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# Selection of forward osmosis draw solutes for subsequent integration with anaerobic treatment to facilitate resource recovery from wastewater

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## HIGHLIGHTS

• An FO draw solute selection protocol is proposed for wastewater pre-concentration.

• Reverse solute flux of inorganic draw solutes may inhibit methane production.

• Ionic organic draw solutes are suitable for integrating FO with anaerobic treatment.

• BMP analysis is a reliable tool for assessing draw solute suitability.

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## ABSTRACT

Forward osmosis (FO) can be used to extract clean water and pre-concentrate municipal wastewater to make it amenable to anaerobic treatment. A protocol was developed to assess the suitability of FO draw solutes for pre-concentrating wastewater for potential integration with anaerobic treatment to facilitate resource recovery from wastewater. Draw solutes were evaluated in terms of their ability to induce osmotic pressure, water flux, and reverse solute flux. The compatibility of each draw solute with subsequent anaerobic treatment was assessed by biomethane potential analysis. The effect of each draw solute (at concentrations corresponding to the reverse solute flux at ten-fold pre-concentration of wastewater) on methane production was also evaluated. The results show that ionic organic draw solutes (e.g., sodium acetate) were most suitable for FO application and subsequent anaerobic treatment. On the other hand, the reverse solute flux of inorganic draw solutions could inhibit methane production from FO pre-concentrated wastewater.

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#### 1. Introduction

The recently recognised value of clean water, energy, and nutrients in municipal wastewater has led to a paradigm shift in urban water management, toward a modern framework that incorporates resource recovery with the traditional sanitation mandate. The value of these resources goes beyond short-term economic outcomes, because long-term human health and environmental benefits can play an even greater role in wastewater management decisions. Water scarcity and environmental pollution have driven water reuse to become an integral function of modern wastewater treatment plants (Shannon et al., 2008). Further efforts to include

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energy and nutrient recovery are justified by the relationship between the stringency of effluent regulations and energy consumption (Iranpour et al., 1999), as well as concerns for worldwide phosphorus security (Koppelaar and Weikard, 2013).

Clean water reclamation from municipal wastewater is well established. However, a greater focus is required to further develop energy and nutrient recovery practices. The dilute nature of municipal wastewater is a major obstacle hindering energy and nutrient recovery. Thus, it is necessary to pre-concentrate municipal wastewater by five to ten-fold to achieve the required strength in terms of chemical oxygen demand (COD) for subsequent anaerobic treatment (Verstraete and Vlaeminck, 2011), through which energy and nutrients can be recovered in the form of biogas (Burn et al., 2013; Nghiem et al., 2014a) and struvite (MgNH<sub>4</sub>PO<sub>4</sub>·6H<sub>2</sub>O) (Garcia-Belinchón et al., 2013; Xie et al., 2014),







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respectively. The most common technique to recover nutrients after anaerobic treatment is via struvite precipitation. In this process, magnesium salt addition is required for struvite formation. However, because of the low ammonium and phosphate concentrations in municipal wastewater, magnesium salt must be added to obtain a concentration well above the stoichiometric ratio to facilitate struvite precipitation. In this instance, the pre-concentration of wastewater will lower the magnesium requirement for struvite formation (McCarty et al., 2011; Xie et al., 2014), thus significantly improving the economics of nutrient recovery (Garcia-Belinchón et al., 2013). The deployment of innovative technologies such as forward osmosis (FO) to pre-concentrate organic matter and nutrients can facilitate anaerobic treatment, thus allowing resource recovery to become economically viable.

FO is a promising technology for the pre-concentration of wastewater and has recently demonstrated potential for direct sewer mining (Lutchmiah et al., 2011; Xie et al., 2013). When applied directly for wastewater treatment, this concentration driven process has several significant advantages, including a high rejection of contaminants and low fouling propensity compared to pressure driven microfiltration. Therefore, FO can concentrate the organic matter and nutrients in wastewater to a small volume for potential integration with anaerobic treatment to facilitate resource recovery. Furthermore, FO provides robust pre-treatment for reverse osmosis (Hancock et al., 2013) or membrane distillation (Xie et al., 2013) for clean water production.

Reverse solute flux is an inherent phenomenon in FO. When integrating FO with a bioreactor, a major technical challenge is the migration of draw solute into the mixed liquor. This can severely affect the biological performance, particularly of the anaerobic treatment process as inhibitory substances are often the major cause of instability and failure of anaerobic treatment systems (Chen et al., 2008). Inorganic salts are widely used as draw solutes for FO, since they are usually inexpensive, capable of generating high osmotic pressures, and are less likely to induce significant internal concentration polarization (ICP). ICP associated with inorganic salts is low because of their small solute size and rapid diffusion: however, these properties often promote a high reverse solute flux (Shaffer et al., 2015). For example, sodium chloride has a high reverse solute flux, and therefore sodium concentrations are likely to exceed the value known to inhibit anaerobic treatment (3 g Na/L)(Feijoo et al., 1995) during wastewater pre-concentration.

Several draw solutes have been investigated with the intention of avoiding or reducing the effects of reverse solute flux on subsequent biological treatment. Lutchmiah et al. (2014) demonstrated that zwitterionic compounds, such as glycine, have a lower reverse solute flux compared to sodium chloride and the potential to increase the methane yield of concentrated wastewater due to their osmoprotectant properties. Bowden et al. (2012) proposed organic ionic salts as substitute draw solutes in osmotic membrane bioreactors (OMBRs), whereby salt accumulation has detrimental effects on biological performance. Other approaches involve comparing the microbial toxicity of draw solutions (Nawaz et al., 2013) or the long-term operation of alternative draw solutions in OMBRs to evaluate effects (Tang and Ng, 2014). Nonetheless, no studies have evaluated the potential impact of reverse solute flux on subsequent anaerobic treatment. This is despite the availability of the well-established biomethane potential (BMP) test, which can be used to simulate the anaerobic treatment process in batch mode to assess the methane production from different substrates (Koch et al., 2015; Mayer et al., 2014; Nghiem et al., 2014b).

In this study, a draw solute selection protocol was developed for FO systems which are integrated with anaerobic treatment. FO flux performance was assessed based on water flux and reverse solute flux. The effect of reverse solute flux on anaerobic treatment was evaluated by BMP analysis of draw solute-impacted substrate.

#### 2. Methods

#### 2.1. Preliminary draw solution selection protocol

A literature review of previous FO studies to pre-concentrate wastewater was conducted to select ten draw solutions to undergo experimental assessment. Firstly, organic draw solutions that have demonstrated a suitably high water flux and the expectation to have negligible impact on anaerobic treatment were considered. Secondly, inorganic draw solutions with low reverse solute flux were considered and sodium chloride was selected as a reference. OLI Stream Analyzer (OLI Systems, Inc., Morris Plains, New Jersey, USA) was then used to simulate osmotic pressure as a function of draw solution concentration, to verify the suitability for further FO experimental assessment and biological screening.

#### 2.2. Materials and chemicals

Cellulose triacetate (CTA) membrane with embedded polyester screen support was acquired from Hydration Technologies Innovation (HTI) (Albany, Oregon, USA). Digested sludge was obtained from a full-scale wastewater treatment plant (Wollongong, Australia) and was used as inoculum for the BMP measurements. All draw solutes used in this study were of analytical grade.

#### 2.3. Forward osmosis system

FO experiments were conducted using a lab-scale, cross-flow FO membrane system (Supplementary Data, Fig. S1). The FO membrane cell consisted of two symmetric flow channels each with length, width, and height of 130, 95, and 2 mm, respectively, and an effective membrane area of 123.5 cm<sup>2</sup>.

The feed and draw solutions were circulated by two variable speed gear pumps (Micropump, Vancouver, Washington, USA) at 1 L/min (corresponding to a cross-flow velocity of 9 cm/s) and was regulated by two rotameters. The working volumes of the feed and draw solution reservoirs were 3 and 2 L, respectively. The draw solution reservoir was positioned on a digital balance (Mettler-Toledo Inc., Hightstown, New Jersey, USA) and weight changes were recorded to determine permeate water flux. For ionic draw solutions, a reservoir containing a highly concentrated solution was also placed on the digital balance and was intermittently dosed into the draw solution to maintain constant osmotic pressure. The conductivity of the draw solution was continuously measured by a conductivity probe (Cole-Parmer, Vernon Hills, Illinois, USA), which was connected to a controller (control accuracy of ±0.1 mS/cm) and a peristaltic pump to automatically regulate the draw solution concentration. For the neutral (covalent) organic draw solutions, concentration was manually controlled by adding the correct volume of highly concentrated solution every 2 h.

#### 2.4. Forward osmosis assessment

The flux performance of each draw solution was evaluated by using the lab-scale, cross-flow FO system to determine water flux  $(J_w)$  and reverse solute flux  $(J_s)$ . FO experiments were conducted according to the standard procedure previously described by Cath et al. (2013). Analytical grade solutes were dissolved in DI water at concentrations corresponding to an osmotic pressure of 30 bar. This osmotic pressure was selected for two reasons. Firstly, seawater has an approximate osmotic pressure of 30 bar and could be used as a readily available and inexpensive NaCl solution. Secondly, higher osmotic pressures were not investigated due to the corresponding increase in draw solute viscosity (particularly

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