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Submerged module of outer selective hollow fiber membrane for effective fouling mitigation in osmotic membrane bioreactor for desalination

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Abstract

This paper investigated the membrane fouling mitigation efficacy and performance of a home-made submerged module containing outer selective hollow fiber thin film composite forward osmosis (OSHF TFC FO) membrane in osmosis membrane bioreactor (OMBR) system treating municipal wastewater for desalination. Initial tests, optimization of draw solution flowrate and pumping mode for the submerged module were carried out before it was applied into the OMBR system. Overall, the OMBR system exhibited high and stable performance with initial water flux of approximately 6.3 LMH using 35 g/L NaCl as draw solution, high removal efficiencies of bulk organic matter and nutrients. Moreover, membrane fouling was effectively mitigated with slow rate of flux decline during 33-day operation of the OMBR system. These results indicated that the submerged membrane module of OSHF TFC FO membrane has stable and reliable performances making it suitable for OMBR supplication without the need of air scouring to prevent membrane fouling.

Keywords: *Osmotic membrane bioreactor; Outer-selective hollow fiber; Forward osmosis membrane; Membrane fouling; Submerged membrane module.*

1 Introduction

Water scarcity has become a critical issue and hence a global challenge. Desalination of saline water (e.g. seawater, brackish water) and wastewater reclamation and reuse are considered reliable and viable solutions to alleviate the severity of water shortage [1] and membrane-based technologies have played a significant role in providing additional water resources to supplement scarce freshwater resources. Reverse osmosis (RO) is the most widely used membrane-based process for both desalination and water reclamation for reuse [2]. Albeit significant improvement has been made to enhance the performance of RO process, RO desalination technology still has major challenges such as membrane fouling [3, 4], high capital cost, energy consumption [5], and environmental impacts of brine discharge [6, 7]. These challenges are hampering the wider reach of the RO technology to and hence efforts are required to find more-effective solutions to mitigate of membrane fouling, reduce energy consumption, and sustainable brine management strategies.

Another emerging membrane-based technology for wastewater treatment and reuse has recently attracted research interests is osmotic membrane bioreactor (OMBR). This is an integrated system of forward osmosis (FO) process and conventional activated sludge (CAS) for treatment of municipal wastewater [8]. The main driving force utilized in OMBR system is the osmotic pressure difference between the low salinity wastewater in the bioreactor (low osmotic pressure) and the highly concentrated draw solution (DS) (much higher osmotic pressure) across a semi-permeable osmotic membrane. Under the osmotic pressure difference, water molecules from the low salinity feed solution (FS) side naturally passes through the highly selective FO membrane towards the DS side thereby diluting the DS. The nonporous osmotic or FO membrane, consisting of a highly selective layer, can provide reliably high rejection of dissolved organics, suspended solids, pathogens, trace organic compounds, and any other contaminants present in the wastewater.

OMBR and RO hybrid system has recently been suggested for simultaneous seawater desalination and water reclamation [9-12]. In the OMBR - RO hybrid system, the OMBR process serves as a very high-quality pre-treatment step, reclaiming water from wastewater to dilute seawater or highly concentrated brine while the RO process works as a post treatment to re-concentrate DS and to produce purified water. Owing to the excellent rejection properties of forward osmosis (FO) membrane, effectively retaining almost pollutants and foulants contained in the contaminated wastewater, OMBR process usually provides high quality pre-treated water for RO process. The integration of OMBR process, therefore, can significantly reduce membrane RO membrane fouling reported severe in most wastewater recycling plants. The dilution of seawater or concentrated brine by reclaimed water also lowers the osmotic pressure of feed solution, thereby reducing the specific energy consumption and enhances water recovery of the seawater RO desalination process. Additionally, RO brine can be recirculated in the closed loop of the OMBR-RO hybrid system serving as the DS for OMBR process, which is a sustainable brine management strategy to minimize the cost and environmental consequence of direct disposal of RO brine [13].

Although FO process is generally considered a low-fouling process, membrane fouling in the OMBR process still can be a critical issue to its operation. Membrane fouling might lead to membrane deterioration, membrane lifetime reduction and water quality decline, significantly increasing the operational and maintenance costs [14]. Membrane fouling mitigation strategies are therefore essential to minimize the consequences of membrane fouling. A number of fouling control methods have been developed including optimization of the system's operating conditions, utilization of new membrane having enhanced anti-fouling property, and development of proper membrane cleaning protocols. Additionally, employment of suitable membrane and module configurations can also be another method to mitigate membrane fouling in OMBR system [15].

Regarding membrane configuration, while many recent research studies have investigated the performance of the OMBR system using flat-sheet plate-and-frame configuration using thin film composite (TFC) and/or cellulose triacetate (CTA) FO membranes [12, 14, 16-28], a few OMBR studies were conducted using hollow fiber FO membrane. Zhang and his colleagues (2015) used a home-made hollow fiber TFC FO membrane having a polyamide selective layer coated in the lumen side of the hollow fiber [29]. The utilization of this inner selective hollow fiber (ISHF) TFC FO membrane in OMBR system poses many serious challenges in terms of membrane fouling control under both membrane orientations including active layer (AL) facing activated sludge (AL – FS) and active layer facing DS (AL – DS). Under the AL – FS membrane orientation mode, the activated sludge flows inside the micro-size hollow fiber membrane with very high potential of fiber blockage and clogging with the sludge or mixed liquor suspended solids (MLSS). Besides AL-FS mode cannot be operated under submerged conditions but has to be operated as an external skid to the activated sludge bioreactor. On the other hand, under the orientation of AL – DS mode, membrane support layer with large-size pores is in direct contact with various foulants and particulates in the activated sludge, resulting in severe irreversible fouling in the pores of support layer. Hence, membrane fouling mitigation under both membrane orientations will be a serious challenge for the OMBR system using ISHF membrane.

To effectively mitigate the membrane fouling in OMBR system, outer selective hollow fiber (OSHF) TFC FO membrane with a polyamide selective layer on the outer surface of hollow fiber is highly desirable for submerged OMBR process. OSHF TFC FO membrane has the edge over the ISHF one including ease of membrane fouling mitigation, low propensity of membrane fouling, and larger effective surface area per unit [26, 30-33]. Another OMBR study has recently been conducted to investigate the efficiencies of fouling mitigation methods using a side-stream module of a home-made OSHF TFC FO membrane [34]. As for the OMBR system using a side-stream (as external skid) membrane module, an additional feed pump is required to circulate the MLSS or FS through the membrane module. This flow circulation of MLSS not only results in additional operation cost but also can have a significant impact on the biological activities of the bioreactor. Kim, Lee and Chang [35] (2001) proved that strong shear generated by the pumps during the recirculation of activated sludge gives rise to serious microbial flocs breakage, lower sludge yield, and reduced overall chemical oxygen demand (COD) removal efficacy of the MBR system.

Submerged FO membrane module is therefore expected to address the challenges of using the side-stream membrane module in OMBR system. Utilization of immersed module eliminates the need of FS pump and minimizes the microbial disturbances [35]. Moreover, submerged OMBR system can take the advantage of air bubbles already used for aeration in the bioreactor of the active sludge process as a mean of air scouring to mitigate membrane fouling and external concentration polarization [24, 36]. Therefore, this study aims to investigate fouling mitigation efficiency of a home-made submerged OSHF-based OMBR system for treating municipal wastewater. For this investigation, the lab-scale OMBR system was operated continuously for a period of 33 days under the AL – FS orientation under submerged condition. The performances of the OMBR system was assessed in terms of FO water flux; salt accumulation in the activated sludge bioreactor and pollutants removal rates. The fouling mitigation efficiency was evaluated according to the flux decline rate over the testing period and flux recovery after the implementation of a fouling control method. Membrane autopsy was then conducted on pristine and fouled membranes with scanning electron microscope (SEM) to obtain the surface and cross-sectional morphologies of fouling cake-layer for a better understanding of the fouling mitigation efficacy.

2 Materials and methods

2.1 *Submerged module of OSHF TFC FO membrane.*

The submerged module and the OSHF TFC FO membranes used in this study were fabricated at Center for Technology in Water and Wastewater, University of Technology, Sydney, Australia. The properties of this membrane was described in our previous studies [34, 37].

2.2 *Optimization of DS flowrate*

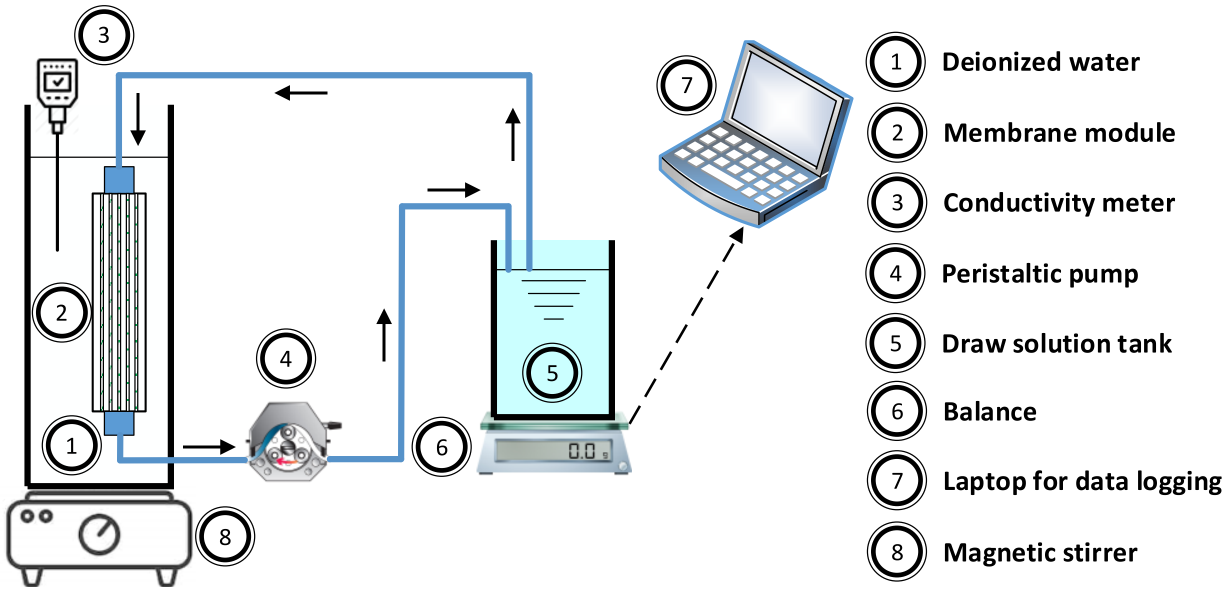

Figure 1. Schematic diagram of a lab-scale FO experiment setup for determination of optimum flowrate of DS stream.

Flowrate of DS stream was firstly optimized using a bench-scale submerged FO testing setup as depicted in **Fig.1.** Various DS flowrates under both sucking and pushing mode were chosen for optimization tests with utilization of deionized (DI) water as FS. Water flux and specific reverse solute flux (SRSF) are two main parameters used for determination of the optimum flowrate of DS stream. The testing conditions are presented in **Table 1**.

Table 1 Testing conditions for determination of the optimum DS's flowrate.

No.	**Feed solution**	**Draw solution**	**Draw solution stream**		**Orientation**	**Testing time (minutes)**
			Flowrate (ml/min)	Mode		
1	DI water	35 g/L	20	Pushing	AL - FS	60
2	DI water	35 g/L	20	Sucking	AL - FS	60
3	DI water	35 g/L	40	Pushing	AL - FS	60
4	DI water	35 g/L	40	Sucking	AL - FS	60
5	DI water	35 g/L	60	Pushing	AL - FS	60
6	DI water	35 g/L	60	Sucking	AL - FS	60
7	DI water	35 g/L	80	Pushing	AL - FS	60
8	DI water	35 g/L	80	Sucking	AL - FS	60

2.3 *The bench-scale submerged OMBR system*

This research work used a lab-scale OMBR system with submerged membrane module as depicted in **Fig. 2**.

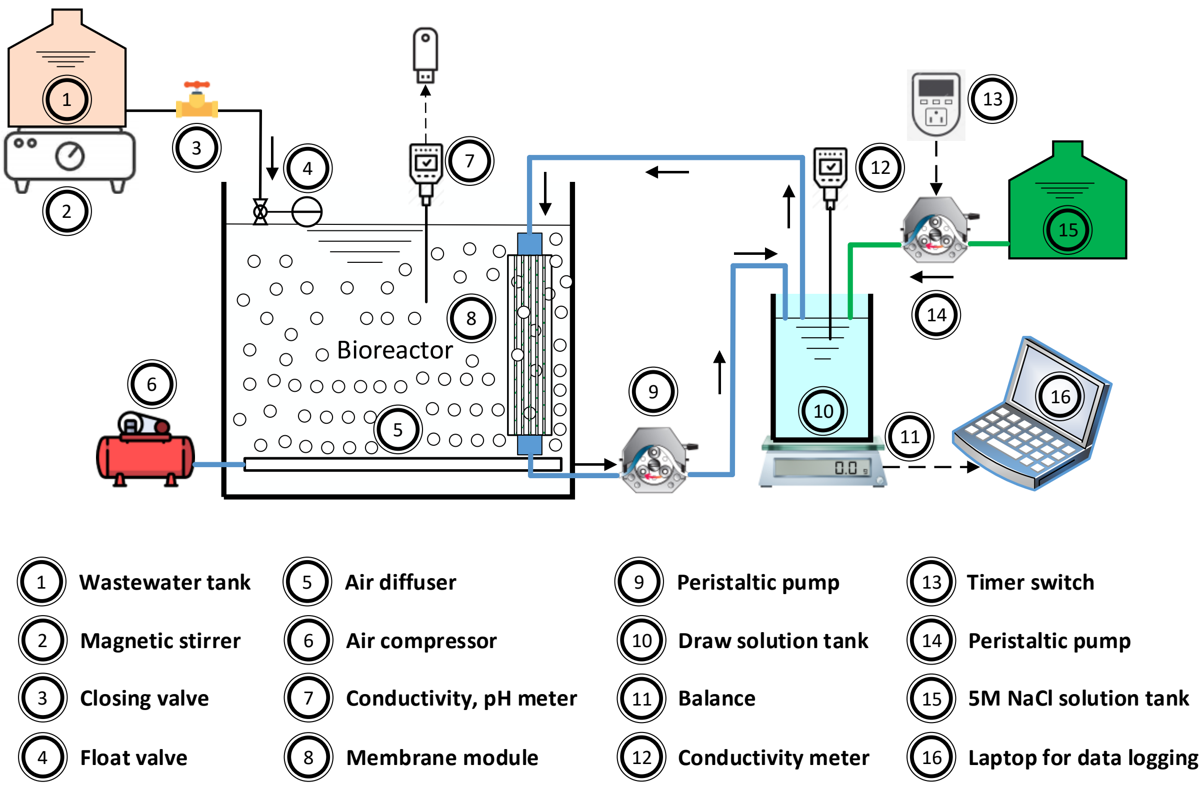

Figure 2. Schematic diagram of the lab-scale immersed OMBR system setup.

2.4 *Draw solution and synthetic wastewater.*

All the chemicals used were of reagent grade supplied by Merck, Australia. Sodium chloride (NaCl) having concentration of 35 g/L was used as DS. Synthetic wastewater influent was continuously supplied into the OMBR system, containing 60 mg/L $(NH_4)_2SO_4$, 10 mg/L $FeSO_4$, 15 mg/L KH_2PO_4, 300 mg/L glucose, 30 mg/L urea, and 50 mg/L yeast. This synthetic influent contained 106.5 ± 5.8 mg/L total organic carbon (TOC), 18.0 ± 1.2 mg/L ammonium (NH_4^+), 27.3 ± 2.2 mg/L total nitrogen (TN) and 3.3 ± 0.2 mg/L phosphate (PO_4^{3-}).

2.5 *OMBR system operation*

This submerged OMBR system utilized the acclimatized activated sludge that was obtained from the recycle water facility at Central Park, Sydney. Acclimatization of the seed activated sludge was continuously conducted over 6 months until the system achieved a stable TOC removal efficacy of more than 90%. Before the OMBR system starts operating, suspended solids concentration in the mixed liquor was adjusted to 8.0 g/L. The synthetic wastewater influent was continuously fed into the OMBR system using a floating valve to maintain the water level in the reactor. To maintain the dissolved oxygen concentration in the bioreactor at a level of more than 3 mg/L for microorganisms, aeration with an intensity of 3 L/min was supplied by an air diffuser (Aqua One, Australia). Regular monitoring of the pH and salinity of the activated sludge in the reactor was performed, during the operation of the OMBR system, using a portable pH and conductivity meter - HQ40D (HACH, Germany). A peristaltic pump (Masterflex, Cole-Parmer, USA) was used to recirculate DS from DS tank through the lumen side of hollow fiber membrane. DS concentration during the testing period was maintained at 35 ± 1 g/L by regularly adding a highly concentrated NaCl solution (5 M) into the DS tank. The regular supplementary of DS was performed by a peristaltic pump which is connected to a programmable timer switch operating based on the monitored DS concentration in the DS tank. All experiments were conducted in a laboratory with a highly controlled environment with an ambient temperature of $22 \pm 1\,^{\circ}$C.

A two-step sequential chemical cleaning was also conducted using two types of cleaning agents including: 0.1% sodium hydroxide (NaOH) + 0.1% sodium dodecyl sulfate (SDS) (w/v) providing an alkaline solution (pH = 12); and an acidic solution comprising of 2% citric acid (w/v) (pH = 3). Chemicals used in this section were at reagent grade and used as received from Sigma Aldrich (Australia). A protocol of chemical cleaning strategy is detailed herein below:

Step 1: The submerged membrane module was placed in a tank filled with DI water and an aeration with an intensity of 3 L/min was introduced to the tank to partially remove fouling cake layer. The tank was then drained off after washing time of 15 minutes.

Step 2: The tank was then filled up with the alkaline solution, this washing step was carried out for 1 hour without aeration. After 1 hour of alkaline washing, the solution was drained off.

Step 3: Step 1 was repeated to remove residual alkaline solution. After 15 minutes of cleaning, the solution in the tank was removed.

Step 4: The tank was filled with acidic solution for 1 hour, no aeration was used in this step. The acidic solution was then drained off at the end of this cleaning step.

Step 5: Step 1 was again repeated to remove the residual of acidic solution before putting the membrane module back to the normal operation.

2.6 ***Analytical methods***

2.6.1 Determination of water flux and specific reverse solute flux

Water flux - J_w (L/m^2 h - LMH) was calculated by Equation [1]:

$$J_w = \frac{\Delta V}{A_m \times \Delta t} \qquad [1]$$

Where: A_m (m^2) is an effective area of FO membrane; Δt (h) is time interval; ΔV is the net volume change of DS solution (L)

When DI water is used as FS, reverse solute flux - J_s (g/m^2 h – gMH) was calculated using Equation [2]:

$$J_s = \frac{\Delta V \times \Delta C_t}{A_m \times \Delta t} \qquad [2]$$

Where: ΔV and ΔC_t are the net changes in the FS volume (L) and FS's salt concentration; Δt (h) and A_m (m^2) are same as in Equation [1].

Subsequently, specific reverse solute flux (SRSF) was determined by Equation [3]:

$$SRSF = \frac{J_s}{J_w} \qquad [3]$$

The experimental water flux J_w (L/m^2 h - LMH) was determined using following equation:

$$J_w = \frac{\Delta V}{A_m \times \Delta t} \quad (1)$$

Where: ΔV is the net increase in the diluted DS volume (L) recorded by a balance over a time interval Δt (h) using an effective area of FO membrane A_m (m^2).

2.6.2 Determination of water quality parameter and pollutants removal efficiency

COD, mixed liquor suspended solids (MLSS), and mixed liquor volatile suspended solids (MLVSS) were measured according to the standard methods for the examination of water and wastewater [38]. Measurement of dissolved oxygen (DO) was carried out using a DO meter supplied by Vernier, USA. Samples were regularly collected from the synthetic wastewater tank, the reactor, and the DS tank for analysis of basic pollutants concentrations. Measurement of TOC concentrations in the collected samples were carried out using a TOC analyzer Analytikjena Multi N/C 2000. Concentrations of NH_4^+, TN, and PO_4^{3-} were measured using corresponding test kits and photometer - Spectroquant, NOVA 60 (Merck, Australia). In order to attain accurate analytical values, samples were pretreated, followed by dilution if necessary to minimize the interference of chloride and ensure the proper range of analytes.

2.6.3 Membrane and fouling cake layer characterization

Virgin membrane and fouled membrane samples were collected at the end of each experiment for morphological structures and elemental composition analysis. Samples were dried at room temperature before being coated with gold in a high vacuum sputter coater (EM ACE600, Leica). Subsequently, membrane samples were analyzed in a field emission scanning microscope and energy diffusive X-ray (EDX) analyzer (FE-SEM, Zeiss Supra 55VP, Carl Zeiss AG).

3 Results and discussion

3.1 *Optimum draw solution flowrate.*

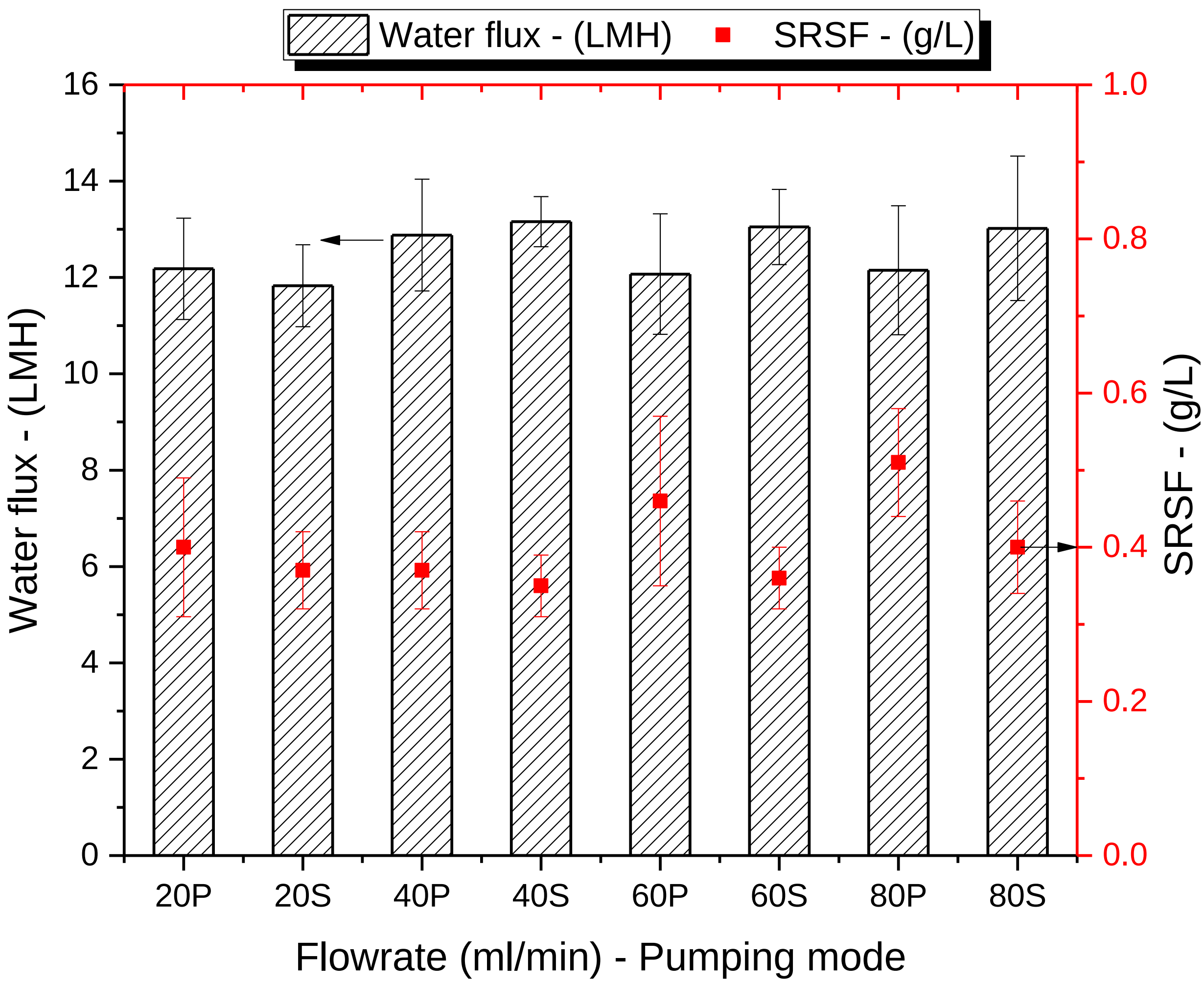

Figure 3. Effects of different DS's flowrates and pumping modes (sucking and pushing) on FO performance (J_w and SRSF). Testing conditions: FS = DI water; DS = 35 g/L NaCl; Temperature = ambient temperature (22 ± 1℃); AL – FS orientation.

Fig. 3 presents J_w and SRSF values of the immersed module of OSHF TFC FO membrane at different DS's flowrate using DI water as FS for the purpose of optimization. Although there was an insignificant difference in the J_w and SRSF values between different DS flowrates, feed flow circulation in suction mode produced slightly better performances with higher J_w and lower SRSF compared to that of pushing mode of FS circulation at same flowrate. J_w slightly increased from 12.2 LMH to 12.9 LMH and from 11.8 LMH to 13.2 LMH while SRSF faintly decreased from 0.40 g/L to 0.37 g/L and from 0.37 g/L to 0.35 g/L with the increase of DS's flowrate from 20 ml/min to 40 ml/min in pushing mode and sucking mode, respectively. The small increase of Jw is likely attributed to the membrane stretching because of the fluid pressure on the lumen side at higher flow rates. Although the crossflow velocity shear on the support layer side of the membrane usually do not have impact on reducing the diluted internal concentration polarization effects, higher DS flowrates in the lumen side facilitated to push the diluted DS stream out and rapidly replaced with a new concentrated DS stream into the hollow fibres. This might help to reduce the severity of dilutive external concentration polarization (DECP), hence improved Jw. Also, the drop of SRSF can be ascribed to the combined effects of a decrease in RSF and the increase in Jw as DS's flowrates escalated.

A clear difference in the variations of J_w and SRSF was observed as DS's flowrate ascended to 60 ml/min and 80 ml/min between two different recirculation modes. In pushing mode, J_w marginally declined to from 12.9 LMH to 12.1 LMH and SRSF quickly rose to 0.51 g/L while J_w remained stable at 13.0 LMH and SRSF slightly bounced back to 0.4 g/L in suction mode. The lower J_w and higher SRSF under pushing mode of DS supply is likely due to slight reduction in the net driving force due to elevated hydraulic pressure on the DS side of the membrane when operated at higher DS flowrates [39]. On the other hand, under the suction mode of DS flow, the presence of vacuum slightly enhances the net driving force that helps improve the water flux [39]. Among the flowrates evaluated, the highest water flux and lowest SRSF were achieved at DS flowrate of 40 ml/min under the suction mode and this flowrate has been therefore considered as the optimum DS flow rates for the operation of the submerged OMBR system in this particular study.

3.2 *Performance of submerged OSHF TFC FO membrane module in OMBR operation.*

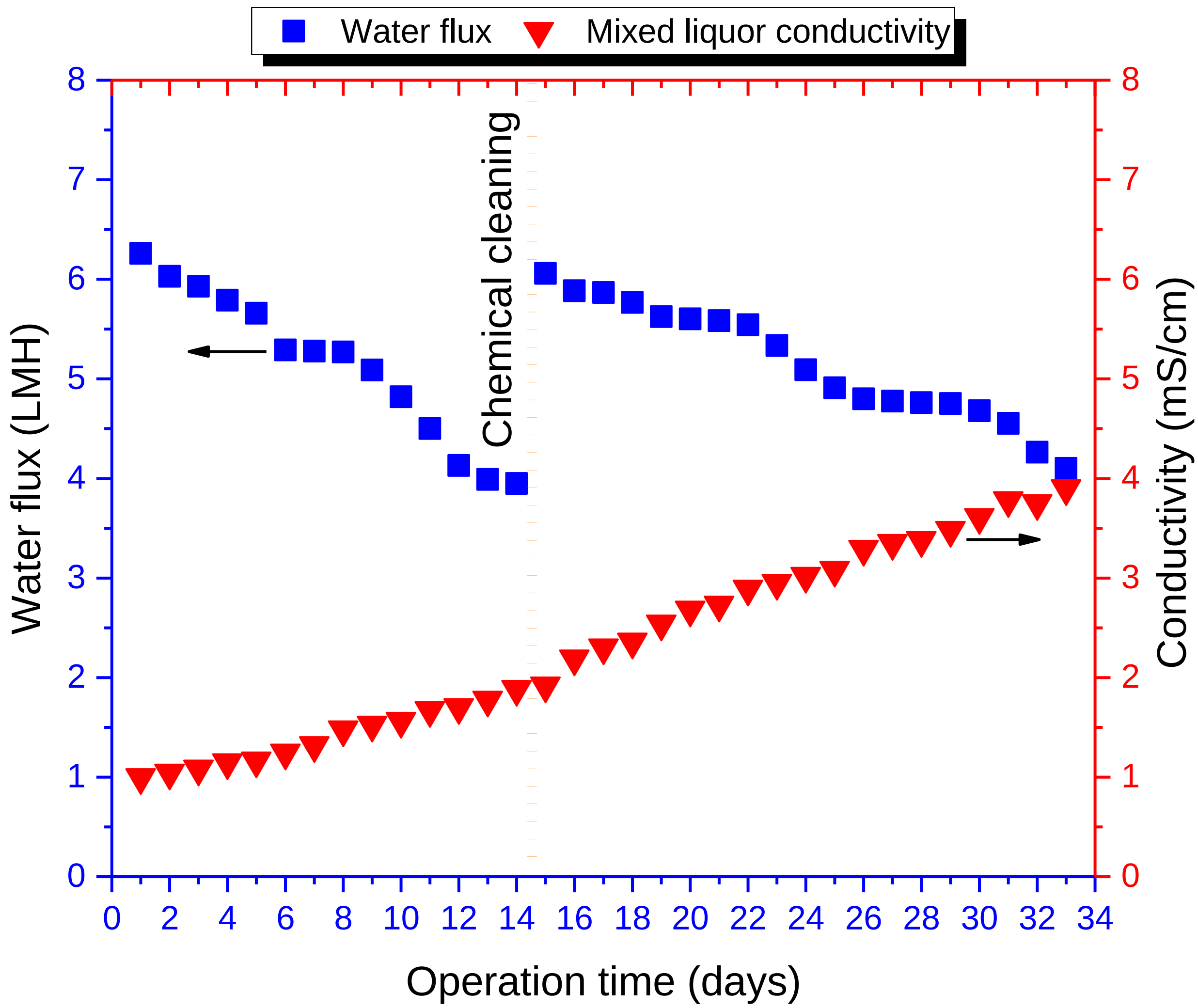

Figure 4. Water flux profile and salinity of activated sludge in the submerged OMBR system over the testing period. Testing conditions: FS = activated sludge; DS = 35 g/L NaCl under suction mode; Temperature = ambient temperature (22 ± 1℃); AL – FS orientation.

Fig. 4 illustrates J_w and mixed liquor conductivity profiles of the submerged OMBR system using the optimum DS flowrate. Similar to previous OMBR studies [24-26], water fluxes produced by the submerged OMBR system gradually and continuously declined while salinity of mixed liquor in the reactor unceasingly increased over the experiment time. Water flux of the OMBR system, initially started at 6.3 LMH, slowly dropped to 4.0 LMH after 14 days of operation. Water flux then recovered to 6.1 LMH after chemical cleaning and then continued to drop to 4.1 LMH at the end of the experiment after 33 days. The flux decline is as a result of the combined effect of the reduction of net osmotic driving force and membrane fouling [12, 40, 41]. Similar result was observed by Pathak., et.al (2018) [26] and this flux drop can be ascribed to severe membrane fouling, forming a fouling-cake layer on the selective layer. This fouling layer works as an additional barrier hindering water permeation through FO membrane. Since the DS concentration of the system was maintained constant at around 35 g/L, the primary reason for reduction in the net osmotic driving force due to buildup of salinity within the bioreactor due to accumulation of salts rejected by the FO membrane and also due to the reverse diffusion of DS.

As presented in **Fig. 4**, the conductivity of the mixed liquor gradually and continuously increased throughout the operation. The initial conductivity of the activated sludge bioreactor was 1.0 mS/cm but increased to 1.9 mS/cm after 14 days of operation and then levelled at 3.9 mS/cm at the termination of experiment after 33 days. The accumulation of salts in the activated sludge bioreactor in the OMBR system is in fact an unavoidable phenomenon because of the high rejection of the feed solutes by the FO membrane and also due to reverse diffusion of draw solutes to the feed reactor [12, 15, 34]. The elevation of mixed liquor's salinity not only leads to the drop of net osmotic driving force, directly resulting in flux decline but also possibly hindering the microbial growth and functionality which affects the efficacy of biological treatment [15]. The salt accumulation before and after the chemical cleaning did not vary which might be a reasonable indicator for the chemical tolerance of FO membrane to alkaline and acidic cleaning agents. Besides, it also shows that FO membrane was not broken or damaged during the operating time since there was no sudden increase in the mixed liquor's conductivity indicating the robustness of the OSHF TFC-FO membrane.

3.3 *The role of aeration as a membrane fouling mitigation method.*

A number of OMBR studies have been conducted at both scales in laboratory and pilot plant, investigating the overall performance of the system using the submerged plate-and-frame membrane modules. These works proves that the submerged OSHF TFC-FO membrane modules are also suitable for OMBR system without the need of an energy the circulation of feed and also it takes advantage of aeration as air scouring to mitigate membrane fouling without the need of additional aeration system [8, 11, 28, 29, 42]. Zhang, et.al., [18] proved that enhanced air scouring rate led to biomass volume and coverage reductions on the membrane surface of 12 times and 31 times. Another FO fouling study using alginate foulant reported that 98% flux recovery can be achieved when periodic aeration was introduced to the FO membrane's selective layer facing FS [43]. Coarse air bubbles were also utilized for air scouring to mitigate membrane fouling in their work by Holloway, et.al., [11] enhancing the performance of the OMBR system with a stable water flux during 125 days of operation.

In this study, instead of using a separate air scouring for membrane surface, the aeration used for aerobic bioreactor of the activated sludge process was employed as a fouling mitigation method for the submerged module of OSHF TFC FO membrane in the OMBR system. The effectiveness of fouling mitigation method was assessed based on the flux decline and flux remained over the operation of the OMBR system.

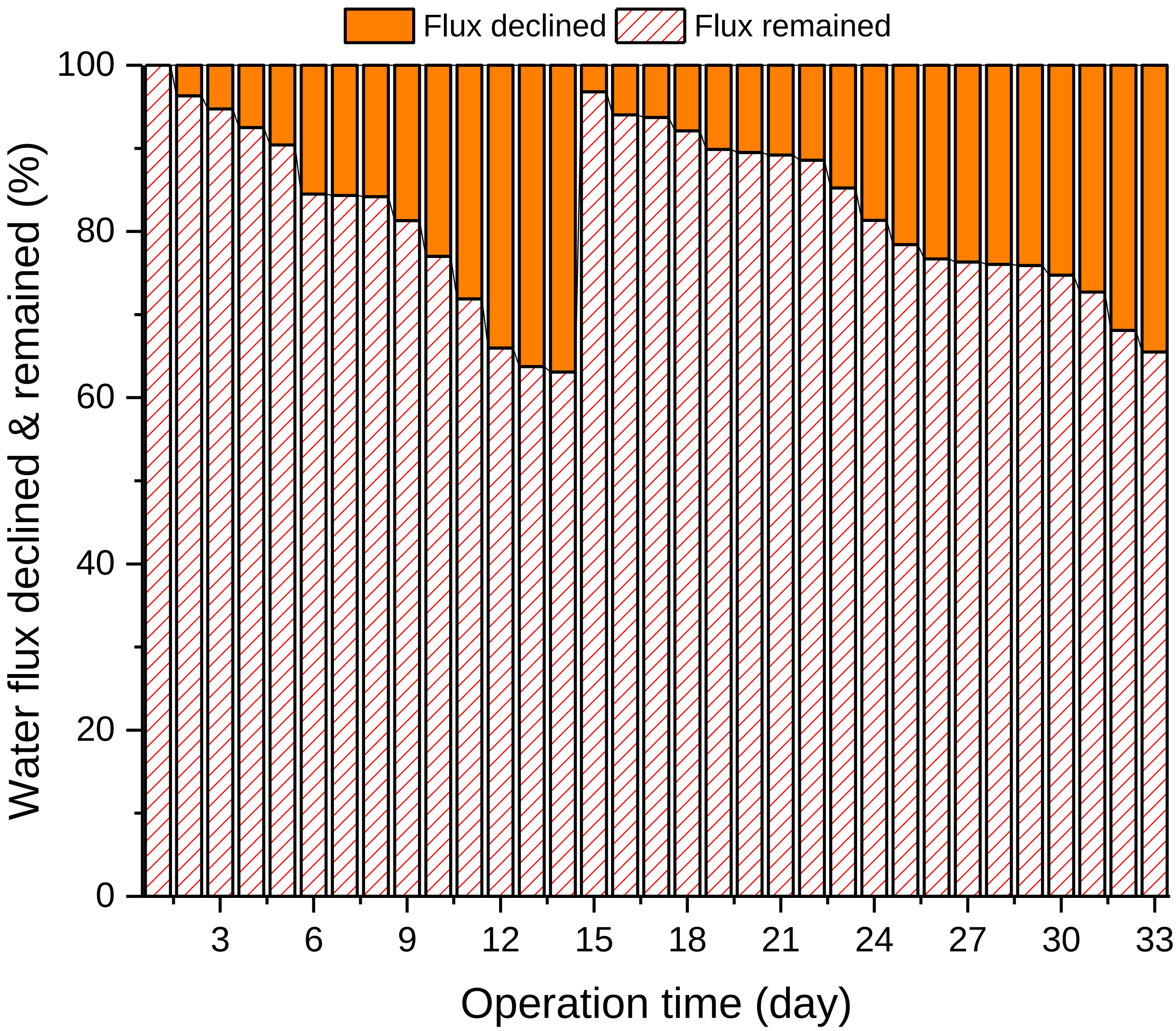

Figure 5. Flux declined and flux remained during the operation of the submerged OMBR system. Testing conditions: FS = activated sludge; DS = 35 g/L NaCl under suction mode; Temperature = ambient temperature (22 ± 1 ℃); AL – FS orientation.

Fig. 5 presents the normalized values in percentage of water flux that remained and declined after the operation of the OMBR system using a submerged OSHF TFC FO membrane module. As can be seen from the **Fig. 5**, water flux slowly declined with a similar pattern earlier in **Fig. 4** before and after two-step chemical cleaning and these results show that the water flux is highly sustained with only about 16% decline after 7 days of operation, which then dropped to 36% after 14 days. After chemical cleaning was subsequently carried out, water flux recovered by 97% of the initial flux before gradually decreasing with a similar pattern in the first 14 days. Although the water flux recovery was not 100% of the original water flux of the FO membrane but this might also be due to the reduced net osmotic driving force as a result of salt accumulation in the FS or MLSS in the bioreactor.

The rate of flux decline in this study was however significantly slower compared to our previous OMBR studies [34, 44] using a side-stream OSHF TFC FO membrane module under similar operating conditions. Using the external membrane module, the OMBR water flux quickly decreased by 80% after 3 days of operation without air scouring and 6-day operation with periodic injection of air bubbles as a mean of fouling mitigation into the membrane module [44]. The difference between these two results might also be due to difference in the aeration time and intensity. In our previous study, using the external cross-flow modules, air bubbles with an intensity of 2 L/min was regularly introduced into the module while in our current work, the membrane module was immersed into the activated sludge where aeration with an intensity of 3 L/min was continuously supplied, providing air bubbles scouring onto membrane surface. These results confirm that for the submerged OSHF TFC FO membrane module, normal air bubbles used in the aerobic bioreactor itself is an effective membrane fouling mitigation method in the OMBR system as evident from the slower flux decline rate. This betterment observed in this study well agrees with previous works on the conventional MBR using MF/UF membrane [45, 46] and in other OMBR systems [11, 36, 43, 47].

The excellent fouling reduction of this submerged OSHF TFC-FO membrane module during the experiment could also be ascribed to the gas-liquid two-phase flow generated by the air bubbles those are aerated into the mixed liquor. In this study, air was sparged into the bioreactor containing a submerged module of OSHF TFC FO membrane. As the air is diffused into the bioreactor, bubbles are formed and move upward driven by buoyancy [48] [49]. These air bubbles then generate a gas–liquid two-phase flow, creating turbulence with wake regions [50] and local vortexes near the membrane surface [45], thereby not only reduces concentrative ECP but also impedes the deposition of cake layer foulants on membrane surface. Moreover, the enhanced turbulence by the slug flows and bubble flows in the mixed liquor, associated with induced surface shear force, partially removed the deposited foulants on the membrane surface [46]. Formation of fouling cake-layer can therefore be hindered, thereby, reducing the severity of cake enhanced concentration polarization (CECP) and potentially biofilm layer formation [29], which ultimately helped minimize rapid flux decline over time. Since the submerged module was immersed in the mixed liquor and directly contacted with the enhanced hydrodynamic turbulence produced by air bubbles, the potential for the damage to the active layer of the of OSHF TFC FO membranes is likely. This damage can be easily detected by the sudden an unexpected increase in the water flux as a result of the membrane damage. Nevertheless, no sudden increase in the water flux was observed during the 33-day operation of the OMBR system, showing that no damage to the thin-film active layer was occurred.

3.4 *Primary pollutants removal efficacy*

Fig. 6 presents the removal rates of four selected pollutants including ammonium NH_4^+, total organic contaminants (TOC), phosphate (PO_4^{3-}), and total nitrogen (TN) by the OMBR system. Samplings were carried out from the synthetic wastewater feed tank (influent), aerated bioreactor (supernatant) and the DS tank. Generally, removal rates of TOC, NH_4^+, TN, and PO_4^{3-} by the OMBNR system were consistently high over the course of 33-day operations. This excellent removal of pollutants and nutrients could be ascribed to the combined effect of biodegradation in the activated sludge process and high rejection by OSHF TFC FO membrane [51]. As can be seen at the **Fig.6-(a)**, a consistent removal of TOC of over 98% was achieved by the OMBR system throughout the operations and these results agree well with our previous works [34, 52]. Other recent studies by Achilli, Cath, Marchand and Childress [8] reported overall TOC removal of 99% while Qiu and Ting [53] observed a stable removal of up to 98%. Some prior OMBR research works also reported high overall TOC removal of over 96% [54] and 98% by the OMBR system [55]. **Fig.6-(b)** exhibits a consistently high (> 99%) removal of NH_4^+ by the OMBR system during the 33 day operation. This excellent removal of NH_4^+ could be ascribed to the combined effects of the biological nitrification by ammonium-oxidizing bacteria and high rejection by FO membrane [34]. Based on this result and our previous OMBR studies using external side-stream OSHF TFC FO membrane module, it has proved that, OMBR system can achieve excellent NH_4^+ removal and this results agree well with some recent OMBR [8, 12, 26, 53, 56-58].

As illustrated in **Fig.6-(c)** the OMBR system has demonstrated to be quite effective in TN removal with up to 80% and a slight increase in the mixed liquor's TN concentration over experiment time. The TN concentration increase in the bioreactor MLSS could be attributed to the accumulation of nitrogen compounds rejected by the FO membrane. Under aerobic condition, TN removal of the

OMBR system is primarily dependent on the biodegradation of nitrogen compounds by microorganism. However, because of the absence of denitrification process in this experimental set-up, nascent nitrogen compounds such as nitrate, a by-product of nitrification process, cannot be transformed to nitrogen gas. The non-degraded nitrogen compounds were then well retained in the reactor by highly selective FO membrane which increased the TN concentration in the supernatant [59]. Similar results of TN removal were also reported in other OMBR studies using different types of flat-sheet membranes such as CTA, TFC, and Aquaporin FO membrane [40, 51]. A consistently high removal r of 92% to 98% of PO_4^{3-} by the OMBR system was observed as presented in **Fig.6-(d)**. Similar to the removal rates of TOC, NH_4^+ and TN, microbial activities and high rejection of FO membrane are the two main factors responsible for PO_4^{3-} removal in the OMBR system. Since activities and metabolism of phosphate accumulating organisms were inhibited by the elevation of the mixed liquor's salinity, rejection by OSHF TFC FO membrane should be the primary reasons for influencing the overall removal of PO_4^{3-}. Having relatively large radius diameter (0.49 nm) negatively charge with multi-valency, PO_4^{3-} ions were almost completely rejected by the FO membrane [25]. Excellent phosphorous removal of OMBR system was also reported by Nguyen, Chen, Nguyen, Ray, Ngo, Guo and Lin [20] and [60] with removal rates of 98% and 99%, respectively. It can be concluded that the OMBR system using submerged module of OSHF TFC FO membrane delivered a stable and high removal rates of the investigated pollutants. This results also proved the durability of OSHF TFC FO membrane, being able to withstand various harsh operating conditions as tested in the aerated biological reactors.

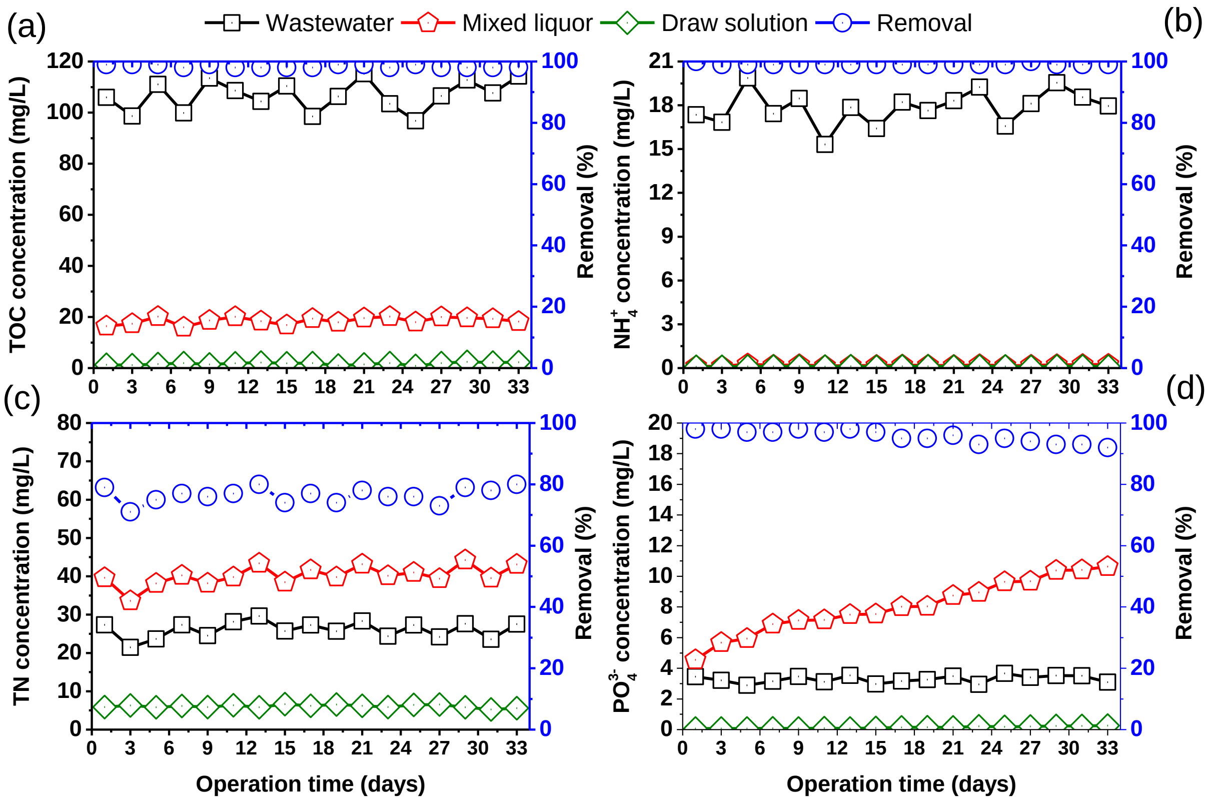

Figure 6. Concentrations and overall pollutant removal efficacies of the submerged OMBR system including (a) TOC; (b) NH_4^+; (c) TN removal; and (d) PO_4^{3-}. Testing conditions: FS = activated sludge; DS = 35 g/L NaCl; Temperature = ambient temperature (22 ± 1 ℃); AL – FS orientation.

3.5 *Membrane and fouling cake layer characterization.*

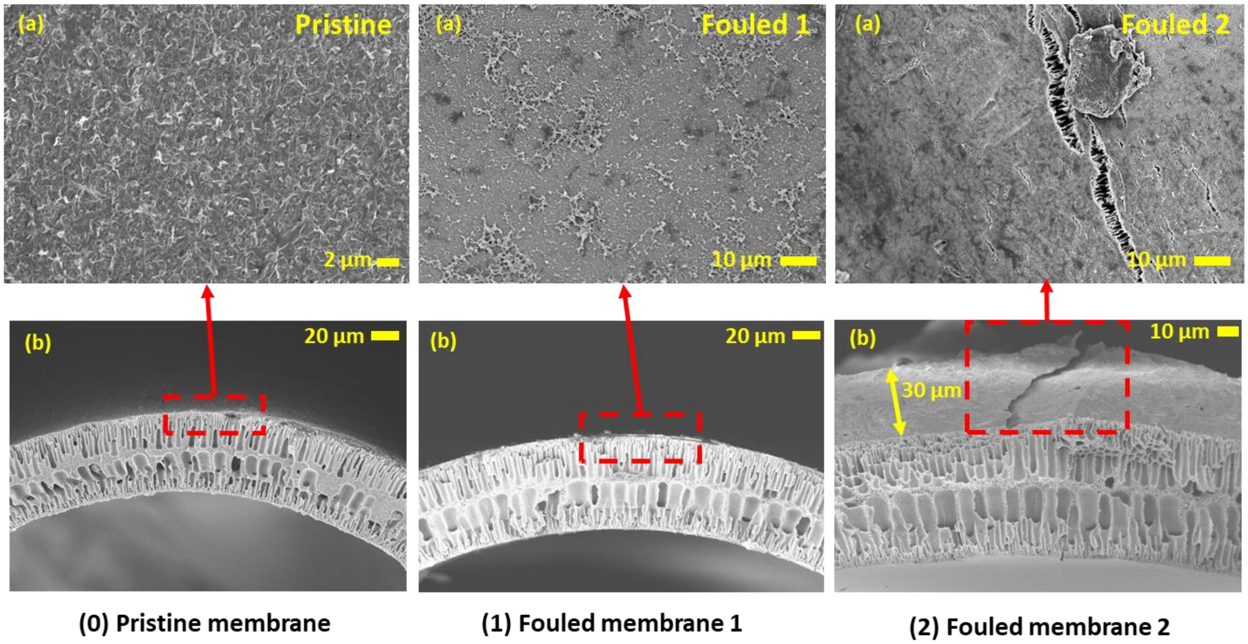

Figure 7. SEM images of pristine and fouled OSHF TFC FO membrane under OMBR operation with (0) pristine membrane; (1) fouled membrane 1; and fouled membrane 2 - (a) outer surface morphology; and (b) cross-sectional morphology.

Table 2 The elemental compositions of pristine and fouled membranes in two operation regimes by energy-dispersive X-ray (EDX) analysis.

Weight %	*C*	*O*	*N*	*S*	*Na*	*Cl*	*Mg*	*Al*	*Si*	*P*	*Ca*	*Fe*
Pristine	55.41	32.64	3.76	8.19								
Fouled 1	50.54	16.54	3.65	3.75	7.18	11.78	0.46	0.37	0.27	2.15	1.02	2.29
Fouled 2	45.15	13.12	5.21	3.99	9.35	11.16	1.5	0.79	0.93	3.12	1.41	4.27

Three membrane samples, including one pristine coupon and two fouled ones (at two different locations of the membrane module) at the end of experiment without washing, were collected to conduct characterization for its surface morphology of selective layer and fouling cake layer. SEM images captured from the surface and cross-section of pristine OSHF TFC FO membrane present a typical ridge-and-valley morphology of polyamide selective layer, confirming that neither deposition of foulants nor formation of fouling cake layer was found on the outer surface of OSHF

TFC FO membrane. However, different foulants and fouling cake layer were seen on the surface SEM images of two coupons of fouled FO membrane, which were also confirmed by their cross-sectional images. Interestingly, it can be visually viewed from the submerged membrane module two distinctive areas of fouling cake layer on the outer surface of each fouled hollow fiber FO membrane. There was one small part of the hollow fiber at one end of the membrane module, where air bubble could not reach to its membrane surface, was completely covered by a relatively thick fouling layer. **Fig.7-(2)** shows the surface and cross-sectional morphology of the fouled membrane with the thickness of the fouling cake layer at about 30 µm. On the other hand, the remaining part of the hollow fiber where it was in direct contact with and being scoured by air bubbles, accumulation of foulants were found scattered on the membrane surface as can be viewed from **Fig.7-(1.a)**. Cross-sectional image **Fig.7-(1.b)** shows the thickness of the fouling cake layer is just around 1 µm. This result indicates that the air bubbles used for aeration of bioreactor can be effectively utilized for the fouling mitigation of the submerged module of OSHF TFC FO membrane without the need for a separate air scouring system. This finding well explains why flux decline was slow in this study and it is in good agreement with our previous study [52] using a side-stream FO membrane module with the regular injection of air bubbles as a fouling mitigation method. Some prior research works in conventional MBR and OMBR also reported the effectiveness of using air bubbles as a mean of fouling control when the submerged membrane module was used [61, 62].

EDX analysis results in **Table 2** shows that the pristine FO membrane comprises of four main elements including carbon (C), oxygen (O), sulfur (S), and nitrogen (N) while the two fouled membrane samples consist of various inorganic elements including Fe, Ca, Ma, Si, P, Al, Cl, and Na. Generally, fouled membrane with thicker fouling cake layer (Fouled 2) contains more composition of each element compared to that of the less fouled membrane (Fouled 1). The presence of Ca, Mg, and Si might be an indicator of the inorganic scaling in association with biofouling, and other elements such as Fe and P might originally come from the synthetic wastewater. High amount of Na and Cl detected in fouling cake layer could be related to reversely diffused draw solutes and accumulated in fouling cake layer.

4 Conclusions

The performances of a submerged OMBR using a OSHF TFC FO membrane was investigated with the aim of using it as a pre-treatment to the RO process for high quality water reuse including its effectiveness to fouling resistance. Generally, the submerged OSHF TFC FO membrane module produced a stably high performance with regards to water flux, activated sludge's salinity build-up, and removal efficacies of four main pollutants such as TOC, NH_4^+, TN, and PO_4^{3-} over 33-day operation of the OMBR system. The study observed that the membrane fouling can be effectively mitigated with the help of the air bubbles used for aeration of the bioreactor as evident from the slow flux decline rate observed in the 33 days of operations. Findings from this study indicates that using the submerged OSHF TFC-FO membrane module, it not only eliminates the additional pumping energy to recirculate feed solution (or the MLSS feed in the external skid) but also the aeration air bubbles are adequate to provide scouring effect on the membrane surface thereby preventing the formation of thick fouling cake layer on the outer surface of the membrane.

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