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A method for the simultaneous determination of transport and structural parameters of forward osmosis membranes

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We present a simple and rapid methodology to characterize the water and solute permeability coefficients (*A* and *B*, respectively) and structural parameter (*S*) of forward osmosis (FO) membranes. The methodology comprises a single FO experiment divided into four stages, each using a different concentration of draw solution. The experimental water and reverse salt fluxes measured in each stage are fitted to the corresponding FO transport equations by performing a least-squares non-linear regression, using *A*, *B*, and *S* as regression parameters. Hand-cast thin-film composite (TFC) FO membranes and commercial TFC FO, TFC reverse osmosis (RO), and cellulose acetate-based asymmetric FO membranes are evaluated following this protocol. We compare the membrane properties obtained with our FO-based methodology with those derived from existing protocols based on an RO experiment followed by an FO experiment. For all membranes, the FO-based protocol gives more accurate predictions of the water and salt fluxes than the existing method. The numerical robustness of the method and the sensitivity of the regression parameters to random errors in the measured quantities are thoroughly analyzed. The assessment shows that confidence in the accuracy of the determined membrane parameters can be enhanced by simultaneously achieving close fitting of the predicted fluxes to experimental measurements (i.e., high R^2 values) and constant water to salt flux ratios in each stage. Additionally, the existing and proposed approaches yield consistently dissimilar results for some of the analyzed membranes, indicating a discrepancy that might be attributed to the different driving forces utilized in RO and in FO that should be further investigated.

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1. Introduction

Forward osmosis (FO) utilizes the osmotic pressure difference developed across a semi-permeable membrane separating two solutions of different concentrations to drive the permeation of water [1]. FO has shown promise in a variety of applications [1–7], and it is also attracting attention as a potential technology to augment water supplies using seawater [1,8,9] and wastewater [10–12]. Where abundant and low value streams can be used without the need for regeneration, such as seawater and wastewater, FO can be employed to concentrate the feed solution (osmotic concentration) or dilute the draw solution (osmotic dilution) [13].

In recent years, a great deal of research has been directed at the fabrication of FO membranes [7,14–29]. These efforts have resulted in the development of substantially improved membranes tailored for the specific needs of FO. In particular, thin-film composite (TFC) FO membranes, consisting of a salt-rejecting, active layer and a porous support, have shown higher water fluxes, reduced salt passage, and enhanced anti-fouling properties [22,29–31]. A convenient and consistent methodology to characterize FO membranes is of critical importance to advance this technology onto its mature phase, facilitating the sharing of data, their interpretation, and comparison.

When describing membrane performance, the literature often reports values of water fluxes, J_w, reverse solute fluxes, J_s, the resistance to solute diffusion in the membrane support layer, *K*, or its inverse parameter, the mass transfer coefficient, $k=1/K$ [32]. However, these quantities are not intrinsic properties of the membrane as they depend on the hydrodynamic conditions at the membrane interface, the concentration and osmotic pressure of the draw and feed solutions, and the type and diffusivity of the solutes. This approach therefore lacks generality, as direct comparisons cannot be made unless the operating conditions are identical.

An alternative approach, adopted for 'tight', salt-rejecting FO membranes, is based on three intrinsic parameters that fully describe membrane systems: the pure water permeability

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coefficient, *A*, and the solute permeability coefficient, *B*, which describe the transport across the membrane active layer, and the structural parameter, *S*, quantifying the mass transport length scale across the membrane support layer. These three parameters are univocal and can be used with the respective governing equations to accurately predict the water and salt flux performance of a membrane sample in any laboratory-scale FO system. Therefore, the values of *A*, *B*, and *S* represent common yardsticks for describing membrane intrinsic characteristics and offer a universal set of criteria for comparing performance, regardless of operating conditions.

The existing approaches to measure *A*, *B*, and *S* of an FO membrane entail the use of at least two separate experiments. Initially, the parameters related to the active layer (*A* and *B*) are measured by applying a trans-membrane hydraulic pressure in reverse osmosis (RO) mode experiments. Subsequently, the membrane is tested using an osmotic driving force [7,15,17–29,33] to determine the support layer structural parameter, *S*. Experiments in the pressure retarded osmosis (PRO) configuration (draw solution facing the active side of the membrane) may also be conducted to complement [17–19,21,24–27] or substitute [14] measurements in FO configuration.

These protocols are cumbersome and laborious, requiring multiple experiments in different experimental setups. Subjecting FO membranes, intended for operation near ambient conditions, to the high pressures typical of RO tests, can result in mechanical damage to the membrane. Furthermore, current methodologies combining RO and FO are based on the notion that transport parameters are universally valid and transferable, an assumption that warrants further examination in light of the fundamentally different permeation driving forces in RO and FO: a *hydraulic* pressure difference applied on the RO membrane active layer versus the *osmotic* pressure difference across the membrane active/support layer interface in FO. These fundamental differences may result in dissimilar observed transport parameters between the RO and FO processes, a phenomenon also suggested in recent studies [34]. It is therefore desirable to formulate a methodology for FO membrane characterization that evaluates the membrane performance under representative driving force and operating conditions, and which, in addition, is both simple (i.e., based on a minimum number of experiments) and reliable.

In this study, we present a method to characterize the intrinsic transport and structural properties of FO membranes in a *single* FO experiment. By changing the concentration of the draw solution in each stage of the experiment, a set of FO water flux and reverse salt flux measurements are obtained. Membrane parameters are determined through non-linear regression, where *A*, *B*, and *S* are treated as adjustable parameters to fit the FO transport equations to the experimental water and salt fluxes. To demonstrate the generality of the method, we characterized four sets of membranes exhibiting a wide range of transport and structural parameters. Our results raise questions about the reliability of current membrane characterization protocols, and point towards further investigations in transport processes in osmotically driven membrane processes.

2. A single FO experiment to characterize osmotic membranes

A single and facile FO experiment is proposed to characterize the intrinsic transport parameters, *A* and *B*, and the structural parameter, *S*, of an FO membrane by measuring the water and reverse solute flux across the membrane under different draw solution concentrations. As depicted schematically in Fig. 1, the FO experiment is divided into a discrete number of stages. The influence of the adopted number of stages on the robustness and accuracy of the methodology will be discussed in Section 5. In our study, the experiments were carried out in four stages.

In the first stage of the experiment, a draw solution concentration $c_{D,1}$ and a feed solution of deionized (DI) water were utilized to measure the FO water flux, $J_{w,1}$, and the reverse solute flux, $J_{s,1}$. At the end of the first stage, a known volume of concentrated draw

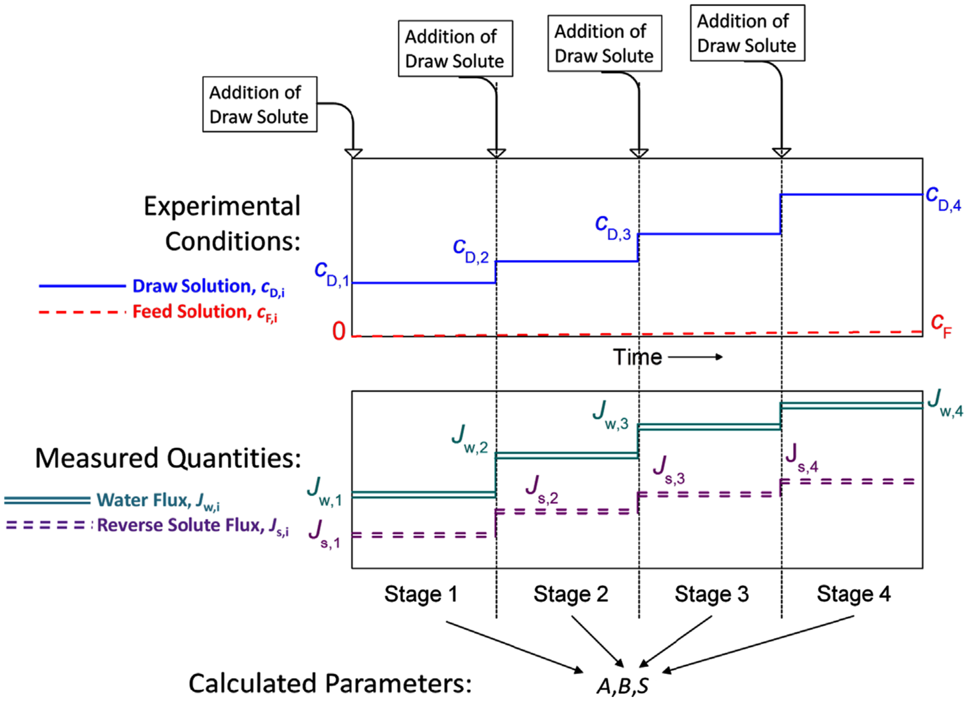

Fig. 1. Protocol of the single FO experiment. Experimental solutions and measured quantities are schematically represented as lines across the time scale for each of the four stages of the experiment. Draw solution concentration (blue), c_D, and feed solution concentration (red), c_F, are represented as single lines in the top plot. Experimental water flux (green), J_w, and experimental reverse solute flux (purple), J_s, are depicted as double lines in the bottom plot. The four stages are separated by a vertical dotted line. The stages allow the calculation of *A*, *B*, and *S*. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

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