



Trace organic contaminant rejection by aquaporin forward osmosis membrane: Transport mechanisms and membrane stability

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ABSTRACT

We investigated transport mechanisms of trace organic contaminants (TrOCs) through aquaporin thin-film composite forward osmosis (FO) membrane, and membrane stability under extreme conditions with respect to TrOC rejections. Morphology and surface chemistry of the aquaporin membrane were characterised to identify the incorporation of aquaporin vesicles into membrane active layer. Pore hindrance model was used to estimate aquaporin membrane pore size as well as to describe TrOC transport. TrOC transport mechanisms were revealed by varying concentration and type of draw solutions. Experimental results showed that mechanism of TrOC transport through aquaporin-embedded FO membrane was dominated by solution-diffusion mechanism. Non-ionic TrOC rejections were molecular-weight dependent, suggesting steric hindrance mechanisms. On the other hand, ionic TrOC rejections were less sensitive to molecular size, indicating electrostatic interaction. TrOC transport through aquaporin membrane was also subjected to retarded forward diffusion where reverse draw solute flux could hinder the forward diffusion of feed TrOC solutes, reducing their permeation through the FO membrane. Aquaporin membrane stability was demonstrated by either heat treatment or ethanol solvent challenges. Thermal stability of the aquaporin membrane was manifested as a relatively unchanged TrOC rejection before and after the heat treatment challenge test. By contrast, ethanol solvent challenge resulted in a decrease in TrOC rejection, which was evident by the disappearance of the lipid tail of the aquaporin vesicles from infrared spectrum and a notable decrease in the membrane pore size.

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1. Introduction

Aquaporins are ubiquitous biological membrane proteins whose identification provided a molecular basis for transmembrane water transport as a molecular water channel (Agre and Kozono, 2003; Gomes et al., 2009). Molecular simulation together with experimental evidence revealed that water molecules formed a single file inside the constriction region of the aquaporin pores, and that water permeation happened as a result of correlated displacements of water molecules in this region (Agre, 2006; Wang et al., 2005).

One remarkable property of aquaporins is an effective water conductance at rates close to 10^9 molecules per second and an excellent exclusion of solutes (Tajkhorshid et al., 2002).

Aquaporin-based biomimetic membranes have been proposed for desalination and water filtration by leveraging the efficient and selective water permeation of aquaporin water channels (Tang et al., 2013). For instance, Zhao and co-workers reported a thin-film composite aquaporin-embedded reverse osmosis (RO) membrane with NaCl rejection of 97% at 5 bar (Zhao et al., 2012). Zhong et al. (2012) employed triblock copolymer vesicle rupture method for aquaporin nanofiltration (NF) membrane. Attributed to the embedded aquaporin vesicles, Li et al. reported high water flux of an aquaporin-based hollow fibre NF membrane (Li et al., 2015). Via layer-by-layer approach, Wang et al. immobilized aquaporin-

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incorporated lipid bilayer on polyelectrolyte membrane to achieve excellent permeability and salt rejection with a high stability (Wang et al., 2015).

Prior aquaporin-based membrane investigations focused on achieving high water flux and solute rejection of a range of ions (e.g., NaCl, MgCl₂ or Na₂SO₄) that are representative in seawater desalination or water filtration. One critical category of chemicals with emerging concerns is trace organic contaminants (TrOCs) that are ubiquitous in wastewater and sewage-impacted water bodies. Despite increasing efforts to understand aquaporin membrane, investigations to examine TrOC transport through the aquaporin membrane are still scarce. In a sole study, Madsen et al. reported more than 97% rejection of three neutral pesticides (atrazine, 2,6-dichlorobenzamide and desethyl-desisopropyl-atrazine) by the aquaporin forward osmosis (FO) membrane (Madsen et al., 2015). Nevertheless, the potential of aquaporin membranes cannot be fully evaluated without a comprehensive examination of TrOCs with varying properties.

Delicate aquaporin vesicles embedded in the membrane active layer may narrow the waste stream sources or require intense pre-treatment. Indeed, as a biological protein, aquaporin is vulnerable to harsh physical or chemical environments, such as, temperature, pH and oxidative compounds. However, the stability of these aquaporin vesicles remains unknown. Li and coauthors examined stability and performance of aquaporin membrane under harsh chemical cleaning using a wide range of chemicals (Li et al., 2017), such as HCl, NaOCl, sodium dodecyl sulfate (SDS) and Alconox. Biological proteins are easily denatured under high temperature and organic solvent conditions, and thus these two extreme scenarios can be applied to inhibit aquaporin within membrane matrix, thereby gaining perspective into the role of aquaporin in membrane transport mechanisms.

In this paper, we investigated the TrOC rejection mechanisms of thin-film composite aquaporin FO membranes and membrane stability under extreme conditions with respect to TrOC rejection. The aquaporin membrane was characterised to shed light on the TrOC transport mechanisms. We also studied the role of varying operating conditions on TrOC rejections by aquaporin membrane. Finally, the aquaporin membrane was challenged by both heat and ethanol exposure to examine the membrane thermal and solvent stability and response to TrOC rejection.

2. Materials and methods

2.1. Aquaporin membrane and representative trace organic contaminants

A biomimetic, aquaporin membrane provided by Aquaporin A/S (Aquaporin A/S, Copenhagen, Denmark) was used. Briefly, the aquaporin membrane is made as a thin film composite membrane where vesicles with embedded aquaporin proteins are stabilized by a polyamide layer supported by a porous polysulfone support layer (Zhao et al., 2013).

A total of 30 TrOCs were selected to represent four major groups of emerging contaminants of significant concern — endocrine disrupting compounds, pharmaceuticals and personal care products, industrial chemicals, and pesticides — that occur ubiquitously in municipal wastewater. Key physicochemical properties of these TrOCs are summarised in Table S1 of the Supplementary Data. A stock solution containing 25 µg/mL of each of the TrOCs was prepared in pure methanol and stored at –18 °C in the dark. The stock solution was introduced into the synthetic wastewater to obtain a concentration of 2 µg/L of each compound. The TrOC stock solution was used within a month.

2.2. Aquaporin membrane characterisation

A detailed aquaporin membrane characterisation — key membrane transport parameters, effective average pore size, and membrane surface properties — shed light on membrane structure-performance relationship as well as the TrOC rejection mechanisms.

2.2.1. Key membrane transport parameters

Key membrane transport parameters were characterised following the protocol previously described by Cath et al. (2013), including pure water permeability coefficient of the active layer, *A*, the salt (NaCl) permeability coefficient of the active layer, *B*, and the structural parameter of the support layer, *S*. The detailed procedure to determine membrane transport parameters were described in the Supplementary Data.

2.2.2. Membrane surface properties

Aquaporin membrane surface properties — surface charge, surface morphology, surface chemistry and surface hydrophilicity — were investigated to shed light on the TrOC rejection mechanisms.

Membrane surface charge was indicated by the zeta potential measurement using a SurPASS electrokinetic analyser (Anton Paar GmbH, Graz, Austria). Zeta potential was calculated from the measured streaming potential using the Fairbrother-Mastin approach that is more accurate for non-conductive samples with irregular shape (Elimelech et al., 1994). All streaming potential measurements were conducted in a background electrolyte solution containing 10 mM KCl. The same electrolyte solution was used to flush the cell thoroughly prior to pH titration using either hydrochloric acid (1 M) or potassium hydroxide (1 M). All measurements were performed at room temperature (approximately 22 °C).

Membrane surface morphology was characterised using a scanning electron microscopy (SEM) (JEOL JCM-6000, Tokyo, Japan). Prior to the SEM measurement, air-dried membrane samples were coated with an ultra-thin layer of gold using a sputter coater. The membrane cross-section morphology was also visualised by transmission electron microscopy (TEM) (JEOL JEM-1230, Tokyo, Japan) at the accelerating voltage of 80 keV. Aromatic acrylic LR white resin was used to embed dehydrated samples in capsules which was described in previous publication (Wang et al., 2012). Briefly, membrane samples were dehydrated by several changes of ethanol (3 times 100% ethanol, 15 min for each step). Samples were then infiltrated in 50, 67, and 100% LR White resin (volumetric %, prepared in ethanol) sequentially, followed by embedding them in fresh LR White resin that was subsequently polymerized at 48 °C for 3 days. After complete polymerization of the resin, thin TEM sections (<100 nm thickness) were cut with a diamond knife using Leica Ultracut S ultramicrotome (Leica, Wetzlar, Germany) and transferred onto copper TEM grids for imaging.

The membrane surface morphology was also resolved by atomic force microscopy (AFM) (Asylum MFP-3D, Asylum Research, Santa Barbara, CA). The AFM imaging was performed with the tapping mode under ambient conditions using silicon cantilever probe tips.

Membrane surface functional groups were identified using a Fourier Transform Infrared (FTIR) spectrometer (Thermo Scientific Nicolet 6700) equipped with an ATR accessory consisting of a ZnSe plate (45° angle of incidence). Absorbance spectra were measured with 64 scans of each sample at a spectral resolution of 2 cm⁻¹. Background measurements in air were collected before each membrane sample measurement. ATR-FTIR spectra were collected for two different spots in each membrane sample.

To obtain information about composition and bonding chemistry of the aquaporin membrane surface layer (with penetration depth from 1 to 5 nm thickness), X-ray photoelectron spectroscopy

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