



Contents lists available at ScienceDirect

Water Research

journal homepage: www.elsevier.com/locate/watres

Silica scaling in forward osmosis: From solution to membrane interface

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ARTICLE INFO

Article history:

Received 3 June 2016

Received in revised form

5 August 2016

Accepted 31 October 2016

Available online xxx

Keywords:

Forward osmosis

Silica scaling

Cellulose triacetate

Polyamide

Silica aggregation

Silica polymerisation

ABSTRACT

Membrane silica scaling hinders sustainable water production. Understanding silica scaling mechanisms provides options for better membrane process management. In this study, we elucidated silica scaling mechanisms on an asymmetric cellulose triacetate (CTA) membrane and polyamide thin-film composite (TFC) membrane. Scaling filtration showed that TFC membrane was subjected to more severe water flux decline in comparison with the CTA membrane, together with different scaling layer morphology. To elucidate the silica scaling mechanisms, silica species in the aqueous solution were characterised by mass spectrometry as well as light scattering. Key thermodynamic parameters of silica surface nucleation on the CTA and TFC membranes were estimated to compare the surface nucleation energy barrier. In addition, high resolution X-ray photoelectron spectroscopy resolved the chemical origin of the silica-membrane interaction via identifying the specific silicon bonds. These results strongly support that silica scaling in the CTA membrane was driven by the aggregation of mono-silicic acid into large silica aggregates, followed by the deposition from bulk solution onto the membrane surface; by contrast, silica polymerised on the TFC membrane surface where mono-silicic acid interacted with TFC membrane surface, which was followed by silica surface polymerisation.

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1. Introduction

Membrane-based water purification processes have played a crucial role in mitigating water scarcity worldwide (Elimelech and Phillip, 2011; Shannon et al., 2008). One promising osmosis-driven membrane process, forward osmosis (FO), could potentially find a wide range of applications in water and wastewater treatment, particularly of challenging and difficult to treat wastewaters (Shaffer et al., 2015; Xie et al., 2016a). This capacity was mainly due to less detrimental and more reversible FO fouling in comparison with pressure-driven membrane process, such as and reverse osmosis (RO) (Lee et al., 2010; Mi and Elimelech, 2010; Xie et al., 2015a,b; Zou et al., 2011). Consequently, there have been several successful demonstrations of FO for the treatment of wastewaters with high fouling propensity with no or limited pretreatment, such as, anaerobic digester concentrate (Holloway et al., 2007; Xie et al., 2014), activated sludge solution (Achilli et al., 2009; Cornelissen et al., 2008), and municipal wastewater (Cath et al., 2005;

Valladares Linares et al., 2011; Xie et al., 2013).

Silica, abundant in brackish groundwater, constrained water recovery and production in membrane filtration due to scaling (Milne et al., 2014). Previous knowledge from RO desalination showed that silica scaling was complicated due to the nature of silica chemistry, and was highly dependent on the operating condition. Various silica scaling morphology was revealed on RO membranes ranging from patches of semi-transparent deposits to opaque, milky to white gel, which indicated the complexity of silica scaling mechanisms. Although previous research efforts were made to gain a better understanding of silica scaling mechanisms and to develop scaling control strategies, silica scaling remains a major unsolved problem for membrane desalination units.

Silica scaling was profiled in FO using silica colloidal particles as well as reactive silica. For instance, Boo et al. (2012) demonstrated severe silica colloidal scaling on FO membrane under elevated reverse salt diffusion. Reactive silica in seawater was attributed to the silica polymerisation on FO membrane surface, which was further aggravated by other natural organic compounds (Li et al., 2012). Silica scaling mechanisms were proposed by Mi and Elimelech (2013) following adhesion force measurement, where the adhesion force between polyamide thin-film composite (TFC)

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membrane was stronger than cellulose triacetate (CTA) membrane. However, this indirect approach cannot accurately reflect the silica scaling mechanisms in the filtration of aqueous solution as well as at the silica-membrane interface. In addition, the underlying chemical origin of silica membrane scaling remains unclear.

In this study, we investigated reactive silica scaling in FO using TFC and CTA membranes. Silica scaling behaviours – water flux decline and scaling layer morphology – were demonstrated. Silica scaling mechanisms were elucidated by characterising silica species in the aqueous solution, and thermodynamic parameters and chemical state of silicon bonds during silica-membrane interaction. Mass spectrometry and light scattering were used to characterise size and structure of silica species in the aqueous solution during silica scaling. Key parameters of silica surface nucleation were estimated using a series of silica filtration experiments. High resolution X-ray photoelectron spectroscopy was used to resolve the chemical origin of the silica-membrane interaction by identifying the specific silicon bonds.

2. Materials and methods

2.1. FO membranes and silica chemistry

An asymmetric cellulose triacetate (CTA) and a polyamide thin-film composite (TFC) forward osmosis (FO) membrane were employed in this study. The CTA membrane was composed of a cellulose triacetate layer with an embedded woven support mesh (Cath et al., 2006; McCutcheon and Elimelech, 2008). The TFC membrane was made of a thin selective polyamide active layer on top of a porous polysulfone support layer (Cath et al., 2013; McGinnis et al., 2013). The CTA membrane surface was abundant with hydroxyl functional groups, while the TFC membrane surface was enriched by carboxylic functional group. Details regarding the membrane surface chemistry can be found in our previous publication (Xie and Gray, 2016). A comprehensive membrane characterisations for both CTA and TFC membranes were provided in the Supplementary Data, including estimated pore size (Table S1), membrane surface zeta potential (Fig. S3), membrane surface chemistry (Fig. S3), membrane contact angle (Table S4), and surface roughness (Fig. S4).

Silica stock solutions with a concentration of 0.2 M Na_2SiO_3 were prepared by dissolving sodium metasilicate ($\text{Na}_2\text{SiO}_3 \cdot 9\text{H}_2\text{O}$, assay >98%, Sigma-Aldrich) in MilliQ water. The stock solution was prepared freshly to avoid undesirable silica condensation (Felmy et al., 2001). The stock solution was dosed into a background electrolyte containing 20 mM NaCl and 1 mM NaCHO_3 at solution pH of 6.5. The reactive silica concentration of the working solution was determined by the molybdate yellow method (Method 8185, Hach DR5000) at wavelength of 815 nm.

2.2. FO setup and silica scaling experimental protocol

The FO membrane cell was made of acrylic plastic and designed to hold a flat-sheet membrane under moderate pressure differential without any physical support. Flow channels were engraved in acrylic blocks that made up the feed and permeate semi-cells. Each channel was 2 mm deep, 90 mm wide, and 120 mm long. Details of this FO filtration setup were provided in our previous publications (Xie and Gray, 2016; Xie et al., 2016b), and can be found at Fig. S1, Supplementary Data. Crossflow rate of 1 L/min (corresponding to crossflow velocity of 9 cm/s) was maintained for both the feed and draw solutions using micro gear pumps. The FO water flux was determined by measuring the weight changes of the feed solution at specific time intervals with a precision balance connected to a computer and a data logging system.

Silica scaling experiment was performed on both CTA and TFC membranes. The same initial water flux of $25 \text{ L m}^{-2} \text{ h}^{-1}$ was achieved for both the CTA and TFC membranes using NaCl draw solution (2.5 M for CTA membrane; and 1.5 M for TFC membrane). Specifically, a new membrane sample, with the active layer facing the feed solution, was placed in the membrane cell before each experiment and stabilised in FO mode with deionised water for 1 h to obtain a stable water flux. Next, the silica scaling experiment was performed for about 24 h to obtain approximately 1600 mL cumulative permeate volume at the conclusion of each experiment. The silica scaling solution contained a reactive silica concentration of 6 mM in a background electrolyte containing 20 mM NaCl and 1 mM NaCHO_3 at solution pH of 6.5, which resulted in a silica saturation index of 1.6. Other experimental conditions were: cross-flow rate of 1 L/min (corresponding to the cross-flow velocity of 9 cm/s), ambient pH (pH 6.5), and temperature of $25.0 \pm 0.1 \text{ }^\circ\text{C}$. Water flux was continuously monitored throughout the fouling experiments by a data logger. A baseline experiment (i.e., feed without silica foulant) was also carried out to correct the flux decline due to the continuous concentration of the feed solution and dilution of the draw solution, as described in our previous publication (Xie et al., 2015b). The feed solution was continuously sampled to quantify the evolution of silica polymerisation. At the conclusion of each scaling experiment, the scaled membrane was air dried in a desiccator for scanning electron microscopy imaging.

2.3. Quantification of silica polymerisation during scaling

Mass spectrometry and light scattering were used to continuously track the growth and polymerisation silica from reactive silica (including a range of low molecular weight monomers, dimers, and trimers), to colloidal silica. Combining these techniques allowed us to capture the entire silica polymerisation process, thereby facilitating the understanding of silica scaling mechanism.

Electrospray ionization mass spectrometry (ESI-MS) was used to identify the evolution of reactive silica species. The ESI-MS spectra were recorded by direct infusion in negative ion mode for 15 min. The feed liquid sample was diluted by methanol (50/50, v:v) to enhance electrospray ionization process and minimize ion suppression. The direct infusion flow of the analyte was 10 $\mu\text{L}/\text{min}$. ESI negative ionization was used with a detector voltage of 3 kV, desolvation temperature of $250 \text{ }^\circ\text{C}$, and heating block temperature of $200 \text{ }^\circ\text{C}$. High purity nitrogen was used as the nebulizing gas at a flowrate of 1 L/min. The time-average MS spectra were reported to identify the silica size and structure.

Dynamic and static light scattering were used to monitor the hydrodynamic radius of silica and weight-average molecular weight of silica. Light scattering experiments were conducted with a multi-angle goniometer setup (BI-200SM, Brookhaven Instruments, NY, USA) with a He-Ne laser with a wavelength of 633 nm as a light source. Dynamic light scattering measurements were obtained with a fixed detector at 90° . For static light-scattering measurements, the normalized scattered light intensity was obtained by altering detector over an angular range of $17\text{--}135^\circ$, corresponding to wave vectors $0.0046 < q < 0.0305 \text{ nm}^{-1}$. The scattering intensities from static light scattering obtained as Rayleigh ratios at an angle θ were processed in a Zimm plot in order to obtain the weight-averaged molar mass. The feed solution samples were monitored continuously over the scaling experiment to record the hydrodynamic radius and weight-averaged molar mass of silica.

2.4. Kinetics and characterisation of silica scaling in FO membrane

A series of silica scaling experiments with varying saturation

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