



Dewatering of *Chlorella pyrenoidosa* using a diatomite dynamic membrane: Characteristics of a long-term operation

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ABSTRACT

Microalgae harvesting has been a primary problem encountered during microalgae utilization because of the low biomass concentration of microalgae and the severe membrane fouling caused by extracellular organic matters (EOM). Our previous study verified the applicability of dynamic membrane technology for algae dewatering. The aims of this study are to investigate the performances of both the diatomite dynamic membrane (DDM) technology, used for algae dewatering during a long-term (96 h) operation period, and the membrane fouling mechanisms, as well as their relationship with algae growth phases. The stable filtration flux reached 12.8 L/m² h, and the ultimate biomass concentration reached 49 g/L. The DDM continued to have advantages in cost, energy consumption and ultimate algae concentration. However, the stability of the DDM was questionable for long-term operations. Algae growth phases had distinct impacts on the EOM concentrations and composition and, therefore, on the membrane fouling mechanisms. The osmotic pressure originating from the EOM might exist during the DDM filtration process and could be largely responsible for the high cake resistance. As the algae growth phase advanced from the logarithmic phase to the decline phase, membrane fouling became much more severe.

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1. Introduction

Microalgae are phototrophic, heterotrophic or mixotrophic unicellular microorganisms that are capable of effectively converting inorganic nutrients, water, carbon dioxide and other substances into organic compounds such as carbohydrates, proteins and lipids [1]. The applications of microalgae are widely focused on biodiesel production [2] and wastewater treatment [3–4]. For such microalgae utilization technologies, the vital and unavoidable process of algae harvesting has significant problems, such as high energy consumption and cell breakage. Low biomass concentration, electrostatic repulsion between negatively charged cells and the excretion of extracellular organic matter (EOM) make the process difficult [5–6]. Algae harvesting consists of one or more solid–liquid separation steps, including concentration and drying processes [5].

In our previous study [7], a new type of diatomite dynamic membrane (DDM) technology for algae harvesting was developed. The dynamic membrane is also called the secondary membrane or the formed-in-place membrane [8]. Unlike traditional membranes, most of the interception for dynamic membrane technology is done by the dynamic cake layer, which is formed by fine particles

on the underlying primary filter surface [9]. Either an ultrafiltration/microfiltration membrane or a porous mesh is commonly used as the underlying primary filter and support materials are chosen based on which is best suited for the different operations [10]. In the wastewater treatment process, activated sludge usually forms the dynamic membrane [11]. The filtration performance, membrane fouling, the cake layer structure and the composition were investigated. The results showed that when compared with widely used algae concentration technologies, such as centrifugation, flocculation, sedimentation and flotation [12], DDM technology possesses the advantages of inexpensive membrane materials and high ultimate algae concentration [13].

To investigate the feasibility of DDM for long-term operation, the operation period was extended to 96 h, based on the results of the 24-h experiments [7]. The operation performance, particularly the influences of varied algae growth phases on the filtration flux, the transmembrane pressure (TMP) and the operation stability for long filtration period were given the greatest attention. The influences of the EOM of different algae growth phases on the filtration behavior and membrane fouling mechanisms were also examined. Comparative analyses were also conducted between short-term (24 h) and long-term (96 h) data to systematically explain the filtration characteristics of DDM and provide information for the further investigation and application of this technology.

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2. Materials and methods

2.1. Algae culture and EOM extraction

Chlorella pyrenoidosa (green algae, Collection No. FACHB-9) was purchased from the Institute of Hydrobiology, Chinese Academy of Sciences. Axenic cultures were conducted in batch mode in 2.5 L conical flasks with 1 L of autoclaved SE medium (NaNO_3 (250 mg/L); $\text{K}_2\text{HPO}_4 \cdot 3\text{H}_2\text{O}$ (75 mg/L); $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ (75 mg/L); $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ (25 mg/L); KH_2PO_4 (175 mg/L); NaCl (25 mg/L); $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ (5 mg/L); Fe-EDTA (1 ml/L); A_5 (1 ml/L); and soil extract (40 ml/L)).

The conical flasks were placed in a light incubator (GZX-300BS-III, CIMO Medical Instrument, Shanghai, China) and cultured at 30 °C, with an illumination of 5000 lx provided for 14 h every day.

For EOM extraction, a 0.6% (w/v) NaCl solution was used to dilute the samples of the cake layers to prevent cell lysis. Then, a 15-min centrifugation at 4000 rpm was utilized to remove diatoms and algae cells. The supernatant was filtered through a 0.45 μm filter to remove cell particles before tests [14].

2.2. Experimental procedure

The schematic diagram of the dewatering process of *C. pyrenoidosa* can be referred from Ref. [7]. The total effective volume of the operation zone was 1.5 L. The dynamic membrane module used a stainless steel mesh with an equivalent aperture of 75 μm as the support layer and had a filtration area of 0.01 m^2 . The diatomite for the DDM was purchased from Zhejiang, China, with a mean particle size of 7.8 μm and a BET specific surface area of 59.4246 m^2/g . The aeration rate was 4 $\text{m}^3/\text{m}^2 \text{ h}$. Before operation, a pre-coating stage of approximately 15 min was conducted, during which the cake layer was formed on the support membrane. After operation, a 15-min air–water backwash could effectively remove the cake layer and restore the membrane flux. Details about the system and operation stages can be found in Ref. [7].

With the filtration stage being as long as 96 h, the algae growth could not be ignored. *C. pyrenoidosa* broth, at different culture times (2, 6, and 10 days), was representative and chosen as the feed. During the operation period, the algae went through the mid-logarithmic phase to the mid-stationary phase, the mid-stationary phase to the late-stationary phase, and the late-stationary phase to the decline phase, respectively.

2.3. Analytical methods

The whole cake layer could be divided into three sub-layers based on its composition and morphology, including the slime layer, the algae layer and the diatomite layer. The diatomite layer was normally regarded as a substitute for traditional membranes. To gain insight into the filtration and fouling mechanisms, these sub-layers were separated using a slide, a surgical blade, and a microtome, and then were sampled for further analyses [7].

OD680 was chosen to measure the growth of *C. pyrenoidosa* with an absorbance measurement (UV-1101 UV/visible Spectrophotometer, China). The total EOM was calculated as the sum of polysaccharides and proteins, which were measured using the phenol–sulfuric method [15] with glucose for calibration and the modified Lowry method [16] with bovine serum albumin (BSA) for calibration, respectively.

The morphology of the sub-layers was analyzed using a scanning electron microscope (SEM515, Philip, Holland). An FTIR spectrophotometer (Nicolet 5700, Thermo Electron Corporation, USA) was used to reveal the functional groups of the cake layers. Luminescence spectrometry (F-4500 FL spectrophotometer, Hitachi, Japan) was used to obtain EEM spectra for the analysis of certain types of organics, especially proteins.

The rate of change of flux was calculated using Eq. (1)

$$\Delta F = \frac{dF}{dt} \quad (1)$$

where ΔF ($\text{L}/\text{m}^2 \text{ h min}$) is the rate of change of flux; F ($\text{L}/\text{m}^2 \text{ h}$) is the filtration flux; and t (min) is the filtration time.

The filtration resistance of the cake layer is calculated by Darcy's law as Eq. (2)

$$R_c = \frac{\Delta P}{\mu J} - R_m \quad (2)$$

where R_c (m^{-1}) is the filtration resistance of the cake layer; R_m (m^{-1}) is the filtration resistance of the support membrane; ΔP (Pa) is the transmembrane pressure (TMP); μ (Pa s) is the viscosity of the permeate; and J (m/s) is the filtration flux. During the filtration, the value of R_m is fixed and only R_c increases.

3. Results and discussion

3.1. Filtration performance of 96-h operation

Fig. 1(a) shows the flux and R_c changes during the first 240 min of filtration. Similar to the results of the 24-h operation, the flux change experienced a rapidly declining stage ($\Delta F > 1.0$), a slowly declining stage ($0.1 < \Delta F < 1.0$) and then a comparatively stable stage ($\Delta F < 0.1$). The flux decline of the 2-day group was less severe than that of the 6- and 10-day groups. Because diluting the

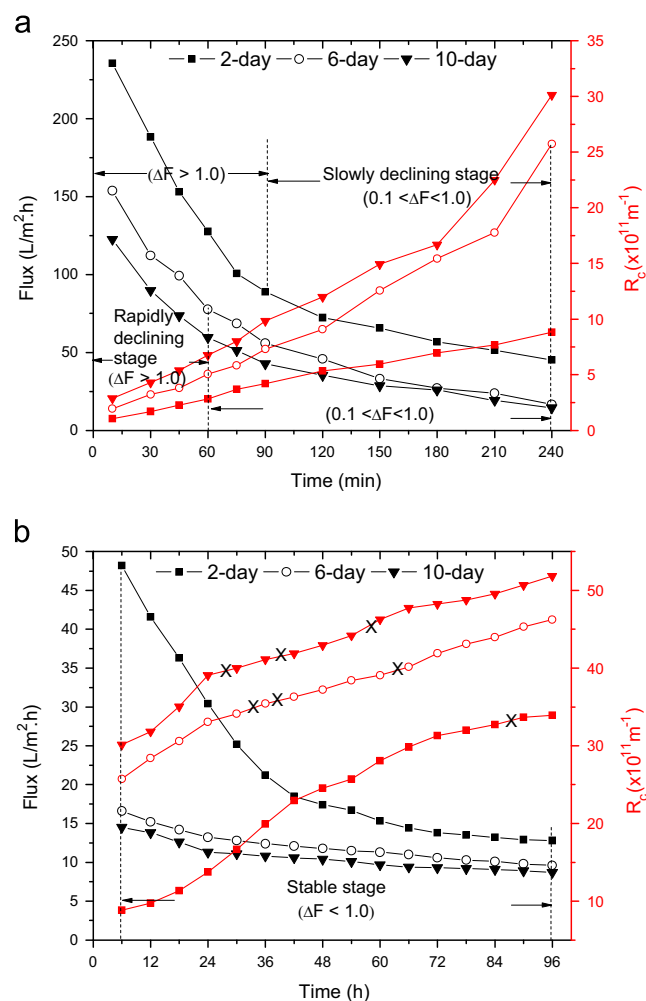


Fig. 1. (a) First 240 min and (b) 96-h flux and R_c variations. (The unit of ΔF is $\text{L}/\text{m}^2 \text{ h min}$.)

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