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Seawater-driven forward osmosis for direct treatment of municipal wastewater

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Abstract. Direct treatment of municipal wastewater by forward osmosis (FO) process was evaluated in terms of water flux decline, reverse salt diffusion, pollutants rejection and concentration efficiency by using synthetic seawater as the draw solution. It was found that when operating in PRO mode (active layer facing the draw solution), although the FO membrane exhibited higher osmotic water flux, more severe flux decline and reverse salt diffusion was also observed due to the more severe fouling of pollutants in the membrane support layer and accompanied fouling enhanced concentration polarization. In addition, although the water flux decline was shown to be lower for the FO mode (active layer facing the feed solution), irreversible membrane fouling was identified in both PRO and FO modes as the water flux cannot be restored to the initial value by physical flushing, highlighting the necessity of chemical cleaning in long-term operation. During the 7 cycles of filtration conducted in the experiments, the FO membrane exhibited considerably high rejection for TOC, COD, TP and NH_4^+-N present in the wastewater. By optimizing the volume ratio of seawater draw solution/wastewater feed solution, a concentration factor of 3.1 and 3.7 was obtained for the FO and PRO modes, respectively. The results demonstrated the validity of the FO process for direct treatment of municipal wastewater by using seawater as the draw solution, while facilitating the subsequent utilization of concentrated wastewater for bioenergy production, which may have special implications for the coastline areas.

Keywords: forward osmosis (FO); municipal wastewater; seawater; membrane fouling

1. Introduction

Environmental pollution and water resource shortage have become two of the most significant problems that faced on a global scale. High pressure driven membrane processes such as reverse osmosis (RO) and nanofiltration (NF) can produce cleaner and safer water from non-conventional water resources, including desalination of seawater and saline water, reuse of treated wastewater,

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purification of impacted surface water, etc. (Do *et al*. 2012, Wu *et al.* 2013, Zheng *et al.* 2014). However, the efficiency and sustainable operation of RO and NF has been limited by the considerable energy consumption and severe membrane fouling induced by the high hydraulic pressure (Boo *et al.* 2012, Menachem Elimelech and Phillip 2011, Motsa *et al.* 2014).

Recently, forward osmosis (FO) has been emerging as an innovative platform technology to address the above-mentioned issues (Kong *et al.* 2015, Liu *et al.* 2013, Shaffer *et al.* 2015, Zhang *et al.* 2015, Zhao *et al.* 2015). Instead of the applied hydraulic pressure, FO employs the osmosis pressure difference between the feed solution and draw solution as the driving force to induce water transport across a semipermeable membrane. As a result, the FO technology exhibits several distinguished advantages such as high rejection of pollutants, i.e. organic micro-pollutants and heavy metal ions, low energy consumption, low fouling propensity, and ease of cleaning (Cui *et al.* 2014, Cui *et al.* 2016, Razmjou *et al.* 2013, Wang *et al.* 2015, Widjojo *et al.* 2013, Xia *et al.* 2015, Zhang *et al*. 2012a). And the FO process has shown excellent application potentials in various field such as seawater/saline water desalination, oily wastewater, shale gas produced water reclamation, landfill leachate treatment, and municipal wastewater treatment. (Dong *et al.* 2014, Phuntsho *et al.* 2013, Qi *et al.* 2015, Shaffer *et al.* 2013, Sun *et al.* 2016, Valladares Linares *et al*. 2012, Zhang *et al.* 2014b). Previous researches have proposed a seawater-driven FO strategy for processing municipal wastewater, and have demonstrated the feasibility for enriching nitrogen and phosphorous in municipal wastewater (Xue *et al.* 2015, Xue *et al.* 2016). Simultaneously, the diluted seawater can be re-discharged to the ocean or used for clean water production via RO, while the concentrate can be used for energy production via anaerobic digestion (Zhang *et al.* 2014c).

On the other hand, along with the increasingly growing demand for clean water all over the world, there is also the corresponding increase in the amount of wastewater needed to be properly treated (Valladares Linares *et al.* 2013). At present, the treatment of municipal wastewater is mainly performed through aerobic biological processes where intensive air bubbling is required. Two problems are associated with these processes: (1) relatively high energy consumption is required for these processes, it is estimated that 0.4-2.1 kW/h of electricity power will be demanded for treating 1 m^3 of wastewater (Zhang 2014c); (2) there are still some kind of pollutants present in the wastewater secondary effluents, such as refractory organics, nitrogen, phosphorous, heavy metals, etc., which may still cause the contamination of receiving water bodies (Valladares Linares *et al*. 2013).

It is reported that today more than 3 billion people live along the coastlines and this number is continuously growing (Valladares Linares *et al*. 2013). As a result, there is an ever-increasing pressure for the sufficient treatment of municipal wastewater to meet the discharge regulation for protection of marine environment. Therefore, it might be a fantastic idea if the FO process can be employed for direct and effective wastewater treatment by using seawater as the natural, abundant and costless draw solution, as suggested by Linares *et al*. (2013), Zhang *et al*. (2014c). In which, the diluted seawater can be discharged back to the ocean, while the concentrated wastewater can be used for biogas production through anaerobic digestion.

In this work, the performance of a commercial cellulose triacetate (CTA) FO membrane for the treatment of municipal wastewater was evaluated with real municipal wastewater as the feed solution and synthetic seawater as the draw solution. The osmotic water flux decline, reverse salt diffusion, pollutants rejection and concentration efficiency of wastewater during the FO process were assessed systematically. The results of this work may provide some insights to the further development of FO-based wastewater treatment technology.

Table 1 Characteristics of the real domestic wastewater

Parameters	Concentrations	
	FO mode	PRO mode
Conductivity (μS/cm)	884.9±42.1	863.0±38.0
TOC (mg/L)	69.4±17.5	80.0±11.7
COD (mg/L)	223.1±44.8	224.6±25.3
TP (mg/L)	8.15±1.04	9.62±1.18
NH_4^+-N (mg/L)	47.0±2.74	47.3±3.62

Table 2 Chemical composition of the synthetic seawater

Salts	Concentration (g/L)	Mass proportion (%)
NaCl	26.73	77.64
$MgCl_2 \cdot 6H_2O$	2.26	6.57
$MgSO_4 \cdot 7H_2O$	3.248	9.435
$CaCl_2$	1.153	3.35
KCl	0.721	2.094
$NaHCO_3$	0.198	0.575
NaBr	0.058	0.168
H_3BO_3	0.058	0.168
Na_2SiO_3	0.0024	0.00697
$Na_2Si_4O_9$	0.0015	0.00473

2. Materials and methods

2.1 Feed and draw solutions

In this work, real municipal wastewater collected from a wastewater pipe of a residential community with a pH of 6.5~7.8 was used as the feed solution of the FO process without any pretreatment. The characteristics of the municipal wastewater were shown in Table 1. For the draw solution, ten inorganic salts were used to prepare the synthetic seawater, as shown in Table 2. This synthetic seawater had an electric conductivity of 47.1±4.05 mS/cm and a pH of 7.70~7.72 at ~20℃.

2.2 Experimental set-up

The FO experiments were carried out with a bench-scale experimental set-up as shown in Fig. 1. The cross-flow membrane cell was a plate-to-frame design, which can accommodate an effective membrane area of 11.2 cm^2. A commercial CTA FO membrane was obtained from Hydration Technology Innovations (HTI) and used in the work. The membrane has an asymmetric structure consisting of a dense active layer and a porous supporting layer embedded in a polyester mesh (Zhang *et al.* 2014c). Thus, two membrane orientations were assessed for the FO process, i.e., the active layer facing the feed solution side (FO mode) and active layer facing the draw solution side (PRO mode). Two peristaltic pumps (Longer, China) were used to continuously recirculate the draw solution and feed solution in the lumen spaces of the membrane cell in a co-current mode.

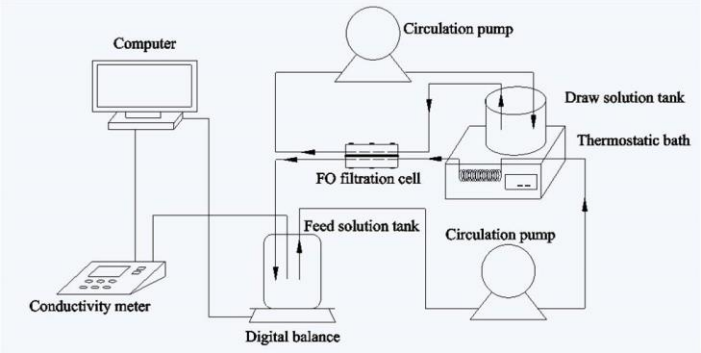


Fig. 1 Schematic diagram of the laboratory-scale FO system for the 7 cycles of filtration

To assess the water flux decline, reverse salt diffusion and pollutants rejection during the FO separation of municipal wastewater, a total of 7 cycles of FO filtration experiments were carried out with 10 h for every cycle. The experiments were started with 2 L of municipal wastewater in the feed solution tank and 2 L of synthetic seawater in the draw solution tank. The feed solution tank was placed on a digital balance (Denver, USA) connected to a computer, and the weight variation of feed solution was measured for calculation of FO membrane water flux. The cross-flow velocity was maintained at 10 cm/s at each side of the FO membrane. The temperature of the feed solution and draw solution was kept at 20±1℃ by using a water bath. At the end of each filtration cycle, the FO membrane was thoroughly flushed with DI water to remove the pollutants accumulated on membrane surface, and then the membrane was soaked in DI water until the next cycle of FO filtration. The concentrated feed solution and diluted draw solution were replaced by fresh municipal wastewater and synthetic seawater respectively for each cycle of the FO filtration experiments.

To assess the concentration efficiency of the seawater-driven FO process for municipal wastewater, the initial volume of feed solution was purposely decreased to 180 mL while other conditions were maintained as the same as mentioned above.

2.3 Analytical methods

Water samples were collected from the FO system after each cycle. Ammonia nitrogen (NH_4^+-N), total phosphorus (TP) and Chemical Oxygen Demand (COD) were detected on the basis of the Chinese NEPA standard methods (CEPB 2002).Total organic carbon (TOC) concentration was measured by TOC analyzer (TOC-5000A, Shimadzu, Japan). The individual reverse salt concentration of Na^+, K^+, Ca^{2+} and Mg^{2+} were analyzed by Inductively Coupled Plasma (Optima 5300 CV, Perkin Elmer Inc).

The osmotic water flux during FO process was calculated from the weight change of the feed solution

$$J_w = \frac{\Delta m}{\rho \Delta t A_m} \tag{1}$$

Where J_W is the FO water flux (L/m^2·h); Δm is the weight change of the feed solution (g) over a predetermined time Δt (h); ρ is the density of permeated water from the feed solution to the draw

solution (approximately 1000 g/L); A_m is the effective membrane surface area (m^2).

The individual reverse solute flux (J_s, g m^{-2} h^{-1} or gMH) of Na^+, K^+, Ca^{2+} and Mg^{2+} from the draw solution to the feed solution was calculated using the following equation

$$J_s = \frac{C_t V_t - C_0 V_0}{\Delta t \cdot A_m} \quad (2)$$

where C_0 (mol L^{-1}) and V_0 (L) are the initial salt concentration and volume of the feed solution, respectively; C_t (mol L^{-1}) and V_t (L) are the salt concentration and volume of the feed solution after filtration with a set time period (Δt) respectively; A_{eff} is the effective membrane surface area (m^2), and Δt is the filtration duration (h).

The conductivity of the feed solution was monitored with a conductivity meter connecting to a computer in order to observe the conductivity change with the extension of filtration time. Then the reverse salt flux was estimated by Eq. (2) after converting the conductivity change to salt concentration change. It should be noted that because Na^+ was found to be the major ion species that reversely diffused from draw solution to feed solution (Section 3.2), for simplifying the calculation the conductivity change in feed solution was converted to the reverse diffusion of NaCl.

The rejection efficiencies of the FO membrane for different pollutants in the feed solution were calculated by Eq. (3)

$$R(\%) = (1 - \frac{C_{t,DS} V_{t,DS}}{C_{0,FS} V_{0,FS}}) \times 100\% \quad (3)$$

Where R is the pollutant rejection efficiency; $C_{t,DS}$ and $V_{t,DS}$ are the pollutant concentration in the draw solution and the volume of draw solution at the end of each FO filtration cycle, respectively; $C_{0,FS}$ and $V_{0,FS}$ are pollutant concentration in the feed solution and the volume of feed solution at the beginning of each FO filtration cycle, respectively.

3. Results and discussion

3.1 Water flux decline

The osmotic water flux patterns of the FO membrane for direct treatment of municipal wastewater were shown in Fig. 2. Generally, the PRO mode exhibited higher water flux than the FO mode, especially for the first 4 cycles. This observation was in consistence with the results of previous studies on FO membrane (Valladares Linares *et al*. 2013, Zhang *et al*. 2014c), which can be explained by the difference in dilutive concentration polarization of the synthetic seawater draw solution between the two modes. In PRO mode, the dilution of draw solution was caused by external concentration polarization (ECP) on the active layer side of the FO membrane; while in FO mode, the draw solution was in contact with the porous support layer and thus internal concentration polarization (ICP) occurred (Aydiner 2015, Phuntsho *et al*. 2013). It has been well documented that the ICP causes a more severe decrease of the effective osmosis pressure than ECP, resulting a lower membrane flux (McCutcheon and Elimelech 2006, Zhang *et al*. 2014a, Zhao *et al*. 2012).

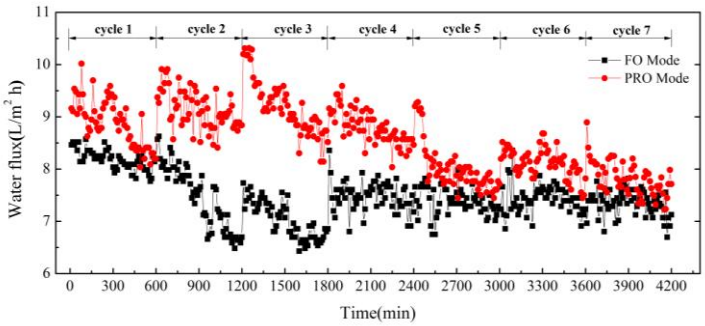


Fig. 2 Water flux patterns for the FO membrane in FO and PRO modes during the 7 cycles of filtration

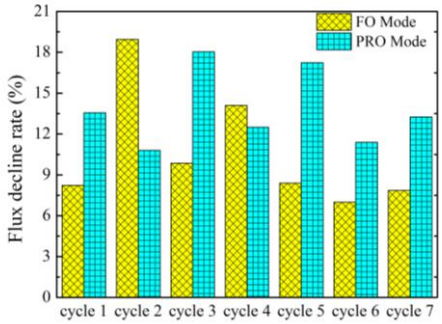


Fig. 3 Water flux decline rate of the FO membrane in FO and PRO modes during the 7 cycles of filtration (calculated as the ratio of average flux for the last hour to that for the first hour in each filtration cycle)

From Figs. 2 and 3, it could also be seen that the membrane flux in each filtration cycle decreased gradually for both the FO and PRO modes. The flux decline might be attributed to three reasons. Firstly, the dilution of draw solution during the FO process caused a decrease of osmotic pressure driving force, the dilution ratio for the 7 filtration cycles was calculated as 4~4.6% for FO mode and 4.4~5.1% for PRO mode. Secondly, an increase of salinity in the feed solution of each cycle was observed as indicated by the conductivity increase (Fig. 4), which might be due to the concentration of municipal wastewater and the reverse salt diffusion during the FO process. However, the increased conductivity was still shown to be negligible as compared with that of the synthetic seawater (47.1±4.05 mS/cm). As in the whole filtration of 7 cycles, the reverse salt flux exhibited stable in FO mode, but gradual decrease in PRO mode. And the feed solution (real municipal wastewater) and draw solution (synthetic seawater) were replaced with fresh ones after each cycle of the FO filtration experiments. Thus, the membrane fouling occurred on the FO membrane due to the accumulation of pollutants in municipal wastewater and the accompanied fouling enhanced concentration polarization might also contribute to the water flux decline (Boo *et al.* 2013, She *et al.* 2012). As shown in Fig. 5, relatively slight decrease in average flux during the 7 filtration cycles was observed for the FO mode. This was because the pollutants in municipal wastewater accumulated on the smooth surface of the membrane active layer in the FO mode, which can be easily removed by the shearing effect of the cross flow. By contrast, more

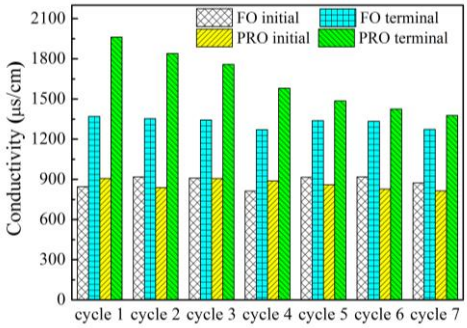


Fig. 4 Variation of conductivity in the municipal wastewater in FO and PRO modes during the 7 cycles of FO membrane filtration

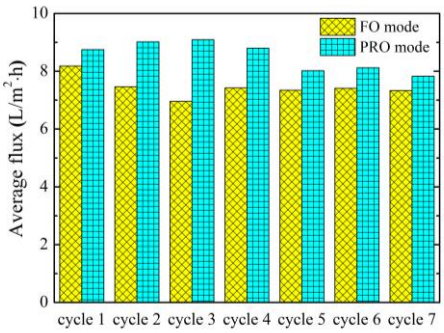


Fig. 5 Average water flux in FO and PRO modes for the 7 cycles of filtration

pronounced average flux decrease was observed for the PRO mode. During the operation in PRO mode, the pollutants in municipal wastewater can transport into the support layer along with the permeation water flow from feed solution to draw solution, which then accumulated in the porous support layer and formed internal membrane fouling. It was noted that the support layer fouling cannot be eliminated by flushing the membrane surface due to the trapping effect of the porous support layer on accumulated foulants. As a result, severe decrease in membrane flux was observed for the PRO mode.

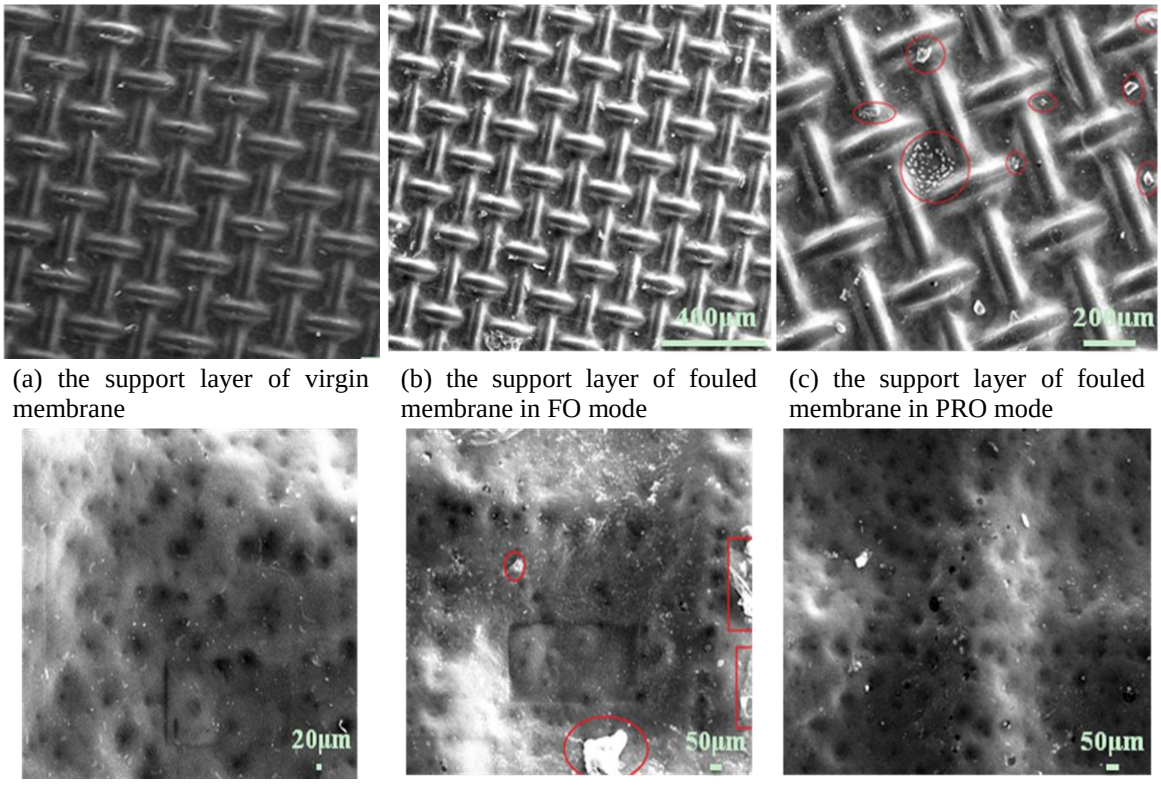
Another observation was that for both FO and PRO modes, the membrane permeability cannot be restored to the initial level after physical flushing, i.e., irreversible membrane fouling had developed on the FO membrane during the 7 cycles of filtration. As shown in Fig. 6, the surface of membrane active layer operated in FO mode was obviously covered by a gel layer, which cannot be removed by the physical flushing conducted at the end of each filtration cycle. By atomic force microscopy analysis (Fig. 7), it was found that the surface roughness of the active layer in FO mode was significantly increased (R_q=23 nm, R_a=17.4 nm) as compared with the new membrane (R_q=4.53 nm, R_a=3.57 nm). On the contrary, the active layer surface of the membrane in PRO mode was shown to be rather clean, something like that of the new membrane. It had been expected that for the PRO mode, the surface of support layer might have been deposited with large amounts of foulants. However, the opposite results was observed from the SEM images (Fig. 6), might be due to that the fouling mainly occurred in the interior of the porous support layer, while

the foulants on the surface was readily eliminated by the cross flow and the physical flushing. The formation of irreversible membrane fouling had also been noticed in a previous FO study for post-treatment of MBR-treated landfill leachate (Dong *et al*. 2014), even though it was claimed that the FO membrane fouling was largely reversible due to the lack of applied hydraulic pressure (Boo *et al*. 2013, Valladares Linares *et al*. 2013), highlighting the necessity of periodical chemical cleaning for the FO membrane during long-term operation.

3.2 Reverse salt diffusion

Due to that the Na^+, K^+, Ca^{2+} and Mg^{2+} were the main ion species present in the synthetic seawater draw solution, the reverse diffusion behaviors of these four ions were evaluated in both FO and PRO mode, as shown in Fig. 8. The most severe reverse diffusion from the draw solution to feed solution had been observed for Na^+ with a cycle-average flux of 3.41 $g/m^2 \cdot h$ in FO mode and 8.87 $g/m^2 \cdot h$ in PRO mode, respectively, followed by K^+ with the cycle-average fluxes of 0.42 $g/m^2 \cdot h$ in FO mode and 0.72 $g/m^2 \cdot h$ in PRO mode, respectively. This was due to the relatively large mass proportion of Na^+ and K^+ in the simulated seawater and the small hydrated ionic radius as compared with the divalent Ca^{2+} and Mg^{2+}.

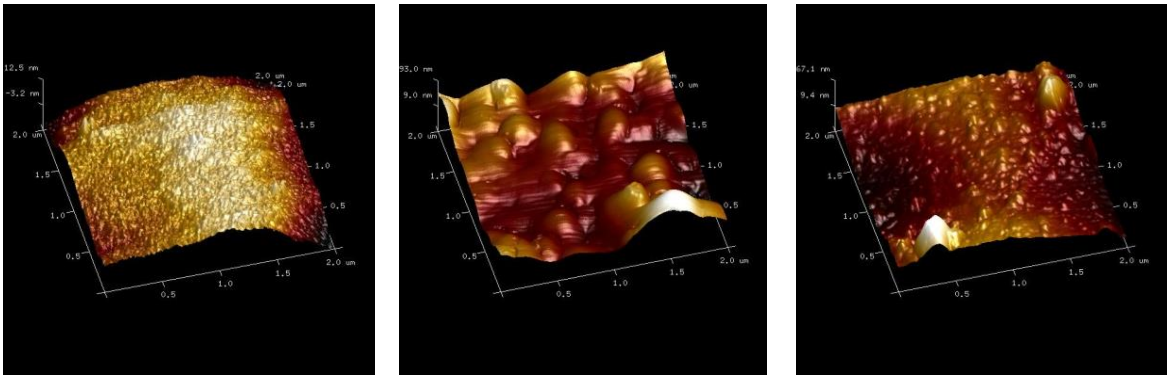
In addition, the reverse solute flux in FO mode during the 7 cycles of filtration was shown to be



(a) the support layer of virgin membrane (b) the support layer of fouled membrane in FO mode (c) the support layer of fouled membrane in PRO mode

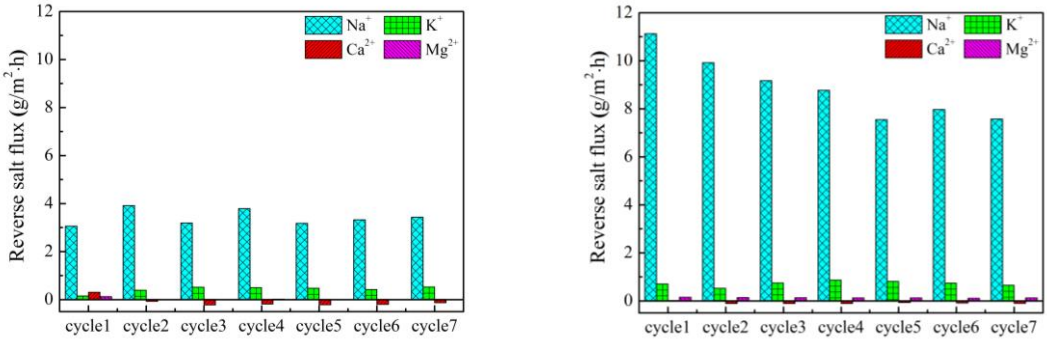
(d) the active layer of virgin membrane (e) the active layer of fouled membrane in FO mode (f) the active layer of fouled membrane in PRO mode

Fig. 6 SEM images of the active layer and support layer of virgin and fouled FO membrane surface



(a) virgin membrane (b) fouled membrane in FO mode (c) fouled membrane in PRO mode

Fig. 7 AFM images of the active layer of the virgin and fouled FO membrane.



(a) in FO mode (b) in PRO mode

Fig. 8 Reverse solute flux of Na^+, K^+, Ca^{2+} and Mg^{2+} ions during the 7 filtration cycles: (a) FO mode; (b) PRO mode

rather stable, while a steady decrease was identified in the PRO mode as illustrated in Fig. 8. This trend can be observed more obviously in the reverse salt flux of NaCl in the two modes by converting from conductivity changes (Fig. 9), which were shown in some kind of consistence with the water flux decline as exhibited in Fig. 2. In PRO mode, the reversely diffused salts was trapped in the cake layer formed in the porous support layer of the membrane, causing an increase in the osmosis pressure as well as the viscosity of the cake layer, which in turn hindered the transport of both the ions and pollutants out of the porous support layer. The cake enhanced concentration polarization reduced the effective osmotic pressure difference across the active layer of the FO membrane (Boo *et al*. 2012, She *et al*. 2012), thus decreasing both the water permeation flux and reverse salt flux across the membrane. Moreover, the reverse diffusion of salts from the draw solution into the feed solution could also increase the salinity of the concentrated municipal wastewater, adversely impacting the subsequent anaerobic digestion for biogas production (Zhang *et al.* 2012b). Further development of high performance FO membrane is required to address this issue from the viewpoint of practical applications of the FO process in wastewater treatment.

It was also noted that for the divalent cation of Ca^{2+}, a forward flux from the feed solution to

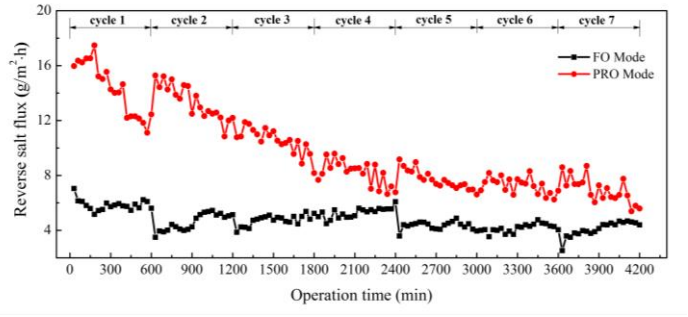
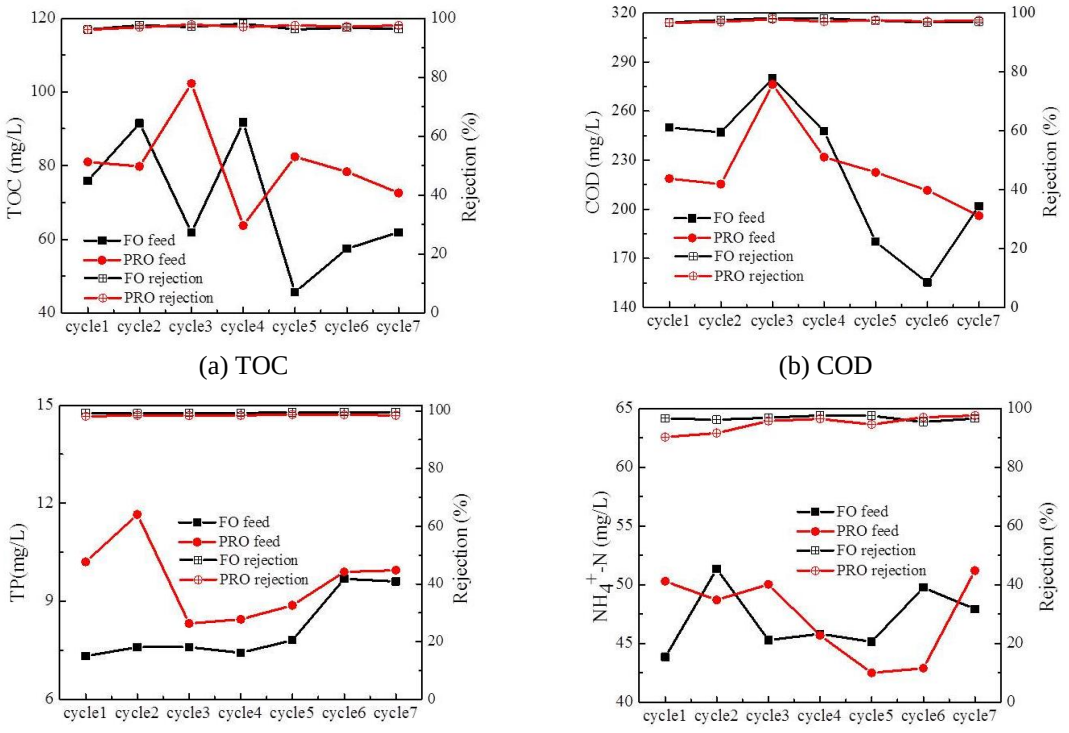


Fig. 9 Reverse salt flux of NaCl by converting the conductivity change with the extension of 7 cycles filtration in FO and PRO modes



(a) TOC (b) COD

(c) TP (d) NH_4^+-N

Fig. 10 The performance for pollutants rejection in FO and PRO modes during the 7 cycles of filtration: (a) TOC; (b) COD; (c) TP; (d) NH_4^+-N

the draw solution rather than the reverse diffusion was observed based on the calculation of eq. (3). However, it was believed that the CTA FO membrane should have a nearly complete rejection of the divalent Ca^{2+} ions. The Ca^{2+} in the municipal wastewater had a high propensity to form bridges between different pollutants as well as between the pollutants and membrane surface (Motsa *et al.* 2014, Tian *et al.* 2013), and the free Ca^{2+} transported to the membrane surface with the permeation water flow can also be readily accumulated in the cake layer, leading to a

significant decrease of Ca^{2+} concentration in the bulk of the feed municipal wastewater. This should be responsible for the observed forward flux of Ca^{2+} during the FO process.

3.3 Rejection of pollutants

Fig. 10 showed the rejection of various pollutants by the FO membrane during the 7 cycles of filtration experiments. No obvious difference for the rejection of TOC, COD and TP was observed between the FO mode and PRO mode. And a rejection rate of TOC and COD generally higher than 97% was achieved. Moreover, the FO exhibited higher rejection for TP about 99.4% in FO mode and about 98.5% in PRO mode. As for NH_4^+-N, a constant rejection with an average rejection rate of 96.6% was achieved in FO mode, which was caused that the active layer was facing the feed solution. In PRO mode, a slight increase in the rejection rate from 90.2% to 97.5% can be identified. It might be due to that the fouling layer formed in the porous support layer enhanced the retention of NH_4^+-N. And owing to the property of FO membrane, the rejection of NH_4^+-N was slightly lower than that of TOC, COD and TP. From the results, it could be concluded that the FO process can achieve excellent rejection for both organic and inorganic pollutants present in the feed solution, guaranteeing the effective treatment of municipal wastewater for protection of marine environment when using the synthetic seawater as draw solution.

3.4 Concentration factor

In the above-mentioned experiments for assessing the water flux decline, reverse salt diffusion and pollutants rejection during the FO process, 2 L of the municipal wastewater and 2 L of the synthetic seawater were used as the feed solution and draw solution, respectively. As a result, only a low concentration ratio of the municipal wastewater was obtained in the FO process (1.042~1.048 for FO mode and 1.046~1.054 for PRO mode in different cycles). However, for effective utilization of the FO concentrated municipal wastewater as a source for biogas production, it is a prerequisite to concentrate the pollutants, especially the organic substances in the municipal wastewater to a proper concentration. Therefore, concentration efficiency of the FO process for the municipal wastewater was evaluated by using 180 mL of municipal wastewater feed solution and 2 L of simulated seawater draw solution (Fig. 11). It could be seen that the volume of the municipal wastewater decreased gradually in both the FO and PRO modes. The

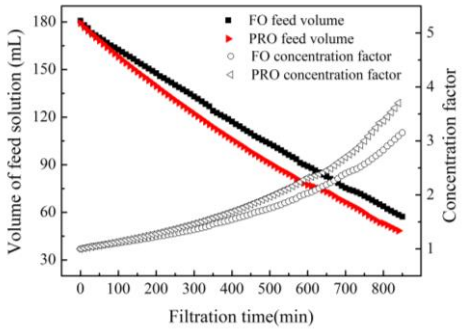


Fig. 11 Volume changes of feed solution and concentration factor as a function of filtration time in FO and PRO modes

PRO mode exhibited relatively higher concentration efficiency as compared with FO mode owing to the higher osmotic water flux of the membrane operated in PRO mode. The concentration experiments were stopped after 14 h of operation due to the significant depletion of feed solution. At the end of the process, a concentration factor of 3.1 and 3.7 was observed for the FO and PRO modes, respectively. This implied that the effective concentration of municipal wastewater can be achieved by the FO process via changing the volume ratio of draw solution and feed solution. Further studies are still required as for increasing the FO concentration efficiency and optimizing the concentration process.

4. Conclusions

In this work, the FO process for the direct treatment of municipal wastewater with synthetic seawater as the draw solution had been investigated, and the following conclusions could be made:

- Generally higher osmotic water flux can be achieved by operating the FO membrane in PRO mode. However, more severe flux decline and reverse salt diffusion were also witnessed for the PRO mode as compared with that for FO mode.
- Irreversible membrane fouling had been observed for the FO membrane in both FO and PRO modes, which cannot be removed by the physical flushing, highlighting the importance of periodic chemical cleaning during long-term operation.
- Na^+ was found to be the major ion species that reversely diffused from the simulated seawater draw solution to the municipal wastewater feed solution, maybe due to its abundance in the simulated seawater and relatively small hydraulic ionic radius.
- The FO membrane exhibited high rejection for TOC, COD, TP and NH_4^+-N; and by optimizing the process conditions such as volume ratio of draw/feed solution and operation time, a high concentration factor can be obtained.

Overall, the FO process may be taken as a new technology for effective treatment and utilization of municipal wastewater, especially for the coastline areas, where the seawater can be employed (Kong *et al.* 2015) as an inexhaustible, costless source of draw solution (Hickenbottom *et al*. 2013).

Acknowledgements

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