Comparison of dual stage ultrafiltration and hybrid ultrafiltration-forward osmosis process for harvesting microalgae (Tetraselmis sp) biomass

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Abstract

In this study, a hybrid ultrafiltration - forward osmosis (FO) process was investigated for harvesting of marine microalgae (Tetraselmis sp). FO was applied as a post-treatment process after the ultrafiltration (UF) process to obtain higher harvesting efficiency while consuming less energy. The UF-FO process was tested using a pilot-scale UF and bench-scale FO setups. The FO process assessed the impact of different flow rates, membrane orientation and feed solution concentration on the process performance. A maximum algal harvesting concentration factor (CF) of 7.0 was achieved using ultra-filtrated algae as feed solution in the FO membrane operating in the FO mode at 2.5 LPM flowrate for 48 hours operation time. The total energy consumption decreased by 46% using a hybrid UF-FO process instead of dual-stage UF. The FO process was inefficient for the harvesting of raw microalgae culture with a concentration ≤ 1 g/l. However, The FO process was an energy efficient post-treatment process after ultrafiltration for further harvesting of microalgae cells.

Keywords: Harvesting of Algae; Forward osmosis; Ultrafiltration; Brine reject; Membrane fouling.

1. Introduction

In recent years, microalgal biomass is increasingly considered as a promising alternative raw material source for biofuel production. Microalgae can be used to produce multiple high-value items such as food supplements, cosmetics and pharmaceutical products. However, algae cells are available in a very diluted culture medium, where their density is similar to water. Harvesting of microalgae is the separation of algae cells from the culture solution. Harvesting of microalgae is considered the most challenging constraint to an industrial scale production process because it stipulate 50% of the total energy of the biomass production process (Lei et al., 2015). Harvesting is usually done using conventional processes such as sedimentation, centrifugation, chemical flotation, electrophoresis and coagulation and flocculation (Buckwalter et al., 2013; Mo et al., 2015). Recently, ultrafiltration (UF) (Shao et al., 2015; Zhang et al., 2013) and microfiltration (MF) (Simstich et al., 2012) have been utilized in harvesting microalgae due to their high separation efficiency and ease of use. However, these pressure-driven membrane filtration processes are amenable to fouling, which increases energy costs (Mo et al., 2015; Rickman et al., 2012). Thus, there is a vital need for a sustainable algae harvesting process to overcome the drawbacks of the existing technologies. Forward osmosis (FO) is an evolving membrane filtration process that has the potential to minimize the total cost of harvesting microalgae by utilizing osmotic pressure differences to concentrate microalgae. In the FO process, fresh water transports from the feed to the draw solution side through a semi-permeable membrane (Noffsinger et al., 2009). Various studies showed the effectiveness of the FO process in harvesting microalgae due to its high efficiency and low energy consumption [reference, I suggest "Separation and Purification Technology, Volume 2042 October 2018Pages 154-161]. Table 1summarizes previous studies on the harvesting of microalgae by the forward osmosis process.

Table 1. Previous studies on harvesting microalgae using forward osmosis

Process	Type of microalgae	Membrane	Mode	Feed Volume (L)	DS	Concentration Factor (CF)	Ref.
Forward osmosis	Chlorella vulgaris	Aquaporin-based polyether sulfone (PES)	AL-FS	0.5	Seawater	4	(Munshi et al., 2018)
Forward osmosis	Scenedesmus obliquus	Cellulose triacetate (CTA)	AL-FS	1	Brine solution	3	(Larronde-Larretche & Jin, 2016)
Forward osmosis	Chlorella vulgaris	Cellulose triacetate (CTA)	AL-FS	1	Brine solution	3	(Larronde-Larretche & Jin, 2017)
Forward osmosis	Chlorella vulgaris	Thin film composite (TFC)	AL-FS	0.5	Urine	1.7	(Volpin et al., 2019)
Electrically-facilitated forward osmosis	Chlorella vulgaris	Thin film composite (TFC)	AL-FS	0.05	Brine solution	1.5	(Son et al., 2017)

Previous studies revealed the influence of membrane orientation and the availability of spacer have significant on the performance of concentrating microalgae by the FO process (Honda et al., 2015). Active layer (AL) facing FS has resulted in a stable flux and better flux recovery by physical cleaning (Honda et al., 2015). Removing the feed spacer improved the harvesting of algal biomass by 27%, using a saline solution (70,000 ppm) DS in the FO process operating in the FO mode (Larronde-Larretche & Jin, 2016). In another study, the FO process was used for dewatering of Scenedesmus acuminates suspensions utilizing a polyamide thin film composite (TFC) with an enhanced surface shearing force (Ye et al., 2018). Shear force provided by mechanical stirring improved the harvesting of microalgae and the average water flux increased by 57.5% at a stirring speed of 1000 rpm using 23.0 g/L of microalgal suspension FS and 2 mol/L of $MgCl_2$ DS (Ye et al., 2018). Previous studies have investigated the performance of different draw solutions in the FO process, such as seawater (Nguyen et al., 2013), brine from desalination plants (Thabit et al., 2019), electrolytes (i.e., $MgCl_2$ and NaCl) and thermolytic salt ammonium bicarbonate (Achilli et al., 2010). Desalination brine is a promising draw solution for the concentration of algae cells because of abundancy. In addition, the desalination brine has high osmotic pressure, around 54 bars, as a result it induces high osmotic pressure gradient in the process (Singh, 2015).

The diluted brine can be discharged back to the sea with a minimal impact on the environment.

This study evaluates the performance of a hybrid ultrafiltration forward osmosis system for the concentration of algal suspension. The FO process will be used as a post-harvesting process after the ultrafiltration process for microalgae. The effects of different flowrates, feed solution concentration and membrane orientation on harvesting microalgae by the FO process was investigated. The performance of forward osmosis was evaluated by measuring water flux, concentration factor (CF) and reverse solute flux. In addition, the energy consumption of the hybrid system was evaluated.

2. Materials and setup

2.1 Ultrafiltration setup

The ultrafiltration (UF) unit shown in Figure 1 was constructed near the 250 L microalgae raceway tank. The unit consisted of two hollow fiber ultrafiltration modules (UF-1 and UF-2). The hollow fiber within the UF modules was made of polyacrylonitrile. UF-1 and UF-2 units have an effective membrane surface area of 25 and 4 m^2, respectively. The modules were connected to tanks and pumps through a system of interconnected (PVC) pipe fittings and valves. Pump 1 was used for transferring microalgae culture from the raceway tank to the feed tank (concentrate tank), whereas pump 2 and pump 3 served as feed pumps for transferring the culture to the inlet of ultrafiltration modules. The power rating for pump 1 was 1.6 kW, whereas the power rating for pump 2 and pump 3 was 1.6 and 0.75 kW, respectively. In the preliminary step, the microalgae culture was concentrated to a desired volume by UF-1 module. The concentrate obtained from the UF-1 module was further concentrated using the UF-2 module. The concentrated microalgae culture obtained from the UF-2 module was collected and stored in a concentrate tank prior to be used as a feed in the FO process.

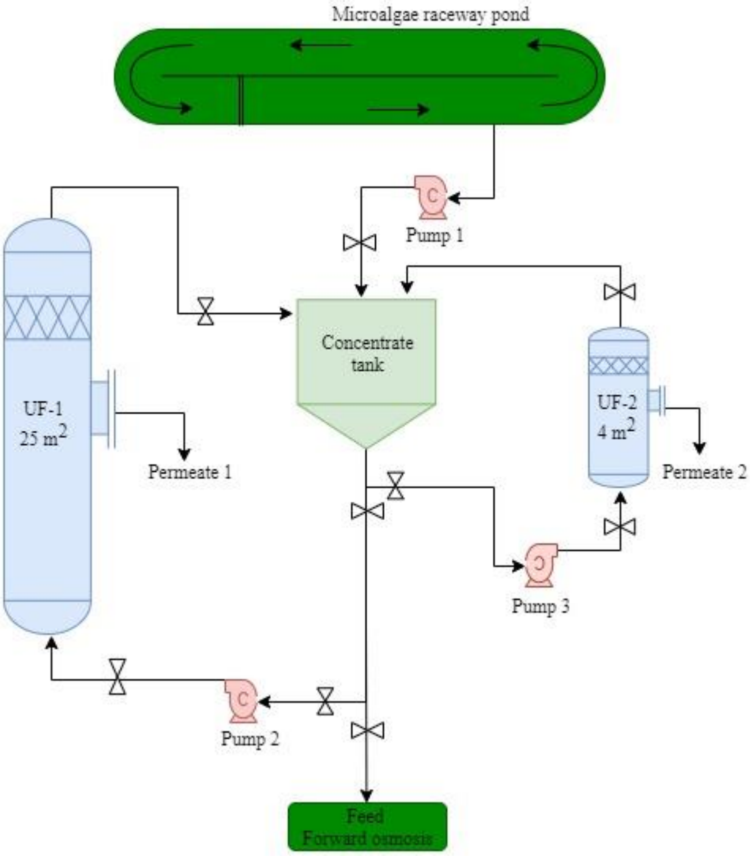

Figure 1. Schematic diagram of the ultrafiltration pilot-scale membrane test skid

2.3 Microalgal cells and growth media

This study used a halotolerant Tetraselmis sp. (Das et al., 2019). Guillard's f/2 medium was used for growing Tetraselmis sp. The used nutrients were of analytical grade except for nitrogen, therefore, commercial-grade urea with 46% of nitrogen was added. Seawater samples were collected from Al Thakhira beach in Qatar and sterilized with a commercial grade 0.02% bleach and kept for one day before using. The used biomass consisted of algae mainly because the algae samples were collected from an open raceway pond where the bacteria growth is minimal. Algal culture samples were checked under a microscope on a regular basis where little or no bacteria contamination was observed. The initial characteristics both algal and ultra-filtered concentrated algae are summarized in Table 2.

Table 2. Initial characteristics of the original algae and the ultra-filtered algae.

Parameter	Original Algae	Ultra-filtered Algae	Standard method
Temperature (℃)	21.2±0.1	22.1±0.1	APHA 2550 Temperature
pH	8.2 ± 0.03	8±0.02	APHA 4500-H+ B. Electrometric Method
Salinity (ppt)	67.68±0.01	64.5±0.02	APHA 2520 B. Electrical Conductivity Method

2.3 Forward osmosis setup

A schematic diagram of the FO setup is shown in Figure 2. CF042 Delrin flat sheet forward osmosis membrane cell was purchased from Sterlitech. The cell dimensions are 12.7 x 8.3 x 10 cm, and the active inner dimensions are 4.6 x 9.2 cm and a slot depth of 0.23 cm. The membrane separated the feed and draw solutions inside the cell. The feed and draw solutions were supplied by two tanks with a capacity of 6 L each. The feed and draw solution were circulated through the membrane cell using two Cole-Parmer gear pumps. The flow rate of the feed and the draw solutions were measured using two flow meters (Sterlitech Site Read Panel Mount Flow Meter). A digital balance (ICS-241 Mettler Toledo) was used to measure the water flux in the FO system. A known quantity of 2 L was used for the feed and draw solution at the beginning of the experiment. The solutions going out from the FO cell were recycled back into the same tanks. Each experiment was running for 2800 min. After 24h of operation, the membrane was washed using distilled water for 30 min to retrieve the initial water flux. A commercial FO membrane (TFC FO membrane, FTSH2O (USA)) was used. Two feed solutions (FS) were used in the FO process namely; microalgae culture with a concentration of 0.43 g/l and ultra-filtered microalgae with a concentration of 15.7 g/l. The draw solution (DS) in the FO system was a concentrated brine from a desalination plant, TDS ~81 g/L. Brine samples were collected from a thermal desalination plant located in Doha, Qatar. The characteristic of brine is summarized in Table 3.

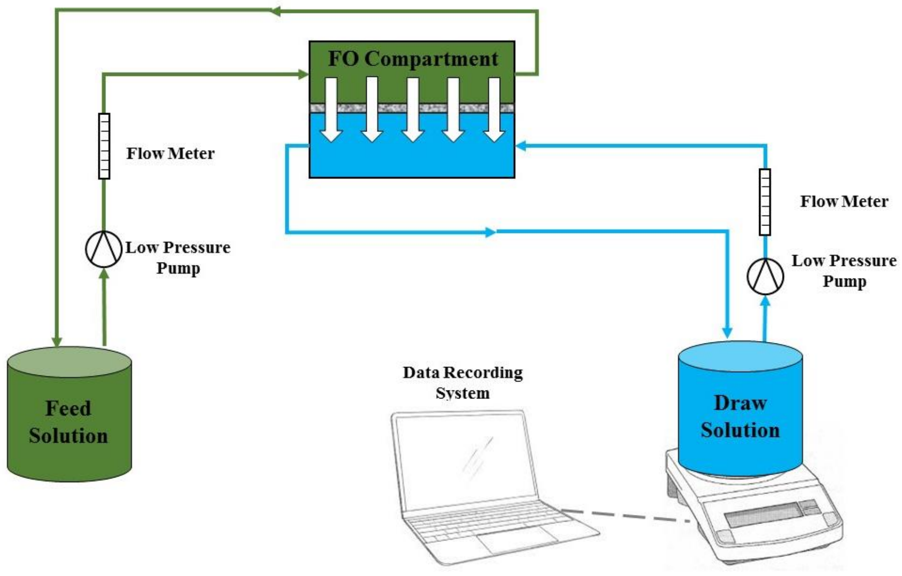

Figure 2. Schematic diagram of the forward osmosis lab-scale membrane test skid

Table 3.Characteristics of brine reject from the desalination process (draw solution)

Parameter	Value	Standard Method
TDS (ppm)	81392 ± 5	APHA 2540 C. Total Dissolved Solids Dried at 180 °C
pH	9.03	APHA 4500-H+ B. Electrometric Method
Turbidity (NTU)	0.25 ± 0.1	APHA 2130 B. Nephelometric Method
EC (mS/cm)	93.6 ± 0.2	APHA 2510 B. Conductivity
Chloride (ppm)	35,266 ± 0.2	APHA 4110 Determination of anions by ion chromatography
Bromide (ppm)	120.3 ± 0.2	
Sulfate (ppm)	5032.2 ± 0.2	
Sodium (ppm)	20982.5 ± 0.2	APHA 3120 Determination of metals by plasma emission spectroscopy
Potassium (ppm)	728.6 ± 0.2	
Calcium (ppm)	723.2 ± 0.2	
Magnesium (ppm)	2511.2 ± 0.2	

3. Results and discussions

3.1 Effect of membrane orientation on water flux

The water flux (J_w) in the FO process was calculated from the following equation:

$$J_w = \left(\frac{V_p}{A_m \times t}\right) \quad (1)$$

Here, V_P is the volume of the permeate (L), A_m is the area of the membrane (m^2), t is the operating time (h). Figure 3 shows the change of water flux with time in the PRO (AL-DS) and FO (AL-FS) modes. Figure 3 shows a gradual decrease in the water flux over time due to membrane fouling, dilution of the DS, and concentration polarization. Algae cells contain lipids, proteins, and carbohydrates, which could accumulate on the surface of the membrane and cause fouling. In the PRO mode, the initial water flux was 9.52 LMH and decreased by 80% after 24 h. After washing the membrane with distilled water for 30 minutes, the water flux was retrieved to 9.51 LMH (i.e. almost 100%), then decreased by 83% at the end of the experiment. In the FO mode, the initial water flux was 14.3 LMH and decreased by 88% after 24 h. After washing with distilled water for 30 minutes, the retrieved water flux was 14.2 LMH (i.e. almost 100%), then decreased by 89% at the end of the experiment. Although the water flux decreased during the FO process in both modes, but it 100% retrieved after washing the membrane with distilled water. This reveals the ability of the FO systems to tolerate biofouling and recover water flux easily after washing. In order to compare the performance of PRO and FO modes, the average water flux was calculated.

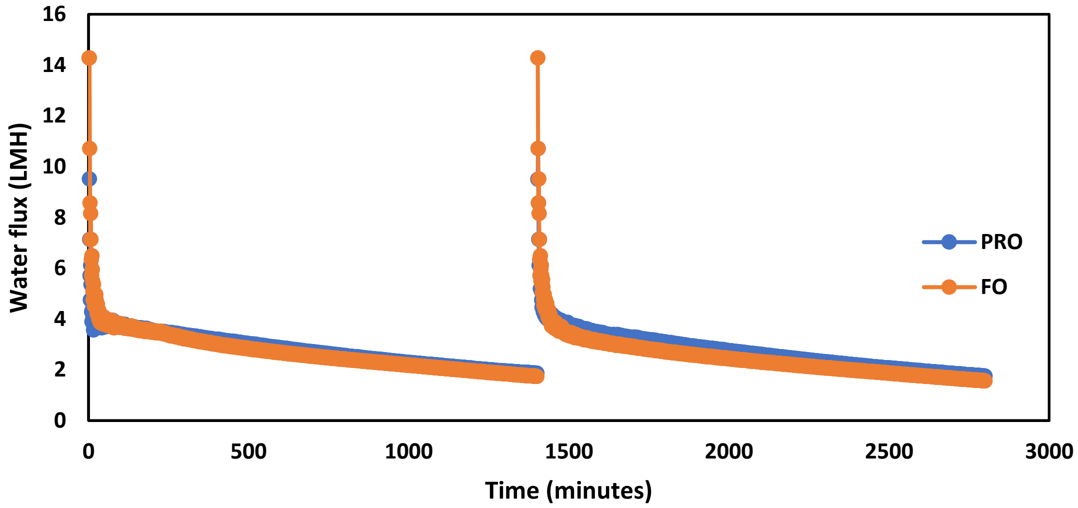

Figure 3. Water flux using algae as the feed solution and brine as the draw solution in FO mode (active layer facing feed solution) and PRO mode (active layer facing draw solution)

Figure 4 shows the average water flux for PRO and FO modes using a flow rate of 2.5 LPM for both the feed solution and the draw solution. In the FO mode, the average water flux during the first 24 hours was 2.74 LMH and declined by 9.2% in the second 24 hours after washing. In the PRO mode, the average water flux was 2.82 LMH in the first 24 hours and declined by 2.2% in the second 24 hours after washing. The average water flux in the PRO mode was higher than the FO mode, which is in good agreement with previous studies (Honda et al., 2015; Mi & Elimelech, 2008; Wang et al., 2010). The reason is that the effect of dilutive concentration polarization at the draw solution side can be mitigated in the PRO mode. The denser and smoother surface of the active layer can improve the fluid shear stress around the membrane surface to reduce diffusion of salt into the membrane and accumulation of salt on the membrane, therefore, reduce both external concentration polarization and internal concentration polarization (Mi & Elimelech, 2008; Valladares Linares et al., 2013; Zhao et al., 2011). As a result, the PRO mode was selected to further evaluate the performance of the FO process.

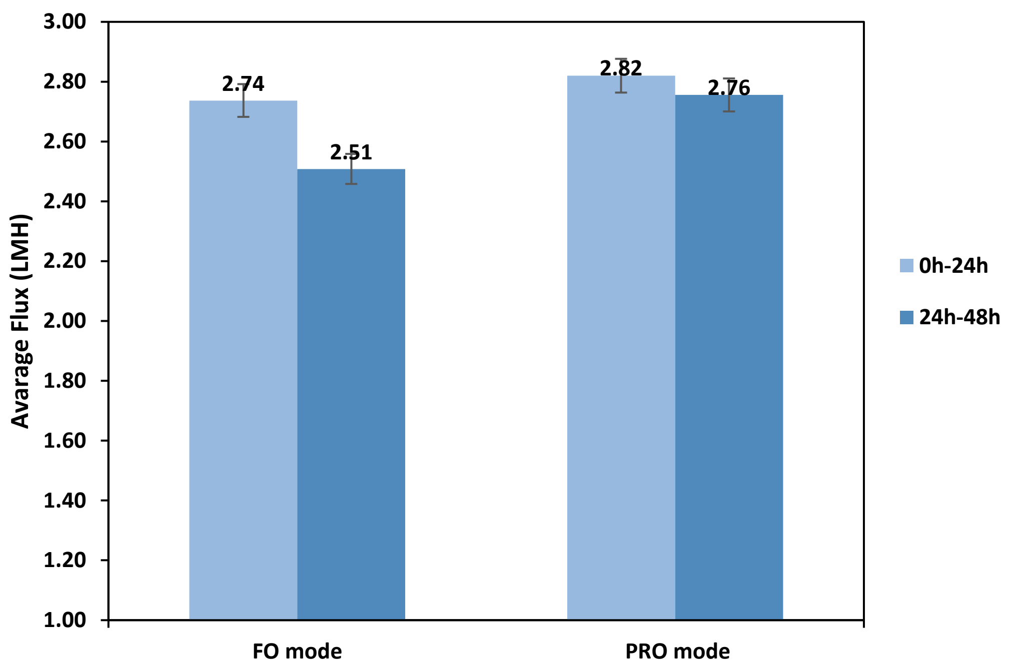

Figure 4. Average water flux using algae as the feed and brine as the draw solution in FO and PRO modes

3.2 Performance of the hybrid ultrafiltration- forward osmosis system

FO performance was evaluated using two different feeds namely: microalgae without ultrafiltration (i.e. concentration of 0.43 g/l) and concentrated ultrafiltered microalgae (i.e. concentration of 15.7 g/l). In the FO process, three different flow rates were used 1.5 LPM for DS and FS (DS:1.5LPM - FS:1.5LPM), 2.5 LPM for DS and FS (DS:2.5LPM – FS:2.5LPM) and 2.5 LPM for FS and 0.8 LPM for DS (DS:0.8LPM – FS:2.5LPM). It is abovious from Figure 5 that the average water flux increased with increasing the flow rate of the DS and FS while using microalgae as a feed solution. The average water flux increased from 1.89 LMH to 2.82 LMH as the flow rates of the draw and the feed solutions increased from 1.5 LPM to 2.5 LPM, respectively. The highest average water flux was 3.06 LMH obtained using a flow rate of 0.8 LPM (DS) and 2.5LPM (FS). The increase of the water flux with the increase of the DS and FS flow rate is due to the minimized concentration polarization effect at higher flow rates (McCutcheon & Elimelech, 2006). Concentration polarization plays a major role in decreasing the osmotic effect across the FO membrane which would decrease the membrane flux (Devia et al., 2015). As the flow rate increase, the turbulence around the membrane surface increases, which in return reduces the effect of concentration polarization and increases the mass transfer coefficient. Furthermore, the increase of turbulence flow at high flow rates would reduce fouling materials deposition on the membrane surface. Explain the applied pressure of 0.5 bar. As shown in Figure 5, the average water flux was almost constant when using concentrated algae as the feed solution. The average water flux was around 1.43 LMH at the different studied flow rates for the feed and draw solutions. This could be due to the constant concentration polarization effect when using concentrated algae as the feed solution. It can be also seen from Figure 5 that at a flow rate of 1.5 LPM for both the feed and the draw solution, the average water flux decreased by 25% when using the concentrated microalgae compared to the unfiltered microalgae. When the flow rate increased to 2.5 LPM for both the feed and the draw solution, the average water flux decreased by 48% when using the concentrated microalgae compared to the unfiltered microalgae. At a flow rate of 2.5 LPM (FS) and 0.8 LPM (DS), the average water flux decreased by 55% when using the concentrated microalgae compared to the unfiltered microalgae. This is due to the fact that the concentrated microalgae has higher density and salinity. The higher density caused more fouling of the membrane and the higher salinity decreased the osmotic pressure gradient (i.e. the driving force) in the FO process.

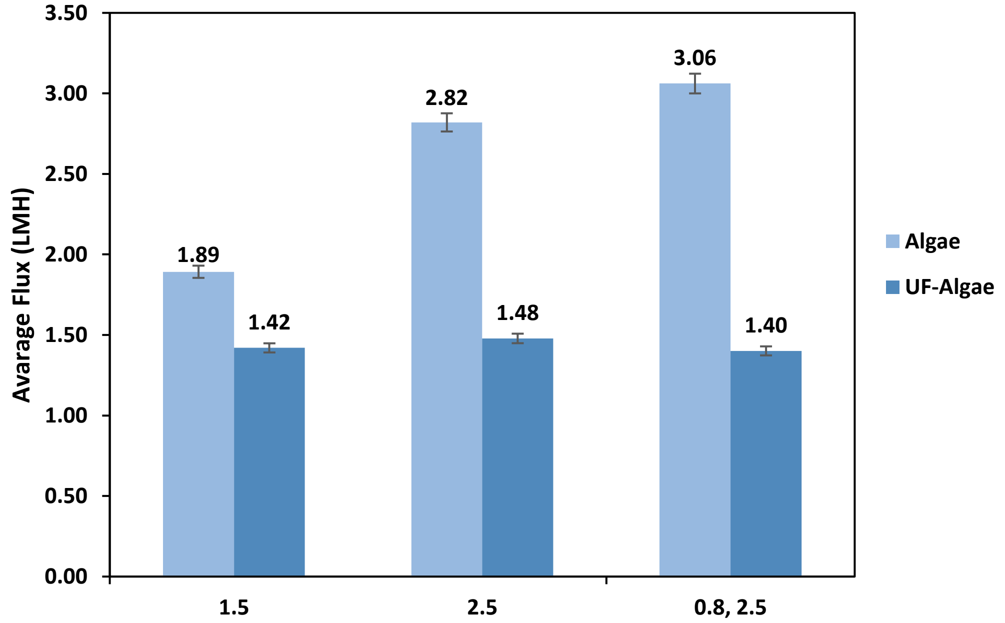

Figure 5. Average water flux of the FO process using microalgae and ultra-filtered microalgae at different flow rates

3.2.1 Concentration Factor (CF)

The main factor used to assess the harvesting process of microalgae is the concentration factor (CF). The CF was calculated using the following equation:

$$CF = \frac{C_f}{C_i} \quad (2)$$

Here, C_f and C_i are the final and initial concentrations of the algae biomass, respectively. Indirectly, the magnitude of CF depends on water flux, membrane area, feed volume and operating period. CF tends to increase with higher water flux, longer harvest durations, less feed volume, and larger membrane area. Figure 6a shows the concentration factor for forward osmosis using unfiltered microalgae as a feed with three different flow rates. It can be seen from Figure 6a that the CF slightly increased as the flow rate increased. The CF increased by 4.9% as the flow rate increased from 1.5 LPM to 2.5 LPM for both the feed and the draw solutions. The highest CF was almost 1.23 obtained using a flow rate of 2.5 LPM (FS) & 0.8 LPM (DS), which is attributed to the highest water flux. The total concentration factor of the hybrid process was also calculated by adding the concentration factors of each process (Figure 6(b)). In the first pass ultrafiltration process, the CF was 4.76. During the second pass ultrafiltration process, the concentration factor decreased significantly to 1.12. In the FO process, the concentration factor was 1.05 using the concentrated microalgae as feed and a flow rate of 1.5 LPM for both the feed and the draw solutions. The CF increased to 1.11 using a flow rate of 2.5 LPM (FS) & 0.8 LPM (DS). The overall concentration factor of the dual pass ultrafiltration process was 5.88 and the total concentration factor increased to 7.0 using forward osmosis. In ultrafiltration, the harvesting efficiency decreased significantly by 76.4% when the concentrated algae was the feed solution instead of microalgae. In the FO process, the harvesting efficiency slightly decreased by 9% when using the concentrated algae instead of microalgae. Single-pass ultrafiltration has been proven an effective technology for harvesting microalgae, given that the CF of ultrafiltration was four times higher than forward osmosis. However, forward osmosis and second pass UF showed similar efficiency for harvesting microalgae. To evaluate the feasibility of each process, the energy consumption was calculated.

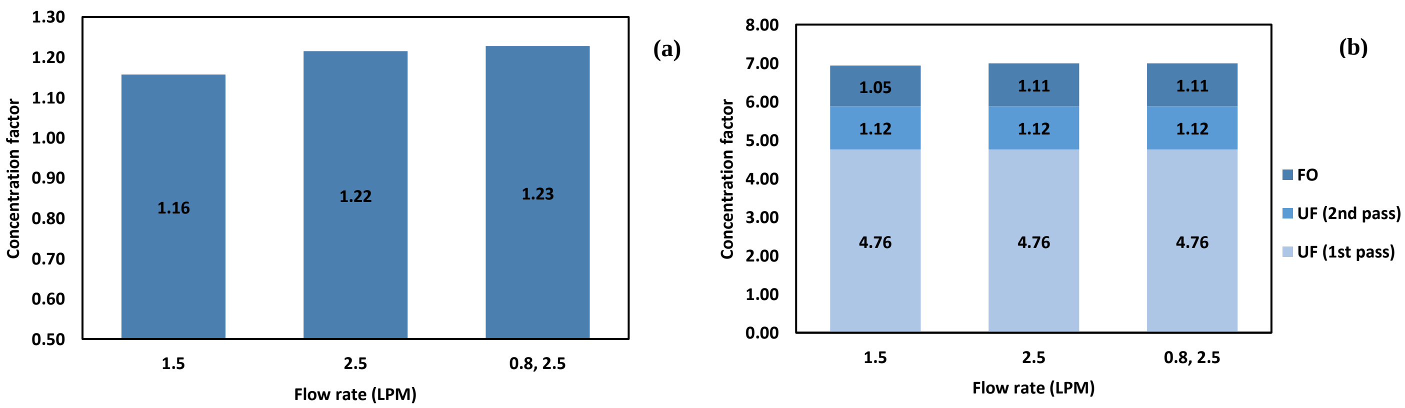

Figure 6. Concentration factor of FO and UF processes at different flow rates using different feed: (a) Forward osmosis using microalgae as feed (b) Microalgae UF 1st pass (concentration = 0.43 g/l), UF 2nd pass (concentration = 15.7 g/l) and FO (concentration = 15.7 g/l).

3.2.2 Specific Energy Consumption

The specific energy consumption of the UF process and FO process have been calculated using equation 3 (Shrivastava & Stevens, 2018) and equation 4 (Lambrechts & Sheldon, 2019) , respectively:

$$E_{s-UF} = \left(\frac{P}{n \times \%R}\right) \quad (3)$$

$$E_{s-FO} = \left(\frac{(Q_s)(\rho)(g)}{36 \, . \, (Q_p)}\right) \quad (4)$$

Here, P is the applied feed pressure (bar), *n* is the pump efficiency, %R is the recovery rate, Q_s is the flowrate of feed and draw solution (m^3/h), Q_s is the flow rate of the permeate (m^3/h), ρ is the density (g/L) and g is the gravitational acceleration (m/s^2). Figure 7a shows the specific energy consumption of the FO process using microalgae as the feed and different flow rates. In the FO process, the specific energy consumption increased from 0.56 Kwh/m^3 to 0.72 Kwh/m^3 at a flow rate of 1.5 LPM and 2.5 LPM, respectively. As shown in Equation 4, the specific energy is a function of FS and DS flow rate, and the permeate flow rate. The low specific energy consumption at such a low flow rate was due to the low pumping power requirements. As the flow rate increased, the specific energy increased because the pumping power increased and the permeate flow rate remained almost constant. Figure 7b shows the total energy consumption of the UF-FO hybrid process. The energy consumption of the first pass ultrafiltration was 4.87 Kwh/m^3. In the second pass ultrafiltration, energy consumption increased by 5.6%. The energy consumption of FO was 0.88 – 1.25 Kwh/m^3 depending of the flow rate of FS and DS. The energy consumption of FO increased as the flow rate increased due to the higher pumping power requirements. The energy consumption of ultrafiltration was four times higher than forward osmosis. The total specific energy consumption was calculated by adding the energy consumption of each process (Figure (b)). The energy consumption of dual-pass UF was 10 Kwh/m^3. However, the energy consumption of the hybrid UF-FO process was 6.12 Kwh/m^3 using a flow rate 2.5 LPM. While achieving the same harvesting efficiency, the energy consumption can be reduced by 46% using a hybrid UF-FO process instead of dual-stage UF.

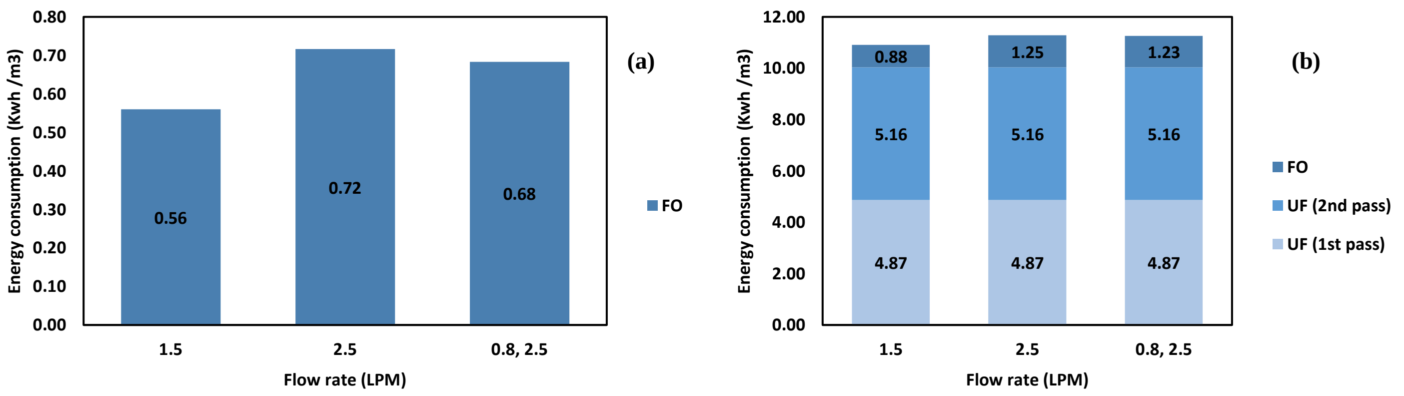

Figure 7. Energy consumption of FO and UF processes at different flow rates using different feed: (a) Microalgae culture (biomass concentration = 0.43 g/l) (b) Microalgae UF 1st pass (biomass concentration = 0.43 g/l), UF 2nd pass (biomass concentration = 15.7 g/l) and FO (biomass concentration = 17.6 g/l)

3.2.3 Reverse solute flux

Reverse solute flux (J_s) is the back diffusion of the draw solute across the FO membrane to the feed solution. RSF must be considered in the FO studies because it might contaminate the feed solution. RSF is calculated using the following equation.

$$J_s = \frac{(C_f V_f) - (C_o V_o)}{t \times A_m} \qquad (3)$$

Here, C_f and V_F are the concentration and feed volume at the end of the experiment, C_o and V_o are the concentration and feed volume at the begging of the experiment. It is obvious from Figure 7, while using microalgae as a feed solution, the RSF flux increased as the flow rate increased. The RSF increased from 0.158 g/m^2.h to 0.261 g/m^2.h as the flow rates of the draw and the feed solutions increased from 1.5 LPM to 2.5 LPM, respectively. At flow rate of 2.5 LPM (FS) - 0.8 LPM (DS), the RSF was 0.170 g/m^2. h. When using ultrafiltered microalgae as the feed solution, the RSF was around 0.111 g/m^2. h at all studied flow rates. Compare with the initial concentration and no contamination of the feed. If the concentration of feed solution is 67 ppt in table 1 (that is 67000 ppm), then the effect of salt back diffusion from DS to FS would be negligible isn't it?

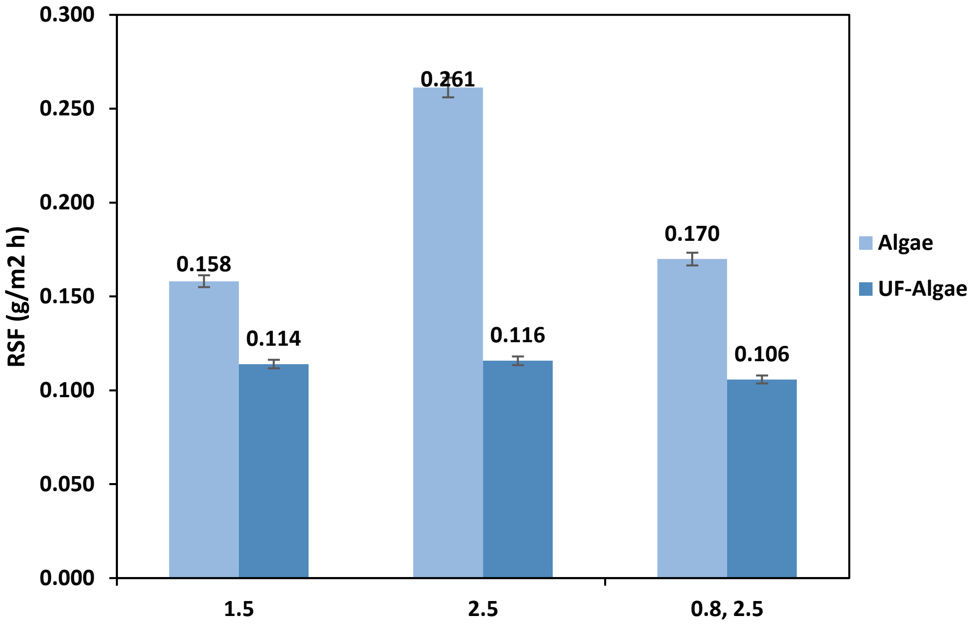

Figure 7. Reverse solute flux of FO process using microalgae and ultra-filtered microalgae at different flow rates

4. Conclusions

In this study, a hybrid ultrafiltration – forward osmosis process was investigated for the enhancement of marine microalgae harvesting. Forward osmosis (FO) was used as a post-harvesting process after ultrafiltration (UF) to attain higher harvesting efficiency and reduce the energy consumption. The FO process exhibited high resistance to fouling where the water flux was completely retrieved after washing the membrane with distilled water for 30 minutes. The average water flux obtained using PRO mode was higher than FO mode. In general, the increase of flow rate increased the average water flux. The increase in flow rate from 1.5 to 2.5 LPM enhanced the average water flux by 33 %, while using microalgae as feed. However, the average water flux was unaffected by the flow rate while using pre-concentrated algae as feed solution, where the average water flux was around 1.40 LMH at different flow rates.

Concentration factors of 4.76 and 1.12 were obtained using first pass ultrafiltration and second pass ultrafiltration, respectively. Applying the FO process increased the concentration factor by 1.05 – 1.23, depending on the feed solution and the flow rates of the feed and the draw solutions. The energy consumption of ultrafiltration was four times higher than forward osmosis, where the energy consumption of the second pass ultrafiltration and forward osmosis were 5.26 and 1.25 Kwh/m^3, respectively. While the harvesting efficiency of forward osmosis and the second pass ultrafiltration were almost the same with a CF of almost 1.12. Forward osmosis was found to be an energy efficient post-harvesting process for microalgae. A maximum total algal harvesting CF of 7.0 was obtained using ultrafiltrated algae as feed, FO mode, 2.5 LPM flowrate ……… and 48 hours operation time. It was found that the energy consumption can be reduced by 46% using a hybrid UF-FO process compared to a dual-stage UF.

Acknowledgments

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