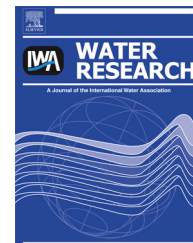




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# Gypsum scaling in pressure retarded osmosis: Experiments, mechanisms and implications

Minmin Zhang<sup>a,b,1</sup>, Dianxun Hou<sup>a,b,1</sup>, Qianhong She<sup>a,b</sup>,  
Chuyang Y. Tang<sup>a,b,\*</sup>

<sup>a</sup> School of Civil and Environmental Engineering, Nanyang Technological University, Singapore 639798, Singapore

<sup>b</sup> Singapore Membrane Technology Centre, Nanyang Technological University, Singapore 639798, Singapore

## ARTICLE INFO

### Article history:

Received 26 June 2013

Received in revised form

19 September 2013

Accepted 27 September 2013

Available online 11 October 2013

### Keywords:

Pressure retarded osmosis (PRO)

Scaling

Internal concentration polarization

(ICP)

Reverse solute diffusion

Gypsum

## ABSTRACT

Pressure retarded osmosis (PRO) is an osmotically-driven membrane process that can be used to harvest salinity-gradient power. The PRO performance (both water flux and power density) can be severely limited by membrane fouling. The current study, for the first time, investigates PRO scaling in a bench-scale pressurized system using calcium sulfate dihydrate (gypsum) as a model scalant. In addition to the bulk feed solution (FS) saturation index ( $SI_{\text{bulk}}$ ), gypsum scaling was found to be strongly affected by the draw solution (DS) type and concentration, the applied hydraulic pressure, and the membrane orientation. The commonly recommended active layer facing draw solution (AL-DS) orientation was highly prone to internal scaling. In this orientation, severe internal concentration polarization (ICP) of scaling precursors induced gypsum clogging in membrane support layer even when the FS was undersaturated (e.g.,  $SI_{\text{bulk}} = 0.8$ ). At higher  $SI_{\text{bulk}}$  values, external gypsum crystal deposition occurred in addition to internal scaling. More severe scaling was observed when the DS contained scaling precursors such as  $\text{Ca}^{2+}$  or  $\text{SO}_4^{2-}$ , suggesting that the reverse diffusion of these precursors into the FS can significantly enhanced gypsum scaling. Increasing applied hydraulic pressure could enhance reverse solute diffusion and thus result in more severe gypsum scaling when the DS contained scaling precursors. A conceptual model, capturing the two important PRO scaling mechanisms (ICP of scaling precursors from FS and reverse diffusion of scaling precursors from the DS), is presented to rationalize the experimental results. Our results provide significant implications for PRO scaling control.

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## 1. Introduction

Pressure retarded osmosis (PRO) is an osmotically-driven membrane (OM) process, where water flows from a low-salinity feed solution (FS) to a high-salinity draw solution

(DS) against a hydraulic pressure (Achilli and Childress, 2010; Ramon et al., 2011; Zhao et al., 2012). It has gained much interest for its potential in harvesting osmotic power arising from two liquid streams with different salinities (Loeb, 2002; Achilli and Childress, 2010; Ramon et al., 2011; Zhao et al.,

\* Corresponding author. Nanyang Technological University, N1-1B-35, 50 Nanyang Avenue, Singapore 639798, Singapore. Tel.: +65 67905267; fax: +65 67910676.

E-mail addresses: [cytang@ntu.edu.sg](mailto:cytang@ntu.edu.sg), [qqiu316@yahoo.com](mailto:qqiu316@yahoo.com) (C.Y. Tang).

<sup>1</sup> These authors contributed equally to this study.

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<http://dx.doi.org/10.1016/j.watres.2013.09.051>

2012). Presumably, one of the limiting factors in PRO operation is membrane scaling. Like pressure-driven reverse osmosis (RO) process, PRO may suffer from the precipitation of inorganic scalants when low-solubility minerals (e.g., calcium sulfate, barium sulfate, and calcium carbonates) in feed solution are supersaturated (Shih et al., 2005; Fritzmann et al., 2007; Antony et al., 2011; Arkhangelsky et al., 2012). Membrane scaling can lead to severe flux decline and even damages to the membrane structure (Arkhangelsky et al., 2012).

Despite the significant advancement of PRO technology in recent years (Achilli et al., 2009; Thorsen and Holt, 2009; Xu et al., 2010; Yip et al., 2011; Chou et al., 2012; Kim and Elimelech, 2012; She et al., 2012b; van der Zwan et al., 2012; Xiao et al., 2012), there is still a lack of systematic investigation on scaling in PRO processes up to date. Some general lessons may be learnt from the RO experience, such as more severe scaling is likely expected for solutions with greater saturation index (SI) (Antony et al., 2011). Other feed water properties, such as pH and temperature, may also affect membrane scaling via their influence on the scalant solubility. Nevertheless, due to the different transport phenomena (e.g., internal concentration polarization (ICP) (Loeb et al., 1997; McCutcheon and Elimelech, 2006; Tang et al., 2010) and reverse solute diffusion (Phillip et al., 2010; Tang et al., 2010; Yip and Elimelech, 2011; Zou et al., 2011; She et al., 2012a; van der Zwan et al., 2012)) involved in the osmotically-driven PRO process, additional scaling mechanisms are likely applicable in PRO. In this sense, the handful number of fouling and scaling studies (Mi and Elimelech, 2008, 2010; Lee et al., 2010; Tang et al., 2010; Jung et al., 2011; Zou et al., 2011; Liu and Mi, 2012) on forward osmosis (FO, a closely related OM process where applied pressure = 0) may provide useful references. Previous studies demonstrated that calcium sulfate dihydrate (gypsum) scaling of cellulose triacetate (CTA) FO membrane is predominately affected by the feed solution composition and controlled by bulk crystals deposition (Mi and Elimelech, 2010; Arkhangelsky et al., 2012; Liu and Mi, 2012). Some recent studies reported that the mechanism of reverse solute diffusion ( $J_s$ ) played an important role in organic/biological fouling of FO – divalent ions in the DS can diffuse through the semi-permeable membrane and interact with foulants in the FS to promote severe fouling (Zou et al., 2011; She et al., 2012a). Up to date, there has not been any specific studies focusing on the effect of reverse solute diffusion on FO/PRO scaling, though one may hypothesize that the reverse diffusion of scaling precursors (such as  $\text{Ca}^{2+}$ ) can similarly interact with their scaling pairs (e.g.,  $\text{SO}_4^{2-}$  and  $\text{CO}_3^{2-}$  in the FS and thereby lead to exacerbated scaling. Furthermore, compared to FO, the mechanism of  $J_s$ -promoted scaling is likely more complicated for PRO, since the rate of reverse diffusion can be greatly affected by the applied pressure (Kim and Elimelech, 2012; She et al., 2012b). Thus, the synergistic effects of reverse solute diffusion and applied pressure on PRO scaling can have important implications for PRO design and operation.

The membrane orientation also plays an important role in membrane fouling/scaling of FO and PRO (Mi and Elimelech, 2008; Tang et al., 2010; Jin et al., 2011, 2012; Zou et al., 2011; Zhao et al., 2012; She et al., 2013). It has been generally recommended that the active layer facing FS (AL-FS) orientation

shall be used for FO applications to avoid severe fouling (Mi and Elimelech, 2008; Tang et al., 2010) and better rejection against feed solutes (Jin et al., 2011, 2012). In contrast, there seems to be a consensus that the active layer facing DS (AL-DS) orientation shall be used for PRO applications to achieve greater water flux, improved power density, and better mechanical stability. Nevertheless, severe ICP of feed solutes can occur in the AL-DS orientation. The more severe ICP in PRO, together with its enhanced reverse diffusion of draw solutes at high pressure, may suggest that PRO may have a significantly different (and more severe) scaling behavior compared that in FO.

The objective of this paper is to provide a mechanistic understanding of PRO scaling using gypsum as a model scalant. The effects of feed solution and draw solution compositions, membrane orientation, and applied hydraulic pressure on PRO water flux and power density were systematically investigated. The mechanisms governing PRO scaling are illustrated. The results of the current study may provide important insight into PRO scaling and scaling control.

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## 2. Material and methods

### 2.1. Membranes

A commercial CTA FO membrane (Hydration Technology Innovations, Albany, OR) was used in our PRO scaling tests. The membrane has a water permeability of  $2.08 \times 10^{-12}$  m/s.Pa, a NaCl permeability of  $1.76 \times 10^{-7}$  m/s, and a structure parameter of 0.48 mm (She et al., 2012b). Membrane samples were stocked in deionized (DI, Millipore) water at 4 °C and soaked in DI water at room temperature for over 24 h before use.

### 2.2. Chemicals and solutions

All solutions were prepared with ACS grade chemicals/reagents as received without further purification. MilliQ water with resistivity of 18.2 M $\Omega$  cm was used throughout the study unless otherwise stated. DSs were prepared using NaCl,  $\text{CaCl}_2$ , and  $\text{Na}_2\text{SO}_4$ . FSs with different bulk gypsum saturation indexes ( $\text{SI}_{\text{bulk}}$ ) from 0.8 to 2.3 were used to investigate gypsum scaling in PRO process. Specifically, the saturation index of calcium sulfate is defined as  $(\text{Ca}^{2+})(\text{SO}_4^{2-})/K_{\text{sp}}$ , where  $(\text{Ca}^{2+})$  and  $(\text{SO}_4^{2-})$  are the activities of the calcium and sulfate ions, respectively, and  $K_{\text{sp}}$  is the solubility constant for gypsum. Detailed composition and properties of the FSs are summarized in Supporting Information S1. The solution osmotic pressures and  $\text{SI}_{\text{bulk}}$  values were calculated using the OLI's Stream Analyzer 3.1 software (OLI Systems, Inc., Morris Plains, NJ).

### 2.3. Bench-scale PRO test system

The schematic diagram and detailed description of the bench-scale PRO system are given elsewhere (She et al., 2012b). Briefly, the system includes a crossflow membrane cell (active membrane area  $\sim 140$  cm<sup>2</sup>) modified from the commercially available GE SEPA II cells (Osmonics, GE, CT). Diamond-shaped spacers were placed in both DS and FS channels to provide support to the membrane and to promote mass transfer (Wang et al., 2010; She et al., 2012b). A high pressure

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