



## Porous forward osmosis membranes for polishing biologically treated wastewater: Condition optimization and draw solution recovery

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### ABSTRACT

Forward osmosis has a great potential to be applied in treating biomass containing streams. However, the reverse salt permeation and the high energy consumption in draw solutes recovery pose two main obstacles. In this work, a porous FO membrane water extraction system is proposed to overcome these shortages. Using polyelectrolytes as draw solution and a porous PAN FO membrane, the system could recover water from a biologically treated wastewater (from a pilot anaerobic fluidized-bed bioreactor) with a stable flux of 7 L/m<sup>2</sup>.h using only 0.94 bar of osmotic pressure generated by 0.8% PSS draw solution. Compared with the dense FO membranes provided by HTI, the porous FO membrane could sustain a much higher flux due to its significantly higher water permeability. Meanwhile, the porous FO membrane has high rejection (99.5%) for PSS polyelectrolytes. Finally, draw solutes could be efficiently recovered (~99%) using a low pressure UF process between each FO cycle.

### 1. Introduction

To address the extensive energy consumption and severe membrane fouling issues faced by conventional pressure-driven membrane technologies, forward osmosis (FO) is a promising technology for applications such as fresh water extraction from high TOC wastewater (e.g. membrane bio-reactor, MBR) or re-concentrating of processed bio-resources (Achilli et al., 2009; Cath et al., 2006). Unlike the conventional hydraulic pressure-driven membrane, the FO membrane relies on the osmotic pressure to extract water from low sanity side to high salinity side. Salts are by far the most popular draw solutes as they bear low cost and can effectively generate osmotic pressure. As a typical application, the FO-MBR (or OMBR) process has advantages of high rejection against organic matter and potentially low fouling tendency (Achilli et al., 2009; Zhang et al., 2012). However, one of the critical issues of the OMBR is the salt accumulation on the feed side due to the reverse solute permeation through the FO selective layer. The salt accumulation has a negative effect on the activity of bacteria, which would cause lower water recovery and nutrient removal efficiencies (Holloway et al., 2014; Holloway et al., 2015; Luo et al., 2018). One effort to remove the accumulated salinity is to treat the mix liquor with a UF

“dialysis” process periodically (Holloway et al., 2014; Holloway et al., 2015), which complicates the overall operations. Another concern for the OMBR is the energy efficiency of the current salt-driven OMBR system is not as economical as expected. Although little energy was consumed on the pumps of operation of FO, the relatively higher cost was paid for draw solution (DS) re-concentration (Shaffer et al., 2015).

Alternatively, a porous FO membrane could be incorporated into OMBR process, which was developed for the first time recently (Qi et al., 2016; Qi et al., 2015). In the porous FO membrane system, salt can diffuse through the FO membrane freely and larger molecular weight solutes are retained to maintain an osmotic pressure difference. In such a configuration, salt cumulation and high energy solute recovery can be avoided as high concentration salt solutions are not used as DS. Alternatively, the potential draw solutes (while not limited to) are polyelectrolytes (Qi et al., 2016; Qi et al., 2015), hydrogel (Li et al., 2013), nanoparticles (Ge et al., 2011; Ling et al., 2011), and temperature-sensitive micellar solutions (Gadelha et al., 2014). The merits of porous FO system includes 1) elimination of the usage of salt DS; 2) extremely low osmotic pressure of DS required due to the high water permeability of the selective layer and 3) extremely low back diffusion of draw solutes due to the relatively large size of draw solutes. In

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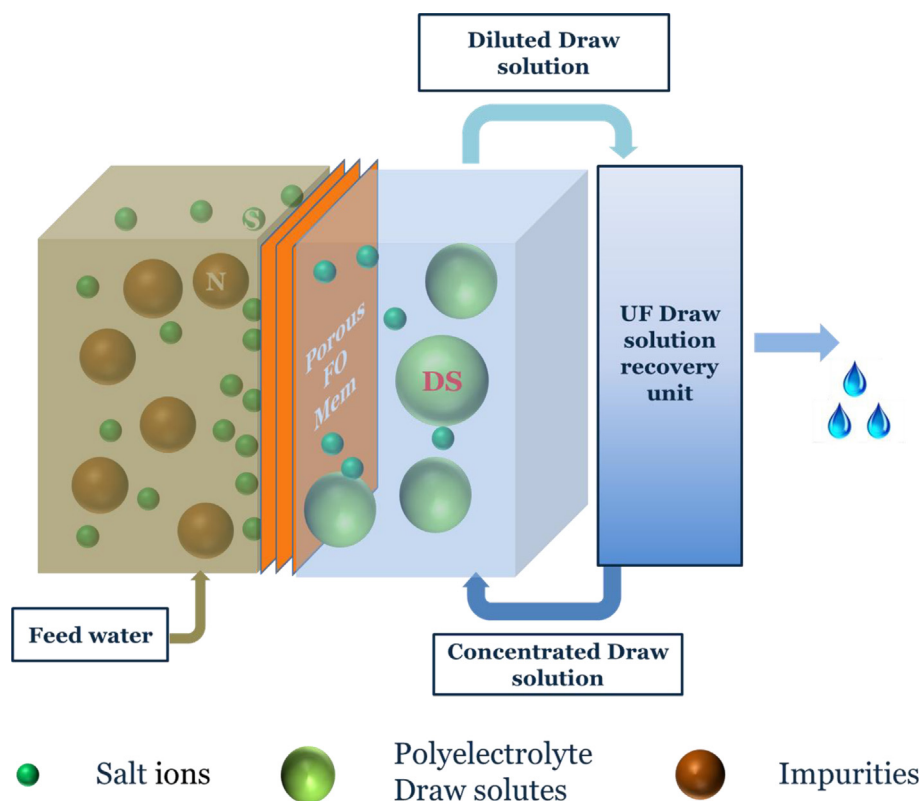


Fig. 1. The schematic drawing of the treatment of wastewater using porous FO system with DS recovery unit.

literature, a water flux of  $\sim 7 \text{ L/m}^2\cdot\text{h}$  can be achieved by using only 0.1 bar osmosis pressure of DS (Qi et al., 2015). However, the membrane performance in realistic applications (such as biologically-treated wastewater and draw solutes recovery) is still to be explored.

Therefore, the current study focuses on evaluating the performance of the porous FO system in real wastewater recovery application. A sewage effluent was collected from a local anaerobic fluidized bed pilot plant in Singapore and used as feed solution (FS). The polyelectrolyte solution was used as DS (Fig. 1). The FO performance was evaluated under various testing conditions, such as different cross-flow velocity (CFV), FS concentration, and DS concentration. The comparison of the FO performance between a commercial HTI FO membrane and the porous FO membrane was also evaluated. Finally, the DS re-concentration was enabled using a UF process. To the best knowledge of the authors, it is the first time to use the real wastewater to evaluate the porous FO membrane performance. This study targets to address the two main dilemmas (reverse salt diffusion and high energy DS recovery) currently faced by the osmotic MBR.

## 2. Material and methods

### 2.1. Chemicals and reagents

The polymer solution was prepared by using polyacrylonitrile (PAN, average  $M_w \sim 150 \text{ kDa}$ , Sigma-Aldrich), N, N-dimethylformamide (DMF,  $\geq 99.8\%$ , Sigma-Aldrich) and lithium chloride (LiCl, Sinopharm). The deionized (DI) water (18 M $\Omega$ ), 10 mM sodium chloride solution (NaCl,  $\geq 99.8\%$ , Sigma-Aldrich), and sewage (collected from a pilot anaerobic fluidized bed bioreactor in a local municipal plant, Singapore) were utilized as FS, respectively. A series concentration of poly (sodium 4-styrene-sulfonate) (PSS,  $M_w \sim 70 \text{ kDa}$ , received as 25–30% aqueous solution from Sigma-Aldrich) solutions were utilized as DS. Unless stated otherwise, the DI water and analytical grade chemicals were used to prepare all solutions.

### 2.2. Membrane fabrication

A UF-like porous FO membrane was prepared according to the reference (Qi et al., 2016). Briefly, a casting knife (Elcometer Pte Ltd, gate height set at 150  $\mu\text{m}$ ) was used to spread the polymer dope solution (consists of 18% PAN, 2% LiCl and 80% DMF) onto a clean glass plate at room temperature ( $23 \pm 1^\circ\text{C}$ ). Then, the cast polymer dope film was coagulated in a tap water bath to form the PAN membranes. After the solvent exchange process, these nascent membranes were then annealed in DI water at  $65^\circ\text{C}$  for 2 mins. This annealing step was used to reduce the membrane pore size by tightening the surface pore structure (Qi et al., 2012a; Qi et al., 2011; Qi et al., 2012b). After cooling down to room temperature, all membranes were soaked in 1.5 M sodium hydroxide (NaOH, Sigma-Aldrich) solution at  $45^\circ\text{C}$  for 1.5 h to partially hydrolyzed PAN. The NaOH treatment introduces negatively charged carboxylic groups to the membrane surface and makes it more hydrophilic (Qi et al., 2011). Finally, the treated membranes were washed with tap water until the pH of the water became neutral. The washed membranes were stored in the  $4^\circ\text{C}$  before usage.

### 2.3. Evaluation of FO performance

A cross-flow FO setup (Qi et al., 2015) was used to evaluate the FO performance. All FO tests were carried out at room temperature (i.e.,  $25 \pm 2^\circ\text{C}$ ). The CFV range was 0.9–7 cm/s for both DS and FS. A new FO membrane was placed in the FO cell with an active filtration area of  $\sim 35 \text{ cm}^2$  for each test. NaCl solution or sewage (located in local Water Reclamation Plant, Hang Zhou, characteristics listed in Table 1) were used as the feed solution (FS). Polyelectrolyte solutions containing 0.2%, 0.4%, 0.8%, 1.6%, and 3.2% PSS were used as the DS. According to van't hof equation, the corresponding osmotic pressure is calculated to be 0.24, 0.47, 0.94, 1.88, 3.76 bar based on the  $\text{Na}^+$  concentration (assuming complete ionization of  $\text{Na}^+$ ) (Qi et al., 2016). Osmotic pressures of the salt solutions were calculated by Morse equation. The

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