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Microalgae filtration by UF membranes: influence of three membrane materials

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ABSTRACT

To evaluate the impact of membrane material on the ultrafiltration performance of microalgae medium, three types of UF membranes: polysulfone membrane GR40PP (PS, MWCO = 100,000 Da), fluoro polymer membrane FS40PP (PVDF, MWCO = 100,000 Da), regenerated cellulose acetate membrane RC70PP (RCA, MWCO = 10,000 Da) were used in this work. Influence of transmembrane pressure (1.3, 1.8, 2.3 bar) and cross-flow velocity (3.86, 4.83, 5.79, 7.72 m/s) on the permeate flux was studied. It was observed that the permeate flux increased with increasing transmembrane pressure for all membranes. Moreover, permeate flux increased as the cross-flow velocity increased. The fluoro polymer membrane showed the most significant improvement of flux (from 83.27 to 136.32 L/m^2h) with increase in cross-flow velocity, which may suggest that the fouling materials attached more weakly on the membrane surface as the cross-flow velocity increased. Hydrophilic RCA membrane had a much lower fouling tendency than hydrophobic PS and PVDF membranes. To maximize flux recovery for the algae-fouled membranes, NaOH, NaOCl, and Ultrasil 10 were applied as cleaning agents. Ultrasil 10 with concentration of 0.5 wt.% was more effective than other agents for membrane cleaning.

Keywords: Microalgae; Ultrafiltration; Membrane materials; Fouling; Cleaning

1. Introduction

Microalgae are widely used in wastewater treatment because of their robustness against variations in wastewater properties and their efficiency to grow and to remove nutrients. Extensive research has been performed to explore the feasibility of using microalgae to treat wastewater, especially for the removal of nitrogen, phosphorus, and chemical oxygen demand from effluents. Microalgae are ideal feedstock for renewable biofuel production as algal cells can accumulate a large amount of oil and have high biomass productivity [1]. However, the lack of an economical and efficient method to harvest algal biomass is a major problem [2]. The fouling due to algae is quite complex because the small size of the algal cells (3–30 μm in diameter). Algal cells change their sizes and morphology, and especially when the extracellular polymeric substance (EPS) attached to their cells [3,4]. Conventional methods, such as coagulation, flocculation, flotation centrifugation and gravity sedimenta-

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tion, have been traditionally used for microalgae separation [5,6]. Membrane technology has received increased attention due to its low-energy consumption, small space occupation, no chemical agents, and high-quality of permeate [7–10].

However, fouling limits the widespread use of membrane separation technology for microalgae harvest due to the decrease in permeate flux and the increase in operating costs associated with routine membrane cleaning [11–14]. The fouling of UF membrane by filtering algae is quite complex, which may be due to algae, bacteria, inorganic colloids, and EPS [15]. Of them, the formation of biofilm on the membrane surface has been regarded as the most serious problem [16,17]. Fouling process, during filtration of microalgae, has been investigated by Liang [15]. Firstly, internal fouling takes place when particles and colloids enter into the membrane channel and deposit or adsorb to the pore walls or entrance causing reversible fouling (reversible fouling can be removed by a strong shear force). Secondly, external fouling occurs when algae cells and bacteria deposited on the membrane surface, EPS was released leading to the formation of a secondary barrier that decreases permeate flux and changes solute selectivity [15].

Fouling control methods, such as optimization of operating conditions [18,19], physical and chemical cleaning [20,21], new membrane development or modification of existing membranes [22,23], have been successfully developed to reduce membrane fouling, especially reversible fouling. However, a lot of work needs to be done in the development and optimization of the membrane process. The selection of membrane and optimum operating conditions are considered as major factors affecting fouling processes in cross-flow ultrafiltration. Membrane characteristics, such as membrane material, pore size and surface roughness are important factors on membrane fouling. It has been widely accepted that hydrophilic membranes exhibit lower fouling potentials than hydrophobic ones [24,25], but hydrophobic membranes are still commonly used in ultrafiltration installations because of the higher resistance to the chemicals [26]. Therefore surface modifications to render the originally hydrophobic polymeric material into more hydrophilic have frequently been used.

The objective of this work was to investigate the effect of membrane material on ultrafiltration performance for the separation of microalgae from a diluted culture medium. The effectiveness of different chemical techniques for cleaning the fouled membranes was also examined.

2. Experimental

2.1. Material and membranes

FACHB-9 *Chlorecla pyrenoidosa* (one type of algae, a globular conformation, size ranging from 3 to 8 μm) cells were cultivated in an open cultivation system, provided by Green Center Algae Innovation Center Lolland, Denmark. The fresh cultures were taken in the middle of exponential growth phase. Then algae cells were placed in refrigerator and stored under darkness at 4 ℃. The pH of the culture was 9.0 ± 0.5.

Three types of commercial UF membranes from Alfa Laval Nakskov A/S were used in the experiments by using Alfa Laval's cross-flow membrane module M10 (a small laboratory-scale membrane module). Performance of different membranes was compared according to the permeate flux and cells retention. The membrane characteristics are shown in Table 1.

2.2. Experimental setup

The M10 module is shown in Fig. 1. The membrane module consists of four plates kept together by four bolts. The module contains four flat-sheet membrane samples operating in series with an effective filtration area of 0.0084 m^2 of each one. Inlet (P_{in}) and outlet pressures (P_{out}) are measured with pressure transducers (D) and (F) mounted on the inlet and outlet of the membrane module. Transmembrane pressure (TMP) was calculated as $TMP = (P_{in} + P_{out})/2$. A diluted chlorella suspension (culture medium (solution with all necessary nutrients) + microalgae cells) was kept in the feed tank (G). The filtration experiments were carried out in circulation mode, meaning that permeate and retentate were recycled in the same feed tank. The experiments were performed at 24 ℃. There is heat exchanger in the filtration system to control the temperature of the feed. The values of membrane fluxes are the averages of the four flat-sheet membrane samples.

The membrane filtration was performed in a batch mode with recycling permeate and retentate back to the feed tank to simulate the continuous operation. After each filtration circulation, the color of the cells did not change from green to brown. Hence, we assume that most cells were not damaged by circulation. In this study, the cross-flow velocity increased from 3.86 to 7.72 m/s. Permeate flow rate was measured by collecting the permeate in a 500-ml measuring cylinder with measuring time of 60 s. The total test time for each membrane test was 4.5 h. After each experiment, the M10 module was cleaned with

Table 1
Membrane type and characteristics

Membrane	Material	MWCO (Da)	pH	Pressure (bar)	Temperature (°C)
FS40PP	Fluoro polymer	100,000	1–11	1–10	0–60
GR40PP	Polysulphone	100,000	1–13	1–10	0–75
RC70PP	Regenerated cellulose acetate	10,000	1–10	1–10	0–60

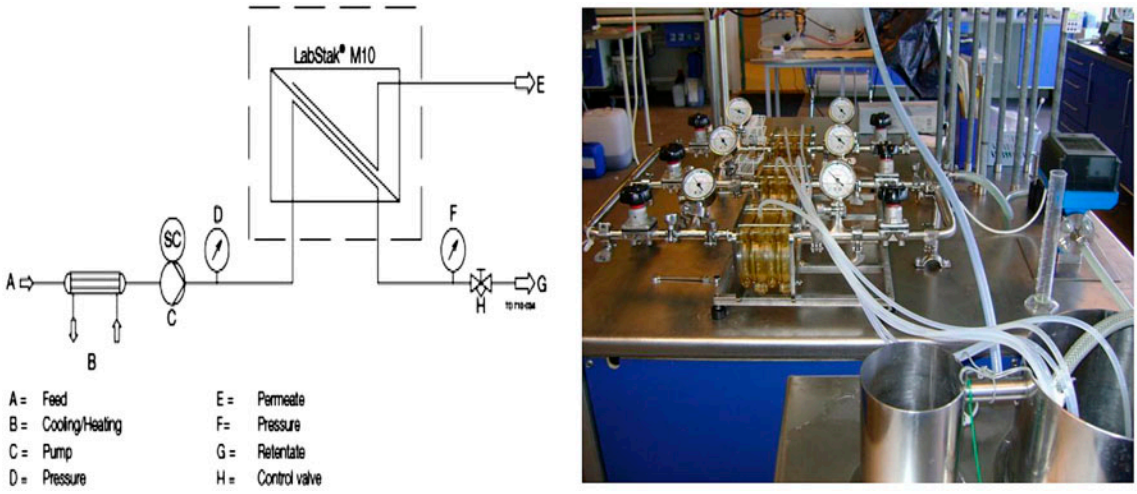

Fig. 1. Cross-flow filtration system: Alfa Laval LabUnit M10 for MF and UF.

cleaning agents Ultrasil 10 (from Henkel, Germany) for about half an hour at 55°C.

The rejection of algal cells was monitored by a UV-vis spectrometer with a wavelength of 665 nm. Since the physical size of algal cells is a few microns, all membranes showed 100% rejection of cells. In order to compare the performance of the tested membranes, all comparative experiments have been carried out with the same cell concentration of 0.68 g/L which has an absorbance of 0.554.

2.3. Membrane fouling and resistance model

Membrane fouling is a major problem in the process of microalgae filtration. Bacteria, inorganic colloids, and EPS deposit onto the membrane surface or adsorbed on the pore walls which caused the membrane pores blocked or becoming smaller, and membrane resistance increased, in turn membrane flux declined.

Based on the attachment, strength of particles to the membrane surface, membranes fouling are divided into reversible and irreversible fouling. Reversible fouling, caused by a gel layer resulted from reversible concentration polarization, can be removed by means of strong shear force of backwashing. Irreversible fouling, caused by irreversible absorption and blockage, needs to be removed by chemical cleaning.

During the ultrafiltration process, permeate flux declines owing to the accumulation of algae and particles on the membrane surface and causing pore clogging. Darcy's law [27] describes solvent passage though the membrane as a function of the applied pressure.

$$J = TMP/\mu Rt \quad (1)$$

where μ denotes the solvent dynamic viscosity (Pa s) and Rt is the total hydraulic resistance (m^{-1}) of the membrane during filtration. TMP is the transmembrane pressure (bar).

The filtration resistance at each step can be calculated by the following equation [18]:

$$Rt = Rm + Rc + Rp \quad (2)$$

where Rm is the intrinsic membrane resistance, Rc the cake layer resistance and Rp is the pore-clogging resistance. The combined value of Rm and Rp was obtained by measuring the resistance of the

membrane after being washed with tap water to remove the cake layer [18]:

$$Rm + Rp = \mathrm{TMP}/\mu Jp \quad (3)$$

$$Rm = \mathrm{TMP}/Jw \quad (4)$$

where Jp is the pure water flux obtained with the used membrane without cake layer (L/m^2 h), Jw is the pure water flux obtained with the virgin membrane.

2.4. Membrane cleaning

The cleaning of membrane after cake deposition was done by water flushing and chemical cleaning. After the filtration of algae, the used membranes were kept in the module, while the pure water (RO water) or cleaning agent solutions were recycled into the feed tank. However, the chemical methods were studied only for PS and PVDF membranes as the water flushing is good enough for cleaning the RCA membrane. Used chemicals were 0.025 N NaOH, 100 ppm NaOCl, 0.025 N NaOH + 100 ppm NaOCl and 0.5 wt.% Ultrasil 10. After each cleaning experiment for 2.5 h, the pure water flux was measured to see the effectiveness of the cleaning method employed. The membrane cleaning effectiveness was evaluated by water flux recovery percent.

3. Results and discussion

3.1. Permeate flux of membranes

To determine the intrinsic membrane resistance (Rm) of UF membranes, pure water fluxes of the membranes were measured at different transmembrane pressures and the hydraulic resistances of the membrane were calculated as the inverse of the slope of the plots of pure water fluxes against the respective transmembrane pressures as shown in Fig. 2. The results demonstrate that the pure water fluxes increased with transmembrane pressure for each membrane. Water permeabilities in L/(m^2 h bar) are 52.1, 177.4, and 270.9 for RC70PP, FS40PP, and GR40PP, respectively. Since FS40PP and GR40PP have much higher MWCO than RC70PP (100,000 versus 10,000), GR40PP showed higher fluxes of more than 200 L/(m^2·h) at 1.5 bar, whereas RCA membrane (RC70PP) showed the lowest fluxes of less than 90 L/(m^2·h) at the same pressure. This can be explained by the differences in pore size and surface porosity.

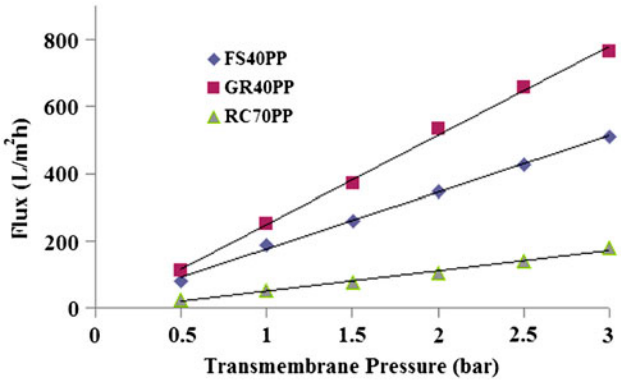

Fig. 2. Pure water flux as a function of transmembrane pressure: Operating temperature 24°C, cross-flow 4.84 m/s.

3.2. Influence of transmembrane pressure

Changes in transmembrane pressure were done by adjusting the inlet and outlet pressure of the membrane module while keeping the feed flow constant. The effect of transmembrane pressure on the permeate flux for filtration of algal medium is presented in Fig. 3. The results show that at a constant transmembrane pressure and cross-flow, higher fluxes were obtained at the beginning of the ultrafiltration process, followed by a rapid decline and finally leveling off. This may be explained by the higher operating permeate flux leading to faster membrane fouling caused by a larger amount of fouling materials being deposited onto the membrane in a shorter time, which possibly results in the quick build-up and compaction of fouling layer on the membrane surface. In addition, EPS released in the culture medium may also lead to the formation of a gel layer, which might cause flux drop. A general trend of increased permeate flux with increasing transmembrane pressure was observed. Higher permeate flux may lead to higher foulant concentration close to the membrane surface due to concentration polarization, which would cause a more densely gel/cake layer and increase filtration resistance.

Owing to higher pure water flux of GR40PP (see Fig. 1), the intrinsic membrane resistance of GR40PP is lower than FS40PP. Theoretically, permeate fluxes of GR40PP could be higher than FS40PP, but this property is lost when fouling occurs. As shown in Fig. 3, the permeate fluxes of GR40PP are likely slightly lower than FS40PP during the ultrafiltration under three different pressures. Permeate flux of RC70PP only showed a slight decline at each transmembrane pressure during the filtration compared with flux changes of FS40PP and GR40PP, which can be explained by the hydrophilicity of RC70PP

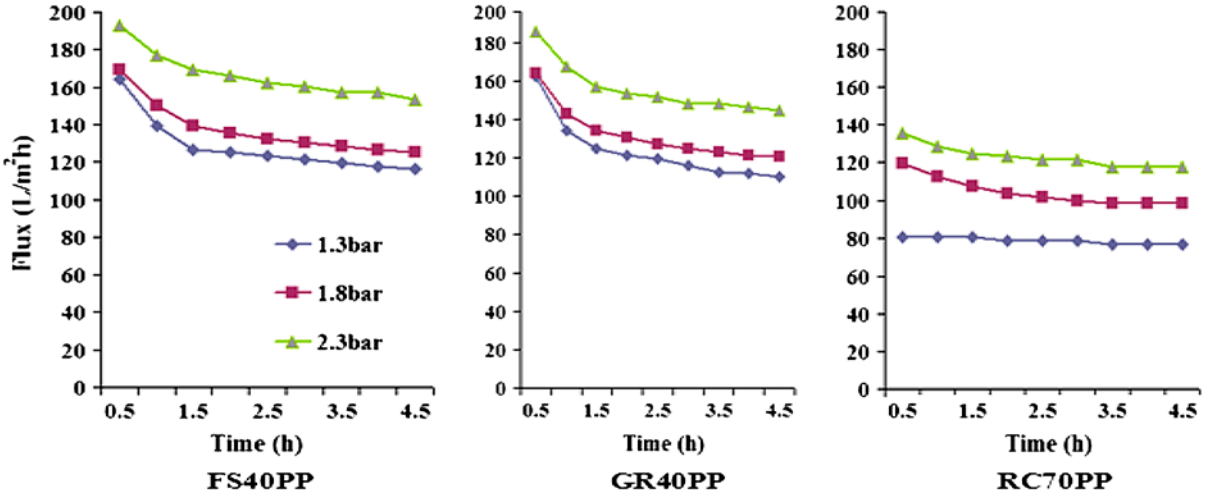

Fig. 3. Effect of transmembrane pressure on permeate fluxes (Temperature = 24 °C, cross-flow = 7.72 m/s).

showing lower fouling tendency. The antifouling property of TC70PP was further demonstrated by the membrane autopsy. After algae filtration tests, the 3 kinds of membranes were removed from the membrane module for visual inspection. It was found that very little algae cell deposition on RC70PP (little green material on the membrane surface) compared to considerable deposition on both FS40PP and GR40PP.

The influence of transmembrane pressure is shown in Fig. 4 in terms of membrane resistances. The intrinsic resistance, *Rm*, keeps constant. Cake layer resistance (*Rc*), increases pronouncedly with TMP, while pore-clogging resistance (*Rp*) increases moderately with TMP. *Rc* always represents the major contribution to the overall fouling resistance for FS40PP and GR40PP. The dramatic increase in *Rc* with TMP for FS40PP and GR40PP is probably due to the compaction of cake layer at higher TMP, resulting in higher resistance [28]. It would thus assume that particle aggregation on the membrane surface plays a key role in fouling phenomena. Further, algae could release extracellular materials leading to more compact cake and higher resistance than algal cell [4,29].

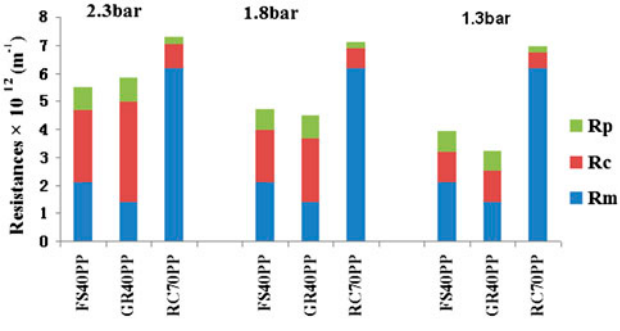

Fig. 4. Influence of transmembrane pressure on the resistances after a 4.5 h ultrafiltration experiment (Temperature = 24 °C, cross-flow = 4.83 m/s).

The total resistance of RC70PP is higher than that of FS40PP and GR40PP as shown in Fig. 4. This is due to the highest *Rm* of the membrane. However, RC70PP exhibited much lower fouling tendency as indicated by lowest *Rc* in Fig. 4. These results must be associated with the pore size and properties of membrane materials. It could also be explained by the fact that the RC70PP is the only hydrophilic membrane and adsorption fouling by protein and dissolved macromolecules is minimized. So RC70PP could be an attractive material for long-term running for concentration of algal medium.

3.3. Influence of cross-flow velocity

Increasing cross-flow velocity may increase the turbulence on membrane surface to reduce solute precipitation and provide a higher shear flow to reduce the concentration polarization, thus reducing fouling. In our experiments, increasing the cross-flow velocity was achieved by increasing the feed flow, while adjusting the inlet pressure and the outlet pressure to keep the same average pressure.

Fig. 5 shows the final permeate flux and cake resistance after 4.5 h of UF filtration at different cross-flow velocities. As the cross-flow increased, the permeate flux increase, suggesting that higher cross-flow velocity makes it more difficult for algae cells to deposit on the membrane surface, thus leading to higher flux. It is also shown in Fig. 5(b) that cake resistance significantly decreases with increasing cross-flow velocity. Similar results were also reported by Salgin [30].

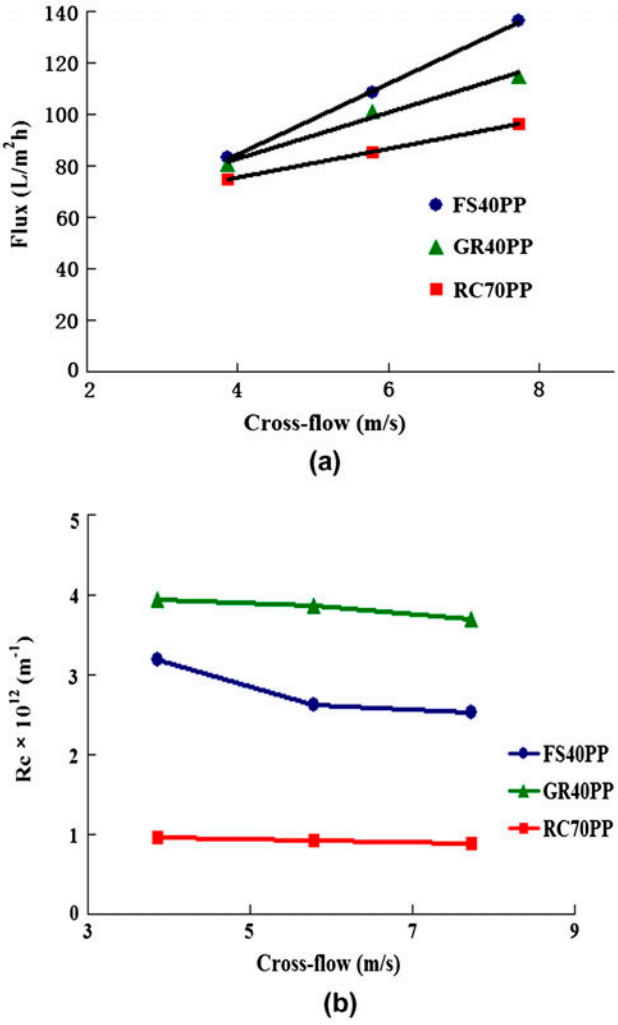

Fig. 5. Permeate flux (a) and cake resistance (b) after 4.5 h of ultrafiltration at different cross-flow velocities. Operation conditions: TMP = 1.8 bar, temperature 24°C.

When cross-flow velocity increased from 3.86 to 7.72 m/s under constant transmembrane pressure of 2.3 bar, RC70PP showed less pronounced flux improvement than the other membranes. This could be explained by the relatively weaker attachment of the cake layer to the membrane surface and the high intrinsic membrane resistance (*Rm*) in the case of the RC70PP membrane. Fig. 5(b) also illustrates relatively moderate decline of cake resistance for RC70PP.

3.4. Cleaning of fouled membranes

After each filtration experiment, the membranes were flushed with pure water. Then three chemical agents (NaOH, NaOH + NaClO, Ultrasil 10) were applied to remove the cake layer and fouling residuals. Water flushing tests were performed on UF module for 1 h, at 20°C and cross-flow velocity of 4.84 m/s and afterwards the pure water fluxes of clean, fouled and cleaned membranes were recorded and compared as shown in Table 2.

Water flushing could achieve 68.15 and 67.74% flux recovery for FS40PP and GR40PP, respectively, which was not effective enough in removing the attached algal cells. However, water flushing was more effective for the RCA membrane RC70PP with a flux recovery of 96.15%, which may be attributed to the hydrophilic property of the membrane surface and less exposure to foulants due to high intrinsic membrane resistance.

To compare the efficiency of three chemical cleaning agents, FS40PP and GR40PP were fouled for 4.5 h under the same conditions (operating pressure of 1 bar, cross-flow of 3.86 m/s and temperature of 24°C) by filtration of algal medium. Then, each cleaning agent was applied individually for totally 2.5 h. Water flux during the cleaning cycle was measured every half an hour. Then, the cleaning cycle continued after water flux measurement.

Fig. 6(a) and (b) shows that the cleaning by NaOH for 2.5 h exhibited relatively less recovery than combined use of NaClO and NaOH. It is probably due to that NaOH could make the fouling layer into a looser and more open structure, which could provide an easier chance for NaClO to break the binding between the foulants and the membrane, and reaching inner layer of fouling materials [31]. However, the results of NaOH or NaOH + NaClO cleaning are only marginally better than water flushing, indicating that caustic and oxidation agents are not effective enough to remove the foulants.

Ultrasil10 is the most effective among these three chemical agents, since only this agent can clean the fouling membranes completely. This is due to the fact that Ultrasil10 is a formulated cleaning agent, which is known to be a caustic-based reagent with the addition of surfactants. However, the water flux of fouled GR40PP cleaned by Ultrasil10 was higher than the clean membrane. The results shown in Fig. 6 also indicate that longer cleaning time is necessary to achieve

Table 2
Experimental results of water flushing on water flux (L/(m² h)) and recovery. Tests conducted at 1.5 bar and 24°C, water flushing for 0,5 h

	FS40PP	GR40PP	RC70PP
Clean membrane	206	215	78
Fouled membrane	128	117	62
Cleaned membrane	141	145	75
Recovery (%)	68.15	67.74	96.15

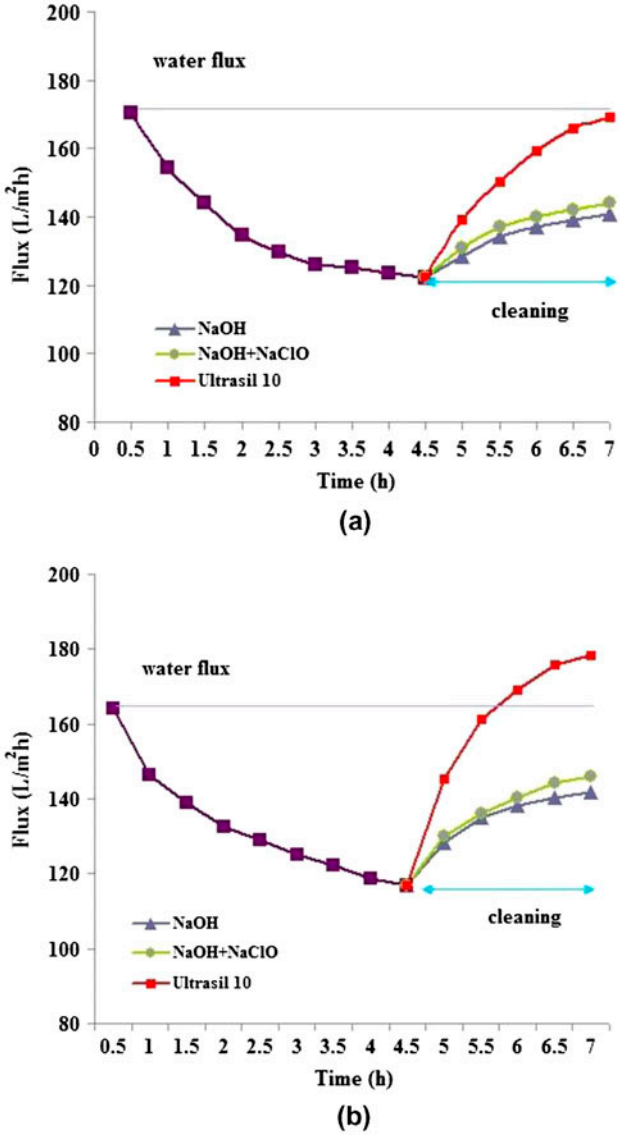

Fig. 6. Change in permeate flux with filtration time for FS40PP (a) and GR40PP (b). The first 4.5 h was under filtration (temperature = 24 °C, cross-flow = 7.72 m/s, TMP = 1.8 bar) of the algal suspension.

better cleaning. Even with best cleaning agent Ultrasil 10, short-time cleaning (e.g. 0.5 h) was not enough to remove foulants. The presence of surfactants in detergent cleaning product Ultrasil 10 could lead to their adsorption on the membrane surface and resulting in less hydrophobic membrane surface, helping to recover the water flux.

4. Conclusion

The permeate flux profiles of the FS40PP and GR40PP membranes were similar, showing fast drop of permeate flux during the initial filtration stage, whereas the RC70PP membrane exhibited the slowest flux decline rate versus test time. Our work suggests very similar performance for FS40PP and GR40PP, indicating there is no preference for membrane material polysulfone or PVDF for this application. The faster permeability decline of FS40PP and GR40PP could be due to the higher initial permeate flux that leads to faster membrane fouling caused by a large amount of fouling materials attached onto the membrane in a shorter time. The RC70PP membrane showed much lower cake layer resistance (*Rc*) after 4.5 h of ultrafiltration, indicating low fouling tendency. The intrinsic resistance (*Rm*) of RC70PP is much higher compared with FS40PP and GR40PP membranes, which is attributed to smallest pore size (lowest MWCO) of the membrane. Flushing with water was effective for cleaning the fouled RC70PP, while chemical cleaning is necessary for cleaning the fouled FS40PP and GR40PP. Applying chemical cleaning agents could achieve satisfied cleaning efficiency for FS40PP and GR40PP, and Ultrasil 10 was shown to be the best cleaning agents.

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