Processing Municipal Wastewaters by Forward Osmosis using CTA Membrane

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Abstract

Direct sewage filtration by forward osmosis (FO) was investigated with the aim of concentrating organic matters in sewage into a small volume of energy source. The results showed that chemical oxidation demand (COD) in the feed sewage solution was concentrated by more than 300%. Although a gradual decline in membrane flux with filtration time occurred, a flux of 3-7.4 L m\textsuperscript{-2} h\textsuperscript{-1} was still produced satisfactorily. The membrane flux decline was caused by both membrane fouling and the decline of osmotic driving force due to the salinity change in both feed solution and draw solution. The membrane fouling analysis indicated that the fouling was mainly attributed to the formation of cake layer on the membrane surface in both membrane orientation, the active layer facing the feed side (AL-FS) and the active layer facing the draw solution side (AL-DS). However, AL-FS outperformed AL-DS in terms of membrane flux and fouling. This study may offer new insight into the development of low-energy wastewater treatment processes and energy recovery.
Highlights
Organic matters in sewage can be effectively concentrated to small volume by FO.
Membrane flux decline is caused by both membrane fouling and the decline of osmotic driving force.
Cake layer is the dominant contributor to the membrane fouling.
This study offers new insight into the development of low-energy wastewater treatment.

Keywords
Forward osmosis; municipal wastewater; energy reduction; membrane fouling; energy recovery

1 Introduction
The treatment of municipal wastewaters is conventionally carried out by aerobic biological processes as schematically depicted in Figure 1a. However, these processes are energy intensive. Treating one cubic meter of municipal wastewater consumes 0.4-2.1 kWh of electricity depending on the scale and process of wastewater treatment plants (WWTP) [1]. This energy consumption is mainly governed by two working functions: a) aeration for providing oxygen and b) sludge dewatering [2, 3]. On the other hand, the calorific value of organic matters in municipal wastewaters is 4.1 kWh per kilogram of chemical oxygen demand (COD) [2]. By recognizing that organic matter in municipal wastewater is a potential energy source, increasing efforts have been devoted to develop low-energy WWTP via recovering the energy present in wastewater itself [4-8]. One potential approach is to replace the aerobic reactor with an anaerobic reactor to produce biogas, which is rich in methane and can be used to generate energy. However, it is not practical to produce biogas from municipal wastewaters via anaerobic digestion (AD) considering the low concentration of organic pollutants [9, 10].

Recently, forward osmosis (FO) has drawn increasing attention due to its potential application in many fields including concentration of various solutions [11-13]. Different from conventional membrane processes, the driving force of FO is the osmotic pressure of draw solution rather than
external hydraulic pressure. Hence, FO may consume less energy than conventional membrane processes if an appropriate draw solution is applied. Several studies have also demonstrated the process advantages of FO in wastewater treatment. For instance, Lutchmiah et al. investigated water recovery from primary effluent of WWTP by FO and found that it was technically feasible to effectively remove most of the pollutants for clean water production [14]. Cath et al. studied FO filtration of secondary and tertiary treated effluent and claimed that FO could be an alternative process for water production [15]. Cornelissen et al. developed an innovative osmotic membrane bioreactor (OMBR) which could not only destroy pollutants but also separate activated sludge from the treated water [16]. Volladares Linares et al. proved that it was feasible to reduce water production cost via combining FO process with seawater desalination [17]. Most recently, Chen et al. investigated a submerged anaerobic membrane bioreactor with forward osmosis membrane (FO-AnMBR) using synthetic wastewater treatment [18]. The results showed that it was feasible to simultaneously destroy organic pollutants and produce biogas by the FO-AnMBR. However, so far less attention has been paid to the energy recovery from wastewater treatment by FO process.

The objective of this study was to investigate the feasibility of FO process to directly concentrate municipal wastewater to a small volume of energy source using a synthetic sea water (NaCl 3.5 wt%) as the draw solute. The key idea presented here is that by replacing the aeration reactor by the FO unit as shown in Figure 1b, the energy consumption in municipal wastewater might be reduced because no aeration is required and less sludge is generated. Moreover, seawater is free and easily accessible. The diluted seawater could be either discharged back to the sea or sent to other processes for fresh water production such as reverse osmosis (RO) [19] and electrodialysis (ED) [20]. Moreover, the small volume of concentrated wastewater can be sent to an anaerobic digester for biogas production. In order to provide practical validity for the concept of this study, real sewage was used as municipal wastewater feedstock. The performance of the commercial FO membrane was systematically
investigated for membrane flux, organic accumulation and membrane fouling. These results along with the new concept may provide insights to the development of low-energy wastewater treatment and energy recovery.

**Figure 1**

2 Experimental Sections

2.1 Chemicals and Materials

Unless otherwise specified, all chemicals and reagents used in this study were of ACS grade, and used without further purification. The FO membrane was a cellulose triacetate (CTA) membrane from Hydration Technology Innovations (HTI). It has an asymmetric structure comprised of a dense functional layer and a porous support layer with an embedded polyester mesh [14, 21]. The sewage was collected from sewer lines at the St Lucia Campus of The University of Queensland, which contained 400-800 mg/L of total suspended solid (TSS) and 300-600 mg/L of chemical oxygen demand (COD). More details about the quality of the sewage are referred to literature [22].

2.2 Forward osmosis concentration of sewage

The FO concentration of the sewage was performed using a bench-scale FO membrane system shown in Figure 2, which consists of a cross-flow membrane cell module and relevant peristaltic pumps and solution tanks. The effective membrane area was 64.6 cm². The cross-flow velocity was 0.14 cm s⁻¹ on each side of the membrane and no spacer was used. The tests were carried out at room temperature 22 ± 2 °C. The feed solution tank containing 800 g of municipal wastewater was placed on a digital balance and the membrane flux was calculated based on the weight changes of the feed solution as a function of time. To minimize the impact of the dilution of the draw solution on osmotic pressure during FO filtration, a total of 4000 mL NaCl solution was used. Its concentration is 3.5 wt.% to mimic the salinity of seawater. The osmotic pressure of the NaCl solution was calculated at 29.2 bars. The FO
membrane was washed with deionized (DI) water and stored in fresh DI water over night before experimental work. There are two different membrane orientations for FO process: (1) the membrane active layer is orientated facing the draw solution (AL-DS) and (2) the membrane active layer facing the feed solution (AL-FS). Both orientations were tested to investigate the influence of membrane orientation on FO performance.

A concentration factor was calculated as follows to determine the concentration extent of the sewage solution:

$$f_c = \frac{W_0}{W_t}$$  \hspace{1cm} (1)

Where $f_c$ is the concentration factor; $W_0$ and $W_t$ are the weight of the sewage at filtration time of zero and $t$.

The mass balance of COD was analyzed to calculate its accumulation ratio of COD in the feed sewage solution as follows:

$$r_{COD} = \frac{C_t V_t}{C_0 V_0} \%$$  \hspace{1cm} (2)

Where $r_{COD}$ is the accumulation ratio of COD; $C_t$ and $C_0$ are the COD concentration at filtration time of zero and $t$; $V_0$ and $V_t$ are the volume of the sewage at filtration time of zero and $t$.

**Figure 2**

2.3 Membrane fouling

The resistance in a series model was applied to evaluate the fouling characteristics of the FO membrane. According to this model, the membrane flux, $J$, can be expressed as follows [23]:

$$J = \frac{\Delta P}{\mu R_t} = \frac{\Delta P}{\mu (R_m + R_t + R_p)}$$  \hspace{1cm} (3)
where \( J \) is the membrane flux (L h\(^{-1}\) m\(^{-2}\)); \( \Delta P \) is the transmembrane pressure (Pa); \( \mu \) is the viscosity of the permeate (Pa s); \( R_t \) is the total resistance (m\(^{-1}\)); \( R_m \) is the resistance due to membrane itself and dilutive concentration polarization of draw solute; \( R_c \) is the fouling resistance due to cake layer (m\(^{-1}\)); and \( R_p \) is the fouling resistance due to pore plugging (m\(^{-1}\)). The experimental procedure to measure each of the resistance values can be found elsewhere [23, 24]. Typically, several testings were conducted as follows: (i) \( R_m \) was obtained through the average membrane flux of the clean membrane in the first hour using 3.5% NaCl as draw solution and DI water as feed solution, (ii) after 17 hours of sewage filtration, the concentrated sewage and diluted draw solution were replaced by DI water and fresh 3.5% NaCl solution. \( R_t \) was then measured from the average flux of the used FO membrane in first hour. (iii) and then, the membrane was removed and flushed with tap water to remove the cake layer. After this cleaning step, the pure water flux of the used membrane was measured again for one hour using 3.5% NaCl solution as draw solution to obtain the sum of \( R_m + R_p \). The fouling resistance, \( R_c \) was obtained by subtracting \( R_m + R_p \) from \( R_t \). Finally, the fouling resistance due to pore plugging \( R_p \) was then obtained by subtracting \( R_c \) and \( R_m \) from \( R_t \).

2.3 Water analysis and membrane characterization

The rejection rates of various pollutants in wastewater by FO membrane have been reported in some recent publications [14, 15, 25, 26]. The present study aimed to investigate the feasibility of FO of concentrating the organic matters from energy recovery points of view. Hence, our water analysis was concentrated on COD which can reflects energy density of sewages. Water samples of 20 mL were withdrawn from the feed and draw solutions before and after the FO filtration. COD concentration of these samples were measured by means of cuvette tests (Merck, Germany) [27]. UV-Vis spectrophotometer was employed to measure the organic concentrations of these samples.
morphologies of the clean and used FO membranes were characterized by SEM (JEOL 6040, 10 kV). For SEM cross section images, the samples were prepared via rapid freezing the FO membranes in liquid nitrogen for 5 min and then fractured into small pieces for characterization. A carbon layer of 20 nm was coated on the samples prior to the characterization.

3. Results and Discussion

3.1 Concentration factor

Figure 3 shows the weight change of the sewage feed solution during FO filtration using commercially available CTA membranes and synthetic saline solution (NaCl; 3.5%) as the draw solute. The weight steadily declined with increasing filtration time because water diffused across the FO membrane to the draw solution side due to the osmotic pressure difference. Meanwhile, the concentration factor gradually increased. It is worth to note that after 17 h of filtration, the weight loss slowed down to equilibrium. The reason is that after 17 h of filtration the feed solution remained in the feed tank was insufficient to completely fill the circulation pipes and the feed chamber of the FO filtration cell causing air bubbles to form in the cell. As a result, the FO filtration process was interrupted which can also be observed in the step-wise increase in the concentration factor at a later stage.

Figure 3

Although the FO filtration was interrupted when the concentration factor reached a value of around 6 due to the limitation of the experiment setup, it is possible to achieve higher concentration factor if the setup is improved, e.g. to increase the size of feed tank or shorten the circulation pipe. In theory, water continually flows to the draw solution side from sewage side until the osmotic pressure difference cross the FO membrane reaches zero. The osmotic pressure of the concentrated sewage can be calculated using Morse equation as follows.
\[ \pi = iMRT \]  \hspace{1cm} (4) 

where \( \pi \) is osmotic pressure (bar), \( i \) is the dimensionless van't Hoff factor, \( M \) is the molarity of sewage constituents, \( R \) is the gas constant \((8.314 \times 10^{-5} \text{ m}^3 \cdot \text{bar} \cdot \text{K}^{-1} \cdot \text{mol}^{-1})\); \( T \) is the absolute temperature (K).

The constituents of sewage generally can be classified into two categories, suspended and dissolved solids. It is reasonable to exclude the suspended solids in the calculation as their contributions to the osmotic pressure are negligible. The total dissolved solids (TDS) in sewage varies from 300 to 2000 mg/L [28], of which about 60% are organic matters while the rest 40% are inorganic pollutants. As the majority of organic matters in sewage are aliphatic hydrocarbon and NaCl is the dominated inorganic ion [28, 29], it is reasonable to assume that all organic pollutants are glucose and all inorganic pollutants are NaCl in order to simplify the calculation. The osmotic pressure of the concentrated sewage as a function of concentration factor is shown in Figure 4. The results show that the osmotic pressure of 3.5% NaCl solution is same as that of the concentrated sewage with a concentration factor of 33 in the worst case scenario (TDS: 2000 mg/L). If it is assumed that the NaCl draw solution is diluted two times during the filtration, the maximum concentration factor can reach 16. This indicates that TDS of the concentrated sewage may be accumulated up to 32 g/L. The calculation is consistent with a recent study, in which FO was proven to be feasible to concentrate activated sludge solution up to 28.5 g/L within 28 h using a 3.6% NaCl solution as the draw solution [30]. Hence, it can be concluded that the osmotic pressure of the 3.5% NaCl solution (seawater) is high enough to be used as the driving force for concentration of sewage by FO membrane.

Figure 4

3.2 Accumulation of organic matter

Organic matters, especially those with a high degree of conjugation, absorb light in UV or visible range. The UV-Vis spectra of the feed and draw solution samples were therefore recorded to determine
the concentrations of organic matters before and after FO filtration. As shown in Figure 5, the intensity of UV-Vis spectra of the concentrated sewage is significantly greater than that of the raw sewage, which provides strong evidences that the organic matters were concentrated by the FO membrane. The UV-Vis spectra of the draw solution both before and after filtration are seen very weak, and there is no obvious change between them. This means that the majority of organic compounds were retained in the feed side during the filtration. Due to the interference of high concentration of NaCl, the organic concentration in the draw solution cannot be measured by a COD or total organic carbon (TOC) analyzer. Hence, the COD concentration of the sewage feed solution before and after FO filtration were measured to evaluate the accumulation of organic pollutants in the feed side. The starting COD value in the raw sewage was 533.6 mg/L which increased to 1642.3 mg/L (equivalent to 308%) after 22 h of filtration in the AL-FS mode. A mass balance calculation shows that about 71.9% COD was accumulated in the feed solution. Same testing and calculation were carried out for AL-DS mode and it was found that a similar amount of COD (69.7%) was accumulated.

The COD and UV-Vis results seem to be incongruous, considering the FO membrane used in the study has a dense functional layer which should achieve a high organic rejection. For instance, a rejection rate of dissolved organic carbons as high as 99% was reported in previous study by the CTA membrane [30]. In another study using municipal wastewater as feed solution, a TOC rejection rate of higher than 97% was achieved [31]. The reason for this inconsistency in this study might be that a large amount of organic compounds formed a cake layer on the membrane surface, which was not taken into account in the mass balance calculation for COD accumulation ratio. The formation of the cake layer was confirmed in the membrane fouling investigation (see Section 3.4). Another possible reason is that some small non-chromophoric organic molecules may diffuse cross the FO membrane and thus cannot be measured by UV-Vis [17, 32].
3.3 Membrane flux

The membrane flux during the filtration was monitored in both AL-DS and AL-FS orientations. As shown in Figure 6a and 6b, the initial membrane flux of AL-DS is 7.4 L m$^{-2}$ h$^{-1}$, being 7.5% higher than that of AL-FS. These results are consistent with other FO membrane studies [14, 17]. It can be explained by the difference of dilutive concentration polarization of the draw solute between the two membrane orientations. The dilutive concentration polarization takes place on the surface of the FO functional layer in AL-DS mode, which is called external concentration polarization (ECP). By contrast, in AL-FS mode, the concentration polarization occurs inside of the porous FO support layer, which is called internal concentration polarization (ICP). Compared with ICP, ECP results in less decline in driving force of FO as it can be alleviated to some extent by the cross flow on membrane surface, which in turn leads to a higher membrane flux. Although salts in sewage might result in ICP in AL-DS mode, their concentration at the initial stage was so low that the ICP effect was neglectable.

Figure 6

Figure 6 also shows that with increasing the filtration time, the membrane flux gradually declined in both membrane orientations. It is noteworthy that although the initial membrane flux of AL-DS was higher than that of AL-FS, the flux declined 28.8% faster than the latter over the filtration period. After 17 h of filtration, membrane flux reduction in the AL-DS was 59.8% but there was only 45.1% reduction in the AL-FS. The decline in membrane flux was also observed in previous FO studies. For example, Valladares Linares et al. observed a significant decline in membrane flux in both FO membrane orientations [17]. In another study using WWTP effluent as feed solution [33], similar membrane flux decline has been reported. Xie et al. also found a steady decline of membrane flux in their study and claimed that the decline was mainly attributed to a decrease of the overall driving force in FO process, which could be caused by either the increasing salinity in feed side or the dilution in
11
draw solution [31]. In the present study, to identify the effect of salinity variation on membrane flux, the conductivity of both draw solution and feed solution before and after filtration was measured and summarized in Table 1. As excess draw solution was used in the present study, only a slight decline of the conductivity in the draw solution was observed. By contrast, the conductivity of the sewage feed solution jumped, which was attributed to the accumulation of feed solutes and reverse diffusion of draw solution. After filtration, the conductivity difference between the draw solution and feed solution decreased by 17.5% and 19.1% in AL-DS and AL-FS, respectively. These should result in a drop of the driving force to some extent. However, membrane fluxes in the two membrane orientations significantly declined by 59.8% and 45.1% as shown in Figure 6. The concentrated sewage and the diluted draw solution were replaced after filtration by DI water and fresh 3.5% NaCl solution, respectively. And then the membrane flux was measured to be 4.8 and 5.0 L m$^{-2}$ h$^{-1}$ in AL-DS and AL-FS. This means the membrane flux can only be partially recovered. Hence, besides the decrease of driving force caused by the change of salinity in the feed and draw solutions, there should some other contributors to the decline of membrane flux. The sewage was directly filtered by FO without any pretreatment so that it contains high suspended solid. It is well known that the organic components in wastewater are liable to attach to membrane surfaces and block membrane pores resulting in a decline of membrane flux in conventional membrane filtrations, such as RO, ultrafiltration (UF) and microfiltration (MF). Although previous studies claimed that FO has less membrane fouling compared with conventional pressure driven membrane filtration process [13], membrane fouling could be an issue in the present study by taking into account of the high organic content in the sewage.

Table 1

Overall, the average flux of the FO filtration of sewage in AL-DS and AL-FS was 5.2 and 5.4 L m$^{-2}$ h$^{-1}$ during 17 hours of filtration, respectively. The osmotic pressure of the draw solution (3.5% NaCl) used in this study was about 29 bars. During the filtration, the transmembrane pressure was roughly 24-
29 bars as the draw solution was gradually diluted. The RO membrane flux in seawater desalination plant is of 10-15 L m\(^{-2}\) h\(^{-1}\) under similar trans-membrane pressure (applied pressure minus the osmotic pressure of seawater). Although RO has higher membrane flux than FO, the feed of RO has to be pre-treated to remove suspended solid and colloids minimizing membrane fouling. It is worth noting no pre-treatment is needed in FO system even though the feed solution (sewage) contains high concentration of organic matters. Hence, the FO fluxes of sewage filtration are satisfactory considering the simplicity of the presses.

### 3.4 Membrane fouling

In order to understand the membrane fouling in the direct FO filtration of sewage, the membrane resistances were measured after 17 h of filtration. As shown in Figure 7, the total membrane resistance is higher in AL-DS than that in AL-FS after filtration, which demonstrates that more severe membrane fouling occurred in AL-DS orientation. The \(R_m\) in AL-FS is higher than that in AL-DS, which provides evidence that the concentration polarization of draw solution in former membrane orientation is worse than that in the latter. However, the membrane resistances attributed to cake layer and pore plugging in the AL-FS are both lower than those in AL-DS. The results are consistent with membrane fluxes shown in Figure 6. Lower membrane resistances in AL-FS resulted in slower decline in membrane flux.

**Figure 7**

The fouled FO membranes used in both orientations were characterized by SEM as shown in Figure 8. It can be seen that a thicker cake layer was formed on the membrane surface in AL-DS compared with AL-FS orientation. This may be attributed to the morphological differences between the support and functional layers of the asymmetric structure CTA membrane shown in Figure 9. The functional layer is smooth whilst the supporting layer is rough. In a previous study, Li et al. [34] found that smoother membrane surfaces resulted in lower membrane fouling rate in line with the findings in this
work. The rough surface may accelerate the accumulation of foulants in the micro recesses which makes the membrane fouling worse [35]. In addition, a number of cracks and defects demarcated by red circles in Figure 9 can be seen on the support layer. As such, foulants may enter into the support layer through these cracks blocking the membrane pores. Previous studies [25, 36] showed that the cake layer and pore plugging could enhance ICP in FO as they reduce the pore size and hinder the back diffusion of feed solute. Hence, the thicker cake layer and worse pore plugging in AL-DS resulted in higher extent of ICP, which can explain why faster membrane flux decline happed in AL-DS. Nevertheless, the $R_p$ is lower than $R_c$ in both membrane orientations, which indicates that cake layer is the dominant contributor to the membrane fouling caused by foulants. Overall, $R_c$ and $R_p$ are less than $R_m$, which means that the key way to improve the FO membrane is to develop low-resistance membrane and reduce the concentration polarization.

4. Conclusions

Direct filtration of sewage by FO process using synthetic saline solution (3.5 wt.%) was investigated in this study aiming to concentrate the sewage into small volume of energy source. The results showed that pollutants in the sewage could be effectively rejected by FO membrane and gradually accumulated in the feed side. A gradual decrease in membrane flux was observed, which is attributed to the membrane fouling and the drop of the overall driving force caused by the build-up of the salinity in feed solution and the dilution in draw solution. Cake layer was the dominant contributor to the membrane fouling. AL-FS outperformed AL-DS in the 17 hours of filtration in terms of membrane flux and membrane fouling. It might be attributed to the asymmetric structure of the CTA FO membrane. Foulants were more liable to accumulated on the surface of support layer in AL-DS than on the surface of functional layer in AL-FS.

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Reference


**List of Tables and Figures**

Table 1. Conductivity of the draw solution and sewage feed solution before and after filtration.

Figure 1. Schematics of municipal wastewater process using (a) conventional aerobic reactor and (b) forward osmosis and anaerobic digestion.

Figure 2. FO setup for sewage filtration.

Figure 3. Weight change of the sewage feed solution and concentration factor as a function of filtration time (membrane orientation: AL-FS).

Figure 4. Osmotic pressure of the concentrated sewage as a function of concentration factor in different scenarios, high TDS (TDS = 2000 mg/L) and low TDS (TDS = 300 mg/L).

Figure 5. UV-Vis spectra of the raw sewage, concentrate and draw solution before and after FO filtration. (Membrane orientation: AL-FS)

Figure 6. Membrane flux variation with filtration time in two membrane orientations. (a) AL-DS, (b) AL-FS.

Figure 7. Membrane resistances of FO filtration of sewage.

Figure 8. SEM images of FO membrane after filtration. (a) Top view image at AL-DS, (b) cross section image at AL-DS, (c) Top view image at AL-FS, (d) cross section image at AL-FS. Scale bar is 50 μm except it is 10 μm in (a).
Figure 9. SEM images of the virgin FO membrane. (a) cross section image, (b) functional layer, (c) supporting layer. Scale bar is 100 μm and membrane defects on the surface are demarcated by red ovals and circles.

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<table>
<thead>
<tr>
<th>sample</th>
<th>Membrane orientation</th>
<th>Conductivity Before filtration (ms cm⁻¹)</th>
<th>Conductivity After filtration (ms cm⁻¹)</th>
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<tr>
<td>Draw solution</td>
<td>AL-DS</td>
<td>48.3</td>
<td>45.9</td>
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<tr>
<td>Draw solution</td>
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<td>42.5</td>
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<td>Al-DS</td>
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<tr>
<td>Feed solution</td>
<td>AL-FS</td>
<td>0.9</td>
<td>4.7</td>
</tr>
</tbody>
</table>
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