Water	Ethylene glycol	Hexadecane	γ^+	γ^-	γ_S	ΔG_{1W}
Virgin	41.0	22.6	14.2	1.95	42.41	44.38	−127.73
Fouled	53.9	44.1	12.8	0.56	35.03	35.69	−115.69
Step A	46.2	39.7	13.5	0.63	43.80	37.28	−123.15
Step B	42.4	42.4	12.7	1.10	44.81	40.91	−126.59

[22]. As shown in Table 1, the contact angle of water on the fouled membrane was the highest (53.9°) and decreased to 46.2° and 42.4° after cleaning with Step A and Step B, respectively, which was approached to that of virgin membrane (41.0°). It means that steps of chemical cleaning provided the recovery of hydrophilicity of the fouled membrane. Furthermore, high wettability can also be defined in terms of higher surface free energy [30]. According to Table 1, all four membrane samples exhibited high electron-donating component (γ^-) and relatively low electron-accepting component (γ^+). The surface free energy (γ_S) of virgin membrane was the highest (44.38 mJ/m^2), while that of fouled membrane was lowest (35.69 mJ/m^2), which confirmed that the fouled membrane surface changed to be hydrophobic due to the foulants. However, the surface free energy was recovered to 37.28 and 40.91 mJ/m^2 after the Step A and Step B, respectively, due to the removal of hydrophobic foulants (it will be further proven in Section 3.3). Furthermore, the total interfacial interaction free energy also exhibited the similar trend that the virgin membrane possessed surface free energy of −127.73 mJ/m^2, while that of fouled membrane was increased to −115.69 mJ/m^2. The negative value of ΔG_{1W} indicates the adhesive van der Waals force between water molecules and membrane surfaces and thus the spontaneous transfer of the water molecules to membrane surfaces [31]. Therefore, water could hardly approach to the fouled membrane surface than the virgin membrane surface. After the chemical cleaning, the total interfacial interaction free energy was decreased to −123.15 and −126.69 mJ/m^2 for Step A and Step B, respectively. It also confirms that the surface of cleaned membrane became more hydrophilic with more acid–base interaction free energy, which indicated the favorable surface properties for the access of water molecules.

3.3. Changes of membrane surface properties

In previous sections, we showed that the chemical cleaning agents could recover the hydrophilicity of membrane surface by the removal of foulants on membrane surface and result in the significant recovery of the initial flux and reverse flux selectivity. For better understanding of the flux recovery, the chemical components of foulants deposited on fouled membrane surface were examined by EDX. The major chemical components from the fouled membrane surface included C (28.09%), O (31.86%), Al (10.48%), and Mn (8.91%). Furthermore, it also included small amount of Si, Fe, S, P, Na, Cl, K, and Ca. To confirm the removal of foulants on membrane surfaces by chemical cleaning agents in FO process, the total solid was analyzed (Fig. 2). Before the chemical cleaning, the amount of total solid collected by silicon knife from the fouled membrane surface was 1,160 mg/m^2, while the residual mass of foulants was reduced to 500 and 60 mg/m^2 after Step A and Step B, respectively. It directly confirms that about 95% of foulants were removed by simple chemical cleanings. It is due to the structure of fouling layer of FO process that the naturally occurring osmotic pressure is the main driving force in FO processes [32]. Consequently, the fouling layer becomes less compact compared to that from pressure-driven membrane processes such as RO and easily removed by simple chemical cleaning.

Images from scanning electron microscopy (SEM) in Fig. 3 also confirm that the application of RO chemical cleaning agents was effective to remove foulants on membrane surfaces operated in FO mode. Fig. 3(a)

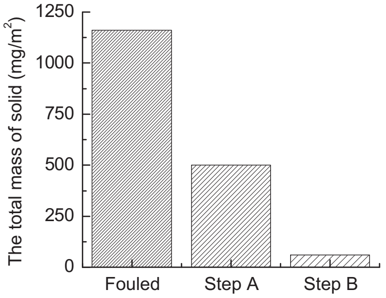

Fig. 2. The mass of foulants on membrane samples.

presents the SEM image of the virgin membrane which indicated a polyamide active layer that is free of foulants, whereas the image of fouled membrane (Fig. 3(b)) shows a complete coverage of the membrane surface by foulants. After Step A (Fig. 3(c)) and Step B (Fig. 3(d)), SEM images indicated the dramatic removal of foulants, while noticeable amount of organic and colloidal matters was still observed on the cleaned membrane surfaces. This observation corresponds with the result from Fig. 2 that the amount of residual solids on the membrane surfaces was measured, and consequently, the normalized flux of cleaned membrane was not fully recovered as shown in Fig. 1(a). In Fig. 3(c) and (d), both organic and colloidal foulants remained on the membrane surface. It has been reported that the colloidal particles can induce more significant flux decline than organic matters in FO processes [33]. Consequently, it will be required to develop the additional cleaning protocol or new chemical cleaning agents to remove colloidal particles for FO processes.

3.4. Changes of membrane surface properties

In order to check the membrane surface properties, Fourier transform infrared spectroscopy (FT-IR) was examined in the range of 450–4,000 cm^{-1}. In Fig. 4, the specific peaks of polyamide (PA) around 1,240, 1,290, and 1,320 cm^{-1} were observed due to the aromatic amines I, II, and III stretching, respectively [34]. The peaks around 1,550 cm^{-1} could be assigned to polysulfonyl group in the porous polysulfone support layer. However, in fouled membrane, these characteristic peaks disappeared due to the deposition and formation of the layer of foulants on membrane surfaces. In addition, the fouled membrane displayed additional signals around 1,030 cm^{-1} which correspond to the CO stretching of carboxylic groups, and around 3,300 cm^{-1} corresponding to N–H stretching from amine groups, which originated from organic matters and/or microorganism deposited on the membrane surface. As the progress of cleaning steps, the characteristic peaks of PA around 1,240, 1,290, and 1,320 cm^{-1} are shown again and become clear. It again

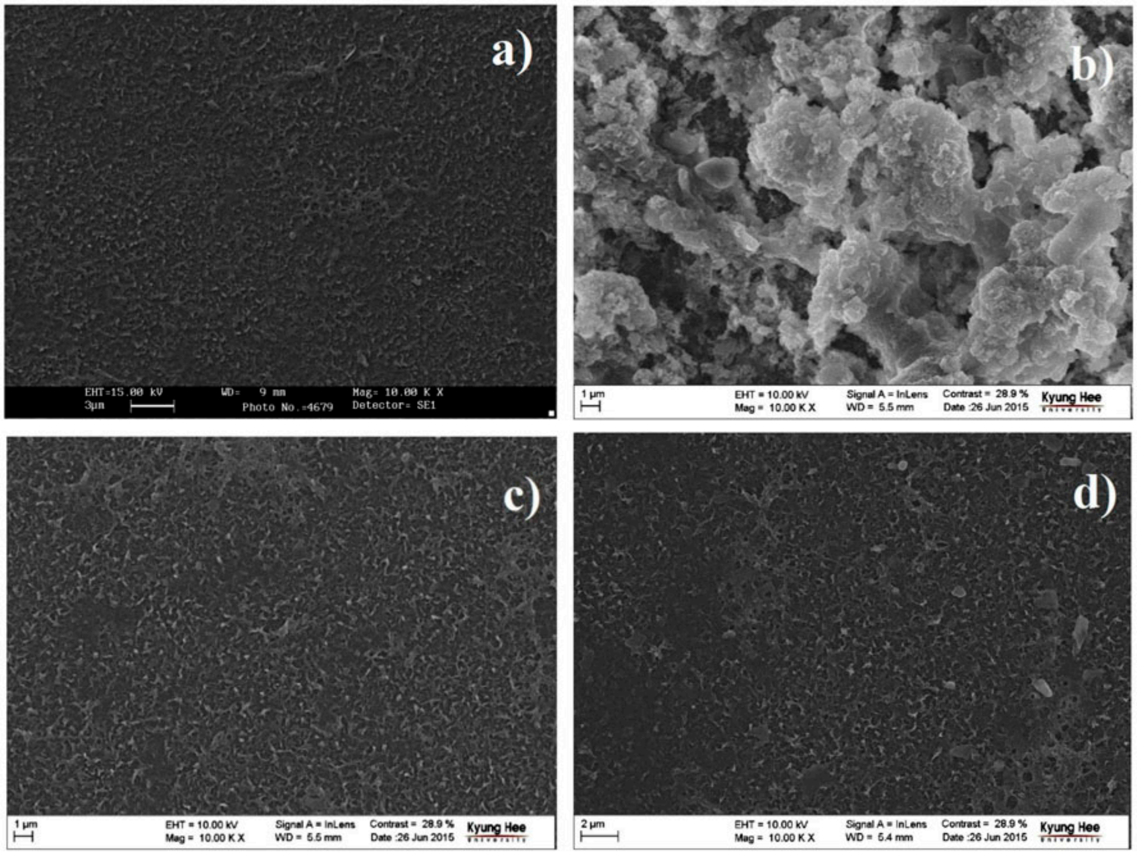

Fig. 3. SEM images of membrane samples: (a) virgin, (b) fouled, (c) after Step A, and (d) after Step B.

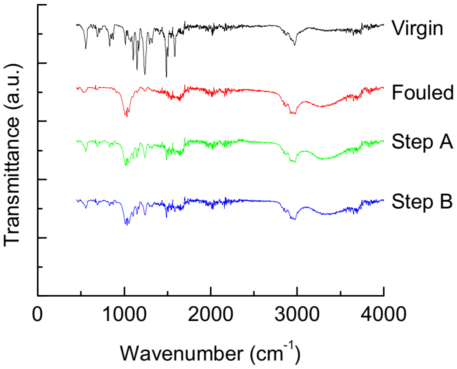

Fig. 4. FT-IR spectrum of membrane samples.

implied the successful removal of deposited foulants and the subsequent recovery of original membrane properties due to the chemical cleaning. Interestingly, characteristic peaks of C–O (1,030 cm^{-1}) and N–H (3,300 cm^{-1}) stretching did not disappear even after the Step A and Step B. It is due to the residual organic and/or colloidal matters on membrane surfaces as shown in Figs. 2 and 3. Overall, the chemical cleaning agents designed for RO process could remove significant amount of foulants deposited on FO membrane surface and could be applied for the cleaning of membranes operated in FO process.

4. Conclusions

In this study, we investigated whether chemical cleaning agents designed for RO could be applicable. It has been found that the chemical cleaning can significantly recover the original membrane performance (flux and reverse flux selectivity) of FO membranes. The foulants on the membrane surface were successfully removed due to the loose structure of fouling layer on FO membrane; thus, the membrane morphology, hydrophilicity, and surface free energy were significantly recovered after the chemical cleaning. Overall, chemical cleaning agents designed for RO could remove various membrane foulants and consequently be applied for the cleaning of membranes operated in FO process. Although about 95% of organic and colloidal matters were removed, advanced operational strategies and new chemical cleaning agents to remove residual colloidal particles should be considered for better FO system in the future.

Acknowledgment

This research was supported by a grant (code 15IFIP-B088091-02) from Industrial Facilities & Infrastructure Research Program funded by Ministry of Land, Infrastructure and Transport of Korean government.

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