Korean J. Chem. Eng., **31**(6), 949-955 (2014)
DOI: 10.1007/s11814-013-0276-4

pISSN: 0256-1115
eISSN: 1975-7220

Optimization of hollow fiber membrane cleaning process for microalgae harvest

Hyuckjin Kwon, Mingshou Lu, and Jinwon Lee[†]

Department of Chemical and Biomolecular Engineering, Sogang University, Seoul 121-742, Korea
(*Received 9 August 2013 • accepted 13 December 2013*)

Abstract−Biodiesel made from microalgae could be a possible replacement for fossil fuel. The separation of microalgae from their culture medium is a critical process in biotechnology. Membrane techniques seem to be effective, reliable, and safe, despite some limitations such as the progressive fouling and the associated decline in flux. Microalgae harvested from membranes have less chemical contamination and therefore membrane systems are more environmentally friendly than other harvesting techniques. We used a microfiltration (MF) system to concentrate the microalgae *Tetraselmis* sp. KCTC12236BP. Two cleaning processes, forward flushing and backwashing, were assessed to minimize the fouling required to concentrate the microalgae *Tetraselmis* sp. KCTC12236BP. It was concluded that backwashing was a more effective method than forward flushing. Furthermore, higher frequencies of backwashing greatly improved the membrane flux. However, even though the optimum cleaning frequency for maintaining high flux was every 5 min, from the perspective of microalgae concentrate and energy consumption, better results were obtained when backwashing was applied every 10 min. Under these conditions the microalgae culture reached 90% concentration in 60 min.

Keywords: *Tetraselmis* sp., Hollow Fiber Membrane, Backwashing, Forward Flushing, Sodium Hypochlorite

INTRODUCTION

Given that the availability of crude oil is limited and that the combustion of fossil fuel results in the production of carbon dioxide and global warming, much emphasis throughout the world has been placed on the development of alternative energy sources using biomass as a replacement for fossil fuels [1-3]. Recently, bio-fuels produced from microalgae biomass have been suggested as a possible candidate [4]. However, harvesting technology is an important factor in the production of biodiesel, and an effective and economic method of microalgae harvesting has yet to be developed [5]. Membrane systems such as microfiltration and ultrafiltration have been the predominant method for producing clean drinking water for several years [6]. Such membrane-based technology also has the potential as a method of harvesting microalgae for biodiesel production [7]. Filtration has many advantages over the conventional methods of centrifugation, flocculation-floatation, and sedimentation [8,9]. However, one of the major disadvantages of using membrane technology is the membrane fouling [10], which is induced by the deposit of inorganic, organic and microbiological substances on both the membrane surface and within the membrane pores [11-13]. Membrane fouling is characterized in general as a reduction of permeate flux through the membrane as a result of increased flow resistance due to pore blockage. This not only shortens the life of the membrane, but also increases the time it takes for microalgae concentration [14-18]. To maintain the economic viability of membrane systems, membrane fouling must therefore be kept to a minimum [15].

We conducted experiments to optimize the concentration of *Tetraselmis* sp. microalgae using a hollow fiber membrane MF system. Two methods of membrane cleaning, forward flushing and backwashing, were first assessed to optimize membrane flux. The effect of different cleaning fluids, seawater or seawater augmented with NaOCl, was also investigated and the frequency of membrane cleaning evaluated by monitoring the average flux to assess the efficiency of cleaning at intervals of 5, 10, 20 and 30 min. Having determined that backwashing was the more effective method of membrane cleaning, we investigated the optimum rate of microalgae concentration and the energy consumption of the different frequencies of cleaning.

MATERIALS AND METHODS

1. Microalgae and Membrane Characteristics

The microalgae used in the experiments was *Tetraselmis* sp. KCTC12236BP obtained from In-ha University and the PF-90M polyvinylidene difluoride (PVDF) hollow fiber membrane with a pore diameter of 0.2 μm obtained from Econity (Econity, Young-in, Korea) was used for the experiments. The MF system was operated at 25 °C (temperature for laboratory). The effective membrane

Table 1. Specifications of the hollow fiber membrane used in the MF

Econity PF - 90M	
Material	PVDF (polyvinylidene fluoride)
Effective membrane area (m^2)	0.07
Pore size (μm)	0.2
Filtration mode	Outside to Inside
Max pressure (bar)	3
Operation temp. (°C)	1~40
pH range	1~10

[†]To whom correspondence should be addressed.
E-mail: jinwonlee@sogang.ac.kr

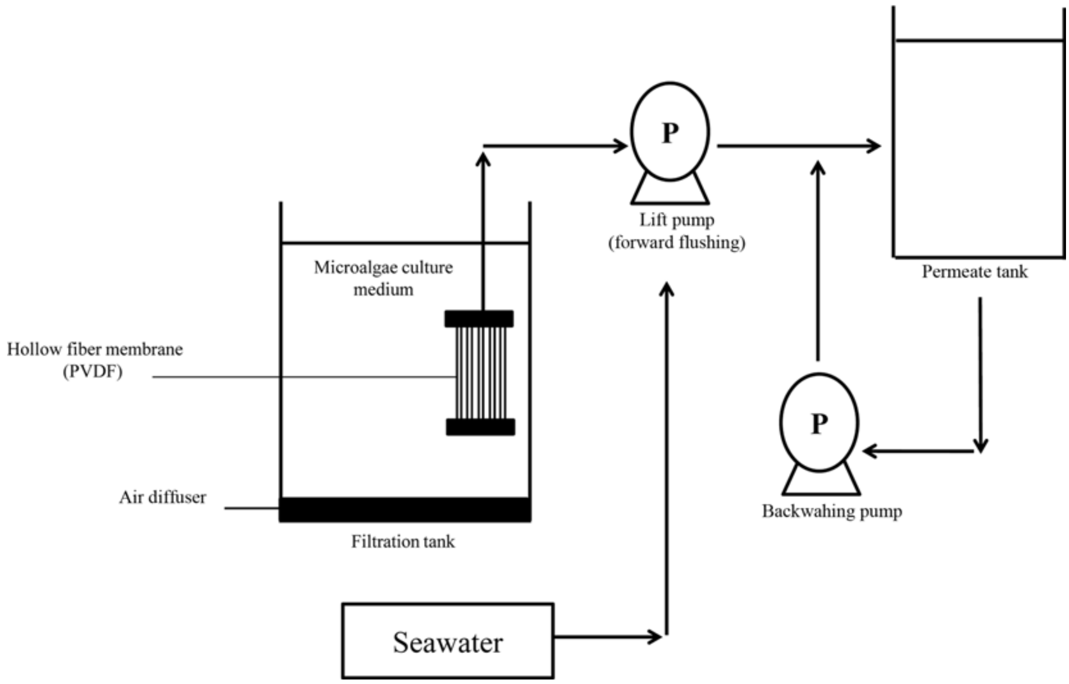

Fig. 1. Schematic diagram the microfiltration system.

area was 0.07 m^2. The membrane physical characteristics are shown in Table 1.

2. Experimental System

A schematic diagram of the MF system is illustrated in Fig. 1. Microalgae culture medium was contained in the filtration tank with hollow fiber membrane module. An air diffuser was used to stir the culture and minimize membrane surface fouling phenomenon. When the hollow fiber filter pores become clogged, they need to be cleaned. Forward flushing and backwashing are the removal methods of a fouling layer of microalgae on the membrane surface through the flow. This, in effect, blasts the clogged particles off the filter. Two peristaltic pumps were used to both maintain the flux and clean the membrane by either forward flushing or backwashing.

3. Method

The purpose of the study was to identify the cleaning conditions which could maximize the process of maintaining high fluxes of microalgae concentration. As a result two cleaning methods were investigated: forward flushing and backwashing. Simultaneously, two cleaning fluids were investigated: seawater or seawater augmented with NaClO (25 ppm) [12]. The forward flushing was started after the membrane module was ejected from the microalgae culture medium tank. When forward flushing was applied in a hollow fiber membrane, microalgae were dropped by flowing on the membrane surface. The cleaning fluids were used for manually conducting forward flushing. In contrast, backwashing experiments were conducted inside of the microalgae culture medium tank. When backwashing was applied in a hollow fiber membrane, microalgae were removed on the membrane surface by reverse flow. The cleaning fluids were used for backwashing by the peristaltic pump. The frequency of membrane cleaning was evaluated with flux recovery percent to assess the efficiency of cleaning at intervals of 5, 10, 20 and 30 min.

To evaluate the effects of each cleaning method, the average flux was determined as follows:

$$F=V/S/T$$

where, F is the average flux ($L/m^2\cdot hr$), S is the effective membrane area (m^2) and T is the time of the concentration process (hr). Further, flux recovery rate (R) in a process is defined as:

$$R=F_{fi}/F_{in} * 100\%$$

where R is flux recovery rate (%), F_{in} is the initial flux rate ($L/m^2\cdot hr$), and F_{fi} is the final flux rate ($L/m^2\cdot hr$).

RESULTS AND DISCUSSION

1. Forward Flushing

The forward flushing experiments were conducted outside of the culture medium tank using two cleaning fluids, seawater or seawater augmented with NaClO, which can facilitate the decomposition of organic substances. The forward flushing was conducted at four different frequencies every 5, 10, 20 or 30 min. The results obtained are represented by the graphs shown in Fig. 2. Among all the four tests using different washing frequencies, the results of flushing every 5 min and 10 min were relatively better especially during the first 30 min. This is because the longer period flushing could not achieve the similar effects since the member fouling increases with time. However, comparison of the various plots indicated that forward flushing with seawater had little effect relative to the data without control, which was operated in the absence of any cleaning, regardless of the frequency of the forward flushing procedure. In addition, Fig. 3 illustrates the forward flushing results utilizing seawater augmented with NaClO. The fluxes were generally increased compared with Fig. 2. This is because the organics adhering to the surface were discomposed by the addition of NaClO. To determine the influence by NaClO addition in seawater, the flux recovery (%) data were

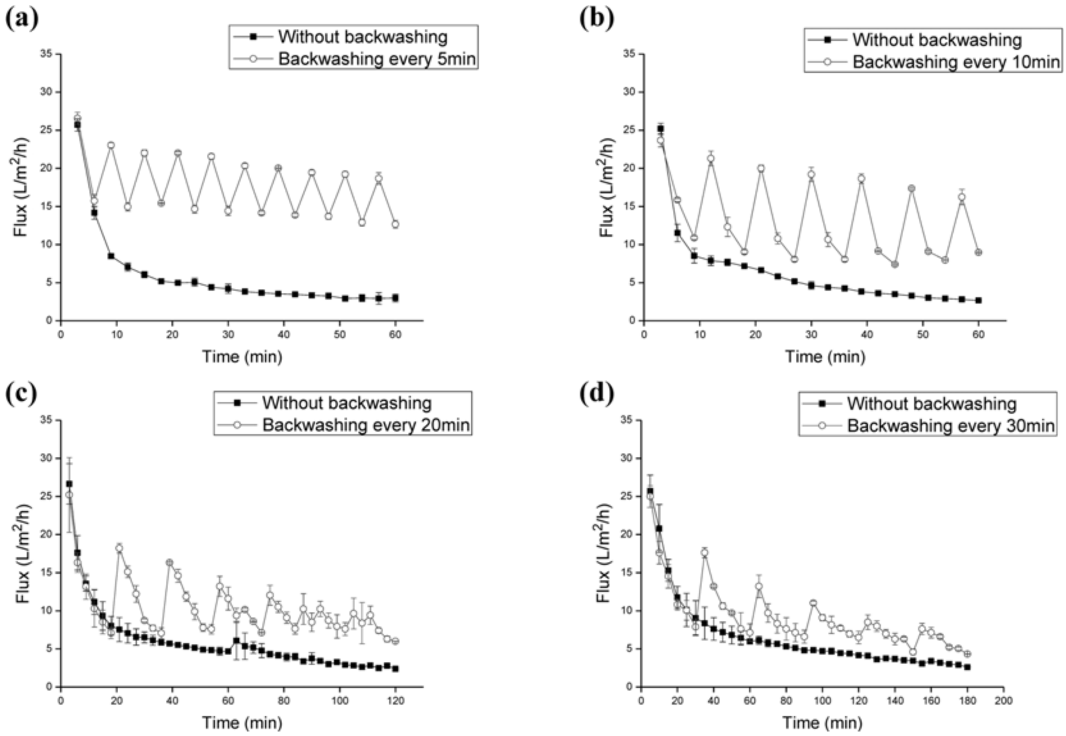

Fig. 2. The rate of membrane flux when forward flushing with seawater was applied at four different frequencies: (a) 5, (b) 10, (c) 20, or (d) 30 min.

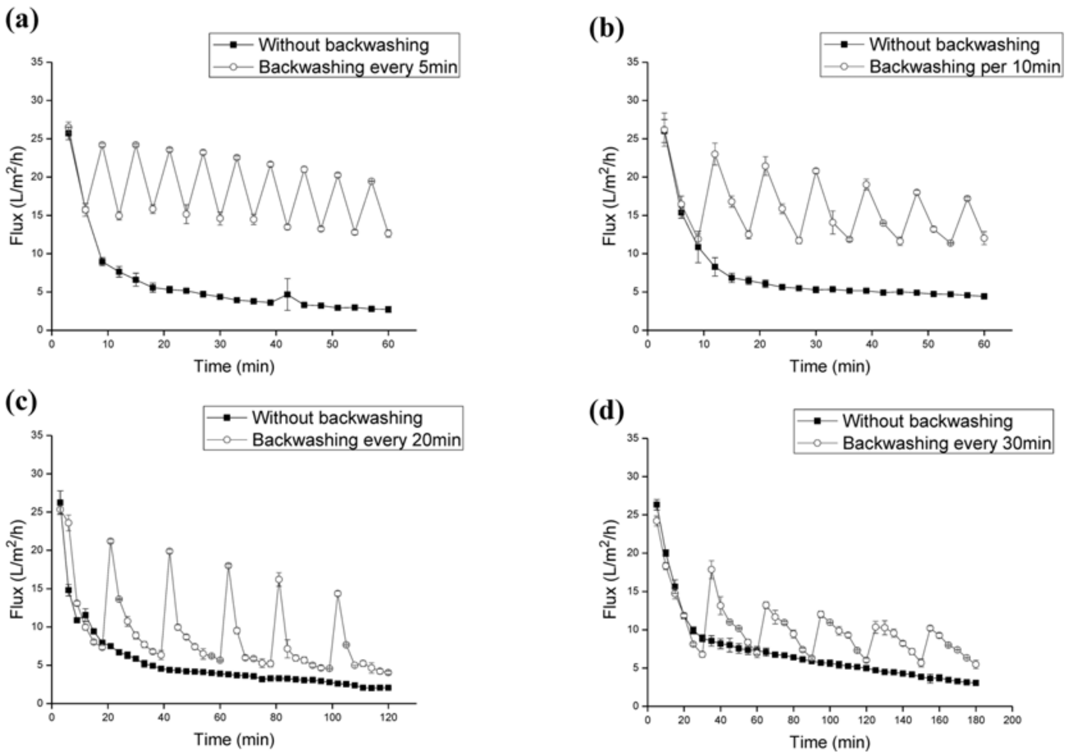

Fig. 3. The rate of membrane flux when forward flushing with seawater augmented with NaClO was applied at four different frequencies: (a) 5, (b) 10, (c) 20, or (d) 30 min.

calculated and shown in Table 2 and Table 3.

Table 2 shows the flux recovery calculated from experiments with seawater only, while Table 3 shows the data with NaClO added seawater experiments. It is clear that the flux recovery levels were higher in Table 2 and the highest recoveries were obtained when the cleaning frequency was set at 5 min for both forward flushing methods.

Table 2. Result of flux recovery percent in forward flushing using seawater

Time (min)	Forward flushing flux recovery (%)			
	Flush every 5 min	Flush every 10 min	Flush every 20 min	Flush every 30 min
6.00	52.81			
11.00	33.3	28.44		
16.00	24.85			
21.00	23.54	23.76	21.14	
26.00	20.81			
31.00	19.25	19.06		16.75
36.00	18.64			
41.00	17.42	16.27	15.32	
46.00	17.02			
51.00	16.74	15.82		
56.00	15.26			
61.00	14.2	13.25	12.36	10.5

Table 3. Result of flux recovery percent in forward flushing using seawater augmented with NaOCl

Time (min)	Forward flushing flux recovery (%)			
	Flush every 5 min	Flush every 10 min	Flush every 20 min	Flush every 30 min
6.00	47.03			
11.00	39.72	34.85		
16.00	28.9			
21.00	26.26	29.89	28.64	
26.00	22.78			
31.00	20.89	20.28		18.6
36.00	20.5			
41.00	18.87	16.91	15.25	
46.00	18.84			
51.00	17.65	14.85		
56.00	17.01			
61.00	16.02	13.25	12.11	10.26

However, since the recovery levels were very low only using forward flushing methods, it is necessary to combine the backwashing techniques together with forward flushing to keep higher flux recovery and simultaneously concentrate microalgae.

2. Backwashing

The results from the backwashing experiments are summarized in Figs. 4 and 5 and Tables 4 and 5. In contrast to forward flushing, backwashing with seawater had a greater effect on membrane flux especially for 5 min and 10 min cleaning frequencies. This effect likely resulted from the cleaning fluid passing through the membrane in reverse and dislodging the microalgae attached to the membrane surface. However, when the rate of backwashing frequency was 20 or 30 min, the effect was less pronounced, and the overall flux decreased rapidly. Compared with Fig. 5(a), (b) and Fig. 4(a), (b), the effects with both backwashing methods were similar for 5 min and 10 min cleaning frequencies. In addition, the seawater augmented with NaClO washing every 20 and 30 min achieved higher fluxes and longer maintenance abilities than those with seawater only experiments also because addition of NaClO made organics decomposition more efficiently to sustain longer effects. Similarly, Table 4 and Table 5 show the flux recoveries for backwashing data. It is obvious that the recoveries decreased with the increase of the

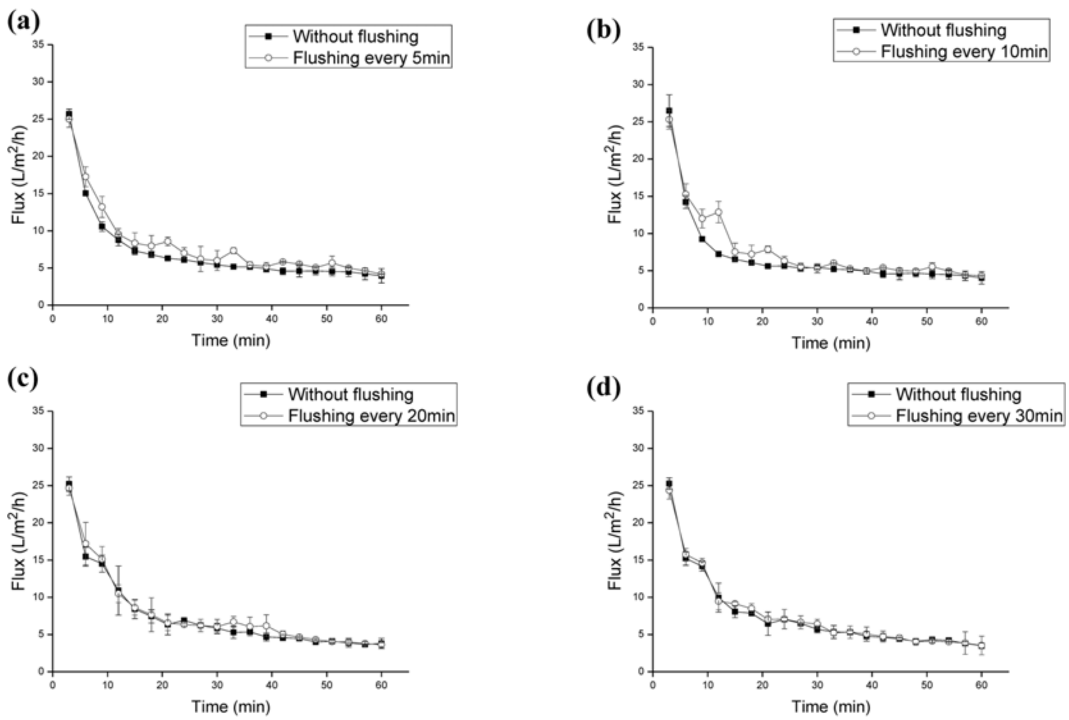

Fig. 4. The rate of membrane flux when backwashing with seawater was applied at four different frequencies: (a) 5, (b) 10, (c) 20, or (d) 30 min.

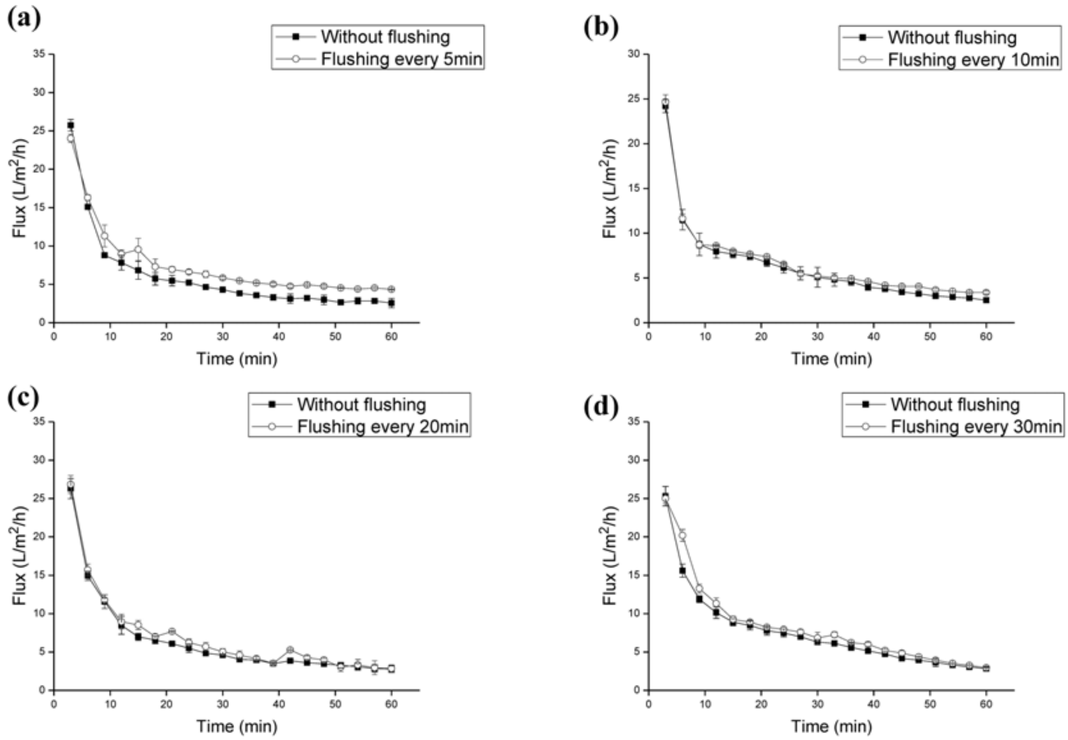

Fig. 5. The rate of membrane flux when backwashing with seawater augmented with NaClO was applied at four different frequencies: (a) 5, (b) 10, (c) 20, or (d) 30 min.

Table 4. Result of flux recovery percent in backwashing using seawater

Time (min)	Backwashing flux recovery (%)			
	Backwash every 5 min	Backwash every 10 min	Backwash every 20 min	Backwash every 30 min
6.00	87.65			
11.00	84.25	82.32		
16.00	82.87			
21.00	81.17	81.07	72.4	
26.00	80.54			
31.00	80.32	78.36		70.2
36.00	78.2			
41.00	76.23	76.52	65.23	
46.00	75.33			
51.00	74.31	72.98		
56.00	73.2			
61.00	72.02	71.02	60.23	52.32

Table 5. Result of flux recovery percent in backwashing using seawater augmented with NaOCl

Time (min)	Backwashing flux recovery (%)			
	Backwash every 5 min	Backwash every 10 min	Backwash every 20 min	Backwash every 30 min
6.00	90.78			
11.00	88.23	86.54		
16.00	86.77			
21.00	86.21	83.63	76.36	
26.00	84.32			
31.00	81.23	80.33		73.65
36.00	80.69			
41.00	78.18	77.25	70.65	
46.00	77.23			
51.00	75.21	73.47		
56.00	74.21			
61.00	74.03	72.89	64.84	60.14

cleaning frequency, and recoveries with augmented NaClO were much higher than that with only seawater. Overall, backwashing was a much more efficient method than forward flushing for maintaining high fluxes. Therefore, backwashing was confirmed as the optimization of cleaning method, and it was selected as the most appropriate cleaning method for the subsequent microalgae concentration and energy consumption experiments.

3. Microalgae Concentration and Energy Usage

The accumulation of the microalgae *Tetraselmis* sp. KCTC12236BP was assessed with different backwashing frequencies--5, 10, 20, 30 min--and the two different cleaning fluids used previously. The percentage of microalgae in the culture was measured at intervals to determine the amount of time required for the culture to reach 90% concentration. Even though the previous experiments had shown that the rate of flux was highest when backwashing was applied most frequently backwashing every 5 min, the optimum frequency for microalgae accumulation was every 10 min, which resulted in a concentration time of 60 min (Fig. 6). This effect could have been caused by the inference of the seawater with the culture medium when backwashing was applied too frequently. If the backwash fre-

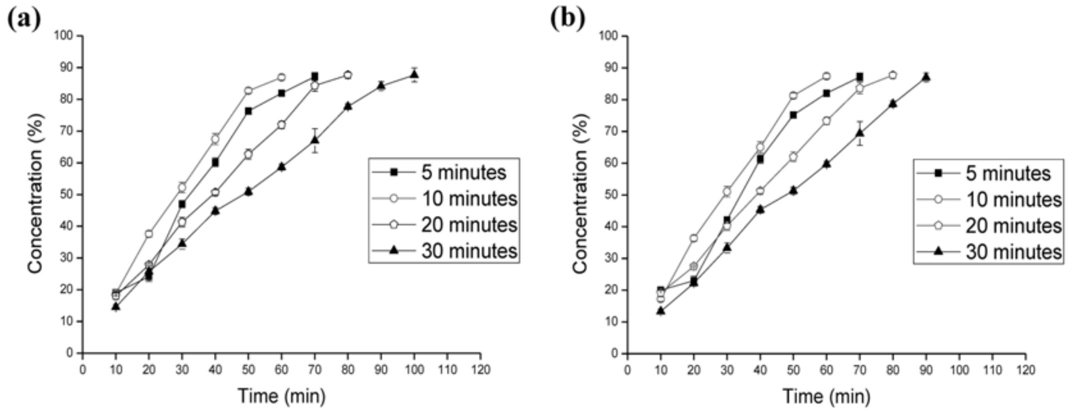

Fig. 6. Time required to achieve 90% concentration of the microalgae *Tetraselmis* sp. KCTC12236BP with different frequencies of backwashing with (a) seawater (b), seawater added NaClO.

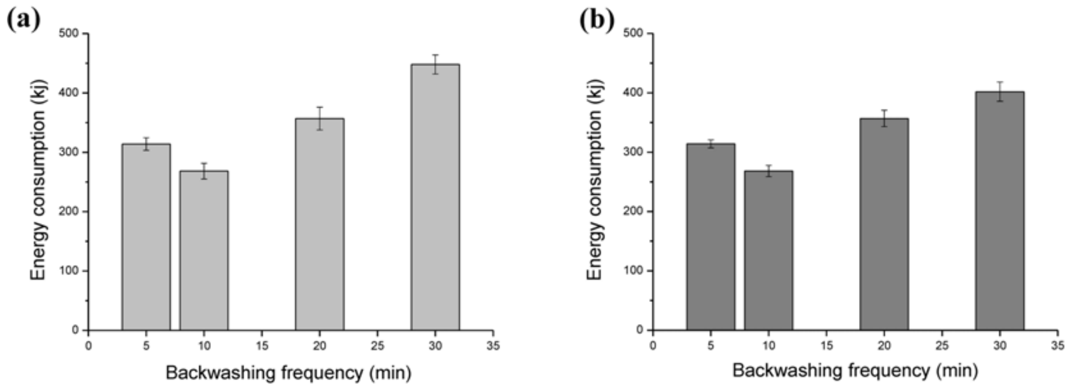

Fig. 7. Energy consumption require to concentrate *Tetraselmis* sp. KCTC12236BP.

quency was too high, the concentration time would be long, which resulted in lower average flux. If the backwash frequency was too low, fouling resistance became higher, which also resulted in a lower average flux.

A similar pattern was observed when the energy consumption of the system was analyzed. Again, the optimum frequency of backwashing was every 10 min rather than every 5 min (Fig. 7). The lower frequencies of cleaning (every 20 or 30 min) resulted in progressively higher energy consumption.

Taken together, these results indicate that although the highest frequency of backwashing (every 5 min) was the most effective at maintaining a high rate of flux, the optimum frequency from the perspective of microalgae accumulation and energy consumption was when backwashing was applied every 10 min. Considering the balance of all these factors, the overall optimum frequency of backwashing for microalgae accumulation was every 10 min.

CONCLUSION

We used a microfiltration (MF) system to concentrate the microalgae *Tetraselmis* sp. KCTC12236BP. The cleaning method of backwashing followed by forward flushing was recommended. Furthermore, higher frequencies of backwashing greatly improved the membrane flux. However, even though the optimum frequency for maintaining membrane flux was every 5 min, from the perspective of microalgae accumulation and energy consumption, better results were obtained when backwashing was applied every 10 min. Because, if the backwash frequency was too high, the concentration time would be long, thus resulting in lower average flux. If the backwash frequency was too low, fouling resistance became higher, which also resulted in a lower average flux. Under these conditions (backwashing every 10 min) the microalgae culture reached 90% concentration in 60 min. Moreover, the seawater augmented with NaClO method resulted in higher flux recovery in all the experiments as well as keeping longer maintenance time. Considering the balance of all these factors, the overall optimum frequency of backwashing for microalgae concentration was every 10 min. The hollow fiber membrane has been proved to be a useful laboratory tool for the concentration of *Tetraselmis* sp. KCTC12236BP by adding backwash.

ACKNOWLEDGEMENT

This research was supported by a grant from Marine Biotechnology Program Funded by Ministry of Oceans and Fisheries, Korea.

REFERENCES

1. M. A. Borowitzka, *J. Appl. Phycol.*, **9**, 393 (1997).
2. Y. Chisti, *Biotechnol. Adv.*, **25**, 294 (2007).
3. A. B. M. S. Hossain and A. Salleh, *Am. J. Biochem. Biotechnol.*, **4**,

250 (2008).
4. L. Brennan and P. Owende, *Renew Sustain Energy Rev.*, **14**, 557 (2010).
5. N. Uduman, Y. Qi, M. K. Danquah, G. M. Forde and A. Hoadley, *J. Renew. Sust Energy*, **2**, 012701 (2010).
6. J. O. Kim, J. T. Jung, I. T. Yeom and G. H. Aoh, *Desalination*, **202**, 361 (2006).
7. N. Rossignol, L. Vandanjon, P. Jaouen and F. Quéméneur, *Aquacult Eng*., **20**, 191 (1999).
8. C. Huang, X. Chen, T. Liu, Z. Yang, Y. Xiao, G. Zeng and X. Sun, *Environ. Sci. Pollut. Res*., **19**, 1416 (2012).
9. T. M. Mata, A. A. Martins and N. S. Caetano, *Renew Sustain Energy Rev.*, **14**, 217 (2010).
10. J. B. Castaing, A. Mass, V. Séchet, N. E. Sabiri, M. Pontié, J. Haure and P. Jaouen, *Deasalination*, **276**, 386 (2011).
11. J. Decarolis, S. Hong and J. Taylor, *J. Membr. Sci.*, **191**, 165 (2001).
12. P. Le-Clech, V. Chen and T. A. G. Fane, *J. Membr. Sci*., **284**, 17 (2006).
13. N. Rossi, M. Derouiniot-Chaplain, P. Jaouen, P. Legentilhomme and I. Petit, *Bioresour. Technol.*, **99**, 6162 (2008).
14. A. L. Lim and R. Bai, *J. Membr. Sci.*, **216**, 279 (2003).
15. H. Liang, W. Gong, J. Chen and G. Li, *Desalination*, **220**, 267 (2008).
16. V. Puspitasari, A. Granville, P. Le-Clech and V. Chen, *Sep. Purif. Technol*., **72**, 301 (2010).
17. P. J. Smith, H. H. Ngo, R. Ben-Aim, H. Nguyen and S. Vigneswaran, *J. Membr. Sci*., **278**, 381 (2006).
18. Y. Xu, J. Dodds and D, Leclerc, *Chem. Eng. J*., **57**, 247 (1995).