Energy efficient harvesting of *Spirulina* sp. from the growth medium using a tilted panel membrane filtration

Insyirah Ismail [a], Kiki Adi Kurnia [b], Shafirah Samsuri [a], Muhammad Roil Bilad [a,c,*], Lisendra Marbelia [d], Noor Maizura Ismail [e], Asim Laeeq Khan [f], Arief Budiman [b], Susilawati Susilawati [g]

[a] *Department of Chemical Engineering, Universiti Teknologi PETRONAS, 32610 Bandar Seri Iskandar, Perak, Malaysia*
[b] *Department of Marine, Faculty of Fisheries and Marine, Universitas Airlangga, Jalan Mulyorejo Kampus C, Surabaya 60115, Indonesia*
[c] *Department of Chemistry Education, Universitas Pendidikan Mandalika (UNDIKMA), Jl. Pemuda No. 59A, Mataram 83126, Indonesia*
[d] *Department of Chemical Engineering, Faculty of Engineering, Universitas Gadjah Mada, Jl. Grafika No. 2, Yogyakarta 55281, Indonesia*
[e] *Faculty of Engineering, Universiti Malaysia Sabah, Jln UMS, Kota Kinabalu 88400, Sabah, Malaysia*
[f] *Department of Chemical Engineering, COMSATS University Islamabad, Islamabad, Pakistan*
[g] *Master of Science Education Program, University of Mataram, Jl. Majapahit No. 62, Mataram 83125, Indonesia*

ARTICLE INFO

ABSTRACT

Membrane fouling is one of the main drawbacks in membrane-based microalgae harvesting. This study assessed the tilted panel to enhance filtration performance of *Spirulina* sp. broth. The influences of the operating parameters including the tilting angle, aeration rate and membrane materials on filtration performance and energy consumption were evaluated. Results showed that the system was effective and energy-efficient for membrane fouling control. The permeability peaked at a tilting of 45° thanks to combination of aeration and panel tilting. The microfiltration performed better than the ultrafiltration membrane due to the effective impact of air bubbles for foulant scouring that maximized the membrane intrinsic property. Small aeration rate of 1.0 L/min offered a high plateau permeability of 540 L/(m^2·hr·bar) in which reversible fouling almost fully absent. The high permeability could be achieved under a low energy input of 0.2 kWh/m^3.

1. Introduction

Spirulina sp.-based bioproducts are rich in bioactive compounds such as protein, vitamins, beta-carotene, and minerals which make it suitable to be utilized extensively in food and beverage productions as well as pharmaceuticals (Silva et al., 2019). Nature derivative products produced through green processing are also more preferred by consumers (Tang et al., 2020). Chemically synthesized products often contain a minor fraction of by-products that impose long-term health issues such as hyperactivity, cancer development, and allergic reaction (Vaz et al., 2016). The sulfated polysaccharide in *Spirulina* sp. is commercialized into tablets, pills for food supplements as it can prevent infection from numerous viruses (HIV-1, herpes virus and cytomegalovirus) and can delay the growth of cancerous cells (Andrade, 2018). These compounds are safe for direct consumption to humans and harmless to the environment.

In spite of its vast potentials, microalgae are known to have a density similar to water and the cell possesses small size which complicates the harvesting process (Bilad et al., 2014a). The *Spirulina* sp. biomass must be harvested prior to further processing. The available options for biomass harvesting methods include coagulation/flocculation, centrifugation as well as membrane filtration. The centrifugation method is energy intensive. On the contrary, membrane filtration is low in energy foot-print and usage of chemicals, offers almost full biomass recovery, and is increasingly more cost-effective (Ríos et al., 2012). Thus, the purity of biomass is guaranteed unlike the excess ions from coagulant and flocculant in the coagulation/flocculation process (Bilad et al., 2012). This way, application of membrane technology for *Spirulina* sp. biomass can enhance the economic feasibility of *Spirulina* sp.-based products.

However, membrane fouling strictly limits filtration of a microalgae broth (Bilad et al., 2012). The severity of fouling varies between micro- and ultra-filtration membranes due to the difference in mass-transfer resistance (Lau et al., 2020). Several types of fouling such as cake

---

layer formation, pore blocking and internal fouling could occur during microalgae harvesting process (Bilad et al., 2014a). The cake layer is the major culprit of membrane fouling, which contributes up to 80% of the filtration resistance (Lee et al., 2001). Therefore, many studies have tried to address the cake layer fouling issue. They include enhancing the operational conditions, conducting cleaning cycles, applying dynamic membrane filtration (DMF) system and air bubbling system (by inducing shear rates) (Eliseus et al., 2017). The cleaning cycles comprise of physical and chemical cleanings, but the latter is known to shorten the membrane lifespan. Besides, the DMF system applied by either vibrating or rotating the membrane poses challenge of high complexity for scaling up (Bilad et al., 2014a).

Air bubbling is one of the proven methods to manage cake layer fouling (Eliseus et al., 2017). In the air bubbling system, the foulant is scoured-off by the air bubbles without damaging the membrane (Hwang et al., 2015). The hydrodynamic forces exerted by the air bubbles onto the membrane surface help to limit the concentration polarization, which in turn reduces the fouling rate (Tian et al., 2010).

Despite of the aforementioned advantages, the conventional air bubbling system in the typical vertically aligned plate-and-frame module has few limitations (Eliseus et al., 2017; Hwang et al., 2015). In the vertical panel, the contact of air bubbles towards the membrane surface is low since the bubbles travel in the center of the two adjacent panels, not on the membrane surface (Eliseus et al., 2017). On the other hand, the horizontal panel requires a large footprint due to difficulties in large-scale module alignment which led (Eliseus et al., 2017) to introduce a tilted panel system. For the inclined tubular membrane, the membrane permeability increases by inclining the module up to 45° in an organic tubular membrane module, beyond which it declines (Cheng, 2002). Meanwhile, a pioneer study using polyvinylidene difluoride (PVDF) membrane in a tilted panel was only done for the tilting angle of up to 20° because at that angle the permeability already reaches the plateau value (Eliseus et al., 2017). So far, no study was conducted on the effect of permeability for the tilted panel system beyond tilting angle of 20° despite a model showed the optimum performance at tilting angle of 45° and no report available on the application for such system for Spirulina sp. feeds. In addition, the study on Spirulina sp. feeds using different membrane materials is still lacking; it is best to extend the tilting angle since to identify the optimum value experimentally.

This study evaluates the filtration performance of PVDF and polysulfone (PSF) membranes in a tilted panel system, evaluated at 0, 20, 45 and 70° of tilting angles for harvesting Spirulina sp. The permeability enhancement was assessed under several operating parameters, namely aeration rate and Spirulina sp. biomass concentration. Lastly, the energy estimation and comparison with other studies were also systematically discussed.

2. Materials and methods

2.1. Preparation and characterization of membrane

The PVDF and PSF membranes were prepared in house by using the phase inversion method with the addition of 1% (w/w) polyethylene glycol (PEG, 20 kDa, Sigma-Aldrich). The dimethylacetamide (DMAC, Sigma-Aldrich) as the solvent was mixed with the polymer at concentration of 15% to form a polymer solution. The bubble-free and homogeneous polymer solution was hand casted at average speed of 2 cm/s at room temperature of 24 °C and air humidity of 70% on a non-woven fabric (Novatexx 24413, Freudenberg-filter, Germany) to avoid shrinkage, followed by immersion in a bath containing water (acting as the nonsolvent) at temperature of 24 °C. The membrane sheet was formed in the coagulation bath, in which the completion of the phase inversion was identified as the point when the sheet floated from the plate. The resulting membrane was then washed using running tap water for 5 min to remove the residual solvent and the membrane was stored wet in water until further use. The effective membrane area for each

panel was 0.014 m^2 (10 cm × 14 cm). The PVDF and PSF based membranes were selected because they are mostly reported in literature for microalgae harvesting. The former represents the microfiltration type, while the latter represents the ultrafiltration type.

The morphology of each membrane was analyzed by using scanning electron microscopy (SEM, ZEISS). The electron digital micrometer screw gauge was used to measure the thickness of the membrane, whereas the capillary flow porometer (Porolux) was used to determine the pore size and distribution. The contact angle of the membrane surface was measured by using the sessile drop method with a drop size of 1 μL.

2.2. Microalgae broth preparation

The inoculum of Spirulina sp. was collected from a full-scale open pond system, then cultivated in a 15-L reactor using Walne's medium (Azma et al., 2011). The pH value of the medium was adjusted to 8. Aeration at a rate of 1.5 L/min was provided as the source of inorganic carbon, while the light as source of energy was provided from 18 Watt Neon lamp continuously. The microalgae broth reached the stationary growth phase after 10 days of cultivation, obtaining biomass concentration of approximately 1.2 g/L. All the filtration tests used the same feed concentration, except for the one for evaluating the impact of Spirulina sp. biomass concentrations (2.2–2.5 g/L). The same microalgae broth could be used for evaluation of one parameter (i.e., effect of tilting angle) as Spirulina sp. has a long stationary phase for about 6 days (de Jesus et al., 2018). Another cultivation was done for evaluating the impact of other parameters.

2.3. Experimental set-up

The filtration performance of microalgae was investigated in a constant-pressure mode using a tilted panel system illustrated in Fig. 1. The air pump was used to supply air bubbles from underneath the membrane panel through a diffuser, as such the air bubbles scour-off the foulant during the filtration. The aeration rate was set constant and was provided continuously without idle phase in the range of 0.5–1.5 L/min (depending on the testes parameter). A vacuum air pump was utilized to create a constant vacuum pressure of 0.1 bar. The filtration was done in 9/1 min cycles of filtration and relaxation. During the relaxation period, the air pump was switched off but the membrane aeration was maintained. The volume of permeate was measured during the relaxation period by using a measuring cylinder. After volume measurement, the permeate was returned into the feed tank to ensure a constant level and condition of the feed.

2.4. Filtration test

The filtration tests were conducted by using the PVDF and PSF membranes to investigate the effect of membrane properties, tilting angle, aeration rate and microalgae biomass concentrations towards the membrane permeability, as well as to compare the filtration performance of both membranes.

To evaluate the filtration performance, the flux (J, L/m^2·h) during filtration was calculated by using Eq. (1), whereas the permeability (L, L/m^2·h·bar) of the membrane was determined by using Eq. (2). In all filtrations, a complete rejection of biomass was achieved because of huge difference in the cell size (40 μm) and the membranes pore sizes (0.04 and 0.42 μm) and thus the rejection is not discussed in detail in the present study. All filtration tests were done at least in duplicate and are presented as mean±standard deviation to ensure the reproducibility of the data.
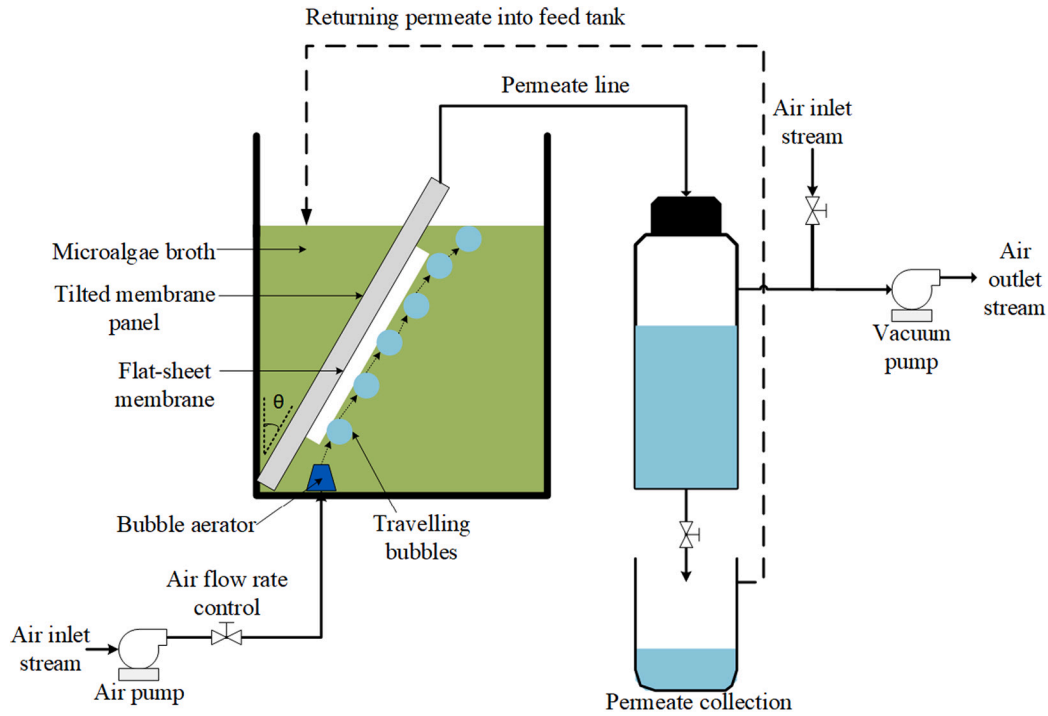
$$J = \frac{V}{At} \tag{1}$$

Fig. 1. Illustration of the lab-scale tilted membrane filtration system.

$$L = \frac{J}{\Delta P} \tag{2}$$

where V is the volume of permeate collected (L), t the duration of filtration (hr), A the effective area of filtration (m^2) and ΔP the trans-membrane pressure (bar).

The experiments to assess the effect of tilting angles were conducted at four tilting angles: 0 (vertical), 20, 45 and 70° for both the PVDF and the PSF membranes. The panel was tilted to the assigned angle, and it was hold using a holder to keep it intact. After placed according to the intended inclination angle, no change of angle was observed during the whole filtration tests. The filtration parameters when evaluating each tilting angle were kept constant at 1.0 L/min aeration rate, 7 L of feed volume and filtration cycle of 9 min on/1 min off.

Prior to the microalgae filtration test, the clean water permeability tests were conducted for 30 min. Afterwards, the microalgae filtration was carried out for 1 h until it reached its about constant permeability values. The permeate volume was measured during the 1 min of relaxation and used to calculate the permeability, resulting in a total of six permeability values in 1-hour of filtration test. Then, the membrane panel was rinsed with tap water and soaked into 1% of sodium hypochlorite for 1 h to restore its permeability value prior to the next filtration test. The results from the tilting angle study were used to select the membrane for further tests.

The filtration tests to evaluate the influence of aeration rate were run at aeration rates of 0.5, 1.0, 1.2 and 1.5 L/min using the PVDF membrane under a constant tilting angle of 45°. The performance was compared only at the tilting angle of 45° because it was found to be the optimum angle from the previous tests. Likewise, each filtration test was carried out for 1 h.

The study on the effect of feed concentration was done using broth with biomass concentrations of 2.15, 2.43, 2.51 and 2.55 g/L. During this tests, the aeration rate and the tilting angle was kept constant at 1.0 L/min and 45°, respectively. The microalgae broth solutions of 2.15, 2.43, 2.51, and 2.55 g/L were obtained by pre-concentrating the microalgae by filtration using another spare PVDF membrane to reach the desired concentration levels.

2.5. Energy estimation

Two different methods were applied to estimate the energy consumption for the filtration in a projected full-scale set-up. The first method utilized the full-scale data from a submerged membrane bioreactor (MBR) operation (Fenu et al., 2010). It was assumed that some equivalent parameters had similar energy consumption. The referenced parameters such as influent pumping (P^*_{IN}), permeate pumping (P^*_{P}), coarse bubble aeration (AER), cleaning in place (CIP) and air compression (AIR) were 0.03, 0.07, 0.23, 0.04 and 0.02 kWh/m^3, respectively. Nonetheless, the applied ΔP from the reference was 0.4 bar whereas in this current study was set at 0.1 bar. Therefore, the permeate pumping (P^*_{P}) was redefined accordingly (Osman et al., 2020). The energy consumption of the filtration was calculated by using Eq. (3) and was further simplified into Eq. (4).

$$E = P^*_{IN} + \frac{\Delta P}{\Delta P^*}P^*_P + \frac{J^*}{J}(AER^* + CIP^* + AIR^*) \tag{3}$$

$$E = 0.048 + \frac{8.12}{J} \tag{4}$$

where E is the estimated energy consumption (kWh/m^3), J^* flux obtained from the referenced study (28 L/(m$^2 \cdot$ hr)) and J the flux obtained from this experiment (54.4 L/(m$^2 \cdot$ hr)).

The second method only projected the aeration energy, according to the method reported elsewhere (Verrecht et al., 2008). To allow full-scale projection, a hypothetical full-scale module assembly was developed. It was assumed that the plate-and-frame module had a dimension of 50 × 100 cm, arranged in double-deck at panel gap of 0.5 cm. Thus, a flow channel of $A_x = 2.5 \times 10^{-3}$ m^2 existed in between the two panels, where feed liquid and air bubbles flow. Due to this configuration, the bubbles could flow over the membrane area of $A = 1.0$ m^2 along the flow channel. The aeration rate of 1.0 L/min through 10 cm width was translated to 5.0 L/min in the large-scale panel, corresponding to the aeration rate along the channel of $U = 8.3 \times 10^{-5}$ m^3/s or $U = 0.033$ m/s. The referenced parameters including the pressure (P), temperature (T), blower efficiency (ξ) and the distance of aerator nozzle from the surface

(y), aerator constant (λ) were assumed to be 20 kPa, 298 K, 0.8, 2 m and ~1.4, respectively. Then, the aeration energy consumption was calculated by using Eq. (5).

$$E_A = \frac{PT\lambda}{2.73 \times 10^5 \xi(\lambda - 1)} \frac{UA_x}{JA} \left[\frac{10^4 y + P^{\left(1 - \frac{1}{\lambda}\right)}}{P} - 1 \right] \tag{5}$$

where E_A is the estimated aeration energy (kWh/m^3) and J is the flux obtained from this study (54.4 L/(m^2 · hr)).

2.6. Statistical analysis

The results on the assessment filtration parameters (membrane, tilting angle, aeration rate and concentration) were analyzed statistically. The significance of overall permeability in each parameter was evaluated using one-way analysis of variance (ANOVA) at 95% confidence intervals ($p < 0.05$). Then, the Tukey HSD post hoc was performed to identify which pairs of mean were significantly different. To allow statistical analysis, each filtration test was conducted twice.

3. Results and discussion

3.1. Membrane characteristics

The summary of PVDF and PSF membranes properties are provided in Table 1. Both membranes were asymmetric, typical of the ones produced using the phase inversion process (Tan and Rodrigue, 2019). The PVDF was microfiltration type and the PSF was ultrafiltration type, as such the impact of pore size on the filtration performance could be clearly compared. Microalgae suspension consisted of algae cells and algogenic organic matters (AOM: polysaccharides, protein etc.) (Zhang and Fu, 2018). The cell size of *Spirulina* sp. was approximately 40μm, much larger than the pore size of the PVDF and PSF the membranes. Theoretically, judging from the pore size of the membranes, the microalgae *Spirulina* sp. could be fully retained. However, some of the AOM smaller than the membrane pore size contained in the culture medium could still pass the membrane pores.

Apart from retaining the microalgal cells, it was also attractive to recover extracellular polysaccharides (EPS, a part of AOM) from the microalgae suspension due to its benefits as the food supplements or its richness in therapeutic compounds. The sulfated polysaccharides were considered as antiviral agent because it could inhibit the growth of viruses (Vaz et al., 2016). *Spirulina* sp. was well known for its capability in secreting abundant amount of AOM (Zhang and Fu, 2018). It was reported that the pilot scale of hybrid microfiltration and ultrafiltration could recover AOM from the culture medium (Li et al., 2011). The microfiltration was used to remove solid residuals and the AOM was then recovered from the microfiltration permeate using the ultrafiltration.

3.2. Comparison of PVDF and PSF membranes performance

Fig. 2 compares the hydraulic performance of the PVDF and PSF

Table 1
Summary of polyvinylidene difluoride (PVDF) and polysulfone (PSF) membranes properties.

Parameters	PVDF	PSF
Clean water permeability (L/(m^2·h·bar))	1740 ± 73	923±54
Pore size (μm)	0.42	0.04
Type	Microfiltration	Ultrafiltration
Contact angle (°)	70.5±3	67.9±1
Thickness (μm)	248	261
Morphology	Asymmetric	Asymmetric

membranes in the tilted panel system operated under variable tilting angles. For the PSF membrane, the tilting angle of 45° showed the highest steady state permeability of 132.5, followed by tilting angles of 70, 20 and 0° of 123.4, 108.9 and 107.1 L/(m^2·h·bar), respectively. This trend was similar to the PVDF membrane that had the highest permeability of 377.4 at tilting angle of 45, followed by tilting angles of 70, 20 and 0° of 344.8, 312.1 and 282.2 L/(m^2·h·bar), respectively. The filtration performance of both membranes increased by tilting the panel up to the tilting angle of 45°, then dropped at the tilting angle of 70°. It is worth noting that the permeability values at 60 min of filtration were considered as quasi steady state value, in which prolonging the filtration would only slightly affect the permeability because of the low rate of membrane fouling. Nevertheless, as the occurrence of membrane fouling was inevitable, a slow decline in permeability was still expected over prolonged filtration time. However, the short-term filtration duration performed in this study was considered sufficient for comparative purpose. Fig. 2a shows the variation of permeability as function of time for PVDF membrane showing the same pattern with the PSF membrane (data not shown).

When comparing the PDVF and the PSF membranes, the one-way ANOVA analysis on the data on the impact of tilting angle obtained the p-values of 0.003 and 0.025, respectively. Those p-values were lower than the significance level of 0.05, indicating that the effect of the tilted panel configurations varied significantly. In this analysis, the means of the permeability values under all tested tilting angles for the two membranes were compared. Further analysis on the effect of tilting angle for the four data points using the Tukey HSD Test resulted in both membranes had Q-critical of 5.76. The PSF membrane had lower Q-statistic as compared to Q-critical for data at 0° vs 20° and 45° vs 70° which implied that the impact of tilting angle was only significant at a range of 20° to 45°. As for the PVDF membrane, the Q-statistic was higher than the Q-critical for data at 0° vs 45° and 20° vs 45° indicating that the tilting angles of 0°–45° affected the permeability significantly.

As the panel was tilted, the magnitude of impact force exerted by the air bubbles on the panel varied. The impact force (F_I) imposed by the air bubbles to the membrane panel was largely influenced by the buoyancy force (F_B) and the tilted angle (θ), in which $F_I = F_B \sin \theta$. Based on this equation, the maximum impact force could be obtained once the tilted panel reached 90° which resembled a sinusoidal pattern as depicted in Fig. 3.

The permeability performance was affected by the impact force exerted by the travelling bubbles. Hence, the permeability of tilted membrane reaching 70° was expected to increase until it reached 90°. However, both membrane permeability performance started to drop at tilting angle of 70°, which contradicted with the sinusoidal pattern of impact force as shown in Fig. 3. The lower permeability at tilting angle of 70° compared to 45° can be explained by the congestion of travelling bubbles along membrane panel. The membrane surface would be overcrowded with air bubbles and disrupting the permeate flow, which lowered the permeability (Nawi et al., 2020). This finding implies that system optimization with respect to aeration rate and tilting angle was required to maximize the impact of aeration. Overcrowding the membrane surface with air bubbles under high aeration rates at higher tilting angle would not only reduce the permeability, but also unnecessarily waste aeration energy. The finding suggests the opportunity to couple panel tilting at high tilting angle with a low aeration rate.

This also implies the permeability reached maximum at the tilting angle of 45° due to good membrane cleaning. The air bubbles could remove the foulant via drag force from the membrane more effectively (Eliseus et al., 2018a). Apart from scouring-off the existing foulant, the bubbles also mitigated the accumulation of the foulant materials on the membrane surface (Eliseus et al., 2017). Therefore, tilting the panel at 45° was the optimal angle as it had the highest permeability attained by both membranes.

When comparing the two membranes, tilting the panel at 45° for the PVDF membrane enhanced the permeability significantly by 34% from
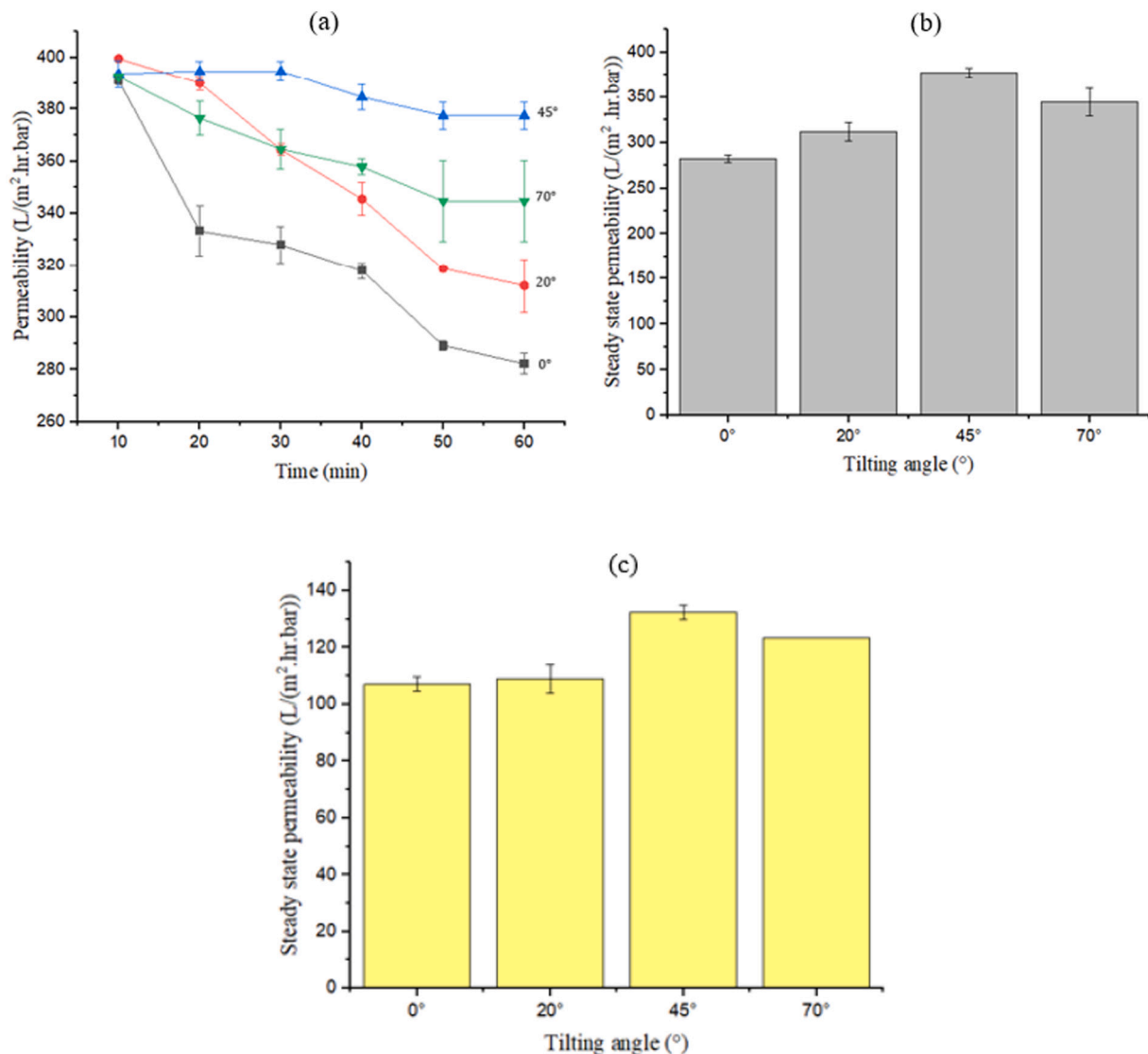
Fig. 2. Effect of tilting angle on (a) PVDF permeability as function of filtration time, (b) steady state permeability of PVDF membrane and (c) PSF membrane. Note that the data for 70° in (c) are identical resulting in zero error.

the vertical panel (tilting angle = 0°). In comparison, PSF membrane panel at a tilting angle of 45° could only improve its permeability from the vertical panel by 24%. This finding was also supported by the one-way ANOVA as it had a p-value of 0.0003 ($p < 0.05$) suggesting that the impact of tilting angle on PVDF and PSF filtration performance were significantly different. Higher permeability of the PVDF membrane can be attributed to its higher pore size than the PSF (Table 1). Membrane with larger pore size had a higher tendency to form irreversible fouling (Lau et al., 2020). Because the PVDF membrane had high fouling propensity, it could still yield remarkably higher permeability than the PSF membrane due to good membrane cleaning. This indicates that the tilted panel system was highly effective in enhancing the permeability of membranes with high (reversible) fouling propensity. Therefore, the PVDF membrane at tilting angle of 45° was selected for further tests.

3.3. Effect of aeration rate

Fig. 4 shows that increasing aeration rate enhanced the permeability and the impact reached plateau at a rate of 1.0 L/min. Beyond plateau permeability, increasing aeration rate no longer enhanced the permeability. It means that the cleaning impact of the air bubbles reached its maximum. The one-way ANOVA conducted in this experiment produced

a p-value of 0.00003 ($p < 0.05$) which proved that increasing aeration rate significantly enhanced permeability. The Tukey HSD Test obtained a Q-critical of 5.76 at $\alpha = 0.05$ was lower than the Q-statistic of 0.5 vs 1.0, 0.5 vs 1.2 and 0.5 vs 1.5 L/min implying that the effect of aeration rate was only significant from 0.5 to 1.0 L/min.

The Tukey HSD Test results suggest that further increment of aeration rate beyond 1.0 L/min would not provide a significant increase in permeability. A phenomenon of plateau permeability was also reported elsewhere (Eliseus et al., 2018a).The finding implies that aeration at a rate of 1.0 L/min was enough to remove all reversible foulant from the membrane surface. Besides, exerting excessive shear force was not advised to preserve the quality of the harvested biomass because excessive bubbling could provoke the microalgae cells into releasing AOM that could aggravate membrane fouling (Zhang and Fu, 2018). Hence, the optimum aeration rate for the *Spirulina* sp. medium tested using tilted panel system in this study was in between 0.5 and 1.0 L/min. It is worth noting that albeit the plateau permeability could be reached at aeration rates of 1.0–1.5 L/min, the value could only reach about 500 L/m².h·bar which was about 68% of the clean water permeability value (Table 1). This implies that the aeration supplied was only capable of removing cake layer (reversible fouling). The irreversible fouling (such as pore blocking, and adsorption) could not be removed effectively by
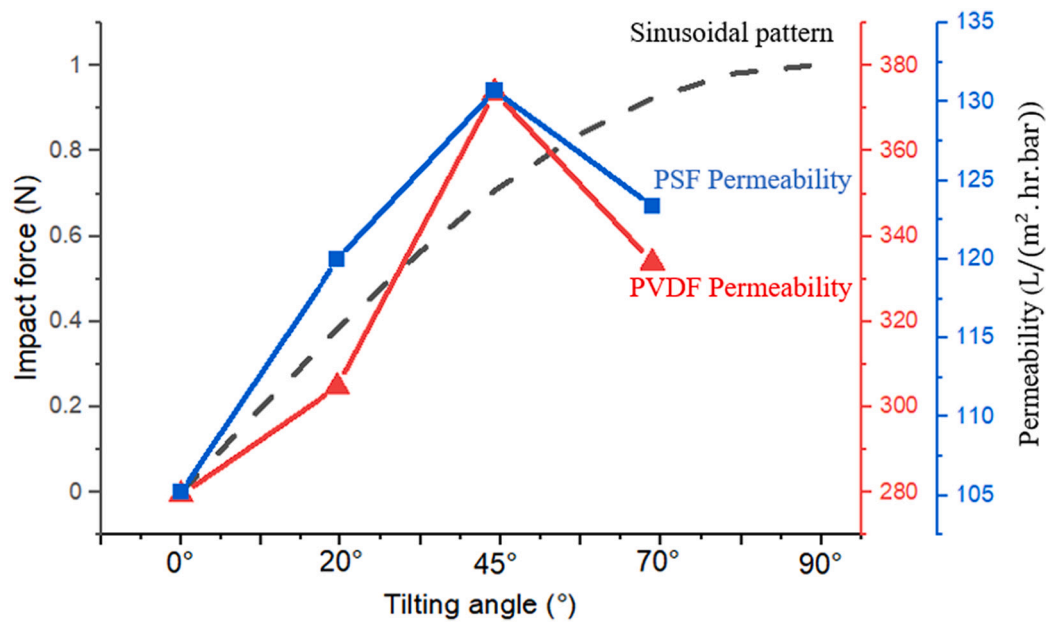
Fig. 3. Permeability at different tilting angle showing the negative impact of overcrowding bubbles at tilting angles above 45°.
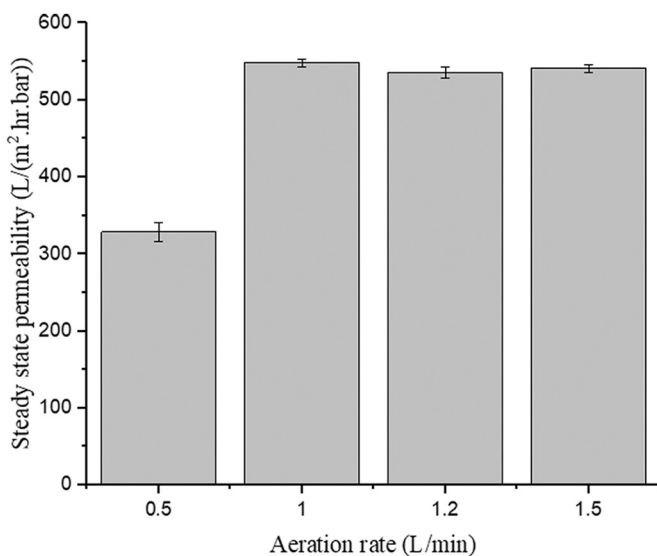

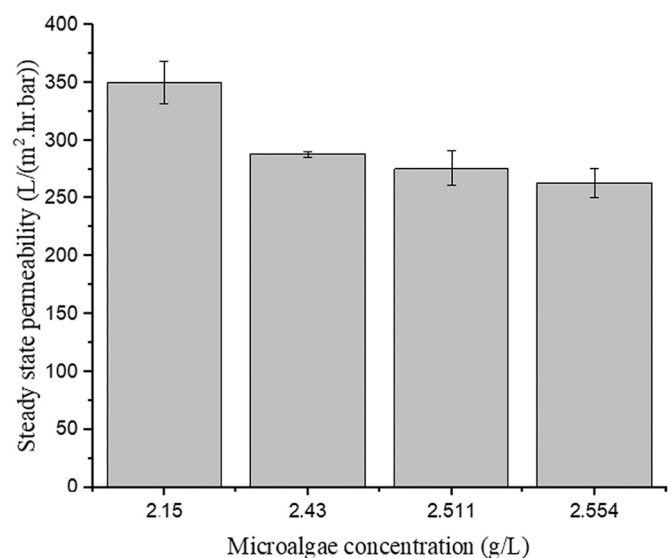**Fig. 4.** Effect of aeration rate on PVDF membrane.

Fig. 5. Effect of microalgae feed concentrations on PVDF permeability.

the induced bubble shear force. Hence, chemical cleaning should be performed in order to restore its pristine membrane permeability.

3.4. Effect of microalgae feed concentration

Fig. 5 shows that the filtration performance was lower at higher biomass concentration. The *Spirulina* sp. broth of 2.15 g/L had the highest permeability whereas the 2.554 g/L had the least permeability. The Tukey HSD analysis between the filtration data of the four concentrations resulted in a Q-critical of 5.76 at $\alpha = 0.05$ which was higher compared to Q-statistic for pair of the groups: 2.43 vs 2.511, 2.43 vs 2.554 and 2.511 vs 2.554 g/L suggesting that within the range of 2.43–2.554 g/L the impact of biomass concentration was insignificant. The Q-critical was lower than Q-statistic only when comparing the 2.15 g/L with the rests, indicating that the influence of concentration was only significantly at a concentration range of 2.15 and 2.43 g/L.

The decreasing trend of permeability at higher concentrations (up to

2.43 in this case) can be explained by the higher AOM concentration accompanied the higher biomass concentration. The presence of AOM was more abundant at higher biomass concentrations causing severe membrane fouling. The biomass fraction alone only imposes a low membrane fouling potential (Discart et al., 2013), but the presence of AOM in the cake layer formation impeded the flow of permeate passing through the cells (Babel and Takizawa, 2010). Besides, high AOM concentration associates with the presence of fouling factor in the form of transparent exopolymer particles (Discart et al., 2015) and an increase in the liquid viscosity.

By considering the negative impact of biomass concentration on the filtration performance, an optimum process design shall be opted. The range of concentration should allow the membrane filtration should operate efficiently, but high enough for effective harvesting since the effectiveness of membrane filtration could be reduced at a very high broth concentration (Bilad et al., 2013). Therefore, it is crucial to identify the optimum concentration for the membrane filtration system

by considering its species, operational conditions and membrane characteristics. The membrane-based harvesting can be considered as primary harvesting step to increase the biomass concentration. The thickening process of the biomass can be further continued using a more appropriate process, resulting in two-stage of harvesting (i.e., membrane filtration followed by centrifugation) (Bilad et al., 2014a).

3.5. Energy consumption estimation

The projection of energy consumption for *Spirulina* sp. filtration using the tilted panel system was done to project the energy input required for biomass recovery. A very low energy input is necessary when the biomass is aimed as biofuel feedstock (Sheng et al., 2017). For higher value end products, the energy input is not the main issue but preserving the yield and quality of the target product becomes the priority.

The energy consumption for the *Spirulina* sp. filtration in this study was estimated to be 0.20 kWh/m^3. The energy input was lower than the other membrane filtration for *C. vulgaris* and *P. tricornutum* of 0.27 kWh/

m^3 and 0.25 kWh/m^3, respectively (Bilad et al., 2012). Application of an optimized tilted panel system for *Euglena* sp. filtration resulted in energy consumption of 0.238 kWh/m^3 (Eliseus et al., 2018b). This finding suggests that despite operating under sub-optimum condition, the energy input for filtration of the *Spirulina* sp. can be lowered. The low energy input can be attributed to the high flux of 54.4 L/m^2·h at low transmembrane pressure −0.1 bar and moderate aeration rate of 1 L/min. The difference in energy input can also be attributed to the relative filtration performance of microalgae species as detailed elsewhere (Baerdemaeker et al., 2013).

To specifically estimate the energy consumption associated with the aeration, the method developed by Verrecht et al. (2008) was applied, which resulted in a very low aeration energy input of 0.03 kWh/m^3. If this value is used to project the full-scale energy consumption, the total energy input can be further lowered because in a common submerged filtration process, the aeration energy makes up majority of the total energy consumption. Nonetheless, further optimization on the operational parameters are still required. For instance, the filtration cycle of 9/1 (9 min on/1 min relaxation) maybe less attractive in the full-scale.

Table 2
Performance comparison with other studies.

Fouling management system		Microalgae species and concentration	Membrane material (pore size)	Flux L/(m^2 · hr)	Permeability L/(m^2 · hr · bar)	[a,b]Energy ** (kWh/m^3)	Ref.
Improved air bubbling system	Tilted panel	1.2 g/L of *Spirulina* sp.	PVDF (0.42 μm)	55.4	554	0.03[a]	This study
			PSF (0.04 μm)	13.3	133	0.20[b]	
						0.03[a]	
	Horizontal panel	1.2–1.4 g/L of *Chlorella* sp.	C-PVDF (0.45 μm)	148	74	–	(Hwang et al., 2015)
			P-PVDF (0.2 μm)	210	105		
	Vertical panel	0.65 g/L of *Chlorella* sp.	MCE (0.22 μm)	11.6–20.5	23.2–41	–	(Alipourzadeh et al., 2016)
	Tilted panel	1 g/L of *Euglena* sp.	PVDF (0.19 μm)	22.5	225	–	(Eliseus et al., 2017)
	Tilted panel	0.6 g/L of *Euglena* sp.	PVDF (0.42 μm)	72	724	0.19[b]	(Lau et al., 2020)
	Finned spacer	1.1 ± 0.1 g/L of *C. vulgaris*	PVDF (0.19 ± 0.01 μm)	87 ± 11	870 ± 11	–	(Razak et al., 2020)
Dynamic filtration system	Membrane vibrations	0.25 g/L of *Phaeodactylum* sp. and	PVDF (0.036 μm)	±21.25–42.5 ±25.5–42.5	212.5–425*	0.02[b]	(Bilad et al., 2013)
		0.21 g/L of *Chlorella* sp.	PVDF (0.013 μm)		255–425*	0.22[b]	
	Axial vibration	0.55 g/L of *C. pyrenoidosa*	PVDF (0.1 μm)	22–64**	220–640	–	(Zhao et al., 2016a)
	Vibration and aeration	0.08 g/L of *Chlorella vulgaris*	PVDF (0.21 μm) PSF (0.13 μm)	32.5	325	–	(Bilad et al., 2014b)
	Axial vibration	0.3 g/L of *C. pyrenoidosa*	PVDF (0.1 μm)	60	85.71	–	(Zhao et al., 2016b)
	Axial vibration and aeration	0.3 g/L of *Chlorella pyrenoidosa*	PVDF (0.1 μm)	238.4	340.57	–	(Zhao et al., 2016c)
	Rotational membrane module	0.06–0.095 g/L of *Phaeodactylum* sp., *Nannochloropsis* sp. and *Chaetoceros* sp.	Ceramic (2 μm) Ceramic (0.5 μm)	460 725 690 430 520 395	230 362.5 345 215 260 197.5	–	(Ríos et al., 2012)
	Vibration shear enhanced process	1.1 g/L of *Dunaliella tertiolecta*	PAN50 PE5 ABS	490 ± 20 164.5 ± 7 18.9 ± 0.2	140 ± 20 47 ± 7 5.4 ± 0.2	–	(Hapońska et al., 2018)
	Rotated disk module	1 g/L of *Parachlorella kessleri*	PES (200 kDa) PAN (500 kDa) PVDF (0.4 μm) PVDF (1.5 μm)	36± 4 29 ± 8 57 ± 20 55 ± 13	90 ± 4 48.3–58 ± 8 142.5 ± 20 91.7–110 ± 13	–	(Villafaña-López et al., 2019)
	Vibration module	1.2 ± 0.2 g/L of *Dictyosphaerium* sp.	12% wt PVDF (0.013 μm)	46**	460	0.21[b]	(Zhao et al., 2020)
Physical cleaning	Submerged disc	10.0 g/L of *Spirulina* sp.	PVDF (10–40 nm)	57–143	95–238.3	12.72[b]	(Kanchanatip et al., 2016)
	Backwashing and ventilation	0.968 g/L of *Scenedesmus* sp.	PVDF (0.2 μm)	130	260	–	(Chen et al., 2012)
	Submerged microfiltration	0.41 ± 0.05 g/L of *Chlorella vulgaris*	PVDF (0.36 μm)	32–50	320–500	0.27[b]	(Bilad et al., 2012)
		0.23 ± 0.06 g/L of *Phaeodactylum* sp.				0.25[b]	
	Backwashing	2.9 g/L of *Chlorella* sp.	HF PVC (0.01 μm)	70	202.9	–	(Zhang et al., 2013)

When no TMP data is available, the permeability was calculated from the reported flux (*) or critical flux (**) by assuming the TMP 0.1 bar. HF (hollow fiber), Polyvinyl chloride (PVC), Commercial PVDF (C-PVDF), Porous PVDF (P-PVDF), Micro cellulose ester (MCE), Commercial polyethersulfone (PE5), Commercial polyacrylonitrile (PAN50), and Self-made acrylonitrile butadiene styrene (ABS). The energy refers to ([a]) aeration energy and ([b]) total energy.

Application of relaxation at high solid concentration can be vulnerable from clogging of the perforated pipe typically used to supply membrane aeration.

3.6. Comparison with other studies

Table 2 summarizes the performance of microalgae filtration in fouling control management from recent literature. To the best of our literature search, there is no report available on *Spirulina* sp. broth filtration. Therefore, the comparison most of the studies were carried out for the filtration of *Euglena* sp. and *Chlorella* sp. as the microalgae feed. The tilted panel system using PVDF membrane in this study showed significantly higher permeability than other fouling control strategies, such as physical cleaning and DMF (Razak and Bilad, 2021). The maximum permeability obtained by the membrane vibration was 460 L/(m^2·hr·bar) (Zhao et al., 2020) and the physical cleaning was 500 L/(m^2·hr·bar) (Bilad et al., 2012). When compared, this study had 20.4% and 10.8% higher. This comparison clearly revealed that the air bubbling system was an effective fouling control strategy for *Spirulina* sp. filtration as compared to others.

Under the air bubbling control system, there were two previous reports that assessed the performance of tilted panel configuration as well. The first study on tilted panel setup yielded a maximum permeability of 225 L/(m^2·hr·bar) which was 59.4% lower than the current study. This could be due to the limited tilting angle at 20°, different type of microalgae, broth concentration, and membrane pore size. In the subsequent study, the permeability obtained was 23.5% greater than this study, in spite of using smaller tilting angle than the current study (20° vs 45°), and identical membrane type and pore size. The other factors that might vary the permeability could be due to different microalgae species and concentration.

The permeability achieved in this study was still lower when compared among other air bubbling control systems. This study obtained a maximum permeability of 554 L/(m^2·hr·bar) which was 36% lower than the maximum permeability gained by the finned spacer membrane filtration of 870 L/(m^2·hr·bar) during filtration of *C. vulgaris* (Razak et al., 2020). Both systems incorporated air bubbling system but with different configurations. Nevertheless, this was not a direct comparison as both systems had different microalgae species, operational parameters, and configurations. In general, it can be seen that the findings on the permeability in this study were within the upper range of the results reported in the literature. Nonetheless, the filtration energy input was the lowest reported so far.

Overall results demonstrate the efficacy of the tilted panel system for fouling control in *Spirulina* sp. biomass filtration. The performance of the system can be enhanced using a more permeable PVDF membrane and can be optimized under certain aeration rate (between 0.5 and 1.0 L/min) and certain biomass concentration. Considering the energy consumption reported in Table 2, PVDF membrane under tilted panel system is economically attractive to boost the economic advantage of *Spirulina* sp. for food purpose. Since the *Spirulina* sp. biomass is marketed as a dry powder, membrane-based process can be used to concentrate the biomass from the cultivation medium to remove the majority of the water prior to thickening and drying processes. The permeate water can be reused for subsequent cultivation in which the excess nutrients can be utilized as well. This water reuse lowers the production cost in term of feed water sterilization as well as reduces the need of the fresh nutrients.

4. Conclusions

This study concludes that the tilted panel system was an effective and energy-efficient for harvest *Spirulina* sp. The permeability peaked at the tilting angle of 45° in accord with the proposed model. Optimal aeration occurred in the range of 0.5–1.0 L/min. Filtration of a higher *Spirulina* sp. concentration yielded a lower permeability. The estimated total energy consumption of the *Spirulina* sp. broth solution was 0.20 kWh/

m^3, the lowest reported so far which can still be reduced further from the independent results of aeration energy estimation of merely 0.03 kWh/m^3.

CRediT authorship contribution statement

Insyirah Ismail performed all the experiments and prepared the manuscript draft; Muhammad Roil Bilad contributed to the original idea of the study, conceptually design the study and revised the manuscript; Lisendra Marbelia, Shafirah Samsuri, and Arief Budiman developed the experimental setup, supervised the experiment and validated the experimental results; Lisendra Marbelia, Kiki Adi Kurnia, Noor Maizura Ismail, Asim Laeeq Khan and Susilawati Susilawati revised the manuscript.

Declaration of competing interest

The authors declare no conflict of interest.

References

Alipourzadeh, A., Mehrnia, M.R., Sani, A.H., Babaei, A., 2016. Application of response surface methodology for investigation of membrane fouling behaviours in microalgal membrane bioreactor: the effect of aeration rate and biomass concentration. RSC Adv. 6, 111182–111189. https://doi.org/10.1039/C6RA23188H.

Andrade, L.M., 2018. Chlorella and Spirulina microalgae as sources of functional foods, nutraceuticals, and food supplements; an overview. MOJ Food Process. Technol. 6, 45–58. https://doi.org/10.15406/mojfpt.2018.06.00144.

Azma, M., Mohamed, M.S., Mohamad, R., Rahim, R.A., Ariff, A.B., 2011. Improvement of medium composition for heterotrophic cultivation of green microalgae, Tetraselmis suecica, using response surface methodology. Biochem. Eng. J. 53, 187–195. https://doi.org/10.1016/j.bej.2010.10.010.

Babel, S., Takizawa, S., 2010. Microfiltration membrane fouling and cake behavior during algal filtration. Desalination 261, 46–51. https://doi.org/10.1016/j.desal.2010.05.038.

Baerdemaeker, T.D., Lemmens, B., Dotremont, C., Fret, J., Roef, L., Goiris, K., Diels, L., 2013. Benchmark study on algae harvesting with backwashable submerged flat panel membranes. Bioresour. Technol. 129, 582–591. https://doi.org/10.1016/j.biortech.2012.10.153.

Bilad, M.R., Vandamme, D., Foubert, I., Muylaert, K., Vankelecom, I.F.J., 2012. Harvesting microalgal biomass using submerged microfiltration membranes. Bioresour. Technol. 111, 343–352. https://doi.org/10.1016/j.biortech.2012.02.009.

Bilad, M.R., Discart, V., Vandamme, D., Foubert, I., Muylaert, K., Vankelecom, I.F.J., 2013. Harvesting microalgal biomass using a magnetically induced membrane vibration (MMV) system: Filtration performance and energy consumption. Bioresour. Technol. 138, 329–338. https://doi.org/10.1016/j.biortech.2013.03.175.

Bilad, M.R., Arafat, H.A., Vankelecom, I.F.J., 2014a. Membrane technology in microalgae cultivation and harvesting: a review. Biotechnol. Adv. 32, 1283–1300. https://doi.org/10.1016/j.biotechadv.2014.07.008.

Bilad, M.R., Marbelia, L., Naik, P., Laine, C., Vankelecom, I.F.J., 2014b. Direct comparison of aerated and vibrated filtration systems for harvesting of Chlorella vulgaris. Algal Res. 6, 32–38. https://doi.org/10.1016/j.algal.2014.09.001.

Chen, X., Huang, C., Liu, T., 2012. Harvesting of microalgae Scenedesmus sp. using polyvinylidene fluoride microfiltration membrane. Desalination Water Treat. 177–181. https://doi.org/10.1080/19443994.2012.692034.

Cheng, T.-W., 2002. Influence of inclination on gas-sparged cross-flow ultrafiltration through an inorganic tubular membrane. J. Membr. Sci. 196, 103–110. https://doi.org/10.1016/S0376-7388(01)00584-1.

de Jesus, C.S., da Silva Uebel, L., Costa, S.S., Miranda, A.L., de Morais, E.G., de Morais, M.G., Costa, J.A.V., Nunes, I.L., de Souza Ferreira, E., Druzian, J.I., 2018. Outdoor pilot-scale cultivation of Spirulina sp. LEB-18 in different geographic locations for evaluating its growth and chemical composition. Bioresour. Technol. 256, 86–94. https://doi.org/10.1016/j.biortech.2018.01.149.

Discart, V., Bilad, M.R., Vandamme, D., Foubert, I., Muylaert, K., Vankelecom, I.F.J., 2013. Role of transparent exopolymeric particles in membrane fouling: Chlorella vulgaris broth filtration. Bioresour. Technol. 129, 18–25. https://doi.org/10.1016/j.biortech.2012.11.034.

Discart, V., Bilad, M.R., Vankelecom, I.F.J., 2015. Critical evaluation of the determination methods for transparent exopolymer particles, agents of membrane fouling. Crit. Rev. Environ. Sci. Technol. 45, 167–192. https://doi.org/10.1080/10643389.2013.829982.

Eliseus, A., Bilad, M.R., Nordin, N.A.H.M., Putra, Z.A., Wirzal, M.D.H., 2017. Tilted membrane panel: a new module concept to maximize the impact of air bubbles for

membrane fouling control in microalgae harvesting. Bioresour. Technol. 241, 661–668. https://doi.org/10.1016/j.biortech.2017.05.175.

Eliseus, A., Bilad, M.R., Nordin, N.A.H.M., Khan, A.L., Putra, Z.A., Wirzal, M.D.H., Aslam, M., Aqsha, A., Jaafar, J., 2018a. Two-way switch: maximizing productivity of tilted panel in membrane bioreactor. J. Environ. Manag. 228, 529–537. https://doi.org/10.1016/j.jenvman.2018.09.029.

Eliseus, A., Putra, Z.A., Bilad, M.R., Nordin, N.A.H.M., Wirzal, M.D.H., Jaafar, J., Khan, A.L., Aqsha, 2018b. Energy minimization of a tilted panel filtration system for microalgae filtration: Performance modeling and optimization. Algal Res. 34, 104–115. https://doi.org/10.1016/j.algal.2018.07.008.

Fenu, A., Roels, J., Wambecq, T., De Gussem, K., Thoeye, C., De Gueldre, G., Van De Steene, B., 2010. Energy audit of a full scale MBR system. Desalination 262, 121–128. https://doi.org/10.1016/j.desal.2010.05.057.

Hapońska, M., Clavero, E., Salvadó, J., Farriol, X., Torras, C., 2018. Pilot scale dewatering of Chlorella sorokiniana and Dunaliella tertiolecta by sedimentation followed by dynamic filtration. Algal Res. 33, 118–124. https://doi.org/10.1016/j.algal.2018.05.007.

Hwang, T., Oh, Y.K., Kim, B., Han, J.I., 2015. Dramatic improvement of membrane performance for microalgae harvesting with a simple bubble-generator plate. Bioresour. Technol. 186, 343–347. https://doi.org/10.1016/j.biortech.2015.03.111.

Kanchanatip, E., Su, B.-R., Tulaphol, S., Den, W., Grisdanurak, N., Kuo, C.-C., 2016. Fouling characterization and control for harvesting microalgae Arthrospira (Spirulina) maxima using a submerged, disc-type ultrafiltration membrane. Bioresour. Technol. 209, 23–30. https://doi.org/10.1016/j.biortech.2016.02.081.

Lau, A.K.S., Bilad, M.R., Nordin, N.A.H.M., Faungnawakij, K., Narkkun, T., Wang, D.K., Mahlia, T.M.I., Jaafar, J., 2020. Effect of membrane properties on tilted panel performance of microalgae biomass filtration for biofuel feedstock. Renew. Sust. Energ. Rev. 120, 109666 https://doi.org/10.1016/j.rser.2019.109666.

Lee, J., Ahn, W.-Y., Lee, C.-H., 2001. Comparison of the filtration characteristics between attached and suspended growth microorganisms in submerged membrane bioreactor. Water Res. 35, 2435–2445. https://doi.org/10.1016/S0043-1354(00)00524-8.

Li, H., Li, Z., Xiong, S., Zhang, H., Li, N., Zhou, S., Liu, Y., Huang, Z., 2011. Pilot-scale isolation of bioactive extracellular polymeric substances from cell-free media of mass microalgal cultures using tangential-flow ultrafiltration. Process Biochem. 46, 1104–1109. https://doi.org/10.1016/j.procbio.2011.01.028.

Nawi, N.I.M., Sivalingam, S., Bilad, M.R., 2020. Fouling control via horizontal membrane panel for activated sludge filtration. J. Eng. Pract. 2, 1–6.

Osman, A., Mat Nawi, N.I., Samsuri, S., Bilad, M.R., Shamsuddin, N., Khan, A.L., Jaafar, J., Nordin, N.A.H., 2020. Patterned membrane in an energy-efficient tilted panel filtration system for fouling control in activated sludge filtration. Polymers 12, 432. https://doi.org/10.3390/polym12020432.

Razak, Nik Nurul Ain Nabilah, Bilad, Muhammad Roil, 2021. Progress in development of Membrane Fouling Control for Microalgae Filtration: a Review. Jurnal Penelitian dan Pengkajian Ilmu Pendidikan: e-Saintika 5 (1), 67–91. https://doi.org/10.36312/e-saintika.v5i1.424.

Razak, N.N.A.N., Rahmawati, R., Bilad, M.R., Pratiwi, A.E., Elma, M., Nawi, N.I.M., Jaafar, J., Lam, M.K., 2020. Finned spacer for enhancing the impact of air bubbles for membrane fouling control in Chlorella vulgaris filtration. Bioresour. Technol. Rep. 11, 100429 https://doi.org/10.1016/j.biteb.2020.100429.

Ríos, S.D., Salvadó, J., Farriol, X., Torras, C., 2012. Antifouling microfiltration strategies to harvest microalgae for biofuel. Bioresour. Technol. 119, 406–418. https://doi.org/10.1016/j.biortech.2012.05.044.

Sheng, A.L.K., Bilad, M.R., Osman, N.B., Arahman, N., 2017. Sequencing batch membrane photobioreactor for real secondary effluent polishing using native microalgae: Process performance and full-scale projection. J. Clean. Prod. 168, 708–715. https://doi.org/10.1016/j.jclepro.2017.09.083.

Silva, N.C., Machado, M.V.C., Brandão, R.J., Duarte, C.R., Barrozo, M.A.S., 2019. Dehydration of microalgae Spirulina platensis in a rotary drum with inert bed. Powder Technol. 351, 178–185. https://doi.org/10.1016/j.powtec.2019.04.025.

Tan, X., Rodrigue, D., 2019. A review on porous polymeric membrane preparation. Part II: production techniques with polyethylene, polydimethylsiloxane, polypropylene, polyimide, and polytetrafluoroethylene. Polymers 11, 1310. https://doi.org/10.3390/polym11081310.

Tang, D.Y.Y., Khoo, K.S., Chew, K.W., Tao, Y., Ho, S.H., Show, P.L., 2020. Potential utilization of bioproducts from microalgae for the quality enhancement of natural products. Bioresour. Technol. 304, 122997 https://doi.org/10.1016/j.biortech.2020.122997.

Tian, J., Xu, Y., Chen, Z., Nan, J., Li, G., 2010. Air bubbling for alleviating membrane fouling of immersed hollow-fiber membrane for ultrafiltration of river water. Desalination 260, 225–230. https://doi.org/10.1016/j.desal.2010.04.026.

Vaz, B. da S., Moreira, J.B., Morais, M.G. de, Costa, J.A.V., 2016. Microalgae as a new source of bioactive compounds in food supplements. Curr. Opin. Food Sci., Food Chemistry and Biochemistry ● Food Bioprocessing 7, 73–77. https://doi.org/10.1016/j.cofs.2015.12.006.

Verrecht, B., Judd, S., Guglielmi, G., Brepols, C., Mulder, J.W., 2008. An aeration energy model for an immersed membrane bioreactor. Water Res. 42, 4761–4770. https://doi.org/10.1016/j.watres.2008.09.013.

Villafaña-López, L., Clavijo Rivera, E., Liu, S., Couallier, E., Frappart, M., 2019. Shear-enhanced membrane filtration of model and real microalgae extracts for lipids recovery in biorefinery context. Bioresour. Technol. 288, 121539 https://doi.org/10.1016/j.biortech.2019.121539.

Zhang, Y., Fu, Q., 2018. Algal fouling of microfiltration and ultrafiltration membranes and control strategies: a review. Sep. Purif. Technol. 203, 193–208. https://doi.org/10.1016/j.seppur.2018.04.040.

Zhang, Wei, Zhang, Wen, Zhang, X., Amendola, P., Hu, Q., Chen, Y., 2013. Characterization of dissolved organic matters responsible for ultrafiltration membrane fouling in algal harvesting. Algal Res. 2, 223–229. https://doi.org/10.1016/j.algal.2013.05.002.

Zhao, F., Chu, H., Su, Y., Tan, X., Zhang, Y., Yang, L., Zhou, X., 2016a. Microalgae harvesting by an axial vibration membrane: the mechanism of mitigating membrane fouling. J. Membr. Sci. 508, 127–135. https://doi.org/10.1016/j.memsci.2016.02.007.

Zhao, F., Chu, H., Tan, X., Yang, L., Su, Y., Zhou, X., Zhao, J., Zhang, Y., 2016b. Using axial vibration membrane process to mitigate membrane fouling and reject extracellular organic matter in microalgae harvesting. J. Membr. Sci. 517, 30–38. https://doi.org/10.1016/j.memsci.2016.06.022.

Zhao, F., Chu, H., Tan, X., Zhang, Y., Yang, L., Zhou, X., Zhao, J., 2016c. Comparison of axial vibration membrane and submerged aeration membrane in microalgae harvesting. Bioresour. Technol. 208, 178–183. https://doi.org/10.1016/j.biortech.2016.02.099.

Zhao, Z., Mertens, M., Li, Y., Muylaert, K., Vankelecom, I.F.J., 2020. A highly efficient and energy-saving magnetically induced membrane vibration system for harvesting microalgae. Bioresour. Technol. 300, 122688 https://doi.org/10.1016/j.biortech.2019.122688.

19 January 2021

Dear Editors of Bioresorce Technology Report

Please find attached a revised manuscript that we would like to submit for the first-time publishing in BITE, entitled "**Energy Efficient Harvesting of Spirulina sp. from the growth medium using a tilted panel membrane filtration**". All authors have agreed to submit the manuscript to Bioresorce Technology Report and declare that it is not under consideration for publication elsewhere. The manuscript preparation follows the guide for author.

This study assesses the filterability of the tilted panel to enhance membrane fouling control for filtration of Spirulina sp. medium. The influences of tilting angles, membrane types (PVDF microfiltration and PSF ultrafiltration), aeration rate, microalgae biomass concentrations, and energy consumption on the filterability were evaluated. The results show that the tilted panel system is an effective and energy-efficient approach for membrane fouling control. This is the first report that explore the application of energy efficient membrane filtration system for non-biofuel purpose which has considerably large market potential.

We believe that this manuscript provides enough relevance to the scope of Bioresorce Technology Report and carries substantial novelty to be considerred for publication.

Hoping on a positive response, I remain yours sincerely,

DR MUHAMMAD ROIL BILAD
Senior Lecturer, Chemical Engineering
Universiti Teknologi, PETRONAS
mroil.bilad@utp.edu.my

Dr Muhammad Roil Bilad

Assistant Professor, Chemical Engineering Department

UNIVERSITI TEKNOLOGI PETRONAS

32610 Seri Iskandar, Perak Darul Rizuan, Malaysia. Tel: +605-368-8000 Fax: +605-365 4075

www.utp.edu.my

25 April 2021

Dear Editors of Polymers MDPI

Please find attached a revised manuscript that we would like to resubmit for publication in Polymers MDPI. During the first submission (**polymers-1195339**), one of the editor reject the submission due to concern over the experimental method. We have attached our response to the comment appended in this letter. All authors have agreed to submit the manuscript to Polymers MDPI and declare that it is not under consideration for publication elsewhere.

This manuscript addressed two environmental issues in form of pollution by cigarette butt waste and emulsified oily wastewater. The cigarette butt waste (consisted mainly of cellulose acetate, CA) was used as main material for fabrication of phase-inverted membrane and use it for treatment of oil/water emulsion. Results show that CA-based membrane from waste cigarette butt offers an eco-friendly material without compromising the separation efficiency, with pore size range suitable for oil/water emulsion filtration with rejection of >94.0%. The CA membrane poses good structural property like that of established PVDF and PSF membranes with equally asymmetric morphology. Hence, this study demonstrates a sustainable approach in addressing issue of oil/water emulsion pollution treated CA membrane from cigarette butt waste.

We believe that this manuscript provides enough relevance to the scope of Polymers MDPI and carries substantial novelty to be considerred for publication.

Hoping on a positive response, I remain yours sincerely,

DR MUHAMMAD ROIL BILAD
Senior Lecturer, Chemical Engineering
Universiti Teknologi, PETRONAS
mroil.bilad@utp.edu.my

Dr Muhammad Roil Bilad

Assistant Professor, Faculty of Applied Science and Engineering

Universitas Pendikan Mandalika (UNDIKMA)
Jl. Pemuda No.59A, Dasan Agung Baru, Mataram (83125), Nusa Tenggara Barat, Indonesia

Response to comment by editor in the first submission

Comment

This manuscript belongs in a membrane science journal since the focus is on membrane performance testing. Furthermore, the testing is flawed because the authors use constant pressure filtration to evaluate fouling of membranes that differ in pure water permeability. Instead they must evaluate performance at constant initial flux or use constant flux filtration. This is a fatal flaw.

Response

The focus of this manuscript is on the utilization of cigarette waste that mainly consisted of cellulose acetate polymer as main material for fabrication of phase-inverted membrane. It is indeed fall under the scope of Polymer MDPI. The resulting membrane was then compared with another phase-inverted membranes prepared from commercial polysulfone and polyvinylidene difluoride polymers. Most of the manuscript contents are on fabrication and characterization of the membrane and only Section 3.9 discuss about the membrane fouling. We disagree on the comment on the testing method. Evaluating membrane filtration can be done either in constant-flux or in constant-pressure (as applied in this work. To allow more fair assessment, the performance is thus evaluated using permeability term to exclude the effect of the testing method. Many, including few of our work published in Polymers MDI, have reported similar assessment methods.

https://doi.org/10.3390/polym13060976

https://doi.org/10.3390/polym13030427

https://doi.org/10.3390/polym12112519

https://doi.org/10.3390/membranes10060121

17 March 2021

Dear Editors of Bioresorce Technology Report

Thank you for the given opportunity to revise our manuscript. We have carefully revised the manuscript in response to the comments by five highly qualified reviewers. The main points of the revisions are detailed as below:

- The introduction section has been extensively revised to to the comment by many reviewers.

- Some additional details on the experiment have been provided

- Highlights has been revised.

- Overall language (including typo errors) has also been improved.

We believe that the revised manuscript has substantially improved, thanks to the insights and comments given by the reviewer. We hope that it meets the high quality standard for publication in Bioresorce Technology

Hoping on a positive response, I remain yours sincerely,

Dr Muhammad Roil Bilad
Assistant Professor, Chemical Engineering Department

Response to Reviewer Comments

Editor comment

Highlights should be maximum 85 characters, including spaces, per bullet point.

Response: All points in the research highlight have been revised to obey the guideline.

Reviewer #2

General comment: Title: Energy efficient harvesting of Spirulina sp. from the growth medium using a tilted panel membrane filtration. The study used a novel tilted panel membrane filtration system for Spirulina sp. harvesting. The language used in this manuscript should be further improved, and authors should go through this paper again to correct some typos. The experiment design is simple and clear. The results in this study are interesting and useful, especially in this specific area. This paper could be accepted if authors can carefully consider the comments below:

General response: Thank you for the positive feedbacks. All comments raised by reviewer have been addressed and revisions have been made in the manuscript to accommodate the reviewer's comments. In addition, the language has been checked over the entire manuscript. The linguistic changes can be traced as the green text in the revised manuscript. The locations of changes in the revised manuscript are provided to ease reviewer.

Comment 1: Highlights part. The unit "LMH/bar" or "L/(m2 hr bar)" should be constant throughout the paper.

Response 1: This error has been corrected.

Location of changes: Point #4 in the research highlight

Comment 2: Abstract Line 23. "the system is an effective and energy-efficient" should be "the system is effective and energy-efficient".

Response 2: The mistake has been corrected.

Location of changes: Line 22-23

Comment 3: Line 356. There is a big blank.

Response: This error has been corrected.

Comment 4: 2.2 part. Which membrane did authors use to concentrate microalgae?

Response 4: Section 2.2 describes the cultivation process of microalgae broth for the study that was conducted prior to the process of concentrating the microalgae. It only involved microalgae cultivation in the Walne's medium, aeration and lights only. The process to concentrate microalgae to prepare the concentrated microalgae solution was done using another PVDF membrane not used for the filtration test.

Location of changes: Lines 179-183.

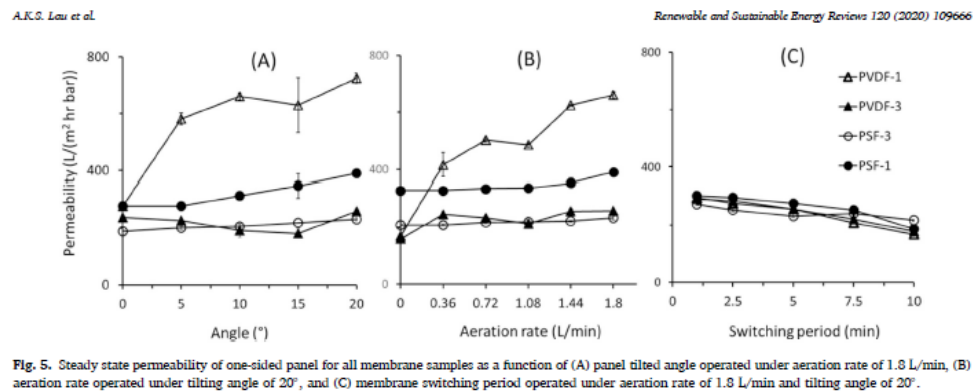**Comment 5**: Similar studies were found in previous studies (https://doi.org/10.1016/j.rser.2019.109666 and http://dx.doi.org/10.1016/j.biortech.2017.05.175). Both studies used tilted membrane panel to harvest microalgae. Authors should show clearly the difference and innovations compared with previous studies. In addition, Lau et al., (2019) (https://doi.org/10.1016/j.rser.2019.109666) also used the same membranes and tilted panel to harvest microalgae, while showing that PSf membrane was better than PVDF membrane (Table 2). Author showed explain the reason why the opposite results were obtained in present study

Response 5: The comment on the importance of explaining the difference and innovation with previous studies on air bubbling system is indeed true. Hence, the additional justification was explained in the revised manuscript. Each of the references focused on different aspects of microalgae harvesting despite their similarity of using the tilted panel system. [10.1016/j.rser.2019.109666] reports the effect of membranes properties on *C. vulgaris* filterability, while [10.1016/j.biortech.2017.05.175] report the tilted system system for membrane fouling control. In the former, the highest permeability is from PVDF membrane followed by PSF as depicted in the figure below. Therefore, no opposite results were obtained against the present study.
Location of changes: Lines 75-91 and Lines 409-416.

Fig. 5. Steady state permeability of one-sided panel for all membrane samples as a function of (A) panel tilted angle operated under aeration rate of 1.8 L/min, (B) aeration rate operated under tilting angle of 20°, and (C) membrane switching period operated under aeration rate of 1.8 L/min and tilting angle of 20°.

Reviewer #3

General comment: In this study, the authors compared membrane performance and energy cost in an algal membrane reactor under different operation conditions, in which a tilted-plate was used to enhance membrane performance. The data have been well organized and manuscript has been well written. The reviewer has several comments need to be further clarified.

General response: Thank you for the positive feedbacks. All comments raised by reviewer have been addressed and revisions have been made in the manuscript to accommodate the reviewer's comments. In addition, the language has been checked over the entire manuscript. The linguistic changes can be traced as the green text in the revised manuscript. The locations of changes in response to each comment (whenever applicable) are also provided.

Comment 1: Research highlights: past tense should be used.
Response 1: The grammar for the highlights have been revised accordingly.
Location of changes: Research Highlight

Comment 2: Figure 1 is confused as the permeate pump should only drive the liquid permeate flow. If air was present in the permeate line, did air entered into the permeate line due to not well sealed connection?
Response 2: As shown in Figure 1, along the vacuum pump line, there is a valve which controls the air inlet stream. The purpose of the air inlet stream is to maintain the transmembrane pressure at -0.1 bar. The air did not enter the permeate line at all and the permeate also did not flow to the pump maybe because of a relatively low vacuum pressure.

Comment 3: The authors should clarify the duration of the membrane filtration experiments performed and when the permeability data were taken.

Response 3: We have included information on the experimental duration and permeate collection method in the revised manuscript.
Location of changes: Lines 138-142, 168-170.

Comment 4: Was the experiments performed in series? If so, any change of other parameters in the algal reactor (such as organic concentrations).
Response 4: The experiments were performed in series. There was no major change in the algal reactor since the experiments were conducted during the stagnant growth phase of *Spirulina* sp. As described in Section 2.2, due to long stationary phase, the microalgae feed was approximately similar (~1.2g/L) in every experiment except for filtration on the effect of concentration (2.15, 2.43, 2.51 and 2.55 g/L). It was impossible to complete all filtration test during the stationary phase (6 days). Hence, each filtration parameter (tilting angle, aeration rate, and concentration) was done using different batches of broth cultivated using the 15-L reactor.
Location of changes: Lines 179-183.

Reviewer #4

Comment 1: Lines 35-65: Many sentences can be removed to remove redundancy and instead more information from literature regarding, membranes types and potential fouling mechanisms during harvesting, can be added.
Response 1: The suggestion given by the reviewer have been acknowledged and amended. A lot of redundant sentences especially in line 35-65 have been removed and more literature on membrane types such as microfiltration, ultrafiltration was included. Besides, the information on potential fouling mechanisms was also discussed.
Location of changes: Lines 35-45

Comment 2: Lines 94-95: One might disagree with this statement since the efficiency is strongly depending one algae and membrane types as well as operating conditions. One would use a less certain expression.
Response 2: We agree with the reviewer point. We have softened the claim in the revised manuscript by stating "aeration as one of many others."

Comment 3: Line 99: The interaction between concentration polarization and fouling layer is much more complicated. Please reconsider this sentence.
Response 3: We agree with the reviewer point. The sentence has been amended: "The hydrodynamic forces exerted by the air bubbles onto the membrane surface help to limit the concentration polarization, which in turn reduces the fouling layer."

Comment 4: Page 7 line 08: PVDF is polyvinylidene difluoride. Please correct all over the text!
Response 4: The spelling errors have been amended.
Location of changes: Lines 84 and 233.

Comment 5: Section 2.1, line 26: Please add more information regarding casting speed, humidity, coagulation bath temperature and time, washing and membrane storing.
Response 5: Detailed information on membrane fabrication parameters have been provided in the revised manuscript.
Location of changes: Lines 102-111.

Comment 6: Section 2.2, line 41: What is lighting rate? – line 46: How long is the stationary phase?

Response 6: The information on lighting rate was added. The stationary phase was about 6 days. We have included the information on those questions in the text.

Location of changes: Lines 122-124 and 129.

Comment 7: Section 2.3., line 54: Since relaxation is critically known to have certain effect on membrane cleaning, authors should describe the conditions clearly (for instance, was air bubbling working or not).

Response 7: Some details on the relaxation condition have been provided to add more clarity to the readers. However, we did not relaxation as a study parameter. As an established membrane fouling control method, its impacts have been well reported.

Location of changes: Lines 128-132.

Comment 8: Also, did the authors notice certain influence on the membrane permeability in the subsequent cycles because of such relaxation.

Response 8: The influence of relaxation on membrane permeability was not thoroughly investigated in this study. Hence, the effect of relaxation was not discussed in detail. We applied the same filtration cycle for every filtration. Due to permeability sampling method (one data per filtration cycle), the influence of relaxation between filtration cycles could not be observed.

Comment 9: Section 2.4, line 69 and in all cases, overall ranges of TOC in permeates should be given to give a general indication for membrane selective properties and influence of membrane fouling on membrane retention.

Response 9: We did not measure the TOC during the experiment and cannot provide further comment on it. In this context, selectivity of microalgae harvesting was indicated by the biomass rejection, which was 100% in all cases.

Location of changes: Please see remark on rejection on lines 152-154.

Comment 10: Section 2.4, line 81: pH?

Response 10: Unfortunately, we did not measure the pH of the leaning solution.

Comment 11: Section 2.4, line 90-91: The text regarding the addition of fresh microalgae broth is confusing and hard to understand. Please revise.

Response 11: Thank you for the valuable input. We have now revised the description.

Location of changes: Lines 181-183.

Comment 12: Section 3.2, line 64: Correct :"queasy" to "quasi"!

Response 12: The typo has been corrected.

Comment 13: Section 3.2, lines 69-71 and Figure 2, permeability curves for PSF membrane at different tilting angles should be added as well. Why were permeability values at 10 min for PVDF membrane very close? Does it reflect low impact of titling angle at early filtration. Besides, it is better to show the values as normalized permeability.

Response 13: We incline to maintain the current for of Figure 2. We prefer to show the actual permeability instead of normalized value. When normalizing the value, it only spot the trend relative to the initial value but completely ignore the magnitude of permeability. The trend of permeability value of PSF has been discussed in more detail. The close values of permeability at the first 10 min is

expected because of the membrane was still clean due to low level of foulant accumulation at the beginning of the filtration.
Location of changes: Lines 258-259.

Comment 14: section 3.4, line 77: Why are both terms EPS and AOM are used in the text?
Response 14: For conciseness, we now revised the manuscript to consistently use the AOM term (to be more general).

Comment 15: Section 3.5: Despite the fact that the energy consumption in this study was estimated to be lower than some lab scale systems reported in literature, one might argue regarding the applicability of the proposed operation protocol (9 min filtration cycle!) in the full-scale application. Please comment. This should be also added to the manuscript.
Response 15: Thank you for the remark on the applicability of the filtration cycle. Additional discussion have been included in the manuscript on this matter.
Location of changes: Lines 392-396.

Comment 16: The term filterability, used all over the text, is not a standard term. It has not been described so far, therefore it is confusing.
Response 16: Thank you for the input. The term "filterability" has been replaced all over the revised manuscript with "filtration performance."

Comment 17: The language is overall good but it still needs revision; many sentences are missing verbs and/or markers. Examples, page 4 lines 23-24, page 4 lines 27-28, page 15 lines 98-99, page 16 line 28 (are -> is), page 17 line 48 (suggests), page 17 line 51 (is -> was) ... etc.
Response 17: We have revised the English of the entire manuscript.

Reviewer #5

General comment: This manuscript regarding Spirulina sp. filtration process offers novel information on the field of membrane-based microalgae harvesting. It is well structured and easy to follow. This manuscript deserves publication. Only some minor comments should be addressed before publication.
General response: Thank you for the positive feedbacks. All comments raised by reviewer have been addressed and revisions have been made in the manuscript to accommodate the reviewer's comments. In addition, the language has been checked over the entire manuscript. The linguistic changes can be traced as the green text in the revised manuscript. The locations of changes in response to each comment (whenever applicable) are also provided.

Comment 1: Abstract (Line 23) The use of the article "an" is incorrect.
Response 1: The grammar error has been fixed.

Comment 2: The introduction part is too long. As the article is not focused on the use of Spirulina for obtaining valuable products, I suggest to summarise and merge paragraphs 2 and 3 (lines 41-64) since they are beyond the topic studied (Spirulina filtration).
Response 2: The paragraph 2 and 3 have been shortened to just be in one paragraph (the first Paragraph) as suggested by the reviewer.
Location of changes: Lines 35-45

Comment 3: L376-384: It would be interesting to show the EPS concentration (protein and polysaccharides) to know the effect of both membranes in the secretion of these compounds.
Response 3: Unfortunately, detailed analysis on the EPS composition was nor performed. We could not provide the information.

Comment 4: L380: "The biomass itself…" Please explain this sentence.
Response 4: It is a typo, it should by "the biomass fraction alone," which has been amended in the revised manuscript.

Comment 5: L387: "should" appears twice in the same sentence.
Response 5: This error has been fixed.

Comment 6: L419, L430: Please make sure that the species' name is in the same format in the whole manuscript.
Response 6: Thank you for the suggestion. This error has been amended.

Reviewer #7

General comment: The authors provided a simple way to mitigate membrane fouling by tilting the membrane panel during the filtration of Spirulina sp. broth. The method seems to be easily applied to the industrial production and may be combined with other anti-fouling method, such as membrane modification and optimization of operating conditions. The experimental design is reasonable and the results are credible. However, some minor problems can be discussed and improved.
General response: Thank you for the positive feedbacks. All comments raised by reviewer have been addressed and revisions have been made in the manuscript to accommodate the reviewer's comments. In addition, the language has been checked over the entire manuscript. The linguistic changes can be traced as the green text in the revised manuscript. The locations of changes in response to each comment (whenever applicable) are also provided.

Comment 1: The introduction is a bit tedious. The authors gave too much information about the micro-algal industry and the importance of Spirulina sp. incubation, but the current algal harvesting processes and the difficulties, especially how to deal with the membrane fouling problem, were not well presented.
Response 1: The suggestions given by the reviewer have been acknowledged and amended. The lengthy explanation of microalgae industry and its importance have been shortened in only one paragraph (the first Paragraph). The current algal harvesting processes such as centrifugation, coagulation/flocculation have been discussed together with its drawbacks in the 2nd Paragraph. As for ways to deal with the membrane fouling problem, several approaches have been mentioned in the third paragraph, such as studying on operational conditions, conducting regular (periodical) cleaning, applying dynamic membrane filtration (DMF) system and air bubbling system (by inducing shear rates. This study focuses more on explaining the air bubbling system in tackling membrane fouling problem which explains why the 4th Paragraph explains solely on that system.
Location of changes: Lines 35-45

Comment 2: It is dubious to use the formula (FI=FBsinθ, line 300) to simulate the effects of aeration on the permeability. According to this assumption, FI is 0 at the condition of θ=0, which implies that aeration should be invalid when the panel is vertical placed. Obviously, this is contrary to the facts.
Response 2: we agree on the exact statement by the reviewer that the formula does not well simulate

the effect aeration on the permeability. The formula was developed to simulate the effect of panel tilting on the permeability of the aerated system, particularly on the impact force. It means that, when the panel is vertical, the impact force has no contribution on the permeability. We have proven the model in our earlier reports and the findings have been consistent.

Detailed Response to Reviewer Comments

Reviewers' comments:

Reviewer #2: The authors have addressed most of the comments; they have also tried to make changes according to the reviewers' suggestions. After revisions, the quality of the manuscript has been adequately enhanced. Therefore, the manuscript could be considered for the publication in the journal.

Reviewer #3: The authors have addressed all of my comments. The manuscript is ready for publication.

Reviewer #4: The authors have fulfilled properly all the comments and the manuscript can be published in the current version.

Reviewer #5: Accept

Reviewer #7: The manuscript has been well improved and should be recommended for publication.

Response: Dear all reviewers. Thank you far valuable inputs to our manuscript. We believe that substantial improvements have been made thanks to the comments by reviewers.

Editor comment: Please refer to the published paper of BITEB for references' format. (Should use abbreviation for journal titles).

Response: The manuscript has been revised acording to reference formate of Bioresource Technology Report.
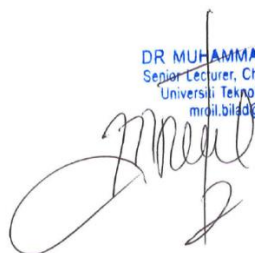
29 March 2021

Dear Editors of Bioresorce Technology Report

Thank you for the given opportunity to revise our manuscript. We have carefully revised the manuscript in response to the comments the editor and by five highly qualified reviewers.

We believe that the revised manuscript has meet the standard on follow the format for publication in Bioresorce Technology

Hoping on a positive response, I remain yours sincerely,

DR MUHAMMAD ROIL BILAD
Senior Lecturer, Chemical Engineering
Universiti Teknologi, PETRONAS
mroil.bilad@utp.edu.my

Dr Muhammad Roil Bilad
Assistant Professor
Faculty of Applied Science and Engineering
Universitas Pendidikan Mandalika