



# The fouling effects of microalgal cells on crossflow membrane filtration



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## ABSTRACT

The main drawback of membrane processes is biological membrane fouling which, in turn, leads to the membrane having an increased amount of transmembrane pressure and energy consumption. The fouling of microfiltration (MF) and ultrafiltration (UF) membranes caused by microalgal cells was investigated in this study. An MF membrane with a pore size of 0.20  $\mu\text{m}$  (made of PVDF) and UF membranes with a molecular weight cutoff (MWCO) of 150, 50, and 30 kDa (made of PES, PESH and RC, respectively) were employed for the filtration experiments. An attenuated total reflection-Fourier infrared (ATR-FTIR) spectroscopy, a scanning electron microscope (SEM), and contact angle device were utilised in order to characterise the membranes fouled by microalgal cells. The results demonstrated that the MF membrane exhibited a faster decrease in flux when the normalised flux was reduced to as low as 0.15. An increase in the crossflow velocity (CFV), on the other hand, can dramatically decrease microalgal cells depositing on the surface of a membrane, thereby leading to higher flux. Moreover, microalgal cells caused both reversible and irreversible fouling for all membranes. In addition, the analysis of the different filtration resistances encountered in membrane filtration involving concentration polarisation, cake layer formation, and the clogging of pores was studied. The experimental results obtained in this study show that the cake resistance ( $R_c$ ) had the highest resistance for the UF membranes and that the concentration polarisation ( $R_{cp}$ ) was dominant in all crossflow velocities for MF membranes.

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## 1. Introduction

Recently, microalgae have attracted much attention due to their potential for being used in a wide variety of processes. They can be used in a vast array of areas, such as in pharmaceutical industries as antibiotics, antioxidants, cosmetics, and food supplements; in the biofuel industry as biodiesel and biogas; as a biotreatment of wastewaters; and in aquaculture and agriculture. Especially in the biodiesel industry, compared to terrestrial crops such as canola, soybean, and oil palm, microalgae have several advantages. For instance, microalgae have a higher photosynthetic efficiency than terrestrial plants [1]. Moreover, they grow approximately 50 times faster than other plants [2]. Microalgae (containing 30% oil by wt.) can give a biomass yield of 58,700 L/ha year, which is much higher than canola or rapeseed (1190 L/ha year), soybean (446 L/ha year), palm oil (5950 L/ha year) and jatropha (1892 L/ha year) [3]. With respect to air quality maintenance and improvement, microalgae can fix carbon dioxide (1 kg of algal biomass absorbs roughly 1.83 kg of  $\text{CO}_2$ ) at higher rate than other plants [4]. In order to minimise the use of fertilizers, wastewaters which are rich in

carbon, nitrogen and other minerals can be used as a cultivation medium [5]. In addition, microalgae can be cultivated on non-arable land, such as deserts and soils with a high salination content [6].

Although microalgae have many advantages, recovering microalgal biomass is a significant problem because of the small size (3–30  $\mu\text{m}$  diameter) of the algal cells, their low concentration in the culture medium (less than  $\sim 1$  g/L dry weight), and the large amounts of water that must be processed in order to recover the biomass.

Several technologies for the harvesting of microalgae cultures have been developed. Centrifugation, coagulation, flocculation, flotation, sedimentation and filtration are well known processes among these technologies [7–9]. All of these technologies, however, are energy-intensive, time consuming, and environmentally harmful. Compared to these methods, membrane technology (e.g., microfiltration (MF) and ultrafiltration (UF)) offers several advantages. For example, membrane filtration allows for the complete retention of microalgae from the culture media while also preserving the structure, properties, and motility of the collected cells [10]. It can remove microorganisms from the used microalgae culture media while recovering residual nutrients, thereby allowing the nutrient media can be recycled [7]. Membrane filtration can potentially reduce the energy consumption from harvesting

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microalgae. Moreover, There is no need for additional chemicals, which simplifies subsequent production of oil and other high value by-products [7].

The most common filtration methods used in the industry are dead-end and crossflow filtration [11]. It has been reported that, for microalgal harvesting, the crossflow filtration offers a high flux due to the high crossflow velocity and high shear conditions exhibited on the membrane surfaces [12,13], while dead-end filtration is a more restricted technique because it damages organisms and the abilities to limited volume processing [11]. In addition, the dead-end filtration is also more vulnerable to the formation of cake layer on the membrane. Thereby leading to a remarkable reduction in membrane permeability. Consequently, crossflow filtration may be the more appropriate technique for harvesting microalgae.

A well-known drawback of membrane filtration is membrane fouling, which leads to an increased transmembrane pressure and energy consumption [14]. There are various mechanisms playing important roles in the occurrence of membrane fouling. These are concentration polarisation, cake layer formation and pore blocking [15]. Using microfiltration membranes, Hwang et al. [16] investigated the effects of membrane pore size on particle fouling, finding that loose membranes, which have larger pores, were subjected to more severe fouling. Qu et al. [17], on the other hand, focused on the effects of membrane pore size and surface hydrophobicity on ultrafiltration (UF) membrane fouling caused by extracellular algal organic matter (EOM). They reported that membranes with larger pores exhibit worse flux reduction but less adsorptive fouling, as well as superior flux recovery [17]. Furthermore, they also stated that cake formation was the main mechanism for the flux decline caused by EOM, independent of membrane pore size and surface hydrophobicity [17]. In addition, Sun et al. [18] has investigated the performance of both microfiltration and ultrafiltration membranes for algae harvesting. Their experiments showed that membrane materials play a very important role in the harvesting of microalgae [18]. Therefore, many researches have proven that membrane properties, which include pore size, surface hydrophobicity, surface charge, and surface roughness, can affect membrane fouling to some degree [16–18].

Although many studies have been carried out in order to investigate the membrane fouling caused by microalgal cells, the mechanism of membrane fouling has not completely been understood and reported yet. The effects of the membrane properties and operating conditions on the flux decline, fouling reversibility, and characterisation of fouled membranes still remains to be investigated. The aim of this study was to investigate the fouling behaviour of microalgal cells on three ultrafiltration and one microfiltration membranes which were made from different polymeric materials and having different pore sizes. In this study, flux decline and reversibility of fouling were examined in terms of various membrane characteristics. Moreover, to better understand how to occurs flux decline in crossflow filtration, various filtration resistances were investigated under different CFVs. Finally, in order to gain more information about the fouled membranes, attenuated total reflection-fourier transform infrared (ATR-FTIR) spectroscopy, a scanning electron microscope (SEM), and a contact angle device were used.

## 2. Materials and methods

### 2.1. Cultivation of the microalgae

Freshwater microalgae (*Chlorella vulgaris*), a unicellular green microalgae, was used as the biological foulant in this study. It was

grown in a treated landfill leachate which was collected from the İSTAÇ A.Ş. Odayeri Leachate Treatment Plant in İstanbul, Turkey. It came from the effluents of its nanofiltration system and consisted of the following components: Colour, 12 PtCo; COD, 1145 mg/L;  $\text{NH}_4^+ - \text{N}$ , 268 mg/L; TKN, 314; Conductivity, 24.5 mS/cm; [19], Fe, 0.29 mg/L; Cu, 0.03 mg/L; and Ni, 0.27 mg/L. 100 mg/L of  $\text{K}_2\text{HPO}_4$  was synthetically added to provide an N/P ratio of 15:1. The seed culture was grown continuously in a 5 L bench-top stirred fermenter (Electrolab Fermac 320) for about 30 days to achieve a sufficient cell density. The cultivation conditions were as follows: mixing=200 rpm; air=0.6 L/min; light intensity=70  $\mu\text{mol m}^{-2} \text{s}^{-1}$  (MIC Lightmeter, Model 98209); light/dark ratio=24:0; temperature=25  $\pm$  2 °C; and pH 7.5  $\pm$  1. The pH of the culture medium was adjusted by adding either 0.1 N HCl or 0.1 N NaOH. The cells were imaged using an Olympus BX51 microscope (magnification of 20  $\times$ ) equipped with a digital camera. The cells were spherical. In addition, the size of most cells ranged from between 2 and 7  $\mu\text{m}$ , with a mean value of 5  $\mu\text{m}$  (data not shown). Furthermore, all experimental materials before use were autoclaved for 15 min at 121 °C.

### 2.2. Determination of the Algae's biomass concentration

The optical density of the cells at 680 nm was measured using a spectrophotometer (WTW PhotoLab 6600 UV-vis Spectrophotometer). To determine the dry biomass weight of the microalgal cultures, 50 ml aliquot of the microalgal culture was filtered over a 0.45  $\mu\text{m}$  fibre glass filter. The filter was dried for two hours at 105 °C to a constant weight. Then, it was cooled in a desiccator for 30 min, and weighed again. Dry biomass weight was determined by subtracting the final filter weight from the raw filter weight and was expressed as g/L. Next, a correlation between the optical density and dry biomass was determined. Then, the amount of dry biomass was calculated from the optical densities of the algal suspensions at 680 nm. The correlation is shown below:

$$\text{Dry weight (g L}^{-1}\text{)} = 0.497 \times \text{OD}_{680} + 0.026, \quad R^2 = 0.9959 \quad (1)$$

Membrane fouling experiments have been conducted with 0.50 g/L biomass concentration so as to compare the performance of the used membranes.

### 2.3. The membrane

Table 1 shows four different commercial membranes (manufactured by Microdyn-Nadir) that were tested, with one MF and three UF membranes. New membranes were used with each test. These were first filtered with deionized (DI) water under 1 bar for 30 min in order to clean the membrane surfaces from accumulated chemicals. Then, the transmembrane pressure was increased to 3 bars for at least 5 min for the purpose of compacting the membrane.

### 2.4. Experimental procedures

#### 2.4.1. Membrane test units

The experiments were performed using a laboratory scale crossflow membrane module. The effective membrane area of the module was 138 cm<sup>2</sup>. Fig. 1 shows a experimental setup of the membrane system. The concentrate was recirculated into the feed tank, while the filtered water was collected into the beaker. The filtrate was weighed by using electronic balance, after which the data were collected with a balance communication software, and then the flux was calculated using a personal computer. All the experiments were performed at 22  $\pm$  1 °C, which was kept using a heat exchanger into the feed tank. The crossflow velocity (CFV)

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