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# The filtration and fouling performance of membranes with different pore sizes in algae harvesting

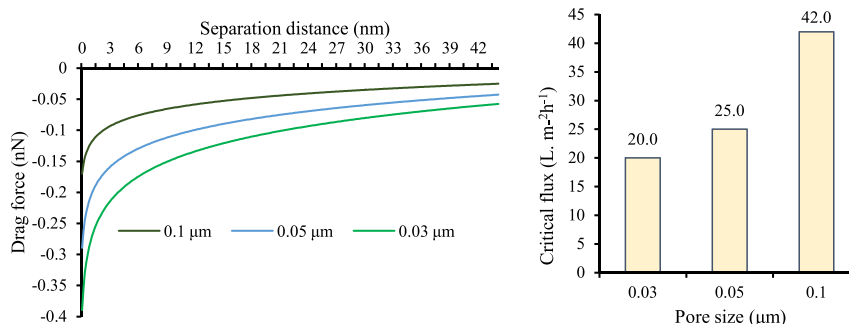
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## HIGHLIGHTS

- Effect of membrane pore size on critical flux was studied.
- Continuous filtration was conducted to find the optimal pore size.
- The drag force revealed how pore size affected membrane fouling.
- Compared with 0.03 and 0.05  $\mu\text{m}$ , 0.1  $\mu\text{m}$  was suitable for harvesting *C. pyrenoidosa*.

## GRAPHICAL ABSTRACT



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## ABSTRACT

In this study, ultrafiltration membranes with three different pore sizes were applied for algae harvesting to investigate filtration performance. The critical fluxes ( $J_c$ ) increased as the pore size increased, and the  $J_c$  of 0.03-, 0.05- and 0.1- $\mu\text{m}$  membranes were 20.0, 25.0 and 42.0  $\text{L} \cdot \text{m}^{-2} \cdot \text{h}^{-1}$ , respectively. During continuous filtration,  $0.7J_c$  was selected as the operation flux and the 0.1- $\mu\text{m}$  membrane had the highest initial flux and final flux. It also had the highest flux decline rate, and therefore, the 0.1- $\mu\text{m}$  membrane was more appropriate for algae separation compared to the 0.03- and 0.05- $\mu\text{m}$  membrane. The mechanism by which pore size influenced filtration performance and membrane fouling was presented from the viewpoint of permeate drag force ( $F_D$ ). A lower  $F_D$  retarded the velocity of algae cells towards the membrane, which could decelerate the deposition of particles on the membrane and thus reduce the membrane fouling rate. As the pore size increased, the membrane hydraulic resistance ( $R_m$ ) decreased, which led to a decrease of  $F_D$ .

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## 1. Introduction

The production of biofuel from algae is a promising new technology. Biofuel, as an alternative to fossil fuels, can reduce global warming caused by fossil fuels and has very promising application prospects because of the renewable and nontoxic properties of microalgae (Davis et

al., 2014; Zhao et al., 2015b). Biofuel produces approximately zero net carbon dioxide and releases fewer gaseous pollutants than fossil fuels (Uduman et al., 2010). Because microalgae can absorb carbon dioxide from the atmosphere for growth, the consumption of biofuel emits carbon dioxide into the atmosphere. Thus, the use of biofuel can play an important role in mitigating the emission of greenhouse gases. Although microalgae can be a good source of biofuel, there are substantial challenges in efficient harvesting and dewatering for commercial use. The challenges of harvesting and dewatering algae derive from the

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nature of microalgae, such as the small size of algal cells (mostly below 30  $\mu\text{m}$ ), the similarities in the densities of microalgae (approximately 1.025 g/L) and water, the concentration of algae in culture solutions at approximately 0.5–3 g/L and the negative surface charge on microalgae that results in dispersed stable algal suspensions, especially during the growth phase (Bhave et al., 2012; Uduman et al., 2010). Due to the nature of microalgae, harvesting is considered a major obstruction to algal biofuel production and a main factor limiting the commercial use of microalgae (Hwang et al., 2015; Milledge and Heaven, 2012; Weschler et al., 2014).

Recently, the application of membrane technology to algae harvesting has drawn growing attention, due to their advantages of high efficiency and stability (Drexler and Yeh, 2014; Zhang et al., 2014; Zhao et al., 2015a). Membrane separation processes can recover microorganisms and yield stable, clean effluent water (Pavez et al., 2015); moreover, membrane technology can remove viruses and protozoans from culture media but retaining the residual nutrients, and thus the cultivation medium can be recycled. As the manufacturing techniques for membranes improve and their applications increase, the cost of membranes steadily falls, making it possible to apply membranes to algae harvesting. Therefore, an increasing number of researchers have studied how to make better use of membranes to harvest microalgae (Ahmad et al., 2013; Rossignol et al., 1999). However, some obstacles must still be overcome in harvesting algae. The primary problem for this process is membrane fouling, which may result in a decline in flux or increase in transmembrane pressure (TMP) (Drews et al., 2006; Rickman et al., 2012; Zhao et al., 2015a). These changes lower harvesting efficiency and increase harvesting cost. Thus, reducing membrane fouling and improving membrane flux are very significant goals in algae harvesting.

Membrane filtration in algae harvesting is a physical separation process whose separation performance depends on membrane pore size. Pores that are too large cannot retain all algae cells, but pores that are too small dramatically reduce permeate flux, which seriously affects harvesting efficiency (Batista et al., 2013). However, the literature shows that the pore size of membranes is not a crucial factor because during algae filtration, the fouling layer on the membrane, caused by the deposition of algae cells and extracellular organic matter, can act as a membrane-selective layer (Batista et al., 2013; Nguyen et al., 2012; Zhou et al., 2014). However, pore size is still an important parameter that influences membrane performance. Selecting an appropriate pore size and operation flux can reduce membrane fouling rate and flux decline rate, which can extend filtration period and reduce membrane cleaning frequency.

In this study, membranes with three different pore sizes (0.03, 0.05 and 0.1  $\mu\text{m}$ ) were used to filter algae to find the appropriate membrane pore size for algae harvesting. Moreover, the mechanism by which membrane pore size influenced membrane fouling and flux was presented and investigated. First, critical flux experiments were conducted to measure the critical flux of the three membranes. Second, the permeate drag force was calculated to theoretically demonstrate how pore size affected membrane filtration. Finally, continuous filtration tests were conducted using membranes with three different pore sizes to obtain the appropriate membrane pore size for filtering algae. This research can provide valuable information for algae harvesting using ultrafiltration membrane technology.

## 2. Materials and methods

### 2.1. Cultivation of microalgae

*Chlorella pyrenoidosa* (*C. pyrenoidosa*, FACHB-9) was purchased from the Institute of Hydrobiology at the Chinese Academy of Sciences. *C. pyrenoidosa* was cultured in Basal medium prepared in sterilized distilled water. The algae was inoculated in 3-L glass flasks and placed in incubators. The cultivation temperature was kept at  $35 \pm 0.5$  °C. The

other cultivation conditions were the same: light intensity 127  $\mu\text{mol m}^{-2} \text{s}$ , light/dark = 14 h/10 h.

### 2.2. Reactor set-up

The filtration was performed in a lab-scale tank, as shown in Fig. 1. The effective volume of the filtration tank was 4 L. A micro-porous pipe was placed below the membrane to reduce membrane fouling and the aeration flow rate was controlled at 6 L  $\text{min}^{-1}$ . Three different hydrophilic PVDF ultrafiltration membranes (pore sizes of 0.03, 0.05 and 0.1  $\mu\text{m}$ , Minglie, Shanghai, China) were employed to determine the appropriate pore size for algae harvesting. The effective filtration area of the membrane was 0.02  $\text{m}^2$ . The filtrate was filtered using a peristaltic pump. The volume of filtrate was measured and recorded using an electric balance and a computer, respectively. The values of the transmembrane pressure (TMP) were recorded using a vacuum meter.

### 2.3. Filtration experiments

#### 2.3.1. Critical flux tests

In filtration, a high flux will result in severe membrane fouling and sharp flux decline, while a low flux adversely influences efficiency. Thus, selecting an appropriate sub-critical flux will prolong membrane service life and reduce membrane cleaning frequency (Bilad et al., 2012c; van der Marel et al., 2009; Wicaksana et al., 2012). Moreover, the value of critical flux can be utilized to compare the propensity towards membrane fouling.

In the present study, an improved flux-step method (IFM) was used to determine the value of critical flux (van der Marel et al., 2009). The difference between IFM and the common flux-step method is that in IFM, the level of the successive membrane flux ( $J_H$ ) increases, including an intermediate flux decrease to an initial low flux ( $J_L$ ) after each  $J_H$  step. In IFM, filtration at  $J_L$  is considered a form of relaxation, although real relaxation filtration should be 0 L  $\text{m}^{-2} \text{h}^{-1}$ . However, a flux larger than 0 L  $\text{m}^{-2} \text{h}^{-1}$  must be applied to measure a value for TMP before and after a  $J_H$  (van der Marel et al., 2009). At low flux, the convective flow towards the membrane is reduced and, due to air scouring, the reversible fouling reduced. For the 0.03-, 0.05- and 0.1- $\mu\text{m}$  membranes, the  $J_L$  of each module was identical (10 L  $\text{m}^{-2} \text{h}^{-1}$ ), with  $J_H$  starting from 10, 10 and 15 L  $\text{m}^{-2} \text{h}^{-1}$  and stepwise increasing by 2.5, 2.5, and 3 L  $\text{m}^{-2} \text{h}^{-1}$ , respectively. The duration for all  $J_L$  and  $J_H$  processes was 15 min. In this research, an arbitrary minimum increase in the TMP of 20 Pa  $\text{min}^{-1}$  was used to determine  $J_C$ . In theory, once the  $J_C$  of all the modules was achieved, the test should stop; however, to better comprehend the effect of temperature on the performance of the membranes, the maximum  $J_H$  of 0.03-, 0.05- and 0.1- $\mu\text{m}$  membranes reached 22.5, 27.5 and 42 L  $\text{m}^{-2} \text{h}^{-1}$ , respectively. In the critical flux tests, there were two phases, namely, the ascending and descending phase. In the ascending phase, with the increase in  $J_H$ , critical flux is achieved; in the descending phase,  $J_H$  decreases stepwise and can achieve fouling condition. The algae in the stationary phase was used for all IFM tests and continuous filtration experiments, and the concentrations were approximately 0.3 g  $\text{L}^{-1}$ . The experiments were performed for several days to reduce the impact of the fast growth of algae on filtration, and we thus selected the stationary phase rather than the exponential phase. To reduce the influence of change in temperature on algae, all filtration experiments were conducted at  $35 \pm 0.5$  °C.

#### 2.3.2. Continuous filtration experiments

**2.3.2.1. Short-term filtration.** Short-term filtration was performed for 1 h because after 1 h, filtration with the 0.03- $\mu\text{m}$  membrane was slowed by serious fouling and filtration could not continue. The operational flux of these three membranes was set as 20 L  $\text{m}^{-2} \text{h}^{-1}$ .

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