Assessing the integration of forward osmosis and anaerobic digestion for simultaneous wastewater treatment and resource recovery

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Assessing the integration of forward osmosis and anaerobic digestion for simultaneous wastewater treatment and resource recovery

Abstract
This study assessed the performance and key challenges associated with the integration of forward osmosis (FO) and anaerobic digestion for wastewater treatment and resource recovery. Using a thin film composite polyamide FO membrane, maximising the pre-concentration factor (i.e. system water recovery) resulted in the enrichment of organics and salinity in wastewater. Biomethane potential evaluation indicated that methane production increased correspondingly with the FO pre-concentration factor due to the organic retention in the feed solution. At 90% water recovery, about 10% more methane was produced when using NaOAc compared with NaCl because of the contribution of biodegradable reverse NaOAc flux. No negative impact on anaerobic digestion was observed when wastewater was pre-concentrated ten-fold (90% water recovery) for both draw solutes. Interestingly, the unit cost of methane production using NaOAc was slightly lower than NaCl due to the lower reverse solute flux of NaOAc, although NaCl is a much cheaper chemical.

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Engineering | Science and Technology Studies

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Authors

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RESEARCH HIGHLIGHTS

• No negative effect on CH$_4$ production at 10 folds wastewater pre-concentration

• At 90% water recovery, CH$_4$ production using NaOAc was 10% more than NaCl as DS

• The unit cost of methane production was highly sensitive to the reverse salt flux

• The unit cost of methane production using NaOAc was slightly lower than NaCl

• Membrane fouling was limited to surface deposition and was readily removed by flushing
Assessing the integration of forward osmosis and anaerobic digestion for simultaneous wastewater treatment and resource recovery

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Abstract

This study assessed the performance and key challenges associated with the integration of forward osmosis (FO) and anaerobic digestion for wastewater treatment and energy recovery. Using a thin film composite polyamide FO membrane, maximising the pre-concentration factor (i.e. system water recovery) resulted in the enrichment of organics and salinity in wastewater. Biomethane potential evaluation indicated that methane production increased correspondingly with the FO pre-concentration factor due to the organic retention in the feed solution. At 90% water recovery, about 10% more methane was produced when using NaOAc compared with NaCl because of the contribution of degradable reverse NaOAc flux. No negative impact on anaerobic digestion was observed when wastewater was pre-concentrated ten-fold (90% water recovery) for both draw solutes. Interestingly, the unit cost of methane production using NaOAc was slightly lower than NaCl due to the lower reverse solute flux and higher methane production.

Keywords: Forward osmosis (FO); reverse solute flux; biomethane potential (BMP) analysis; draw solution selection; sewer mining.
1. Introduction

In a circular economy, wastewater is considered as a source of water, energy, and nutrients, rather than a waste. As such, there is a growing demand for low impact wastewater treatment systems that provide water reuse and are able to recover nutrients and energy (Desmidt et al., 2014; Puyol et al., 2016). This demand has driven the development of innovative technologies to tap into the resource potential of wastewater. Membrane-based technologies have been essential for advanced water purification in reuse applications (Shannon et al., 2008; Xie et al., 2016). Similarly, anaerobic digestion has evolved as a key technological pathway for the realisation of energy and nutrient recovery from wastewater (Frijns et al., 2013; Verstraete et al., 2009).

Anaerobic digestion is a promising platform for low energy wastewater treatment and resource recovery. Indeed, the conventional activated sludge process requires significant electrical energy consumption for aeration. Anaerobic digestion has been widely used for the treatment of sludge originating from wastewater treatment plants, however, there are several technical challenges associated with applying anaerobic digestion for direct wastewater treatment. One such difficulty is the dilute nature of wastewater that significantly increases the digester heating requirement per unit of biogas production and thus influences the economic viability of the process. In addition, methane loss due to dissolution in the effluent is significant at a low production rate. For low-strength wastewater, processes that pre-concentrate chemical oxygen demand (COD) and nutrients (e.g. phosphorus) represent one avenue to improving the economics of biogas recovery from anaerobic treatment units (Jin et al., 2017; Wan et al., 2016).

High retention membranes such as forward osmosis (FO) can be strategically integrated with anaerobic digestion to achieve simultaneous wastewater treatment and resource recovery (Ansari et al., 2017; Wang et al., 2016). The major advantages of FO compared to other membrane processes include, low hydraulic pressure operation, low fouling propensity, easy cleaning, and a high rejection of a broad range of contaminants. FO can also be coupled with a draw solution regeneration process such as membrane distillation (MD) and reverse osmosis to directly extract clean water from raw wastewater, while simultaneously concentrating wastewater organics for subsequent anaerobic digestion (Luo et al., 2017; Nguyen et al., 2016; Shahzad et al., 2017). Anaerobically digesting FO pre-concentrated...
wastewater can produce biogas, which can be utilised by a combined heat and power engine to produce electricity and thermal energy. Surplus electricity can be supplied to the grid and the produced thermal energy can be used for MD and the anaerobic process. This latter process also converts biologically bound phosphorus into a soluble form, thus allowing phosphorus recovery as struvite (MgNH₄PO₄·6H₂O) or hydroxyapatite (Ca₁₀(PO₄)₆(OH)₂).

Interest in combining FO with anaerobic treatment has significantly increased in recent years due to the potential advantages of low-energy wastewater stabilisation and resource recovery. Recent studies have investigated FO-anaerobic integration in terms of draw solution selection (Kim et al., 2016), process configurations (Qiu et al., 2016; Wang et al., 2017b; Zhang et al., 2017), membrane cleaning (Wang et al., 2017a), trace organic contaminant removal (Kim et al., 2017), microbial composition (Wu et al., 2017), and energy dynamics (Onoda et al., 2017). However, there is a lack of studies which thoroughly assess the key FO operating parameters that essentially govern anaerobic digestion performance.

Water recovery and the selected draw solution can influence the composition of pre-concentrated wastewater in terms of organics retention and salinity accumulation. The incompatibility between high salinity and anaerobic microorganisms represents the most prominent challenge associated with integrating FO with anaerobic treatment. Salinity accumulation is inherently associated with the FO process. However, appropriate draw solution selection can potentially reduce the amount of solute diffusing into the feed solution. On the other hand, water recovery determines the accumulation of existing dissolved solutes in wastewater. Determining the influence of these FO operating parameters on anaerobic treatment is imperative to evaluate the feasibility and optimise biogas production from FO pre-concentrated wastewater.

This study aims to evaluate the process performance and investigate the key challenges associated with integrating FO with anaerobic treatment. Specifically, this study optimises the FO concentration factor (i.e. system water recovery) to balance the organic content and salt concentration in pre-concentrated wastewater and their combined effects on methane production. Representative inorganic and ionic organic draw solutes, namely sodium chloride (NaCl) and sodium acetate (NaOAc) were compared in terms of FO membrane performance and the digestibility of pre-concentrated wastewater. Optimised parameters and cleaning
techniques are applied to mitigate salinity accumulation (i.e. alternative draw solute) and
membrane fouling (i.e. physical flushing).

2. Materials and methods

2.1 Forward osmosis system

The lab-scale FO system used in this study consisted of a cross-flow membrane cell with an
effective membrane area of 50 cm$^2$. The membrane cell comprised of two symmetric flow
channels for the feed and draw solutions to contact the membrane. Each flow channel had
length, width, and height dimensions of 100 mm, 50 mm, and 3 mm, respectively. The flat-
sheet membrane was positioned between two rubber gaskets and two semi-cells made of
perspex. The feed and draw solutions were circulated through the membrane cell channels via
two variable-speed gear pumps (Micropump, Vancouver, Washington, USA). The pump
speed was adjusted to maintain the system cross-flow velocity, and the circulation flow rate
was regulated using two rotameters. A diamond shaped spacer with a thickness of 1 mm was
placed within the draw solution flow channel to improve mixing.

The flux dynamics of the system were determined according to the standard procedure
described by Cath et al. (2013). The weight change of the draw solution tank was measured
using a digital balance (Mettler-Toledo Inc., Hightstown, New Jersey, USA) to determine the
permeate water flux. The osmotic pressure of each draw solution was kept constant during
each FO experiment by controlling the solution conductivity. The draw solution conductivity
was continuously measured using a conductivity probe (Cole-Parmer, Vernon Hills, Illinois,
USA). A peristaltic pump connected to a controller dosed highly concentrated stock solution
(5 M) into the draw solution as the measured conductivity fell below the specified range at a
control accuracy of (±0.1 mS/cm). This re-concentration system was also placed on a digital
balance to ensure accurate flux measurements due to weight changes.

2.2 Biochemical methane potential experimental set-up

The biochemical methane potential (BMP) experimental set-up consisted of 16 fermentation
bottles (Wiltronics Research, Ballarat, Victoria, Australia). Each BMP bottle was filled with
500 mL of inoculum and 250 mL of the simulated FO pre-concentrate. The fermentation
bottles were submerged in a water bath at a constant temperature of 35.0 ± 0.1 °C (Ratek
Instruments, Boronia, Victoria, Australia). Each bottle was sealed with a rubber bung
attached to a water filled S-shaped air lock, and flexible plastic tubing was used to transfer biogas to the collection gallery. The gas collection gallery included 16 inverted 1000 mL plastic measuring cylinders, filled with a 1 M NaOH solution. The NaOH solution sequestered the CO$_2$ and H$_2$S in the biogas, whilst the CH$_4$ gas displaced the NaOH inside the cylinder. Daily measurements of CH$_4$ gas production were recorded.

2.3 Materials and chemicals

Wastewater (after primary sedimentation) and digested sludge were obtained from the Wollongong Wastewater Treatment Plant (WWTP) in New South Wales, Australia. The wastewater was used as a feed solution for FO pre-concentration experiments, whilst the digested sludge was used as the inoculum for the BMP experiments. Basic characteristics of the solutions are summarised in Table 1.

<table>
<thead>
<tr>
<th>Table 1</th>
</tr>
</thead>
</table>
| Draw solutions were prepared using analytical grade NaCl or NaOAc. The draw solution concentration was determined by OLI Stream Analyzer (OLI Systems, Inc., Morris Plains, New Jersey, USA) calculations to achieve an equivalent osmotic pressure of 30 bar (similar to that of seawater).

To accurately assess the effect of FO water recovery and draw solution on methane production, BMP experiments were conducted using a synthetic wastewater solution. The actual concentrate originating from the FO system was not used in the BMP experiments, as the liquid volume produced by the lab-scale FO system was too small. Instead, a synthetic solution was made to simulate the pre-concentration of wastewater components, as well as the contribution of reverse draw solute flux. The concentrated stock solution was prepared to contain 4 g/L glucose, 1 g/L peptone, 0.35 g/L urea, 0.175 g/L KH$_2$PO$_4$, 0.175 g/L MgSO$_4$, 0.1 g/L FeSO$_4$, and 2.25 g/L NaOAc. This stock solution was then diluted to accurately simulate the COD of the initial primary effluent as well as the experimentally measured COD amount in FO pre-concentrated wastewater at 50, 80 and 90% water recovery. A pre-determined amount of analytical grade NaCl or NaOAc was then added to the synthetic feed to simulate salinity increase corresponding to each water recovery values as calculated from the FO experimental results. Pure nitrogen gas was used to flush the BMP bottles and a 1 M
sodium hydroxide (NaOH) solution was used to absorb the carbon dioxide (CO$_2$) and hydrogen sulphide (H$_2$S) from the biogas.

A thin film composite (TFC) FO membrane was used in this study and was supplied by Porifera (Porifera Inc., Hayward, CA). This had a polyamide active layer with a porous polysulfone layer for support. The membrane was positioned in FO mode (i.e. active layer facing the feed solution) for all experiments.

2.4 Experimental protocol

For the FO experiments, wastewater from the Wollongong WWTP was used as the feed solution. Analytical grade NaCl or NaOAc was dissolved in DI water to obtain the final concentration of 0.65 or 0.72 M, respectively, corresponding to the osmotic pressure of seawater (approximately 30 bar). The system water recovery was calculated based on the ratio of the cumulative permeate volume and the initial feed solution volume. The FO system was operated continuously until 90% of water had been recovered from the feed solution. The initial volume of wastewater feed solution was 2 L, corresponding to a total concentrate volume of 0.2 L. The water flux was continuously monitored, whilst the wastewater conductivity, pH, and temperature were frequently measured. At specific time intervals, samples of 10 mL volume were withdrawn from the feed solution for COD analysis to represent the organic content in solution. The circulation flow rates were maintained at 1 L/min giving a cross-flow velocity of 16.7 cm/s.

At the conclusion of the experiment, the membrane was flushed at a higher cross flow velocity for 30 minutes. This was achieved by replacing the feed and draw solutes with DI water and doubling the cross-flow velocity (i.e. 33.4 cm/s). After flushing, fresh wastewater was used as the feed solution to verify the water flux recoverability at the initial conditions.

After experimentally determining the pre-concentrated wastewater characteristics (i.e. COD and salinity), a synthetic wastewater solution and each draw solute was used to simulate the wastewater at 50, 80, and 90% water recovery. The COD results from the FO experiments were used to represent the COD increase in wastewater. The synthetic wastewater solution described in Section 2.1 was prepared to obtain the COD value at each corresponding water recovery, and also provided the expected salinity related to only FO rejection of feed water.

Alternatively, the contribution of reverse solute flux was provided by adding a specified...
amount of either NaCl or NaOAc to the synthetic wastewater solution. This reverse solute flux contribution ($Salt_{RSF}$) was estimated using salinity measurement assuming: $Salt_{RSF} = Salt_{Total} - Salt_{WW}$, where $Salt_{Total}$ is the measured salt concentration at each water recovery value and $Salt_{WW}$ is the calculated salt concentration from the wastewater due to FO rejection. This concentration ($Salt_{WW}$) was calculated using a mass balance, assuming complete rejection of any salts in wastewater as equivalent NaCl. The salinity of the feed solution was determined using electrical conductivity measurements and calibration curves were then used to determine salt concentration.

The simulated FO pre-concentrate was mixed with digested sludge in each BMP bottle. An inoculum volume of 500 mL and a substrate volume of 250 mL was selected, corresponding to an inoculum/substrate ratio of 2:1. A reference condition was used to represent the methane production of the inoculum, and real wastewater (i.e. FO feed solution with 0% water recovery) was also used as a separate condition for comparison to the synthetic wastewater. Prior to the BMP experiment, the bottles were purged with nitrogen gas, sealed, and submerged in the water bath. The flexible plastic tubing was connected to the biogas collection gallery. All BMP experiments were conducted in duplicate and biogas measurements were recorded daily. The contents of each bottle was characterised before and after the BMP experiment in terms of pH, conductivity, and COD.

### 2.5 Analytical methods

Standard methods were used during the analysis of basic water quality parameters. The temperature, pH, and electrical conductivity were monitored using an Orion 4-Star pH/conductivity meter (Thermo Scientific, Waltham, MA). COD samples were analysed using a Hach DBR200 COD Reactor and Hatch DR/2000 spectrophotometer (program number 435 COD HR) following the US-EPA Standard Method 5220 D. Total solids (TS) and volatile solids (VS) of the primary effluent were determined within three days after sample collection. All samples were stored at 4 ºC in the dark.

### 2.6 Draw solute cost

Replenishment costs were calculated based on the pure water performance of the FO system at the draw solute concentration corresponding to 30 bar osmotic pressure. The replenishment cost only considered the loss of salt due to reverse draw solute flux. Losses from the draw
solute recovery process (i.e. RO or MD) were assumed to be insignificant. The initial cost of
draw solution was also neglected as it can be reused in the process. Current average
wholesale price of NaCl and NaOAc was used. The cost of draw solute replenishment per
ML of permeate produced by the FO system was determined and a system water recovery of
90% was evaluated. Next, experimentally determined values of methane production and the
draw solute replenishment costs were used to calculate the unit cost of methane production
for each draw solute.

3. Results and discussion

3.1 Pre-concentration performance using thin film composite membrane

Pre-concentrating wastewater with the TFC FO membrane resulted in a substantial increase
in COD (i.e. approximately eight-fold) at a water recovery of 90% (Figure 1). Organic matter
enrichment for NaOAc was higher than NaCl, due to the contribution of organic reverse draw
solute flux. For both draw solutions, the maximum COD was slightly lower than the
theoretical COD amount. As no fouling mitigation strategy was implemented for this
experiment, it is possible that surface deposition of organics was an important fouling
mechanism, and thus, resulting in a lower bulk COD concentration than theoretically possible
(i.e. ten-fold). In practice, the fouling layer can be re-suspended into the feed solution during
membrane cleaning, and thus contribute to the feed COD amount.

[Figure 1]

Similar to the enrichment of COD in pre-concentrated wastewater, the level of salinity also
increased as the FO experiment progressed (Figure 2). Wastewater conductivity increased for
two reasons. The natural salinity of the wastewater (approximately 1 mS/cm) accumulated
within the feed solution, due to rejection by the membrane and the concentrating effect. The
reverse diffusion of the draw solute into the feed solution also contributed to salinity
accumulation. The relative contribution of these two mechanisms is shown in Figure 2 and
compared to the theoretically calculated conductivity increase due to the concentration of
wastewater (i.e. ignoring reverse draw solutes flux). NaOAc exhibited a similar increase in
conductivity compared with the theoretical wastewater salt accumulation, owing to the small
amount of reverse solute flux (2.2 g/m²h) compared with NaCl (12.4 g/m²h). Deviation from
the theoretical salt accumulation behaviour was likely due to the impact of flux dynamics and
membrane fouling on salt rejection at high water recoveries. In contrast, the reverse solute
flux of NaCl contributed to salinity accumulation by approximately 50% higher than NaOAc.
The results highlight the potential negative impacts associated with using highly diffusive
inorganic draw solutions, such as NaCl.

[Figure 2]

3.2 Effect of forward osmosis concentration factor on methane production

Variations in wastewater characteristics at FO water recoveries of 50, 80, and 90% were
simulated in batch anaerobic BMP experiments (Table 2). For both draw solutions, the
conditions were simulated based on the experimentally determined values for salt
concentration (i.e. conductivity) and organic content (i.e. COD) during the FO wastewater
pre-concentration experiments. Wastewater COD was simulated using synthetic wastewater
and the remaining conductivity requirement was supplied with the relevant amount of each
draw solute (i.e. NaCl or NaOAc). Higher FO system water recovery resulted in an increase
in both conductivity and COD (Table 2). It has been reported that conductivity and COD
could have adverse and opposing effects on methane production by anaerobic treatment
(Appels et al., 2008). COD loading up to 1,000 mg/L can significantly benefit the anaerobic
process in terms of methane conversion. Conversely, high conductivity solutions can
seriously affect methanogenic health and inhibit methane production.

[Table 2]

The cumulative methane production over a period of 30 days demonstrated the varying effect
of FO water recovery and draw solute selection on the digestibility of pre-concentrated
wastewater (Figure 3). Firstly, the methane production of real wastewater (i.e.
unconcentrated) was only slightly higher than the reference condition (i.e. inoculum only)
and can be attributed to marginal difference in total COD for these two conditions, as well as
due to variations in the inoculum characteristics (Table 2). This result demonstrates the
difficulties associated with digesting low-strength wastewaters for the purpose of biogas
recovery. In all cases, pre-concentrating wastewater using FO improved the total wastewater
COD, thus tended to increase methane production over the evalutation period. For both NaCl
(Figure 3A) and NaOAc (Figure 3B), the cumulative methane production increased as the
system water recovery was maximised. Overall, a minor effect of the reverse draw solute flux
on methane production was observed. This was likely due to the presence of sufficient biodegradable matter in the pre-concentrated wastewater, or because of the applied inoculum/substrate ratio of 2:1, which may have masked the total salinity.

Methane production increased linearly with increasing pre-concentration factor and indicates an improvement in digester performance owing to the FO process. At the pre-concentration factor of ten (i.e. 90% water recovery), methane production was improved by approximately five and seven times for NaCl and NaOAc, respectively (Figure 4). Comparing the two draw solutions, NaOAc could produce a larger amount of methane compared with NaCl (i.e. approximately 10%), due to lower reverse solute flux and degradable nature of NaOAc. Therefore, in terms of concentrated wastewater digestibility, no apparent negative effect on anaerobic treatment was observed when wastewater was pre-concentrated by ten times (equivalent to 90% water recovery) and with an inoculum/substrate ratio of 2:1. Although FO reverse solute flux of inorganic draw solutions has been reported to negatively affect anaerobic treatment (Li et al., 2017), these results show that careful selection of FO operating parameters and digester loadings could potentially improve the process performance. In effect, pilot-scale assessment is necessary to evaluate the feasibility of operating at a high FO system water recovery and to determine the optimum anaerobic digester loading rate.

When comparing this process to the direct digestion of raw wastewater, a number of additional advantages of using FO to pre-concentrate wastewater exist. These include a substantially reduced digester volumetric loading (i.e. 10% of initial wastewater volume) and therefore, a smaller amount of anaerobic effluent. Furthermore, FO pre-concentration can provide a foulant-free draw solution for a subsequent desalination process to recover fresh water.

### 3.3 Unit cost of methane production

The costs associated with replenishing the draw solute as a result of reverse solute flux are shown in Table 3. Table 3 also includes the unit cost of methane production for both NaCl and NaOAc in terms of FO draw solute replenishment. Although the wholesale price of NaCl
is significantly lower than that of NaOAc, the high reverse solute flux of NaCl resulted in a slightly higher replenishment cost. The unit costs of methane production using NaOAc and NaCl were $0.53 and $0.64 per m$^3$ of methane, respectively. At 90% water recovery, there was about 10% increase in the volume of methane produced using NaOAc in comparison to NaCl (section 3.2). However, this contribution is insignificant compared to the difference in reverse solute flux between NaOAc and NaCl (Table 3). Results in Table 3 indicate that the unit cost of methane production is highly sensitive to the reverse solute flux. Further improvement in FO membrane fabrication is expected and can lower the cost of methane production from wastewater. It is noteworthy that Table 3 can be only used to compare the unit cost of methane production between NaOAc and NaCl. The calculation in Table 3 did not take into account the potential revenue from clean water production and further research is necessary for an overall economic analysis of methane production from pre-concentrated wastewater by anaerobic digestion.

|Table 3|

### 3.4 Water flux decline and flux recoverability

At the same osmotic pressure, water flux decline was evaluated for both NaCl and NaOAc (Figure 5). Although the initial water flux of NaOAc (16.6 L/m$^2$h) was slightly lower than that of NaCl (17.4 L/m$^2$h), both draw solutes exhibited a similar flux decline in the initial stages of the experiment. Subsequently, NaOAc fouling was more severe and indicated the possible interaction between the draw solute and membrane fouling layer (Luo et al., 2016; She et al., 2012). The total experimental duration to achieve 90% water recovery for NaCl and NaOAc was 65 and 72 hours, respectively. Despite the observed membrane fouling, 30 minutes of in-situ membrane flushing could completely recover water flux, indicating that no significant irreversible fouling occurred and that fouling was limited to surface deposition (Figure 5). The results in this study show that the rate of membrane fouling using the TFC membrane was higher compared with the CTA membrane used in a previous study (Ansari et al., 2016). This can mostly be attributed to the significantly larger initial water flux of the TFC membrane.

|Figure 5|
4. Conclusion

Pre-concentrating wastewater using the TFC FO membrane effectively concentrated COD by approximately eight-folds. Although the resultant pre-concentrated wastewater solution was highly saline, no apparent effect on methane production was observed for both draw solutes at the maximum water recovery value (i.e. 90%) during biomethane potential assessment. Overall, the pre-concentrated wastewater containing NaOAc resulted in a higher methane production to that of NaCl. Additionally, the unit cost of methane production using NaOAc was slightly lower than NaCl. FO membrane fouling was limited to surface deposition, thus, allowed for effective cleaning via membrane flushing at a high cross flow velocity.

Acknowledgements

This research was supported by the Australian Government Research Training Program Scholarship (Ashley Ansari) and the Australian Research Council’s Discovery Project funding scheme (project DP140103864).

5. References


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### List of Tables

**Table 1.** Characterisation of real wastewater and digested sludge inoculum (average concentration ± standard deviation from at least three samples).

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Units</th>
<th>Wastewater</th>
<th>Digested sludge</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chemical oxygen demand (COD)</td>
<td>mg/L</td>
<td>288 ± 10</td>
<td>4,000 ± 60</td>
</tr>
<tr>
<td>Electrical conductivity (EC)</td>
<td>µS/cm</td>
<td>977 ± 4</td>
<td>5,230 ± 8</td>
</tr>
<tr>
<td>Total solids (TS)</td>
<td>%</td>
<td>0.07 ± 0.02</td>
<td>1.7 ± 0.5</td>
</tr>
<tr>
<td>Volatile solids (VS)</td>
<td>%</td>
<td>0.03 ± 0.01</td>
<td>1.1 ± 0.3</td>
</tr>
</tbody>
</table>

**Table 2.** Variation in pre-concentrated wastewater conductivity and COD simulated in BMP experiments for NaCl and NaOAc. The calculated total COD in each BMP bottle (750 mL) is also shown. Two BMP experiments were performed and each condition was conducted in duplicate.

<table>
<thead>
<tr>
<th>Condition</th>
<th>FO water recovery (%)</th>
<th>Pre-concentrated wastewater conductivity (µS/cm)</th>
<th>Pre-concentrated wastewater COD (mg/L)</th>
<th>Total COD in each BMP bottle (mg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reference</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>4,000</td>
</tr>
<tr>
<td>Real wastewater</td>
<td>0</td>
<td>977</td>
<td>288</td>
<td>4,072</td>
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<tr>
<td>Synthetic wastewater + NaCl</td>
<td>50</td>
<td>2,449</td>
<td>540</td>
<td>4,135</td>
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<tr>
<td></td>
<td>80</td>
<td>7,846</td>
<td>1,079</td>
<td>4,270</td>
</tr>
<tr>
<td></td>
<td>90</td>
<td>16,750</td>
<td>2,280</td>
<td>4,570</td>
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<tr>
<td>Synthetic wastewater + NaOAc</td>
<td>50</td>
<td>1,889</td>
<td>540</td>
<td>4,675</td>
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<tr>
<td></td>
<td>80</td>
<td>6,122</td>
<td>1,079</td>
<td>6,306</td>
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<tr>
<td></td>
<td>90</td>
<td>8,900</td>
<td>2,280</td>
<td>7,588</td>
</tr>
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</table>
Table 3. Draw solute replenishment cost and unit cost of methane production using NaCl and NaOAc. Draw solute replenishment costs were based on the average wholesale salt cost and the pure water flux performance ($J_w$ and $J_s$) for each draw solution at 30 bar osmotic pressure. Draw solute cost per methane produced was determined at 90% FO water recovery.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Units</th>
<th>NaCl</th>
<th>NaOAc</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water flux ($J_w$)</td>
<td>L/m²h</td>
<td>18.1</td>
<td>16.9</td>
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<tr>
<td>Reverse solute flux ($J_s$)</td>
<td>g/m²h</td>
<td>12.4</td>
<td>2.2</td>
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<tr>
<td>Specific reverse solute flux ($J_s/J_w$)</td>
<td>g/L permeate</td>
<td>0.69</td>
<td>0.13</td>
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<tr>
<td>Salt cost</td>
<td>$/kg</td>
<td>0.05</td>
<td>0.3</td>
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<tr>
<td>Replenishment cost</td>
<td>$/ML permeate</td>
<td>34.25</td>
<td>39.23</td>
</tr>
<tr>
<td>Specific methane production at 90% FO water recovery</td>
<td>L CH₄/L substrate</td>
<td>0.48</td>
<td>0.66</td>
</tr>
<tr>
<td>Unit cost of methane production</td>
<td>$/m³ CH₄ produced</td>
<td>0.64</td>
<td>0.53</td>
</tr>
</tbody>
</table>
List of Figure Captions

**Figure 1:** Pre-concentration of wastewater COD using NaCl and NaOAc draw solutions with the TFC FO membrane. Theoretical COD increase is shown assuming 100% COD retention. Experimental conditions: primary effluent feed solution (2 L); $\pi = 30$ bar draw solution; cross-flow rates of both feed and draw solutions were 1 L/min (corresponding to a cross-flow velocity of 16.7 cm/s).

**Figure 2:** Variation in wastewater conductivity for NaCl and NaOAc draw solutions. Theoretical salt accumulation ($Salt_{Acc}$) from natural wastewater salinity only (i.e. excluding reverse draw solute flux) is shown assuming 100% salt retention Experimental conditions as in Figure 6.1.

**Figure 3:** Average cumulative methane production over the 30 day evaluation period at various wastewater (WW) pre-concentration stages using (A) NaCl and (B) NaOAc FO draw solutions. Error bars represent n=4 measurements, including two BMP experiments with each condition performed in duplicate.

**Figure 4:** Specific methane production over the experimental period, indicating no negative effect of pre-concentrated wastewater up to 90% water recovery. Experimental conditions as in Figure 6.3. Error bars represent n=4 measurements, including two BMP experiments with each condition performed in duplicate.

**Figure 5:** Water flux decline and recoverability during FO pre-concentration with TFC membrane. After achieving 90% water recovery, membrane flushing was performed for 30 min using DI water at double the experimental cross-flow velocity (i.e. 33.4 cm/s)). Experimental durations corresponding to 90% recovery were 65 and 72 hours for NaCl and NaOAc, respectively. Initial water flux was 17.4 L/m$^2$h for NaCl and 16.6 L/m$^2$h for NaOAc. Experimental conditions as in Figure 1.
Figure 1

Figure 2
Figure 3
Figure 4

Specific Methane Production (mL CH$_4$/L substrate) vs Pre-concentration Factor

Figure 5

Water Flux (L/m$^2$h) vs Water Recovery (%)

Membrane flushing