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

Abstract

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

1. Introduction

33	In a circular economy, wastewater is considered as a source of water, energy, and nutrients,
34	rather than a waste. As such, there is a growing demand for low impact wastewater treatment
35	systems that provide water reuse and are able to recover nutrients and energy (Desmidt et al.,
36	2014; Puyol et al., 2016). This demand has driven the development of innovative
37	technologies to tap into the resource potential of wastewater. Membrane-based technologies
38	have been essential for advanced water purification in reuse applications (Shannon et al.,
39	2008; Xie et al., 2016). Similarly, anaerobic digestion has evolved as a key technological
40	pathway for the realisation of energy and nutrient recovery from wastewater (Frijns et al.,
41	2013; Verstraete et al., 2009).
42	Anaerobic digestion is a promising platform for low energy wastewater treatment and
43	resource recovery. Indeed, the conventional activated sludge process requires significant
44	electrical energy consumption for aeration. Anaerobic digestion has been widely used for the
45	treatment of sludge originating from wastewater treatment plants, however, there are several
46	technical challenges associated with applying anaerobic digestion for direct wastewater
47	treatment. One such difficulty is the dilute nature of wastewater that significantly increases
48	the digester heating requirement per unit of biogas production and thus influences the
49	economic viability of the process. In addition, methane loss due to dissolution in the effluent
50	is significant at a low production rate. For low-strength wastewater, processes that pre-
51	concentrate chemical oxygen demand (COD) and nutrients (e.g. phosphorus) represent one
52	avenue to improving the economics of biogas recovery from anaerobic treatment units (Jin et
53	al., 2017; Wan et al., 2016).
54	High retention membranes such as forward osmosis (FO) can be strategically integrated with
55	anaerobic digestion to achieve simultaneous wastewater treatment and resource recovery
56	(Ansari et al., 2017; Wang et al., 2016). The major advantages of FO compared to other
57	membrane processes include, low hydraulic pressure operation, low fouling propensity, easy
58	cleaning, and a high rejection of a broad range of contaminants. FO can also be coupled with
59	a draw solution regeneration process such as membrane distillation (MD) and reverse
60	osmosis to directly extract clean water from raw wastewater, while simultaneously
61	concentrating wastewater organics for subsequent anaerobic digestion (Luo et al., 2017;
62	Nguyen et al., 2016; Shahzad et al., 2017). Anaerobically digesting FO pre-concentrated

63 wastewater can produce biogas, which can be utilised by a combined heat and power engine 64 to produce electricity and thermal energy. Surplus electricity can be supplied to the grid and 65 the produced thermal energy can be used for MD and the anaerobic process. This latter 66 process also converts biologically bound phosphorus into a soluble form, thus allowing 67 phosphorus recovery as struvite (MgNH₄PO₄·6H₂O) or hydroxyapatite (Ca₁₀(PO₄)₆(OH)₂). 68 Interest in combining FO with anaerobic treatment has significantly increased in recent years 69 due to the potential advantages of low-energy wastewater stabilisation and resource recovery. 70 Recent studies have investigated FO-anaerobic integration in terms of draw solution selection 71 (Kim et al., 2016), process configurations (Qiu et al., 2016; Wang et al., 2017b; Zhang et al., 72 2017), membrane cleaning (Wang et al., 2017a), trace organic contaminant removal (Kim et 73 al., 2017), microbial composition (Wu et al., 2017), and energy dynamics (Onoda et al., 74 2017). However, there is a lack of studies which thoroughly assess the key FO operating 75 parameters that essentially govern anaerobic digestion performance. 76 Water recovery and the selected draw solution can influence the composition of pre-77 concentrated wastewater in terms of organics retention and salinity accumulation. The 78 incompatibility between high salinity and anaerobic microorganisms represents the most 79 prominent challenge associated with integrating FO with anaerobic treatment. Salinity 80 accumulation is inherently associated with the FO process. However, appropriate draw 81 solution selection can potentially reduce the amount of solute diffusing into the feed solution. 82 On the other hand, water recovery determines the accumulation of existing dissolved solutes 83 in wastewater. Determining the influence of these FO operating parameters on anaerobic 84 treatment is imperative to evaluate the feasibility and optimise biogas production from FO 85 pre-concentrated wastewater. 86 This study aims to evaluate the process performance and investigate the key challenges associated with integrating FO with anaerobic treatment. Specifically, this study optimises 87 88 the FO concentration factor (i.e. system water recovery) to balance the organic content and 89 salt concentration in pre-concentrated wastewater and their combined effects on methane 90 production. Representative inorganic and ionic organic draw solutes, namely sodium chloride 91 (NaCl) and sodium acetate (NaOAc) were compared in terms of FO membrane performance 92 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

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The lab-scale FO system used in this study consisted of a cross-flow membrane cell with an effective membrane area of 50 cm². 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 flatsheet 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

2.2 Biochemical methane potential experimental set-up

balance to ensure accurate flux measurements due to weight changes.

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

(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

123 attached to a water filled S-shaped air lock, and flexible plastic tubing was used to transfer 124 biogas to the collection gallery. The gas collection gallery included 16 inverted 1000 mL 125 plastic measuring cylinders, filled with a 1 M NaOH solution. The NaOH solution 126 sequestered the CO₂ and H₂S in the biogas, whilst the CH₄ gas displaced the NaOH inside the 127 cylinder. Daily measurements of CH₄ gas production were recorded. 128 2.3 Materials and chemicals 129 Wastewater (after primary sedimentation) and digested sludge were obtained from the 130 Wollongong Wastewater Treatment Plant (WWTP) in New South Wales, Australia. The 131 wastewater was used as a feed solution for FO pre-concentration experiments, whilst the 132 digested sludge was used as the inoculum for the BMP experiments. Basic characteristics of 133 the solutions are summarised in Table 1. 134 [Table 1] 135 Draw solutions were prepared using analytical grade NaCl or NaOAc. The draw solution 136 concentration was determined by OLI Stream Analyzer (OLI Systems, Inc., Morris Plains, 137 New Jersey, USA) calculations to achieve an equivalent osmotic pressure of 30 bar (similar 138 to that of seawater). 139 To accurately assess the effect of FO water recovery and draw solution on methane 140 production, BMP experiments were conducted using a synthetic wastewater solution. The 141 actual concentrate originating from the FO system was not used in the BMP experiments, as 142 the liquid volume produced by the lab-scale FO system was too small. Instead, a synthetic 143 solution was made to simulate the pre-concentration of wastewater components, as well as 144 the contribution of reverse draw solute flux. The concentrated stock solution was prepared to 145 contain 4 g/L glucose, 1 g/L peptone, 0.35 g/L urea, 0.175 g/L KH₂PO₄, 0.175 g/L MgSO₄, 146 0.1 g/L FeSO₄, and 2.25 g/L NaOAc. This stock solution was then diluted to accurately 147 simulate the COD of the initial primary effluent as well as the experimentally measured COD 148 amount in FO pre-concentrated wastewater at 50, 80 and 90% water recovery. A pre-149 determined amount of analytical grade NaCl or NaOAc was then added to the synthetic feed 150 to simulate salinity increase corresponding to each water recovery values as calculated from 151 the FO experimental results. Pure nitrogen gas was used to flush the BMP bottles and a 1 M

152 sodium hydroxide (NaOH) solution was used to absorb the carbon dioxide (CO₂) and 153 hydrogen sulphide (H₂S) from the biogas. 154 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 155 polysulfone layer for support. The membrane was positioned in FO mode (i.e. active layer 156 157 facing the feed solution) for all experiments. 158 2.4 Experimental protocol 159 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 160 161 concentration of 0.65 or 0.72 M, respectively, corresponding to the osmotic pressure of 162 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 163 164 was operated continuously until 90% of water had been recovered from the feed solution. The 165 initial volume of wastewater feed solution was 2 L, corresponding to a total concentrate 166 volume of 0.2 L. The water flux was continuously monitored, whilst the wastewater 167 conductivity, pH, and temperature were frequently measured. At specific time intervals, 168 samples of 10 mL volume were withdrawn from the feed solution for COD analysis to 169 represent the organic content in solution. The circulation flow rates were maintained at 1 170 L/min giving a cross-flow velocity of 16.7 cm/s. 171 At the conclusion of the experiment, the membrane was flushed at a higher cross flow 172 velocity for 30 minutes. This was achieved by replacing the feed and draw solutes with DI 173 water and doubling the cross-flow velocity (i.e. 33.4 cm/s). After flushing, fresh wastewater 174 was used as the feed solution to verify the water flux recoverability at the initial conditions. 175 After experimentally determining the pre-concentrated wastewater characteristics (i.e. COD 176 and salinity), a synthetic wastewater solution and each draw solute was used to simulate the 177 wastewater at 50, 80, and 90% water recovery. The COD results from the FO experiments 178 were used to represent the COD increase in wastewater. The synthetic wastewater solution 179 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. 180 181 Alternatively, the contribution of reverse solute flux was provided by adding a specified

182 amount of either NaCl or NaOAc to the synthetic wastewater solution. This reverse solute 183 flux contribution ($Salt_{RSF}$) was estimated using salinity measurement assuming: $Salt_{RSF}$ = 184 $Salt_{Total} - Salt_{WW}$, where $Salt_{Total}$ is the measured salt concentration at each water 185 recovery value and Saltww is the calculated salt concentration from the wastewater due to 186 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 187 188 solution was determined using electrical conductivity measurements and calibration curves 189 were then used to determine salt concentration. 190 The simulated FO pre-concentrate was mixed with digested sludge in each BMP bottle. An 191 inoculum volume of 500 mL and a substrate volume of 250 mL was selected, corresponding 192 to an inoculum/substrate ratio of 2:1. A reference condition was used to represent the 193 methane production of the inoculum, and real wastewater (i.e. FO feed solution with 0% 194 water recovery) was also used as a separate condition for comparison to the synthetic 195 wastewater. Prior to the BMP experiment, the bottles were purged with nitrogen gas, sealed, 196 and submerged in the water bath. The flexible plastic tubing was connected to the biogas 197 collection gallery. All BMP experiments were conducted in duplicate and biogas 198 measurements were recorded daily. The contents of each bottle was characterised before and 199 after the BMP experiment in terms of pH, conductivity, and COD. 200 2.5 Analytical methods 201 Standard methods were used during the analysis of basic water quality parameters. The 202 temperature, pH, and electrical conductivity were monitored using an Orion 4-Star 203 pH/conductivity meter (Thermo Scientific, Waltham, MA). COD samples were analysed 204 using a Hach DBR200 COD Reactor and Hatch DR/2000 spectrophotometer (program 205 number 435 COD HR) following the US-EPA Standard Method 5220 D. Total solids (TS) 206 and volatile solids (VS) of the primary effluent were determined within three days after 207 sample collection. All samples were stored at 4 °C in the dark. 208 2.6 Draw solute cost 209 Replenishment costs were calculated based on the pure water performance of the FO system 210 at the draw solute concentration corresponding to 30 bar osmotic pressure. The replenishment 211 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.

230 [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.

246 [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.

260 [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 evaluation 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.

275 [Figure 3]

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water.

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 approximatley 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 intial 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

296 [Figure 4]

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 m³ 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.

314 [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/m²h) was slightly lower than that of NaCl (17.4 L/m²h), 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.

329 [Figure 5]

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