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Microalgal biomass dewatering using forward osmosis membrane: Influence of microalgae species and carbohydrates composition

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ABSTRACT

The potential application of forward osmosis (FO) in microalgae dewatering requires an improved understanding of the factors that control membrane fouling which can reduce dewatering performance in terms of water flux through membrane and algae recovery. The aim of this study was to elucidate the influence of algae cell wall carbohydrate composition on the FO dewatering performance using three types of draw solutions (sea salts, $MgCl_2$ and $CaCl_2$). Experimental results suggest that the interaction between microalgae and back diffused draw solutes plays a key role. *Scenedesmus obliquus* with fructose and abundant glucose and mannose in its cell wall showed strong response to the back diffusion of calcium ions which encouraged *S. obliquus* to produce more extracellular carbohydrates and formed a stable gel network between algal biomass and extracellular carbohydrates, leading to algae aggregation and severe loss in both water flux and algae biomass during FO dewatering with Ca^{2+}-containing draw solution. *Chlamydomonas reinhardtii* without fructose but great galactose showed a similar response to the calcium back diffusion but to a lower extent. Both *S. obliquus* and *C. reinhardtii* did not cause obvious membrane fouling but dramatic algae biomass loss at the end of FO filtration with $MgCl_2$ draw solution due to their interaction with back diffused Mg^{2+} ions which led to the deposition of algae flocs onto membrane surface and/or feed spacer. *Chlorella vulgaris* without fructose was the most suitable microalgae species to be dewatered by FO with algae recovery over 81% and negligible flux decline regardless of which draw solution was applied. The findings improve mechanical understanding of FO membrane fouling by microalgae; have significant implications for the algae species selection; and are critical for the development and optimization of FO dewatering processes.

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1. Introduction

Microalgae are recently considered as an excellent renewable energy feedstock due to their high-yield production without impinging on food crops [1]. In addition, microalgae are able to serve the roles of carbon dioxide sequestration and wastewater remediation by nutrients fixation [2]. Algae biomass can be also used to produce a variety of high-value products such as cosmetics, antioxidants and food supplements [3]. Despite such promises, due to their similar density to water [4], and usually very diluted cultures [5], separating and concentrating algae cells from the culture medium (algae dewatering) is one of the technical and economical bottlenecks to large-scale microalgae production. Microalgae can be dewatered by conventional methods (such as centrifugation, flotation, chemical flocculation and sedimentation) and advanced pressure-driven membrane processes (such as microfiltration and ultrafiltration). The conventional methods are either highly energy intensive or require harmful chemical addition. Compared to conventional methods, membrane filtration requires lower energy but is prone to fouling which greatly reduces the algae dewatering efficiency and overall process sustainability. Thus, there is an innovation demand for more energy efficient and environmentally sustainable algae dewatering alternatives to overcome the many drawbacks of existing technologies.

Forward osmosis (FO), an emerging membrane separation process, has recently been considered as a promising and sustainable dewatering technology [6,7]. FO is based on the natural tendency of water to flow through a semi-permeable membrane from a feed solution of lower osmotic pressure (higher water chemical potential) to a draw solution of higher osmotic pressure (lower water chemical potential). The driving force for water movement is the osmotic pressure difference across membrane. The absence of external hydraulic pressure allows FO to offer many advantages including (1) low energy consumption if an appropriate draw solution is applied [8], (2) lower fouling propensity and higher cleaning efficiency and (3) greater recovery of unbroken algae cells.

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In our previous study [6], we proposed to integrate FO and seawater reverse osmosis (SWRO) processes to significantly improve the energy efficiency and environmental sustainability of algae dewatering and existing desalination. Briefly, highly concentrated SWRO brine (osmotic pressure ~ 5.4 MPa) is used as draw solution to pull clean water through the FO membrane from algae culture in the feed side while the FO membrane with superior separation efficiency retains all algal biomass and thus enhances the algae concentration. During the FO dewatering process, most of membrane fouling is reversible by simple hydraulic flushing without any chemical addition. Meanwhile, the high quality FO permeate water mixes with the brine and substantially reduces its concentration. The diluted brine can be disposed back to the sea with minimal environmental impact or sent to the SWRO desalination process to further recover clean water. The dilution of brine significantly reduces the required hydraulic pressure and thus overall energy consumption during SWRO desalination process.

Despite the low fouling tendency of FO, FO dewatering performance may still be adversely impacted by membrane fouling, resulting in lower water flux and algae recovery efficiency, shorter membrane life and greater operating cost [6]. FO fouling is reported to be governed by membrane characteristics and orientation, draw solution concentration and chemistry, feedwater composition, and hydrodynamic conditions [9]. Compare to pressure-driven membrane process, FO has a unique mass transfer phenomenon called draw solute back diffusion that may play an important role in membrane fouling. In our previous study, FO was applied for *Scenedesmus obliquus* dewatering with various types of draw solution [6]. We found that the back diffusion of Ca^{2+} ions into the algae suspension on the feed side of membrane encouraged algae to release more carbohydrates and induced more severe fouling in comparison with Mg^{2+} and Na^{+}. For example, significant water flux decline and algae biomass loss were observed due to membrane fouling when $CaCl_2$ was used as draw solution. In contrast, Zou et al. reported a great membrane fouling when $MgCl_2$ was used as draw solution to concentrate microalgae *Chlorella sorokiniana* [10]. These different observations can be attributed to the different characteristics (such as surface chemistry, cell size and morphology) of different algae species.

During the FO dewatering process, membrane fouling is caused by the deposition of algae cells and their released extracellular polysaccharides (EPS) on the membrane surface. Such fouling layer can be further fortified by the back diffusion of certain draw solutes. For example, Ca^{2+} ions bind specifically with the carboxylate functional groups at the interface of algae cells/EPS, and form egg-box-shaped gel network [11–14]. Clearly, algae surface chemistry (such as charge, functional group and free energy) determined by cell wall composition can impact the interfacial forces between algae cell and membrane in the aqueous media [15]. Cell shape and size may further influence the cake layer structure and compactness [16–18]. In addition, during the FO process, the cross-flow velocity in feed channel lifts algae particles away from membrane and thus reduces the cake layer formation [19]. However the shear force caused by feed pump may induce cell rupture and EPS production, both of which can enhance membrane fouling. Different algae species may have different sensitivity and response to this hydraulic stress and the salt stress caused by draw solute back diffusion, and thus are expected to exhibit different impacts on membrane fouling and overall algae dewatering efficiency. To realize a feasible FO dewatering process, selection of optimal microalgae species is essential. However, to date, very little is known about the role of algae species-dependant characteristics (such as cell wall composition and stress sensitivity/response) on FO membrane performance.

The objective of this work is to compare the FO dewatering performance in terms of water flux behaviour and algae dewatering efficiency between three freshwater microalgae species (*Scenedesmus obliquus*, *Chlamydomonas reinhardtii*, and *Chlorella vulgaris*). Specific aims were to (1) examine the role of cell wall carbohydrate composition and demonstrate that it is a key factor affecting FO performance; (2) investigate the effect of hydraulic stress on the EPS production from the three algae species; and (3) identify the most suitable algae species for FO dewatering. Based on the results, important mechanisms and factors that govern FO fouling are discussed and elucidated. The findings of this study will provide important insights into the efficient operation of FO for algae dewatering in terms of optimal algae species selection, fouling control and FO system design, assisting the future development of FO technology for more effective microalgae dewatering.

2. Materials and methods

2.1. Microalgae species, cultivation and characterization

Three freshwater microalgae species (*Scenedesmus obliquus*, *Chlamydomonas reinhardtii*, and *Chlorella vulgaris*) were investigated in this study. They were selected due to their high lipid content, reaching over 50% of the dry weight [20,21], as well as excellent potential for wastewater treatment [22,23], and CO_2 capture [24,25]. Pure cultures of them were obtained from the Culture Collection of Algae and Protozoa (CCAP, UK). Each species was individually cultivated in modified BG-11 medium with compositions described in our previous study [6]. Algae suspensions were continuously stirred with air injected (75 L/h) at room temperature (25 ± 1 °C). Illumination was provided by fluorescent lamps (100 μmol photons/$m^2 \cdot s$). The algae growth was periodically monitored by optical density measurement with a spectrophotometer (Helios Zeta, Thermo Scientific, UK) at 435 nm wavelength [26]. The pH of each algae suspension was maintained at 7 ± 0.5 for the optimum algae growth. After 14 days of cultivation, 2–3 g dry weight/L of each algae species was obtained. This stock suspension was diluted with BG-11 medium to prepare the feed water for FO dewatering experiments and hydraulic stress tests.

Based on microscopic observation (Olympus IX71, Olympus Corporation, Tokyo, Japan), *S. obliquus* has an ellipsoidal shape and is around 5 μm in width and 10 μm in length; *C. reinhardtii* has a circular shape with a diameter of around 10 μm and possesses two flagella; and *C. vulgaris* has a circular shape with a diameter of around 5 μm. In the BG-11 medium, *S. obliquus*, *C. reinhardtii* and *C. vulgaris* exhibited negatively zeta potential of 15.45 ± 1.87 mV, 19.07 ± 0.75 mV, and 16.8 ± 1.15 mV, respectively (Zetasizer nano, Malvern Instruments Ltd., UK).

2.2. FO membrane

In all FO experiments, a new flat sheet cellulose triacetate (CTA) membrane (Hydration Technologies Innovation, Albany, OR, USA) was used. The membrane has an active layer made of CTA supported by an embedded woven mesh to enhance its mechanical strength [27]. Total membrane thickness is around 50 μm [28]. The membrane active layer exhibited a slightly negative zeta potential of approximately −10 mV in 10 mM KCl at pH of 7 ± 0.5 [29].

2.3. Experimental setup and protocols for algae dewatering

All algae dewatering experiments were conducted using a bench-scale FO membrane setup (Fig. A1, Appendix) that has been described in our previous study [6]. A flat sheet membrane coupon was housed in a plate-and-frame membrane cell with two identical channels on both sides of the membrane. Diamond-patterned spacers were placed on either side of the membrane for additional support. Counter-current flow was applied with cross-flow rate on each side of the membrane controlled by a variable-speed peristaltic pump (Cole-Parmer, Vernon Hills, IL, USA). To prevent microalgae sedimentation, a magnetic stirrer was used to provide mixing in feed tank. Draw solution tank was placed on a digital scale (Denver Instrument, USA) which was interfaced with an automatic data acquisition system to determine permeate water flux. The temperature of both feed and draw solutions was kept constant using a re-circulating water chiller (Fisher Scientific,

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