



Increasing the vibration frequency to mitigate reversible and irreversible membrane fouling using an axial vibration membrane in microalgae harvesting



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ARTICLE INFO

Keywords:

Vibration membrane
Algae
Frequency
Membrane fouling
EOM

ABSTRACT

During algae harvesting using membrane technology, membrane fouling caused by the deposition of algae cells and extracellular organic matter (EOM) poses a major challenge. In this study, axial vibration membrane (AVM) filtration was conducted at 0, 5 and 10 Hz. As the frequency increased, AVM could effectively reduce the reversible fouling caused by the deposition of algae cells on the membranes. With the increase of frequency from 0 to 10 Hz in the 2-h filtration experiments, the amount of algae deposited on the membranes sharply decreased from 8.64 to 0.03 g/m². For the reversible EOM on the membranes, with increasing frequency, the protein and polysaccharide contents exhibited declining trends, and no humic-like material was observed. It was also found that both low-MW (< 1 kDa) and high-MW (> 200 kDa) EOM easily caused reversible membrane fouling. The irreversible EOM adhesion to the membrane consisted of protein, polysaccharide and humic-like material. With the increase of frequency the MW peak of irreversible EOM had a right shift tendency. At 0, 5 and 10 Hz, EOM with MW of 3.5, 4 and 5 kDa, respectively, was more easily adsorbed on the membranes and caused irreversible membrane fouling.

1. Introduction

Microalgae are considered as a promising biofuel resource, which have the potential to alleviate the greenhouse gas emissions by the use of fossil fuels [1–3]. However, inefficient microalgae harvesting is still a critical limiting factor of algal biofuel production for commercial use [4–6]. Membrane filtration is appropriate to harvest microalgae, as it can completely retain microalgae cells by size exclusion [7–10]. With the improvement of membrane technique, more and more researchers have studied to make better use of membranes in algae harvesting [11–13]. Despite this ability, membrane fouling is a formidable challenge in algae harvesting with membrane filtration. Membrane fouling can result in a remarkable decrease of flux; in addition, it can also lead to frequent membrane cleaning, thus reducing the filtration efficiency and increasing the filtration cost. Therefore, it is significant to reduce membrane fouling and enhance membrane flux. Increasing the shear rate at the membrane surface is an efficient method to mitigate membrane fouling. Many vibration membranes have been utilized to mitigate membrane fouling and increase the permeate flux, as they can create high surface shear rates with high vibration frequencies or

amplitudes [14–17].

Recently, the line vibration membrane (axial vibration membrane or transverse vibration membrane) have been investigated in algae harvesting by more and more researchers [11,18,19]. The line vibration membrane could effectively control membrane fouling even at a small amplitude or frequency [20–22]. In addition, the shear action induced by the line vibration membrane acts only near the membrane surface and thus can reduce the impact of shear on algae [23]. Although vibration membranes can reduce reversible membrane fouling caused by algae cells deposited on membrane surface during the filtration of microalgae, EOM can still be deposited on the membrane surface or in the pores, resulting in irreversible membrane fouling, which may lead to a lower cleaning efficiency and compromise the long-term performance of membranes [13,15,18,19]. Reversible fouling is considered to be a loosely bound fouling layer that can be removed by physical methods [24,25]. On the contrary, irreversible membrane fouling is caused by strong adhesion to the membrane in the form of membrane pore plugging, biofilm formation and gel layer build-up, which can only be removed using chemical methods [26,27]. Generally proteins and polysaccharides in EOM are considered to be the major foulants, and

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they have great potential for membrane fouling as they can be adsorbed on the membrane surface or particularly, inside membrane pores, leading to severe irreversible membrane fouling [28,29].

In the previous studies, we found that with increasing vibration frequency, the deposition of algae cells on the membrane surface was obviously reduced, but EOM could still be adhered to the membranes at a high frequency [19,23]. However, the effect of increasing frequency on reducing the adherence of EOM on the membranes has not been investigated so far. In addition, the effect of increasing frequency on the reversible and irreversible membrane fouling of microalgae has not been comprehensively studied either. Understanding these phenomena comprises the main objective of this study. Although algal deposition is the main cause for reversible fouling, as previously reported [26], for AVMs, reversible EOM might also be a significant factor, resulting in reversible fouling at high frequencies, which requires further verification. In this study, we also determine the EOM content in the reversible and irreversible layer with different frequencies. Finally, the difference of the main components between reversible and irreversible EOM is compared.

2. Materials and methods

2.1. Cultivation of algae

Chlorella pyrenoidosa (*C. pyrenoidosa*, FACHB-9) was seeded from the Institute of Hydrobiology at the Chinese Academy of Sciences. It was cultured with a sterile Basal medium in a 25 L glass box inside of an incubator. The cultivation temperature was maintained at 30 ± 0.5 °C, light intensity was $127 \mu\text{mol}/\text{m}^2 \text{ s}$ and light/dark was 12 h/12 h.

2.2. Experimental setup

In this study, an axial vibratory membrane (AVM) was used to study the effect of increasing frequency on mitigating membrane fouling in the filtration of microalgae. The membrane system was vibrated using a servo-motor (60FSM-04030, USA), and the frequency was adjusted by a digital servo-drive (FDS15A-400X, USA). The tank of this system had a 50-L working volume. A hydrophilic PVDF membrane (Minglie, China) was used in the filtration, with nominal pore size of $0.1\text{-}\mu\text{m}$ and total effective membrane area of 0.02 m^2 . The filtrate was sucked using a peristaltic pump (BT100-LJ, Kejian, China). The variation of the flux was recorded by an electronic balance connected to a computer. The transmembrane pressure (TMP) was measured by a vacuum meter. The flow diagram of AVM system had been provided in our previous study [19], thus it was not presented here. In the experiments, the vibrations were conducted at different frequencies of 0, 5 and 10 Hz, with a same amplitude of 1 cm. In the filtration, $30 \text{ L}/\text{m}^2\text{h}$ were arbitrarily selected for the operating flux and the algae concentration was maintained at about $0.4 \text{ g}/\text{L}$.

2.3. Measurement of the fouling rate

After filtration, the fouled membrane was cut by a sharp knife and two fouled membranes were obtained. One fouled membrane was directly used for determining the water flux before rinse to evaluate the total membrane fouling using a cup-type filtration vessel driven by nitrogen gas at 0.05 MPa [30]. Then, the membrane was rinsed using pressurized water for scanning electron microscopy (SEM). The other membrane was flushed using water, and the algae were flushed into a beaker to determine the contents of algae and EOM (reversible EOM)

on the membrane. After rinse, the membrane was used to determine the water flux to compare irreversible fouling at different frequencies. Finally, the membrane was soaked in a $0.5 \text{ g}/\text{L}$ NaOH solution (50 mL) and placed in a shaker at 100 rpm for 2 h to separate the EOM from the membrane, which was determined to be irreversible EOM [31].

In this study, membrane fouling is divided into reversible, irreversible and total membrane fouling. The flux of the new membrane during water filtration with a cup-type filtration vessel is referred to as J_0 , the flux of the fouled membrane before rinse is J_1 , and the flux of the rinsed membrane is J_2 . The total fouling rate (TF), irreversible fouling rate (IF) and reversible fouling rate (RF) can be computed as follows [29,32]:

$$TF = 1 - J_1/J_0 \quad (1)$$

$$IF = 1 - J_2/J_0 \quad (2)$$

$$RF = TF - IF \quad (3)$$

In this study, the total fouling rate represented the flux decline rate of the fouled membrane before rinse compared with the new membrane; the irreversible fouling rate represented the flux decline rate of the fouled membrane after rinse compared with the new membrane; while the reversible fouling rate represented the flux decline rate caused by the reversible foulants.

2.4. Analytical methods

An algae solution was centrifuged at 4000 rpm for 15 min with a high-speed centrifuge and then filtered in a $0.45\text{-}\mu\text{m}$ filter to acquire the reversible EOM. The anthrone-sulfuric acid method was used to determine the polysaccharide content: 1 g anthrone and 25 mL ethyl alcohol were put in 75% sulfuric acid (500 mL) to make the anthrone-sulfuric acid solution; 1 mL sample solution was in a test tube, and 10 mL anthrone-sulfuric acid solution was added; then, the tube was kept in a boiling water bath for about 15 min; after cooling to room temperature for 10 min in a water bath, the absorbance was measured by a spectrophotometer (UV-1101, China) at a wavelength of 620 nm; glucose was used as standard of carbohydrate. The protein content was measured by the modified Lowry method: the Lowry reagent was purchased from Lida Co., Ltd (Shanghai, China), and the reagent contained solution A and solution B; 0.1 mL sample solution was in a tube, and 0.5 mL solution A and 3 mL solution B was added successively, and then mixed evenly; the solution was kept at room temperature for 10 min and then its absorbance was measured at a wavelength of 750 nm; the bovine serum albumin was used as standard of protein. The algae concentration was determined by the OD_{680} method. TOC was determined using a total organic carbon analyzer (TOC-V_{CPH}, Shimadzu, Japan). The fluorescence excitation-emission matrix (EEM) spectra were measured using a Fluorescence Spectrophotometer (F-4500, Hitachi, Japan). Prior to EEM analysis, the pH of the reversible and irreversible EOM solutions was adjusted to 7.0 ± 0.1 [28]. The molecular weight (MW) distribution of EOM was analyzed by high performance size exclusion chromatography (LC-10 CE, Shimadzu, Japan). SEM analysis was used to observe the changes of EOM on the membranes (XL30FEG, PHILIPS, Holland).

3. Results and discussion

3.1. Long-term filtration

3.1.1. Filtration experiment

Long-term filtration tests were conducted to estimate the performances of the AVM system at different frequencies, and the TMP

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