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Towards Efficient Membrane Filtration for Microalgae Harvesting: A Review

(Menuju Filtrasi Membran yang Cekap untuk Penuaian Mikroalga: Kajian Semula)

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ABSTRACT

Microalgae contain pigments such as chlorophyll and ß-carotene that are found to be useful as ingredients in human foods. Downstream processing is the key step to obtain the pigments but before that, harvesting process is required. The purpose of harvesting is to separate the microalgae cells from its culture media. There are many harvesting methods including membrane filtration. Besides simple in operation, membrane filtration technology involves no chemicals, no phase changes and offers complete solid retention. However, high flux requirement and reducing operating and maintenance cost of membrane filtration are challenges for microalgae harvesting. Another issue with membrane filtration is membrane fouling. One of the common approaches used currently in anti-fouling harvesting is by modifying the configuration of the filtration system itself by adding auxiliaries such as air scouring and vibration into the filtration systems. Thus, this review covers recent microalgae species that have been harvested by membrane filtration and their efficiency. Chlorella vulgaris, Nannochloropsis oculata and Scenedesmus sp. are among the species that have been harvested using membranes. Techniques that can be applied for tailoring membrane filtration to be a universal harvesting method for all microalgae species are also highlighted. Having this in mind, factors such as membrane formulation and types of membrane making process which play significant roles in determining the efficiency of harvesting were discussed.

Keywords: Microalgae; harvesting; filtration; membrane; efficiency

ABSTRAK

Microalgae mengandungi pigmen seperti klorofil dan ß-karotena yang didapati bermanfaat sebagai bahan makanan manusia. Pemprosesan hiliran adalah langkah utama untuk mendapatkan pigmen tetapi sebelum itu, proses penuaian diperlukan. Tujuan penuaian adalah untuk memisahkan sel-sel mikroalga dari media kulturnya. Terdapat banyak kaedah penuaian termasuk filtrasi membran. Selain mudah beroperasi, teknologi filtrasi membran tidak melibatkan bahan kimia, tiada perubahan fasa dan menawarkan penahanan lengkap pepejal. Walau bagaimanapun, keperluan fluks yang tinggi dan pengurangan kos operasi dan penyelenggaraan filtrasi membran adalah cabaran untuk menuai microalga. Satu lagi masalah dengan filtrasi membran adalah membran tersumbat. Salah satu pendekatan umum yang digunakan saat ini dalam penuaian anti-tersumbat adalah dengan mengubah konfigurasi sistem filtrasi itu sendiri dengan menambah bantuan seperti pengarahan udara dan getaran ke dalam sistem filtrasi. Oleh itu,ulasan ini merangkumi spesies mikroalga terkini yang telah dituai oleh filtrasi membran dan kecekapan mereka. Chlorella vulgaris, Nannochloropsis oculata dan Scenedesmus sp. adalah antara spesies yang telah dituai menggunakan membran. Teknik-teknik yang boleh digunakan untuk menyesuaikan filtrasi membran menjadi kaedah penuaian sesuai untuk semua spesies mikroalga juga ditekankan. Memikirkan ini, faktor-faktor seperti pembentukan membran dan jenis proses pembuatan membran yang memainkan peranan penting dalam menentukan kecekapan penuaian dibincangkan.

Kata kunci: Microalga; penuaian; filtrasi; membran; kecekapan

INTRODUCTION

Microalgae are eukaryotic unicellular organisms that can be found in saline or freshwater bodies. There are numerous microalgae species around the world but only a handful such as *Scenedesmus*, *Chlorella*, *Heamatocuccus* and *Nannochloropis* algae are known for their useful products. *Nannochloropsis* is known in biodiesel making because of its high percentage of triglyceride yield in relation to overall lipid content (Brennan and Owende, 2010). Previously, animal fats and vegetable oils were used for biodiesel production but they are not practical due to food competitor issues and large area requirement. Besides that, microalgae such as *Dunaliella* sp. *Chlorella* sp. and *Scenedesmus* sp. contain various pigments molecules like β-carotene, chlorophyll and carotenoids that have been used as colorants in cosmetic and food (Shah et al. 2016) for a long time. Meanwhile, *Haematococcus pluvialis* contains astaxanthin. Astaxanthin is used in cosmetics products, food supplements and pharmaceutical industries because of its free radical scavenging capacity and powerful antioxidant activity (Khanra et al. 2018). Since microalgae holds economic value in many industrial applications hence, they are sometimes cultivated indoor.

Harvesting of cultivated microalgae is necessary so as, to obtain their biomass before further processing them into valuable products. There are many methods of microalgae harvesting but centrifugation is commonly used for lab and pilot scale production. Centrifugation applies high rotational and shear forces to separate microalgae and consumes huge amounts of energy if it was used for vast production. Normally, centrifuge is adjusted to maximize capture efficiency where the energy is consumed. According to Barros et al. (2015), high solid capture of 94% consumed 20 kWh of energy and 17% of solid capture consumed only 0.80 kWh but obviously is less efficient. Meanwhile, belt filter system can be used for up-scale harvesting. A belt-filter system separation is based on gravity drainage followed by compression of filtered material. However, belt filter system is only suitable for high concentration algae culture. A study reveals that a belt filter system can recover microalgae suspension with minimum concentration is 6 g dry wt/L because when 4 g dry wt/L of microalgal suspension was used, the percent of microalgae recovered dropped significantly due to leakage in the filter section (Sandip, Smith & Faddis 2015).

Membrane filtration is another type of harvesting method. The concept of membrane filtration separation is molecular sieving through membrane pores which can be divided into nanofiltration, reverse osmosis, microfiltration (MF) and ultrafiltration (UF). Since ages, membrane filtration has emerged as a promising tool for separation process especially in water treatment (Teow et al. 2017) and water desalination (Lee, Dayou & Karunakaran 2018). This is because it is easy in operation, requires only low operating pressure and temperature and does not require any chemical addition. A desirable membrane filtration process is one with high selectivity and flux and possesses good antifouling properties. Hence, the efficiency of membranes is always determined based on their flux, the percentage of microalgae rejection, concentration factor and volume reduction factor. Moreover, membrane technology offers the possibility of managing the membrane. This paper reviews on recent approaches in microalgae harvesting using membrane filtration and its efficiency. *Chlorella vulgaris*, *Nannochloropsis oculata* and *Scenedesmus* sp. are among the species that have been harvested using membranes. Moreover, techniques that can be applied for tailoring membrane filtration to be a universal harvesting method for all microalgae species are discussed in future prospect section.

ISSUES

The function of membrane filtration for microalgae harvesting is limited by many factors. Membrane filtration always suffers from low flux especially at high biomass concentration which is time consuming rendering its practicality. Compared to other methods, centrifuge offers high recovery rate and can produce high concentrated algae. Therefore several studies have suggested to combine membrane filtration as preliminary separation before centrifugation for microalgae harvesting (Baerdemaeker et al. 2013; Monte et al. 2018; Bilad et al. 2012). Another factor that has an adverse effect to membrane flux is fouling. The main foulants in microalgae harvesting are the algae cell itself, algae debris and extracellular polymeric substances (EPS). Algae cells and debris have been said to cause more fouling than EPS (Liu et al. 2017). Therefore, higher biomass concentration always causes faster algae cake builds up and rapid flux decline (Chen, Huang and Liu 2012). High shear stress and velocity especially in the cross-flow filtration produced cell debris (Monte et al. 2018). Cell debris that is weakly deposited on the membrane surface can be washed away easily (Hilal et al. 2007). However, cell debris that have sizes very close to that of the membrane pore size will plug into the pore passage (Bilad et al. 2014). EPS are secreted in the form of high molecular weight polymer. It is the major component that helps to maintain the characteristic and properties of the microalgae. The main components in EPS are carbohydrate, proteins, humic substances and nucleic acids. Many factors can influence the release of EPS into the water such as culture process, growth phase and the extraction method (Sheng et al. 2010). Additionally, water with high salinity secreted more EPS (Mishra and Jha 2009). EPS varies in composition, thus results in complex fouling reaction (Lin et al. 2014).

Table 1 depicts a summary of studies relating to microalgae fouling. Most of the findings in Table 1 reveals that algae cake build-up leads to fouling and there are many factors that can influence the rate of fouling (Baerdemaeker et al. 2013). Microalgae concentrations, type of membrane materials, pore size of the membrane and the filtration operating conditions are amongst factors that influence the fouling rate. Fouling causes flux decline but one can determine the threshold of sustainable flux through critical flux measurement whereby at flux values above the critical flux value, fouling will become prominent. Therefore, to avoid fouling occurring operation at sub-critical flux or near critical flux is important (Wicaksana et al. 2012). The membrane pore size and size of microalgae are also factors to be considered; a membrane with average 0.1 μm pores showed the highest average flux with negligible pore fouling over the various range of biomass concentrations when the algae used have the size range from 2-6 μm. In such cases there is no advantage in using a small pore size (less than

TABLE 1. Recent studies of membrane filtration for microalgae harvesting

Focus	Species	Material	Finding	References
Role of algae cake deposition in fouling	*Chlorella* sp.	Cellulose ester, Polyvinylidene difluoride	Algae cake deposition was independent of the membrane material and it was compressible with a compressibility index of 0.439	Babel and Takizawa, 2010
Examining the role of different type of resistance in membrane flux	*Chlorella* sp.	Cellulose acetate	Cake resistance was the major cause of flux reduction compared to resistance due to concentration polarization and pore blocking	Ahmad et al. 2012
Importance of hybrid integrating harvesting system to endure fouling issue	*Nannochloropsis* sp.	Polyvinylidene fluoride	A membrane with average 0.1 μm pores showed the highest average flux with negligible pore fouling over the various range of biomass concentrations. Since, the algae used have the size range from 2-6 μm there is no advantage in using a small pore size membrane (less than 0.1 μm)	Bhave et al. 2012
Investigating the effect of air ventilation into algae broth and backwashed for fouling control	*Scenedesmus* sp.	Polyvinylidene Fluoride	Periodically air ventilation that gives algae solution aerated and backwashing has reduced the membrane fouling to some extent but somehow it did not helped in increasing the membrane recovery ratio	Chen et al. 2012
Developing Carman–Kozeny (C–K) model to predict flux decline	*Chlorella* sp.	Cellulose acetate	The Carmen-Kozeny model result was in good agreement with result obtained from the experiment and can be used to predict flux decline. It was also found that the mean flux can be determined by the model in a condition where the transmembrane pressure (TMP) is between 1-1.5 bar	Ahmad et al. 2013
Effect of polymer, algae cell concentration and species in fouling	*Nannochloropsis oculata, Phaeodactylum Tricornutum, Isochrysis* sp., *Chlorella vulgaris, Pavlova lutheri*	Polyvinyl chloride, Polyethersulfone,- Polyvinylpyrollidone, Polyvinylidene fluoride	The membrane critical flux was depends on microalgae species and concentration, membrane material, pore size and operating conditions. The use of backwashing has helped to improved membrane flux	Baerdemaeker et al. 2013

(*continue*)

Table 1. (*cont'd*)

Focus	Species	Material	Finding	References
Investigating vibrated and aerated membrane system for fouling control	*Chlorella vulgaris*	Polyvinylidene fluoride	Vibration system offers better anti-fouling compared to aerated system due to direct impact on the membrane body that cannot be achieve by aeration. However, both systems were unable to prevent severe pore blocking in the early stage of filtrations	Bilad et al. 2014
Coagulant assisted filtration to reduce fouling	*Chlorella vulgaris*	Polyvinylidene fluoride	The used of coagulation method in microalgae harvesting has made possible for the use of highly porous membrane	Discart et al. 2015
Effect of temperature on EPS secretion and membrane fouling	*Chlorella pyrenoidosa*	Polyvinylidene fluoride	High temperature had increased the membrane flux and lowered the membrane fouling due to the decline in algae solution viscosity. Moreover, EPS was unlikely to release at high temperature particularly when temperature increase from 15°C to 35°C	Chu et al. 2016
Effect of pore sizes on microalgae harvesting using F_D formula	*Chlorella pyrenoidosa*	Polyvinylidene fluoride	The reason > 0.1 µm pore sized membranes are prone to fouling because of increase in permeate drag force (F_D). F_D is the function of membrane resistance. Increase in membrane resistance lead to increase in F_D that accelerated the deposition of particles on the membrane and increase the membrane fouling rate	Zhao et al. 2017

TABLE 2. Performance of UF/MF membranes for microalgae harvesting

Author	Membrane	Velocity (m/s)	TMP (bar)	Filtration flux (L/m²h²)	Harvesting efficiency	Auxiliary used
Ahmad et al. 2012	MF	4.0	1.5	≈ 130	N/A	–
Castaing et al. 2010	MF	N/A	0.3	29 (critical flux)	99%	Air blower
Rossignol et al. 1999	UF	2.5	0.5	40	N/A	–
Hwang and Wu 2015	MF	1	2	≈ 105	100%	Rotating disk
Hwang et al. 2015	UF	1	2-3	96	100%	–
Castaing et al. 2011	MF	N/A	0.3	108	99%	Air blower
Pavez et al. 2015	UF	1.5-2	N/A	≈ 60 (critical flux)	N/A	–
Sun et al. 2013	UF	5.79	1.8	≈ 120	N/A	–
Frappart et al. 2011	UF	1	1	>100	100%	Rotating disk
Bilad et al. 2013	MF	N/A	N/A	>50	100%	Vibrating machine

Note: N/A= information not available

0.1 µm) membrane as this can reduce flux drastically (Bhave et al. 2012). In other studies (Chen et al. 2012; Bilad et al. 2014) fouling can be controlled using air bubble scouring and vibration. These techniques are further discussed in the next section.

CURRENT APPROACH

One of the common approaches used currently in anti-fouling harvesting is by modifying the configuration of the filtration system itself. As mentioned in Table 2, adding auxiliaries such as air scouring and vibration into the filtration systems has been an option. First of all, UF and MF systems require low operating pressure. Therefore most of UF/MF systems in microalgae harvesting were operated at low TMP (Table 2). The highest TMP is only at 3 bars while the highest crossflow velocity is at 4.0 ms^{-1}.This is one of the advantage when using UF and MF membranes because a very high TMP and velocity can cause severe fouling. High velocity and TMPare attained via pumping through a highly restrictive valve. Strong pumping induced high shear on microalgae. Shear is responsible for broken cells and release of microalgae products. The sheared algae can cause more drastic flux decline than non-sheared microalgae (Ladner et al. 2010). Nevertheless according to Ahmad et al. (2012) increase of flow velocity is better than increase of TMP as the result obtained shows that a decrease in the TMP and increase in velocity has decreased cake layer formation. Overall, anti-fouling microalgae harvesting involves filtration system that is similar to most of filtration system used in other separation process with advance technology.

Some of the results listed in Table 2 are obtained using the modified system. Castaing et al. (2010) applied hollow fiber submerged filtration system with aeration effect for *H. triquetra* harvesting. A blower was set at the bottom of the hollow fiber membrane to generate bubbles and it was found to slow down the fouling occurrence. Critical flux achieved for the harvesting was 29 L/h.m^2 after 180 min of filtration under 0.3 bar TMP. Bilad et al. (2013) performed a flat sheet submerged filtration for harvesting *Phaeodactylum tricornutum* and *Chlorella vulgaris*. The filtration system was equipped with vibrator machine. The vibrations generated from the vibrator machine were from magnetic repulsion. The vibration was only subjected to the area of the membrane. The critical flux achieved was slightly higher than achieved by Castaing et al. (2010) which was above 50 L/h.m^2. Thus, it was proven that the vibrated system is better than aerated system in order to diminish fouling effect. Even Nurra et al. (2014) demonstrated microalgae harvesting using vibrated filtration system in pilot scale. A pilot plant with six photo bioreactors with a total capacity of 53,000 L was developed for cultivation, harvesting, cell disruption and lipid extraction. The harvesting process was performed using a membrane vibrating set-up from New Logic Research Inc., model id VSEP Series LP. Results from membrane filtration achieved microalgae filtration at 28.5 L/h/m^2/bar using a polyethersulfone with a molecular weight cut-off of 7000 Da. To support microalgae biomass demand, centrifugation was used in parallel with membrane filtration to harvest microalgae. In the centrifugation process a total of 28,100 L was treated in 11 batches. Each batch had duration of 3 hours approximately at a recirculating flow rate of 1000 L/h. The total concentrated volume obtained was 20.3 L and the total dry biomass obtained was 2.640 kg.

Additional, Kim et al. (2014) used cross-flow electro-filtration system as a step to anti-fouling harvesting. A platinum plate has been placed on the opposite side of the electro-membrane with 5 mm distance to cause water electrolysis during filtration and served as the counter anode. The electro-membrane used had caused electrical repulsion between the membrane surface and microalgae cell and fouling decreased which was indicated by the high concentration factor achieved. Kim et al. (2015) and Hwang and Wu (2015) performed microalgae harvesting using a cross-flow cell equipped with rotating disk. The rotation from the rotating disk can generate sheer stress on membrane surface to mitigate the algae fouling. However, these systems are expensive due to current energy and limiting space in the rotating disk system.

FUTURE PROSPECT

Most of the techniques used for efficient microalgae harvesting were focused on configuration improvement. However, other technique that can be considered in the future is to utilize hydrophilic membrane during microalgae filtration. Generally, hydrophilic membranes offer better fouling resistance since many foulants are hydrophobic in nature. It has been known that a hydrophilic membrane is compulsory in water or wastewater filtration. Table 3 shows several researches on hydrophilic membrane applications that have been used to filter protein and humic acid. Proteins and humic acids are among components in EPS. Table 3 also depicts that Hwang et al. (2015) is the only study that utilizes a hydrophilic membrane to dewater microalgae besides us. In the study, PVDF membranes were embedded with hydrophilic PEGylated polymeric particles and were used to filter *Chlorella* sp. and results revealed that the hydrophilic membranes indeed have anti-foulant properties depicted by the high flux recovery ratio (FRR) of 100%.

In a recent study (Khairuddin, Idris and Hock 2019), antifouling membranes were fabricated for microalgae harvesting purpose. The antifouling membranes were fabricated using polyethersulfone (PES) as the base polymer, multiwall carbon nanotubes (MWCNT) and lithium bromide (LiBr) as additives. Findings revealed that membrane fabricated with 1wt% MWCNT, 5wt% LiBr and 18 wt% PES via thermal induced phase separation (TIPS) process possessed an excellent filtration performance and anti-fouling effect. 28 g/l *Nannochloropsis* sp. has been fully retained using the fabricated membrane with average flux 28.9 L/m^{2}h.bar. Furthermore, the membrane demonstrated

TABLE 3. Application of hydrophillic membranes to improve flux and antifouling properties

Targeted foulant	Hydrophilic membrane	Filtration type	Contact angle (°)	Filtration flux (L/m^2h)	Flux recovery ratio (%)	References
Protein	Sulfonated polyphenylsulfone blend with nanoparticle titanium oxide (SPPSu/TiO_2)	UF	67	60	85	Arockiasamy et al. 2017
	Crosslinking calcium alginate/polyacrylamide (CA/PAM) on nanofiltration membrane	UF	N/A	≈23	85.84	Zhang et al. 2015
	PES blend with mesoporous carbon nanoparticles (PES-MCN)	UF	50	25	66.5	Orooji et al. 2017
	Polyvinyl chloride blend with zinc oxide (PVC/ZnO)	UF	54.5	≈140	92	Rabiee et al. 2015
	Poly (ether imide) blend with N-phthaloyl chitosan (PEI/NPHCs)	UF	72.7	70.2	88.6	Kanagaraj et al. 2015
Humic acid	Polysulfone blend with zinc oxide and graphene oxide (PSf/ZnO-GO)	UF	39.6	≈43	>90	Chung et al. 2017
	Polysulfone blend with titanium dioxide and graphene oxide (PSf/TiO_2/GO)	UF	50.7	85	97.0	Kumar et al. 2016
Proteins, polysacch-arides and microotganism	PES blend with amphiphilic block copolymers	UF	> 30	≈110	<90	Zhao et al. 2014
Chlorella microalgae	PVDF membranes embedded with PEGylated polymeric particles	UF	55	96	100	Hwang et al. 2015
Nannochloropis sp. microalgae	PES blended with LiCl and fuctionalized MWCNT	UF	33.8	28.9	100	Khairuddin et al. 2019

excellent anti-fouling effect owing to its higher membrane hydrophilicity indicated by the low contact angle value of 33.8°. Durability study performed exhibited 100% flux recovery rate after rinsing with tap water. The membrane demonstrated no fouling detrimental effects even after five cycles via consistent flux (Khairuddin, Idris & Hock 2019).

Most membranes are made via non-solvent induced phase separation (NIPS). It is the process by which a polymer solution inverts into a swollen three dimensional macromolecular network or gel comprising of a polymer rich phase and this phase will solidify to form a solid membrane structure whilst the liquid polymer lean phase forms the voids (Bonyadi, Chung and Krantz 2007). There is also another method called thermally induced phase separation (TIPS). TIPS method is where a homogenous solution is prepared at high temperature and then is cooled rapidly by immersion in the coagulation water bath at room temperature where the membrane is formed. Preparation of membranes from TIPS methods were found more significant in pore size distribution, permeation and mechanical strength compared to NIPS (Jung et al. 2016). In the past few years, reports on membrane fabrications via TIPS were more concentrated on the mechanism and membrane morphologies rather than TIPS effect on the membrane performance (Mannella et al. 2015;Wu et al. 2017;Liu et al. 2017b).

Therefore, it might be worth to study TIPS membrane application in microalgae harvesting. Previously, Venault et al. (2016) have applied vapour-induced separation (VIPS) membrane for microalgae harvesting. They managed to obtain an anti-biofouling membrane with a FRR of 76.91% but the membrane permeability was enhanced by the hydrophilic effect from additive used. Normally, membrane porosity has more impact on permeability compare to hydrophilic effect. Unlike TIPS, VIPS caused non-crystallized membrane or membrane with interconnected layer structure (Carretier et al. 2016). This is because VIPS process is based on water vapor exposure to form the membrane. During VIPS, solvent inside polymer solution evaporated slowly to the air while water from humid air diffused faster into the polymer film. Slow exchange between air and polymer produced membrane that is dense in structure (Venault et al. 2016). Thus, here are some advantages of applying TIPS method:

1. It is possible to used high amount of additives in polymer solution but achieve porous membrane with TIPS method. Normally, increase in polymer components lead to high viscosity which subsequently lowers the membrane porosity. However as reported by many (Tavajohi et al. 2014; Wu et al. 2013) even though the polymer concentration had increased, water permeation achieved was still promising.
2. TIPS method works well with polymer solution containing nanoparticle additive. Li et al. (2016) employed TIPS method to synthesize PVDF membrane with silicone oxide and graphene oxide nanoparticles. The temperature did not change the function of nanoparticles and the nanoparticles were found uniformly dispersed.
3. TIPS method can also be used to fabricate hollow fiber (HF) membrane. Jeon et al. (2018) fabricated their HF membranes using polymer solution which was mixed in a vessel that was heated at high temperature under a nitrogen atmosphere. The solution was kept stirred but at a lower speed and was fed to a spinneret by a gear pump under a nitrogen atmosphere. The hollow fiber was then extruded from the spinneret and wound on a take-up winder and quenched through coagulation bath at room temperature.
4. TIPS process does not require extreme temperature. According to a literature (Xu et al. 2015), too high casting temperature (above solvent boiling temperature) did not further improve membrane flux. This is because part of solvent was evaporated which ultimately causes the polymer to move upwards to the top layers thus creating very large voids in the sub layer but dense structure at the top layer.

CONCLUSION

Membrane filtration is a promising harvesting tool for microalgae that allows 100% of microalgae retention. In most cases, fouling and low flux inhibit the use of membrane filtration. These issues have led several researchers to focus on enhancing filtration via several methods such as through configuration improvement and the use of hydrophilic membranes. However, most of the work involved configuration improvement were successful in reducing fouling but not in putting membrane filtration as the reliable harvesting method. Thus, the use of membrane with more advance hydrophilic materials is expected to become more common in the future for microalgae harvesting application. The possibility of using hydrophilic membranes with suitable morphology, stability, and permeation properties can be obtained from TIPS process.

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