

Performance Evaluation of Flocculation and Membrane Filtration for Microalgae Harvesting

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ABSTRACT

Microalgae (MA) has huge potential use as feedstock for producing biodiesel. However, harvesting of MA is still a major obstacle. This paper presents a performance evaluation of flocculation and membrane processes for harvesting of MA. The experiments were conducted using *Chlamydomonas sp.*, which was cultured in an open pond. Chitosan was used as flocculants, while microfiltration and ultrafiltration membrane were used during filtration using membrane. The results showed that the harvesting efficiency of MA using flocculation was within the range of 74.2-81.2%. The harvesting of MA using membrane processes resulted in efficiency within the range of 86.8-91.1% and around 99% for MF and UF, respectively. The harvesting efficiency of the combination of flocculation and MF was comparable with UF only i.e. ~99%. The performance of flocculation process was influenced by the concentration of the flocculant, the agitation rate and the agitation time. Flocculation installed before MF membrane improved the resulting normalised flux of microfiltration membrane as well as increased harvesting efficiency.

Keywords: Microalgae harvesting, membrane filtration, flocculation, fouling, microfiltration, ultrafiltration

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INTRODUCTION

The global energy crisis has encouraged an effort to create renewable energy. Biodiesel has been concerned as one of renewable energy types to replace fossil fuel. Biodiesel is produced from fatty acid derived from edible oil or non-edible oil, which can be processed by esterification and/or trans-esterification (Chisti, 2010; Hu et al., 2008). Microalgae has been explored as the raw material for biodiesel production (Hu et al., 2008). Microalgae produces more oil than terrestrial plants such as corn, soybean, jatropha, coconut and

oil palm (Chisti, 2010). Although the cultivation of algae is relatively easy, the harvesting of microalgae is rather difficult because of its small size, its density is close to water, the concentration of the media is very dilute, the surface is negatively charged and there is a lot of dispersed algonenic organic matter in the surrounding environment (Bilad, Arafat, & Vankelecom, 2014; Danquah, Ang, Uduman, Moheimani, & Fordea, 2009; Kim et al., 2013; Zhang, Hu, Sommerfeld, Puruhito, & Chen, 2010). Furthermore, the energy required for the harvesting process can be higher than the energy produced by the microalgae itself (Chisti, 2010; Tran et al., 2013; Uduman, Danquah, Forde, & Hoadley, 2010).

The method of harvesting depends on the properties of microalgae such as density, size and value of the desired product (Vandamme, Muylaert, Fraeye, & Foubert, 2014). Several microalgae harvesting techniques have been developed, such as flocculation, centrifugation, ultrasound, floatation and membrane filtration. Among them, flocculation is commonly used due to its simple operation (Danquah et al., 2009; Kim et al., 2013; Zhang et al., 2010; Tran et al., 2013; Uduman et al., 2010; Vandamme et al., 2014). Flocculants are added to merge algae cells into large flocks such that they can be easily separated. As an example, Vandamme, Foubert, Meesschaert and Muylaert (2010) used cationic starch as flocculants for microalgae harvesting. The flocculation efficiency was reported to be 80-90%, with flocculants doses within the range of 10-20 mg/l. However, the flocculation method requires a large space to accommodate sedimentation and high operating costs (Ryll et al., 2000).

Harvesting of microalgae by centrifugation with high speed rotation was conducted by Chen, Yeh, Aisyah, Lee and Chang (2011) to obtain algae cells. This technique was effectively used to separate microalgae cells from their liquid medium. However, according to Knuckey, Brown, Robert and Frampton (2006), the force of gravity and high friction leads to damage of cell structure. In addition to the weakness of the centrifugation method, there is also the high operating cost to be considered (Ryll et al., 2000). Bosma, Spronsen, Tramper and Wijffels (2003) applied ultrasound for the harvesting of microalgae. Ultrasonic irradiation was imposed on the microalgae at a specific wavelength causing cavitation bubbles. After operation, sedimentation of cells was rapidly formed due to the force of gravity. The disadvantage of this technique was that the operation was difficult and the investment costs were high (Bosma et al., 2003). Kurniawati, Ismadji and Liu (2014) conducted harvesting of microalgae by floatation using saponin and chitosan. This method was very effective for separating microalgae and algonenic organic matters. However, it required further processing and investment in expensive equipment.

Ultrafiltration and microfiltration have been proposed for microalgae harvesting (Zhang et al., 2010; Rickman, Pellegrino, & Davis, 2012; Bilad, Vandamme, Foubert, Muylaert, & Vankelecom, 2012). Membrane filtration has advantages that include being environmentally friendly (Zhang et al., 2010); providing separated water and nutrients that can be reused to grow microalgae (Ahmad, Yasin, Derek, & Lim, 2012); allowing for a continuous process using low energy (Rickman et al., 2012; Ahmad et al., 2012; Bhave, Kuritz, Powell, & Adcock, 2012); and being run on a low operating cost compared to other processes such as centrifugation and flocculation (Ahmad et al., 2012; Grima, Belarbi, Fenandez, Medina, & Chisti, 2003; Weschler, Barr, Harper, & Landis, 2014). However, the main problem of membrane filtration is the

formation of fouling, which causes a decrease in flux and affects the efficiency of harvesting. Some of the strategies conducted to overcome fouling include membrane modification (Hwang, Park, Oh, Rashid, & Han, 2013), fluid management (Kim, Jung, Kwon, & Yang, 2015), membrane washing (Huang et al., 2012) and chemical pre-treatment (Babel & Takizawa, 2011).

In summary, flocculation and membrane filtration have been proposed as promising techniques for harvesting of microalgae. Each technology has its own advantages and disadvantages. Flocculation has lower separation efficiency than MF and UF but the complexity of membrane operation includes fouling, which restricts the use of membrane filtration. By contrast, flocculation can still be found in practical application. It is well known that UF has higher separation efficiency than MF. However, in terms of flux and energy used, MF should be more competitive than UF. Thus, it is not easy to determine the best process that should be chosen i.e. flocculation, microfiltration, ultrafiltration or their combination. More attention is given to performance comparison between ultrafiltration and a combination of flocculation and microfiltration.

Furthermore, there also seems to be considerable disagreement among the different reports. This is because the performance of this process is influenced by many factors such as coagulation type, coagulation condition, filtration performance, type of microalgae and microalgae concentration. This paper systematically evaluates the performance of flocculation, membrane filtration and their combination for harvesting of microalgae. The chitosan as flocculants was chosen because of high cationic charge density, long polymer chain, non-toxic, biodegradable, little problems for subsequent application of the recovered biomass and the recycling of the culture medium (Xu, Purton, & Baganz, 2013). It is expected that high harvesting efficiency will be achieved and fouling as a severe problem of membrane process will be reduced by combining flocculation and membrane filtration.

MATERIALS AND METHODS

Materials

Chlamydomonas sp. culture obtained from an open pond in the premises of the Department of Chemical Engineering, Diponegoro University, was used as the feed. Chitosan was purchased from Biotech Sorendo Indonesia. Sodium hydroxide and hydrogen chloride were purchased from Merck. Two commercial polyethersulfone (PES) ultrafiltration (UF) membranes (with molecular weight cut-off of 10 and 100 kDa) and a commercial regenerated cellulose (with MWCO 10 kDa) were used. All UF membranes were supplied by Alfa Laval (Denmark). In addition to the UF membrane, a commercial PES microfiltration membrane (with the average pore size $\sim 0.8 \mu\text{m}$) obtained from Membrana GmbH (Germany) was used.

Quantification of microalgae cells

The algae was cultivated at room temperature ($25 \pm 1^\circ\text{C}$) and at a constant pH of 7. Turbidity was used as a representation of microalgae concentration. Turbidity was measured using the Nephelometric method (SM 2130 B). Figure 1 shows the correlation of turbidity (x) and number

of microalgae cells (y) resulting from the experiment. Furthermore, it can be expressed in the following linear correlation (Equation 1).

$$y = 3.443 * 10^7 x \quad [1]$$

where y is the number of microalgae cells and x is turbidity.

Harvesting of Microalgae by Flocculation

Harvesting of microalgae by flocculation was performed using a jar test equipped with an agitation system. In this experiment, chitosan was used as the flocculant. The initial concentration of microalgae (C_{A0}), the dose of flocculants (C_c) and the agitation time were varied. Harvesting efficiency was determined using the following equation:

$$\text{Efficiency} = \frac{C_{A0} - C_A}{C_{A0}} \times 100\% \quad [2]$$

where C_{A0} and C_A are the microalgae concentration before and after flocculation, respectively.

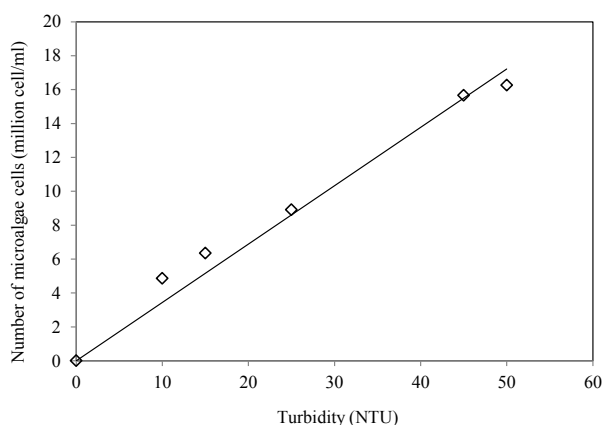


Figure 1. Correlation of turbidity and the number of microalgae cells

Harvesting of Microalgae by Membrane Filtration and the Combination of Flocculation and Membrane Filtration

In membrane filtration, either microfiltration or ultrafiltration membrane was used in filtration experiments. The filtration was performed by cross-flow filtration with feed and bleed mode. The membrane was firstly compacted by pressurising distilled water in a feed tank into the membrane cell using a pump. Thereafter, the pressure was lowered to the operating pressure for filtration. The pure water flux was then measured as initial pure water flux (J_0). In a filtration experiment, distilled water was replaced with a solution of microalgae of a certain concentration.

The microalgae solution was pumped at a certain pressure into the membrane cell. Both the retention and permeate flux were not returned to the feed tank. The value of the permeate flux (J) was measured for 120 minutes. All the experiments were conducted at room temperature ($28 \pm 2^\circ\text{C}$) and at a constant trans membrane pressure (300 kPa for UF and 20 kPa for MF). The permeate flux was measured by collecting the permeate volume for a certain period of time and calculated using Equation [3]. To take into account the heterogeneity of the membranes, the normalised flux (J/J_0), which is the ratio of the permeate flux to the initial pure water flux of the same membrane used, was used to express flux behaviour.

$$J = \frac{V}{A \times t} \quad [3]$$

where J is permeate flux ($\text{L}/\text{m}^2\text{h}$), V is permeate volume (L), A is membrane surface area (m^2) and t is filtration time (h).

In the experiments using a combination of flocculation and membrane filtration, flocculation of microalgae solution was firstly performed. First, microalgae was flocculated using the optimum condition obtained from the flocculation experiment. Thereafter, the supernatant of the flocculated microalgae solution was flowed into the feed tank for membrane filtration. The following experiment protocols were similar to the previous membrane filtration using an MF membrane. The harvesting efficiency was determined using Equation [2], where C_A is the MA concentration in the permeate stream (both for the membrane filtration only and the combination of flocculation and membrane filtration) instead of the MA concentration after flocculation. Efficiency measurement was conducted every 15 minutes of filtration (at 15, 30, 45, 60, 75, 90, 105 and 120 min) and the results were then averaged.

RESULTS AND DISCUSSION

Harvesting of Microalgae by Flocculation

The flocculation process is influenced by concentration of flocculant, agitation speed and agitation time. Figure 2 shows clearly that chitosan can be used as a flocculant for the harvesting of microalgae. Further, flocculation efficiency is influenced by both flocculant concentration and agitation rate. The increase in chitosan concentration, firstly, increases flocculation efficiency. However, further increase in chitosan concentration decreases flocculation efficiency. In this study, a similar phenomenon was observed in the effect of agitation rate i.e. the increase in agitation rate increased flocculation efficiency but further increase decreased efficiency. Several mechanisms of coagulation using chitosan have been explained in previous publications (Tran et al., 2013; Vandamme et al., 2014). In this case, the amine groups of chitosan were protonated to NH_3^+ , leading to electrostatic attraction of the microalgae cells, which finally forms microalgae aggregates.

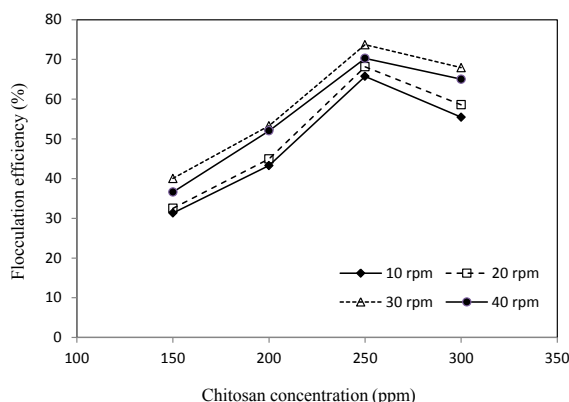


Figure 2. The effect of chitosan concentration on flocculation efficiency at various stirring rates for 15 minutes of agitation. Microalgae concentration was 2.96×10^{10} cells/ml

It is seen that the best flocculation efficiency was 74%, obtained at the concentration of chitosan of 250 ppm with an agitation rate of 30 rpm. As explained by Sundstrom and Klei (1979), at a low agitation rate, the energy required to make a large flock is not enough, whereas at a high agitation rate, the high energy can break the already formed flocks.

Figure 3 shows the effect of agitation time on flocculation efficiency. It is seen that the increase in agitation time increased efficiency. However, excessive agitation i.e. agitation beyond 35 minutes broke the flocks and therefore, decreased flocculation efficiency. The best flocculation efficiency was obtained from the flocculation where the agitation rate was 30 rpm for 35 minutes.

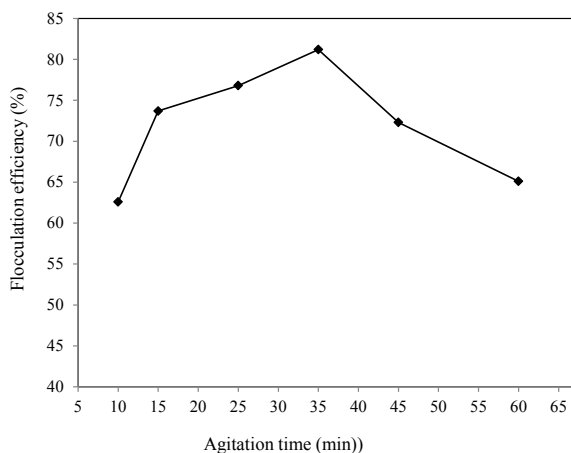


Figure 3. The effect of agitation time on flocculation efficiency at concentration of chitosan 250 ppm. Microalgae concentration was 2.96×10^{10} cells/ml

In order to know the ratio of microalgae concentration and chitosan concentration used for flocculation, the concentration of the microalgae in the feed solution was varied. As clearly seen in Figure 4, flocculation efficiency was influenced by both microalgae and chitosan concentrations. As the microalgae concentration was decreased the chitosan concentration required to obtain optimum flocculation efficiency decreased. Excessive addition of chitosan compared to microalgae caused the formed microalgae flocks to become unstable. Furthermore, these unstable flocks were deflocculated and their sizes became smaller. This phenomenon has been explained by Yoon et al. (2005) and Matos, Benito, Cambiella, Coca and Pazos (2010). Figure 4 suggests that the optimum flocculation of microalgae using chitosan was obtained at the ratio of microalgae and chitosan within the range 10^{11} to 1.4×10^{11} cells/mg of chitosan. This means that 1 to 1.4×10^{11} cells of microalgae need 1 mg of chitosan to obtain 75-80% flocculation efficiency.

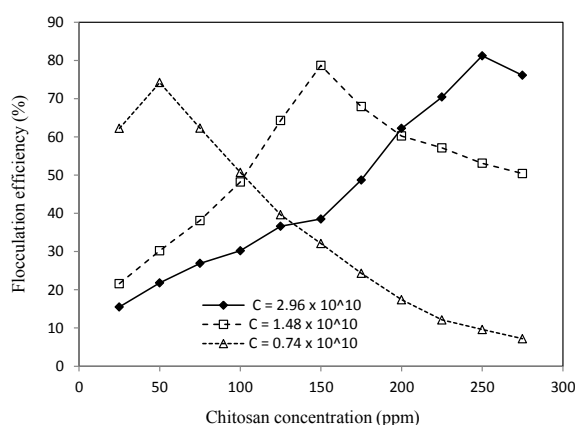


Figure 4. The effect of chitosan concentration on flocculation efficiency at various concentrations of microalgae. The agitation rate and agitation time were 30 rpm and 35 min, respectively

Harvesting of Microalgae by Membrane Filtration

In this experiment, MF and UF membranes were used for filtration of the microalgae solution. The membrane was operated at a pressure of 20 kPa for MF and 300 kPa for UF. The performance of membrane filtration was determined by measuring permeate turbidity and permeate flux. The results are presented in Figure 5.

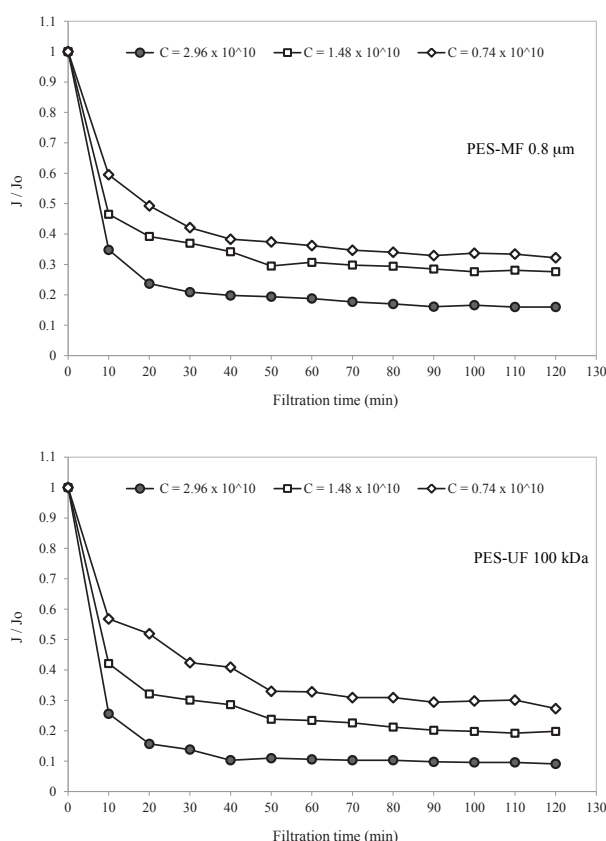


Figure 5. Normalised flux profile during filtration of various microalgae solutions (cells/ml) using microfiltration membrane (top panel) and ultrafiltration membrane (bottom panel)

Both MF and UF membranes showed rapid flux decline at the beginning of filtration and after about 40 minutes, they displayed a steady state flux. The concentration polarisation, which is an accumulation of retained microalgae on the membrane surface, is believed to have contributed to this rapid flux decline. This explanation is evidenced by the results that demonstrated the higher concentration of microalgae the lower normalised flux. As the concentration of microalgae was increased the number of microalgae cells accumulated at the membrane-solution interface increased. The contribution of concentration polarisation on flux decline has been reported in many previous publications (e.g. Zularisam, Ismail, & Salim, 2006; Rickman et al., 2012; Susanto, Roihatin, & Widiassa, 2016). The contribution of concentration polarisation and fouling to the flux decline was investigated by stopping the filtration after 10 minutes of filtration for 10 seconds. The contribution of fouling was evidenced by the fact that the flux could not be restored to its initial value after the filtration was restarted (data not shown). The contribution of concentration polarisation was proven by the higher flux after the filtration was restarted than the flux before stopping the filtration.

Comparing the MF and UF membranes showed that for the same concentration of microalgae, the normalised flux of the MF membrane was higher than that of the UF membrane. This indicates that fouling of PES-UF 100 kDa for harvesting of microalgae was higher than

the MF membrane. In order to further investigate the effect of membrane characteristics, various UF membranes were used. The results are presented in Figure 6. In addition to fouling, concentration polarisation investigation (the method used was similar to the method that yielded the results seen in Figure 5) showed that MF demonstrated a lower concentration polarisation effect on flux reduction than all the UF membranes.

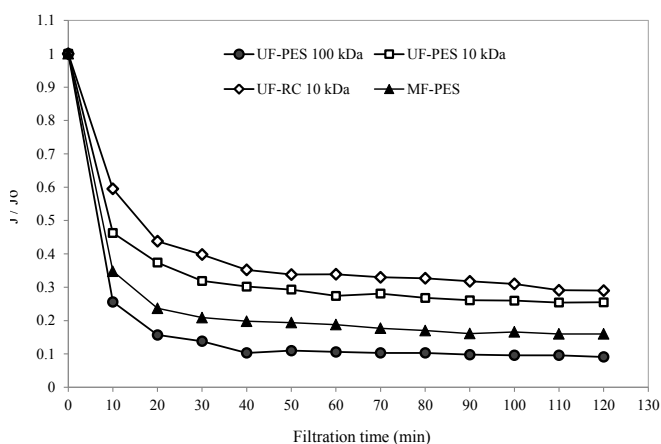


Figure 6. Normalised flux profile during filtration of microalgae solution (2.96×10^{10} cells/ml) using various membranes. The experiments were conducted at a constant pressure of 300 kPa for all UF membranes and 20 kPa for the MF membrane

Figure 6 shows that normalised flux behaviour was influenced by both membrane pore structure and membrane material. For the same membrane material (see UF-PES 100 kDa vs. UF-PES 10 kDa vs. MF-PES), it was observed that the membrane having the smallest pore size showed the highest normalised flux. The increase in membrane pore size decreased normalised flux, indicating higher fouling had taken place. However, regardless of the resulting efficiency, if the membrane used had a very large pore size (see PES-MF membrane), the normalised flux would increase, indicating less fouling. These phenomena can be explained by the fouling mechanism. Fouling occurs via pore narrowing, complete pore blocking and gel layer formation. Initially, if we increase membrane pore size, the possibility of complete blocking will be higher. However, if the membrane pores are too large compared to the dimension of the microalgae, the possibility of complete blocking will be lower. For membranes that have a large pore size, the possibility of the microalgae to access membrane pores is higher, leading to higher pore narrowing than pore blocking. It should be kept in mind that fouling by complete blocking reduces flux more significantly than pore narrowing.

Comparing the membranes, which had the same pore size but were made of different materials (see UF-PES 10 kDa and UF-RC 10 kDa), showed that the UF-RC 10 kDa had higher normalised fluxes than the UF-PES 10 kDa. This suggests that the fouling during filtration of microalgae was influenced by membrane material i.e. hydrophilic material was more resistant towards fouling than hydrophobic material.

Table 1 shows the harvesting efficiency of the different processes used. It is seen that using the MF membrane showed a higher efficiency than did the flocculation technique (~90% vs. ~80%). The increase in concentration of microalgae slightly increased harvesting efficiency. All the UF membranes demonstrated higher efficiency than the MF membrane. There was no significant difference in harvesting efficiency among UF membranes. All UF membranes showed very high harvesting efficiency i.e. higher than 99%.

Table 1
Comparison of Harvesting Efficiency for Different Methods

No	Microalgae concentration ^a	MF			UF			Flocculation + MF ^d	
		250 ^b	25 ^b		PES-100	PES-10	RC-10	25 ^b	250 ^b
1	~2.96 x 10 ¹⁰	81.2	15.5	91.1	99.6	99.8	99.2	98.2	99.2
2	~2.96 x 10 ¹⁰	78.7	21.6	89.3	99.5	n.dc	n.dc	99.1	99.7
3	~2.96 x 10 ¹⁰	74.2	62.2	86.8	99.2	n.dc	n.dc	99.4	99.6

^ain cells/ml; ^bflocculant concentration in ppm; ^cnot done; ^dcombination of flocculation and MF

Harvesting of Microalgae by a Combination of Flocculation and Microfiltration

Flocculation as a pre-treatment process was expected to improve harvesting efficiency of the microfiltration membrane as well as to reduce fouling. In this experiment, flocculation was combined with MF. The results are presented in Figure 7.

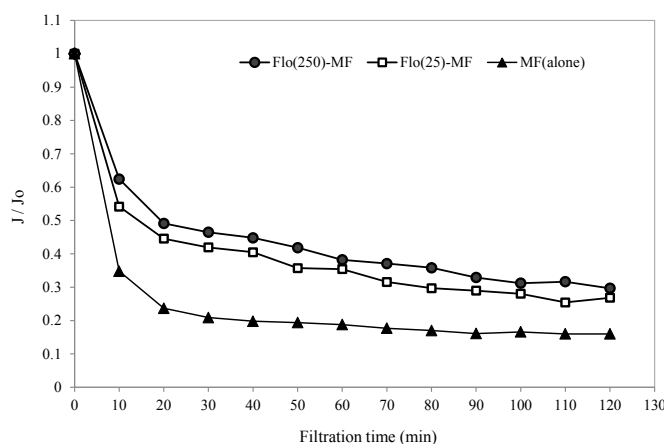


Figure 7. Normalised flux profile during microfiltration with and without flocculation. The concentration of microalgae was 2.96 x 10¹⁰cells/ml. The number inside the brackets indicates chitosan concentration used in ppm. The experiments were conducted at a constant pressure of 0.2 kPa

It is clearly seen that using flocculation before using a microfiltration membrane increased the normalised flux, indicating less fouling had occurred. This means that the existence of flocculation was able to reduce fouling in microfiltration. Larger particles of microalgae were formed by flocculation so that the possibility of complete blocking of membrane pores was

smaller, leading to higher flux. In addition, a cake layer formed by the flocculated particles on top of the membrane surface was more porous than the original particles (Barbot, Moustier, Bottero, & Moulin, 2008). Even though a very low concentration of chitosan was used (25 ppm), a significant fouling reduction was observed. A similar phenomenon was reported by Matos et al. (2010), who integrated flocculation and ultrafiltration to filter activate sludge.

In addition to reducing fouling, flocculation can also increase harvesting efficiency of microfiltration membranes from 86-91% to 98-99.7% on the one hand (Table 1). On the other hand, a microfiltration membrane could also increase harvesting efficiency of the flocculation technique. Interestingly, with a very low concentration of chitosan, the harvesting efficiency could reach 99%, which is comparable to using an ultrafiltration membrane.

In order to further compare the harvesting efficiency of flocculation, microfiltration, ultrafiltration and the combination of flocculation and microfiltration, the experiments using a feed with similar total loading were conducted on the same conditions detailed in Table 1 (flocculation using 250 ppm, MF, UF PES 100 kDa, flocculation 25 ppm and MF). The results showed that the process efficiencies were similar to the results presented in Table 1. The mutual effect of flocculation and MF with respect to flux and harvesting efficiency was observed. Further, the combination of flocculation (using 25 ppm) and MF showed a harvesting efficiency that was comparable with that of using a UF membrane i.e. 99.3% (for flocculation and MF) compared to 99.6% (for UF).

CONCLUSION

The performance of flocculation, microfiltration, ultrafiltration and the combination of the flocculation and microfiltration processes for microalgae harvesting was evaluated in this paper. The important parameter in harvesting of microalgae using flocculation was the ratio of microalgae concentration to chitosan concentration, agitation rate and agitation time. Overall, the best harvesting efficiency resulted from flocculation was 81%. Harvesting using a MF membrane showed higher efficiency than using the flocculation process (~90% vs. ~80%). All the UF membranes demonstrated a higher efficiency than the MF membrane i.e. higher than 99%. The flux behaviour and harvesting efficiency of membrane processes were influenced by membrane pore structure and membrane material. Integrating flocculation and using a MF membrane could give a mutual effect i.e. increasing both harvesting efficiency and permeate flux of the MF membrane and reducing significantly the amount of flocculants used.

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