

Evaluation of the efficiency of cleaning method in direct contact membrane distillation of digested livestock wastewater

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Abstract. This study investigated effects of physical and chemical cleaning methods on the initial flux recovery of fouled membrane in membrane distillation process. A laboratory scale direct contact membrane distillation (DCMD) experiment was performed to treat digested livestock wastewater with 3.89 mg/L suspended solids, 874.7 mg/L COD, 543.7 mg/L nitrogen, 15.6 mg/L total phosphorus, and pH of 8.6. A hydrophobic PVDF membrane with an average pore size of 0.22 μm and a porosity of 75 % was installed inside a direct contact type membrane distillation module. The temperature difference between feed and permeate side was maintained at 40°C with the feed and permeate stream velocity of 0.18 m/s. The results showed that the permeate flux decreased from 22.1 $\text{L}\cdot\text{m}^{-2}\cdot\text{hr}^{-1}$ to 19.0 $\text{L}\cdot\text{m}^{-2}\cdot\text{hr}^{-1}$ after 75 hours of distillation. The fouled membrane was cleaned first by physical flushing and consecutively by chemicals with NaOCl and citric acid. After the physical cleaning the flux was recovered to 92 % as compared with the initial clean water flux of the virgin membrane. Then 94 % of the flux was recovered after cleaning by 2,000 ppm NaOCl for 90 minutes and finally 97 % of flux recovered after 3 % citric acid for 90 minutes. SEM-EDS and FT-IR analysis results presented that the foulants on the membrane surface were removed effectively after each cleaning step. The contact angle measurement showed that the hydrophobicity of the membrane surface was also restored gradually after each cleaning step to reach nearly the same hydrophobicity level as the virgin membrane.

Keywords: membrane distillation; wastewater treatment; membrane cleaning; flux recovery rate

1. Introduction

Membrane distillation (MD) is a process to extract water vapor driven by the partial pressure difference between the feed and permeate side through a hydrophobic membrane (Nejati 2015, Wang 2015). The partial pressure difference is established by the temperature gradient across the membrane, which induces the movement of vapor from the feed to permeate side and accelerates phase separation (Lawson and Lloyd 1997, Curcio 2005, Alkudhiri 2012, Cmacho 2013). The

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MD process requires a lower temperature difference as compared with traditional distillation processes as well as a lower pressure difference than the reverse osmosis membrane process (Chang 2015). Additionally, a high quality of permeate can be expected because only pure water vapor passes selectively through the membrane (Zoungrana 2016). In the membrane distillation process, a strong hydrophobic membrane should be used to transport only clean water vapor but prevent the movement of water in a liquid state (Lin 2014). Water vapor passed through the membrane is condensed in the liquid state again at the low temperature permeate side. In conditions where the temperature gradient between the feed and permeate side is maintained constantly, this causes continuous vapor movements without the establishment of saturated vapor pressure inside the whole system and the condensed water at the permeate side cannot move back to the feed side (Kim 2013). Therefore, when only pollutants that are less vaporizable than water exist in feed solution, theoretically almost 100% pure water can be produced in the permeate side (Gryta 2010). Additionally, it has been reported that a permeate flux is not significantly influenced by the pollutant concentration in feed solution and thus theoretically almost no serious fouling is expected in MD process (Lawson 1997, Lagana 2000, Pangarkar 2014, Zarebskar 2014). For these reasons, there have been many attempts to apply an MD process to various fields of water treatment such as seawater desalination (Shirazi 2012, Lee 2015), recycling of water (Wu 1991, Bader 2005), the beverage industry of milk and juice manufacturing (Yun 2006, Gunko 2006, Alves 2006), as well as pharmaceutical and medical products processing (Sakai *et al.* 1986, Ding 2010). However, with contrary to the theoretical expectation a previous research showed a serious fouling could be occurred even in MD process in case the feed solution included a high concentration of suspended (Kim 2016). This study observed that high suspended solids induced a cake layer formation on the surface of membrane and consequent pore blocking, which prevented water vapor transport through membrane pores, leading to the flux decline.

And the cleaning strategy has been also studied by many researcher in membrane distillation system. For example, Wang *et al* reported that 2% HCl and 2% NaOH solution for 30 min each is used for recovering the fouled membrane using recirculating cooling water. Also, fouled membrane of membrane distillation experiment using tap water is cleaned by 5% HCl solution, polyphosphate antiscalant, and 5 different type of antiscalant solution by Gyta, and He *et al.*, respectively. Gas bubbling is studied as fouled membrane cleaning method using high salt concentration and traditional Chinese medicine (TCM) extract by Chen *et al.*, Ding *et al.*, respectively. However, in the study of MD applications, system maintenance and management study are still lacking for treating wastewater that includes a high concentration of suspended solids, organic matters, and nutrients such as nitrogen and phosphorus (Gryta. 2008, David 2015). If membrane fouling proceeds in an MD system, it has been known that a “wetting” phenomenon occurs, which results in the change of the membrane surface characteristics from hydrophobic to hydrophilic conditions and thus, the selectivity on the liquid water from gaseous water vapour is deteriorated. Therefore, studying a fouling mechanism and fouling control strategy in an MD system is still important for a sustainable maintenance of an MD system and extension of membrane life time (Lim 2003).

In this study, a physical and chemical cleaning was performed on the fouled membrane in MD process and the flux performance was evaluated after every cleaning step to analyse the contribution of each cleaning method on the total initial flux recovery. To achieve this objective, a laboratory scale direct contact membrane distillation (DCMD) experiment was performed to treat digested livestock wastewater. The permeate flux decline was observed for 3 days and clean water flux was measured for virgin, fouled, and cleaned membranes, respectively. The membrane surface

Table 1 Characteristics of feed wastewater used in this study (n=3)

Components (mg/L)				
SS	COD	T-N	T-P	pH
3.89±0.3	874.7±42.2	543.7±60.8	15.6±0.7	8.6±0.3

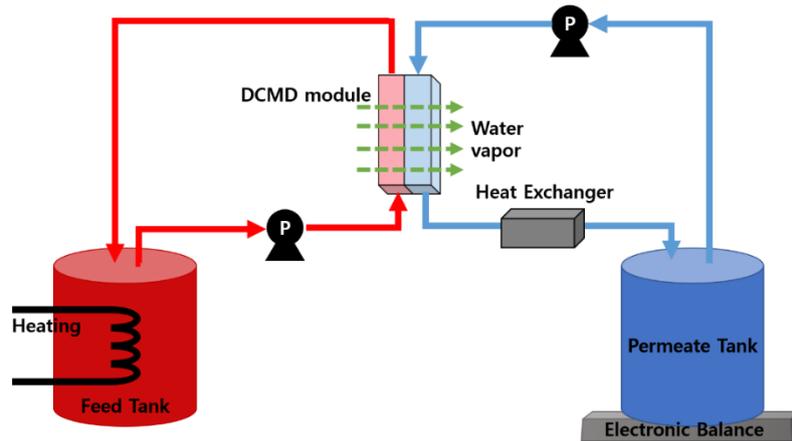


Fig. 1 Schematic of laboratory scale DCMD system

before and after cleaning was observed with the contact angle measurement, Scanning Electron Microscope (SEM)-Energy Dispersive Spectroscopy (EDS), and Fourier Transform Infrared Spectroscopy (FT-IR) analysis.

2. Materials and methods

2.1 Preparation of feed

Anaerobically digested wastewater was collected from a real plant in Gyeonggi-do province and used as a feed solution after the following pretreatment; the collected wastewater was settled for one hour and the supernatant was centrifuged at 2,000 rpm and 4°C for 15 minutes to remove debris such as animal hair, leaves, fine sand and unknown remains. The supernatant after centrifugation was again filtered through a GF/C filter with average pore of 1.2 μm. After the centrifugation and filtration the collected solution still included a high concentration of organic matters, nitrogen, phosphorous and other suspended solids. The components measured three times concentration of this study result in Table 1. as presented in Table 1. The prepared feed solution was stored in a refrigerator at 4°C.

2.2 MD system and membrane

A transparent acrylic DCMD (Direct contact membrane distillation) module was manufactured. The distance between the membrane and acrylic module is 5 mm. The inlet port and outlet port were connected to the tube using a 63.5 mm connector. A hydrophobic PVDF (polyvinylidene

fluoride) membrane (Millipore GVHP, USA) was purchased, cut to fit the size of module and placed in the middle of module. The effective area of membrane was $1.75 \times 10^{-3} \text{ m}^2$. The average pore size, thickness and porosity of separation membrane were $0.22 \text{ }\mu\text{m}$, $125 \text{ }\mu\text{m}$ and 75% respectively.

The schematic of MD system was illustrated in Fig. 1. The feed and permeate tank were positioned lower than the membrane module to prevent the establishment of hydrostatic pressure difference inside membrane module. The feed and permeate stream were circulated through the module counter-currently with the flow velocity of 0.18 m/s using pumps (Cole-Parmer console drive gear pump). The de-ionized water was used for permeate solution and the temperature was maintained at 20°C with a heat exchanger and cooling water circulation device. The feed solution was heated by a water bath to maintain a constant temperature of 60°C . Thus, the temperature difference between the feed and permeate side was 40°C .

A change in the mass of treated water (that is, the mass of permeate solution) was observed using the electronic scale (Ohaus arg4202) in every 3 minutes and the flux was calculated as

$$J = \frac{dm}{\rho \cdot A \cdot dt} \quad (1)$$

Where, J is the permeate flux ($\text{L/m}^2/\text{hr}$), m is the treated water mass (kg), ρ is the density of water according to the temperature, A is the effective area of separation membrane (m^2) and t is the distillation time (hour). And in the result and discussion part, value is indicated by average flux what measured every 3 minute of each section like clean water test, flux maintain section.

Additionally, morphological and compositional characteristics of fouled membrane surface was investigated by SEM-EDS (Hitachi S-4200, Japan), FT-IR (ThermoMattson, USA), and contact angle (KRUSS, Germany) measurements.

2.3 Membrane cleaning and flux recovery

The cleaning on the fouled membrane was performed consecutively by three different methods which included a physical cleaning to remove the cake layer formed on the membrane surface and chemical cleaning using two different chemicals to remove a residual organic and inorganic foulants after physical cleaning as shown in Table 2. In the physical cleaning, de-ionized water was used to flush the surface of fouled membranes after taken out from the module for three minutes with the flow-rate of 6 L/min . Then, the flushed membranes were taken out from the module and submerged in $2,000 \text{ mg/L NaOCl}$ solution for 90 minutes, and rinsed with de-ionized water. The membrane was subsequently submerged in 3% citric acid solution for 90 minutes and rinsed again. And flux recovery test after each step of cleaning was conducted with same DCMD system with de-ionized water.

The recovery rate of flux performance after each step of cleaning was calculated as below

Table 2 Cleaning condition used in this study

Type of fouling	Type of cleaning	Target foulants	Cleaning agent
External	Physical	Cake layer	De-ionized water
Internal	Chemical	Residual organics	$2,000 \text{ mg/L NaOCl}$
Internal	Chemical	Residual inorganics	3% citric acid

$$\text{Recovery rate of flux performance (\%)} = \frac{J_t}{J_0} \times 100 \quad (2)$$

Where J_0 is the clean water flux of the virgin membrane and J_t is the clean water flux measured instantly after each cleaning step. The contribution of each cleaning step on the flux recovery was evaluated by calculating the difference of recovery rates before and after each cleaning step.

3. Results and discussion

3.1 Permeate flux and cleaning of fouled membrane

Fig. 2(a) shows the flux decline in direct contact membrane distillation process for 75 hours. And as mentioned in the materials and methods, we maintained at 60°C on the temperature of feed by use of water bath, heat exchanger, and cooling circulator. The temperature of permeate side at 20°C was controlled by a heat exchanger and cooling circulator. As result, the temperature difference between the feed and permeate side was maintained almost constantly at 41.6±0.45°C for 3 days of distillation. The clean water flux of the virgin membrane was measured by 22.1±0.15 LMH. As membrane distillation started, the flux was maintained at 22.3±0.24 LMH for initial 24 hours but then the flux decreased gradually to reach 19.0±0.22 LMH after next 69 hours. The flux decline was caused by the cake formation with the suspended solids deposition on the membrane surface, which induced the pore blocking to hinder water vapor from transporting through the membrane pores.

The clean water flux measured after each cleaning step was 20.3±0.24, 21.1±0.20 and 21.2±0.24 LMH for a physical flushing, chemical cleaning with NaOCl, and chemical cleaning with citric acid, respectively. The calculated recovery rate of flux performance after each cleaning method were 92, 94, and 97%, respectively (Table 3). As the final recovery rate of flux performance was 97%, the irreversible fouling portion was about 3%. The contribution of chemical cleaning by NaOCl and citric acid on the recovery rate of flux performance was estimated by 2% and 3%, respectively (Fig. 2(b)), indicating that the majority of foulants could be removed just by the first physical flushing on the surface of membrane. Membrane fouling is usually classified into an external and internal fouling (Tijing 2015), and the above results implied that the external fouling was the main cause of flux decline in this experiment since the flux could be recovered to 92% by the surface flushing. Therefore, the cake layer formation on the membrane surface should be considered as significant even in membrane distillation process and this external fouling interrupted the movement of water vapor, which caused the decreasing of permeate flux

Table 3 Clean water flux and recovery rate of flux performance after each cleaning step ($n=30$)

Permeate flux	Clean water flux (L/m ² /h)	Recovery rate of flux performance
Virgin membrane	22.1±0.15	-
Fouled membrane	19.0±0.22	-
Physical cleaning	20.3±0.24	92
Physical + NaOCl cleaning	21.1±0.20	94
Physical + NaOCl cleaning + citric acid cleaning	21.2±0.24	97

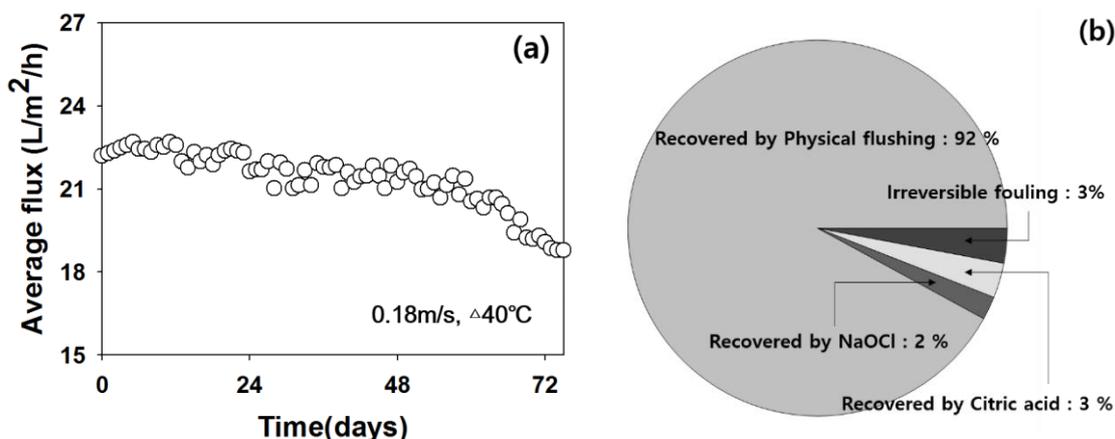


Fig. 2 (a) Flux decline for 3 days distillation and (b) contribution of each cleaning method on flux recovery

(Woo 2015). Generally external surface fouling is reversible which can be cleaned by physical cleaning. But some fouling like internal or pore blocking is irreversible because of compacting of foulants (Tijing 2015, Hoek 2008). An irreversible fouling existence means cleaning methods in this study do not completely recover the initial capacity (Al-Amoudi 2007).

3.2 Membrane surface analysis

Fig. 3 shows that the SEM images of the membrane surface after each cleaning step. As compared with the virgin membrane (Fig. 3(a)), Fig. 3(b) indicated that much of foulants was deposited to form a cake layer and covered the entire surface of the membrane. However, as the membrane cleaning proceeded physically and chemically, the cake layer was gradually removed. As the driving force of the MD system is the temperature difference, the higher temperature difference results in the higher flux in the permeate side (Yun 2006). However, even in a membrane distillation, the membrane surface could be covered with foulants (cake layer) in higher flux, which diminished the effective pore area as well as reduced the temperature gradient between the feed and permeate sides, and decreasing permeate flux (Camacho 2013).

The elementary composition of the membrane surface was analyzed by EDS (Fig. 4). The numerous inorganic matters such as C (66.6%), O (25.1%), Mg (0.4%), Si (0.5%), P (2.4%), S (1.0%), Ca (3.5%), and Fe (0.6%) were accumulated on the fouled membrane (Fig. 4b), while the virgin membrane was composed of only C (56.3%) and F (43.7%) (Fig. 4(a)). The analysis results showed that the inorganic and non-volatile matter in the feed solution was deposited on the membrane surface as distillation proceeded. Additionally, the foulants on the fouled membrane surface were effectively removed by each step of cleaning. Especially, Ca, which is known as a major component of inorganic scaling (Rahimpour 2009) was removed significantly after the chemical cleaning with citric acid, which indicated that citric acid was very effective to remove an inorganic foulant of potential scaling formation in the membrane distillation process (Warsinger *et al.* 015).

Fig. 5 presents the FT-IR analysis of the virgin membrane, fouled membrane, the membrane after physical cleaning, the membrane after chemical cleaning using 2000 mg/L NaOCl, and the membrane after chemical cleaning using 3% citric acid. In comparison with the virgin membrane,

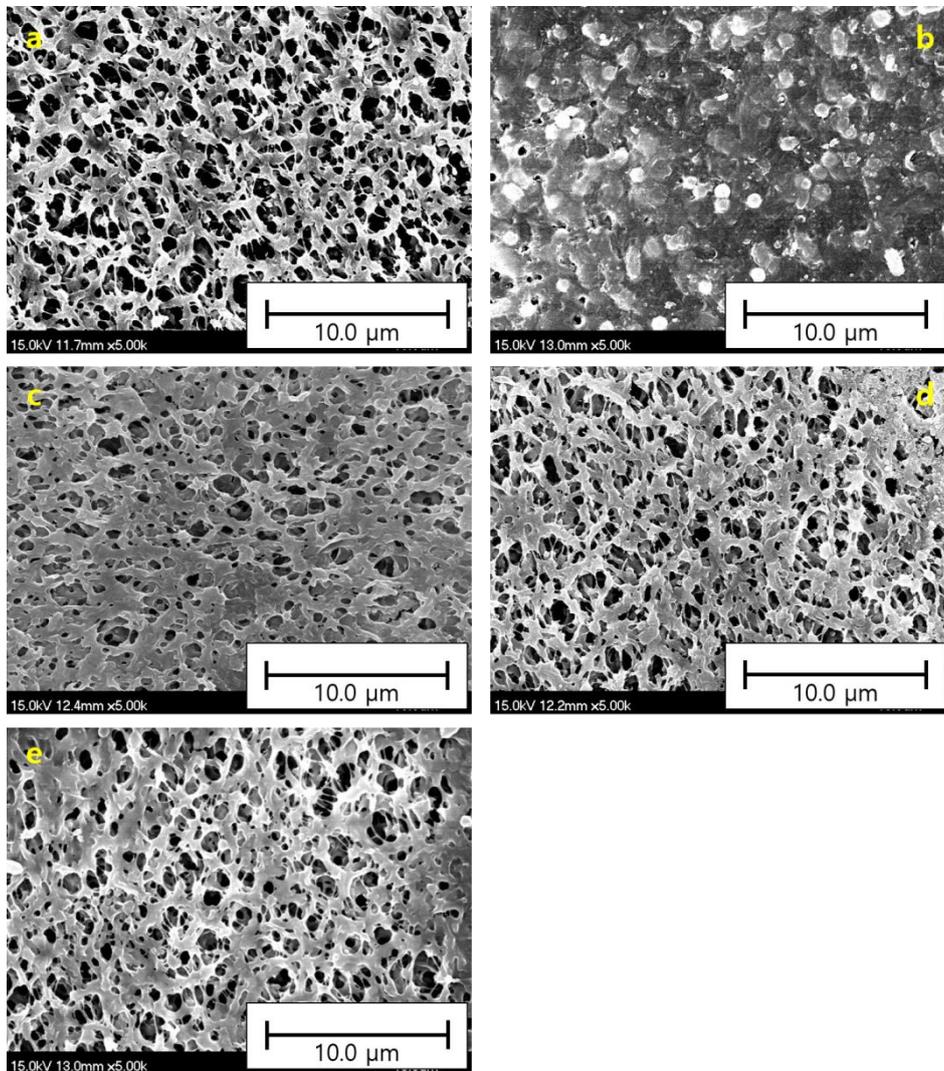


Fig. 3 SEM images on the surface of (a) virgin membrane, (b) fouled membrane (c) after physical cleaning (d) after chemical cleaning using 2000 mg/L NaOCl (e) after chemical cleaning using 3% citric acid

the fouled membrane showed a clear peak at 3300 cm^{-1} and 3000 cm^{-1} . This means that N-H group and C-H group was built up at the membrane surface due to fouling (Jackson 1995). Also, the peak at 1650 cm^{-1} , 1540 cm^{-1} and 1050 cm^{-1} was observed, indicating that C=O, N-H and C-O group form the cake layer respectively. The membrane after physical cleaning showed nearly similar peaks with the virgin membrane. These results implied that the physical cleaning was considerably effective in removing the organic matter in the cake layer formed on the surface of the membrane.

Fig. 6 shows the contact angle measurement on the membrane surface after each cleaning step. The contact angle of the virgin membrane was measured by 119.9° , indicating the virgin membrane had a strong hydrophobicity. While the fouled membrane surface showed the contact angle of 50.2° , which indicated that the membrane surface was changed to be hydrophilic due to

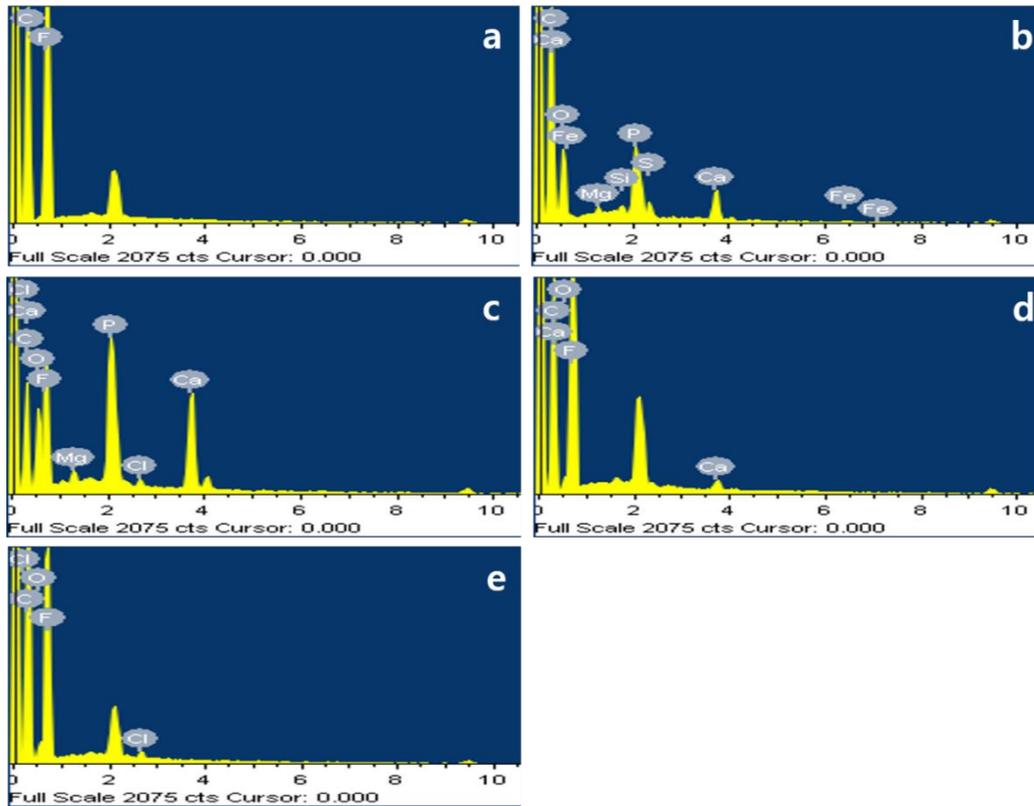


Fig. 4 Fouling components analysis by EDS: (a) Virgin membrane, (b) Fouled membrane (c) Membrane after physical cleaning (d) Membrane after chemical cleaning using 2000mg/L NaOCl (e) Membrane after chemical cleaning using 3% citric acid

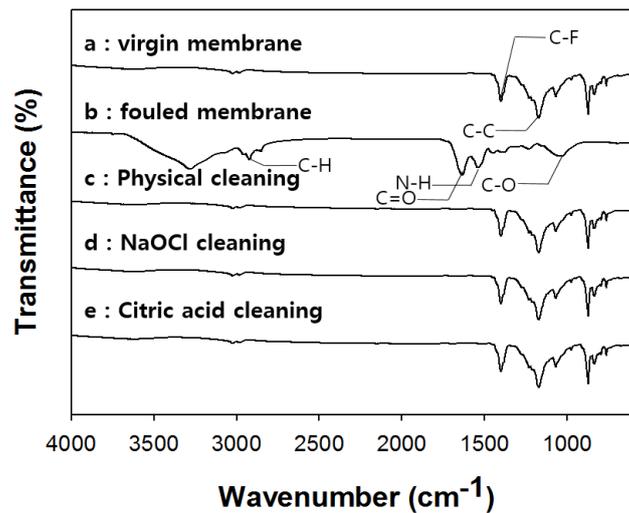


Fig. 5 FT-IR analysis after each cleaning step: (a) Virgin membrane, (b) Fouled membrane (c) Membrane after physical cleaning (d) Membrane after chemical cleaning using 2000 mg/L NaOCl (e) Membrane after chemical cleaning using 3% citric acid

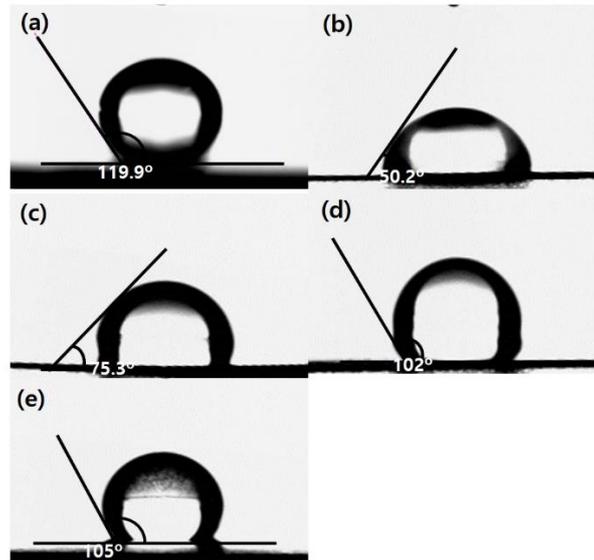


Fig. 6 Contact angle measurement after each cleaning step: (a) Virgin membrane, (b) Fouled membrane (c) Membrane after physical cleaning (d) Membrane after chemical cleaning using 2000 mg/L NaOCl (e) Membrane after chemical cleaning using 3% citric acid

the cake layer formation as membrane fouled. With the hydrophilic surface of membrane, a wetting phenomenon could occur. However, in this study the contact angle was measured directly for the fouled surface of membrane, which just become an indication of hydrophobicity and hydrophilicity of the cake layer (foulants). And the ultimate change of hydrophobicity on membrane surface should be discussed by measuring the contact angle of membrane surface after the cake layer was removed. As noted in Fig. 6, the contact angle of fouled membrane was restored after each cleaning step to the level of hydrophobicity.

5. Conclusions

In this study, physical and chemical cleaning was performed on the fouled membrane in the membrane distillation process and the cleaning efficiency was evaluated by analyzing the flux recovery rates after each cleaning step. A laboratory scale direct membrane distillation process was applied to treat an anaerobically digested wastewater for 3 days with a constant temperature difference of 40°C between the feed and permeate sides.

The fouled membrane was cleaned by physical flushing using de-ionized water, and then consecutively by the chemical cleaning with 2,000 mg/L NaOCl and 3% citric acid for 90 minutes, respectively. After the physical cleaning the flux was recovered by 92% as compared with the initial clean water flux of the virgin membrane, which indicated that the majority of the cake layer on the membrane surface could be removed. The contribution of NaOCl and citric acid on the total flux recovery was evaluated by 2 and 3%, respectively. The irreversible fouling was finally estimated to be 3%. The SEM-EDS and FT-IR analysis results implied that the foulants on the membrane surface were removed effectively after each cleaning step. The contact angle

measurement also presented that the hydrophobicity of the membrane surface was restored gradually to the same extent as the virgin membrane after each cleaning step.

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