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Effect of chemical cleaning conditions on the flux recovery of MF membrane as pretreatment of seawater desalination

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ABSTRACT

Flux recovery through chemical cleaning was investigated using mini-module for application in desalination pretreatment using seawater as feed. This study focused on the causes of membrane fouling by evaluating the cleaning efficiency with several chemical agents at various conditions. Sodium hydroxide and sodium hypochlorite (NaOCl) as alkaline chemicals, and citric acid, nitric acid, sulfuric acid, and oxalic acid ($C_2H_2O_4$) as acidic chemicals were used in the experiment. Each chemical was prepared at concentrations of 0.1, 1, 3, and 5%. The effect of chemical combination was tested with cleaning sequences of alkaline–acid–alkaline and acid–alkaline–acid. The surface characteristics of the membrane were also investigated using scanning electron microscopy and tensile strength analysis. Prior to the chemical cleaning, flux of fouled membrane was measured using artificial seawater additive humic acid and sodium alginate. Duration of chemical cleaning was set at 4 h and then the flux was checked. For the control, pure water flux was measured using virgin hollow fiber membranes. Among the chemicals, NaOCl showed the highest flux recovery rate of 27.9% at 1% concentration. For the acidic chemicals, the highest recovery rate was 79.3% at 3% $C_2H_2O_4$ in humic acid fouled membrane. While for the combination chemical cleaning, 97.6% flux recovery rate was recorded. On the other hand, in the case of sodium alginate fouling membrane, NaOCl showed the highest flux recovery rate of 30.3% at 0.1% concentration. For the acidic chemicals, the highest was 68.8% recovery rate at 0.1% $C_2H_2O_4$. For the series type chemical cleaning, 98.0% flux recovery rate was recorded. Recovery efficiency increased as the concentration of chemicals and cleaning time increased. Organic or biofilm was considered as the main foulant as observed from the experimental results.

Keywords: Chemical cleaning; Hollow fiber membrane; Microfiltration; Recovery rate; Seawater; Desalination; Pretreatment

1. Introduction

Many regions of the world suffer from the shortage of fresh water resources for potable, industrial, and agricultural purposes. One of the alternative ways to make fresh water is to utilize seawater so-called desalination. Desalination process includes multi-stage flash, multi-effect distillation (MED), and reverse osmosis (RO) [1,2]. RO process is considered because

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only water can pass through the semi-permeable membrane and reject solute. Seawater is fed to the RO system by applying pressure to get drinking water. Not only effect on RO process's energy is three times higher than distillation, but also construction and system operation are easier than other desalination processes [3].

RO process uses various pretreatment methods such as coagulation and flocculation, media filtration, multi-media filtration (MMF), and microfiltration (MF)/ultrafiltration (UF) in desalination process [4]. There are several advantages in MF/UF method for pretreatment of RO process. First, silt density index (SDI) value can be obtained between 2 and 4, which is more stable compared to other methods. Second, other methods need larger foot prints than MF/UF method. Third, MF/UF has stable flux. Lastly, it is easy to operate auto system. However, MF/UF needs much electric power, cleaning of membrane, operating cost, and higher initial capital [5–7].

MF membrane should have stable flux, water quality, and SDI value, which can be run in RO membrane [8]. But, membrane can be fouled by foulants attached on the membrane surface, which decreases flux and increases pressure. For these reasons, physical cleaning is needed to be done periodically such as backwashing, aeration, air-scrubbing, and chemical-enhanced backwashing [9–13]. However, physical cleaning should be limited for long-term operation. Thus, operation for more than six months needs chemical cleaning with various chemical agents. It takes almost one day to perform CIP (Cleaning In Place) for every six months operation. Two kinds of chemical agents were used [14,15]. First, for alkaline, sodium hydroxide (NaOH) and sodium hypochlorite (NaOCl) were tested. Second, oxalic acid ($C_2H_2O_4$), citric acid ($C_6H_8O_7$), nitric acid (HNO_3), and sulfuric acid (H_2SO_4) were used as acidic agents [16–24].

In this study, artificially fouled membranes were used. Foulants consist of humic acid and sodium alginate. Humic acid is an organic compound, while sodium alginate is an inorganic compound. Membrane module fouled by humic acid and sodium alginate was subjected to chemical cleaning. This study aims to investigate the next suitable chemical agent for membrane cleaning.

2. Materials and methods

2.1. Membrane and dead-end filtration

Polyvinylidene fluoride (PVDF) hollow fiber membrane was used in this study, which is widely employed in MF and UF. Advantages of PVDF membrane include high mechanical strength, high thermal stability, low cost, and high chemical resistance [25,26]. The fouled membrane was made from a laboratory treating artificial seawater [27]. Artificial seawater [27] with humic acid and sodium alginate at 10 ppm was used as feed. Specification of the hollow fiber membrane is summarized in Table 1. Dead-end filtration experiment was conducted at constant pressure of 0.5 bar. The initial flux using the virgin membrane was recorded with an average value of 130 LMH. The flux of the fouled membrane was observed to decrease significantly obtaining about 27 LMH [7,15,18,27].

The schematic diagram of the lab-scale MF system is shown in Fig. 1. First, artificial seawater flowed into the feed tank. When chemical cleaning started, the valve was closed for the feed tank and the cleaning agent was pumped which was present in the chemical cleaning tank. It passed through the valve and its flow was controlled by a gear pump. The pressure was set at 0.5 bar monitored by a pressure gage [15,18].

2.2. Single chemical cleaning

Chemical cleaning was conducted using NaOH, NaOCl, H_2SO_4, HNO_3, $C_6H_8O_7$, and $C_2H_2O_4$ as cleaning agents, and diluted to make different percent concentrations of 0.1, 1, 3, and 5%.

To determine the effectiveness of each chemical for single cleaning on flux recovery, each chemical was passed on the surface of the mini-module at 30 min contact time and 10 min rinsing with artificial seawater. Thereafter, flux recovery was measured using artificial seawater for 30 min. The experiment was repeated at different duration (1, 2, and 4 h). The flux was calculated using the equation

$$\mathrm{Flux(LMH)} = \frac{Q}{A} \times \frac{\omega_T}{\omega_{25}} \times \frac{0.5\,\mathrm{bar}}{\mathrm{TMP}} \tag{1}$$

Table 1
Specifications of MF membrane used in experience

Shape	Hollow fiber module
Pore size (μm)	0.04
Material	PVDF
Average filtration flux (L/m^2 h)	130
Membrane area (m^2)	2.26×10^{-3}
Dimension (π × D × 1 × unit)	(π × 150 mm × 1.2 mm × 4 unit)
Operating pressure, bar	0.5

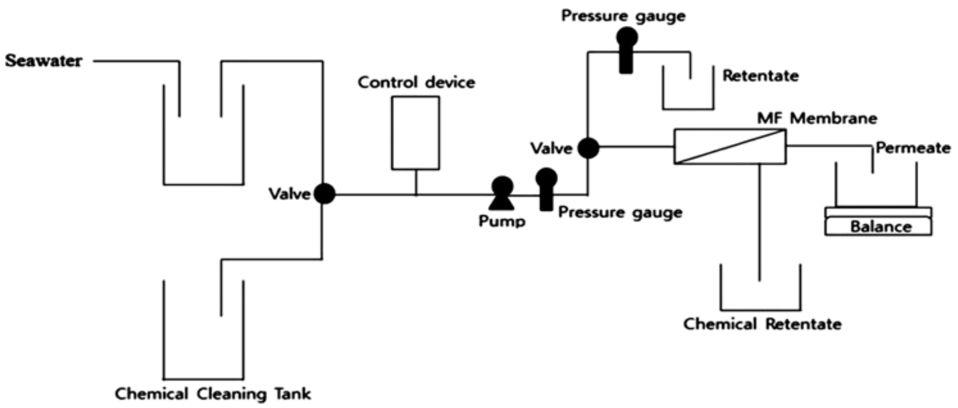


Fig. 1. Schematic diagram of the dead-end filtration system in this experiment.

where Q is the filtration flow rate, A is the effective area of the membrane, ω_T is the viscosity at actual temperature, and ω_{25} is the viscosity at 25°C. The equation used to calculate the recovery rate is as follows,

$$\text{Recovery rate } (\%) = \frac{\text{Flux}_\text{A} - \text{Flux}_\text{F}}{\text{Flux}_\text{I} - \text{Flux}_\text{F}} \times 100 \qquad (2)$$

where Flux_A is the flux after chemical cleaning, Flux_F is the flux of the fouled membrane, and Flux_I is the initial pure water flux [15].

2.3. Combination chemical cleaning

The chemical cleaning was also conducted by subjecting the fouled membrane with different chemical agents in series. Two sequences were tested, acid–alkaline–acid and alkaline–acid–alkaline. First, the mini-module was made using the fouled membrane obtained from the pilot plant. Then the initial flux was measured using artificial seawater. This was followed by chemical cleaning for 1 h duration using either acid or alkaline agent. Second cleaning was conducted for 2 h duration, followed by another last cleaning for 1 h. Immediately after the chemical cleaning, the flux of the cleaned membrane was measured using DI water, and then the percentage recovery rate was calculated. The duration of the chemical cleaning was altered from 4 h total cleaning time to 8 h, with cleaning sequence of 2 h–4 h–2 h. Through this, the effect of the cleaning agent on the recovery rate can be investigated at two different contact time. Moreover, the effect of temperature on membrane cleaning was also investigated by changing the temperature from 23 to 2°C, and the experiment was carried out with the same procedure [28,29].

3. Results

3.1. Results of single chemical cleaning

Six chemical cleaning agents were used in this study: NaOH, NaOCl, H_2SO_4, HNO_3, $C_6H_8O_7$, and $C_2H_2O_4$. Each chemical agent was prepared at 0.1, 1, 3, and 5% concentration, and the pH of each solution is listed in Table 2. Normally, chemical cleaning operated at pH higher than 12 which could damage the PVDF membrane. However, to investigate the effect of pH at harsh conditions, the experiment was still conducted even if the pH of the solution exceeds pH 12 [30–32].

As shown in Fig. 2 and Table 3, among the cleaning agents, NaOH obtained the highest recovery rate of 19.3% with cleaning solution concentration of 0.1% with humic acid as foulant. In the case of sodium alginate, cleaning solution concentration of 5% shows the highest recovery rate at 28.5%. NaOCl can also be an alternative based on percentage recovery rate of 45.0% humic acid and 48.4% sodium alginate. However, with 0.1, 1, 3, and 5% NaOH over pH 12, browning of the mini-module was observed due to the partial dissolution of the epoxy on the potting site making it

Table 2
pH of cleaning solutions at different percent concentrations

Chemical	0.1%	1%	3%	5%
NaOCl	11.10	11.76	12.10	12.23
NaOH	12.82	13.13	13.44	13.89
H_2SO_4	1.72	0.78	0.56	0.34
HNO_3	1.59	0.75	0.34	0.12
$C_6H_8O_7$	2.41	2.24	2.12	1.90
$C_2H_2O_4$	2.29	1.48	1.22	1.08

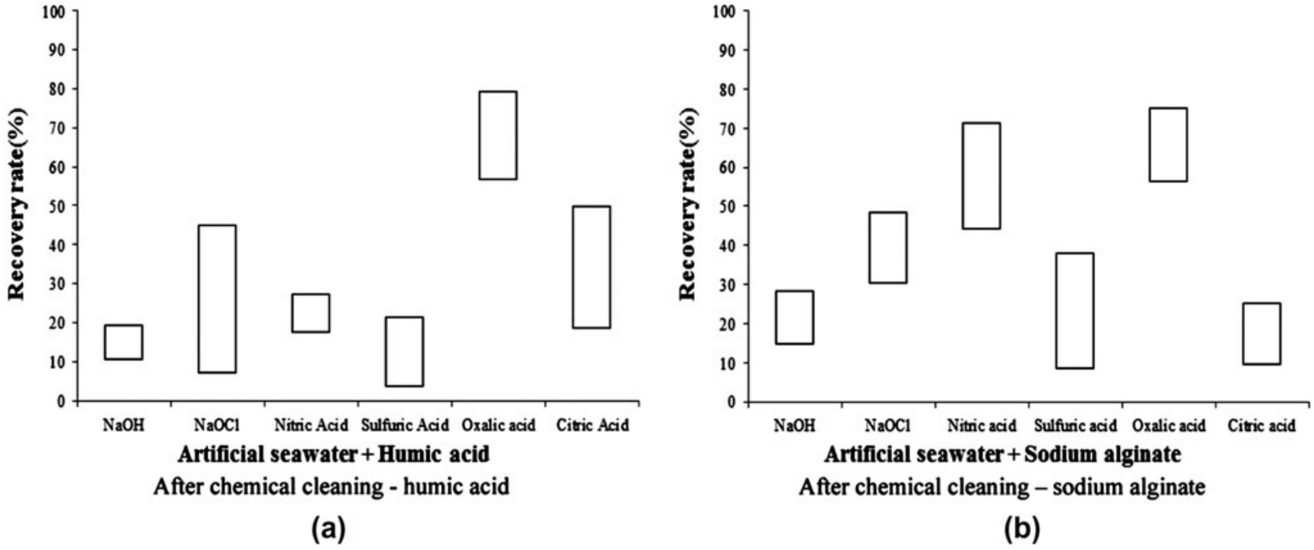


Fig. 2. Range of recovery rate for single chemical cleaning.

undesirable to use. Two types of alkaline agents were compared. NaOCl recorded higher recovery rate than NaOH. Membrane fouling was visually observed during the experiment carried out at 5% NaOCl. But, with 3% and 5% NaOCl over pH 12, performance stability was not reached. For NaOH, fouling on the membrane surface was not detected with cleaning solution concentration. Thus, cleaning agents with pH values more than 12 could damage the membrane and increase the tendency of membrane fouling.

As shown in Fig. 2 and Table 3, percent recovery of NaOH and NaOCl were recorded at different ranges. NaOH got narrow recovery range whereas NaOCl obtained a wide range, which indicates that NaOH has nothing to do with the concentration. On the contrary, concentration is relevant to NaOCl.

For the inorganic cleaning agents, the highest percent recovery rate was obtained using 3% H_2SO_4 with 21.5% recovery rate. However, 3% HNO_3 showed better performance with 27.4% recovery rate with humic acid as foulant. In case of sodium alginate, H_2SO_4 obtained the highest recovery rate of 38.1% at 5%, while HNO_3 got the highest recovery rate at 5% that recorded 71.3%. Thus HNO_3 performed better than H_2SO_4. Using HNO_3, it was shown that sodium alginate foulants treated better than humic acid foulants.

As compared to $C_6H_8O_7$, the cleaning performance of $C_2H_2O_4$ was better attaining an average percent recovery rate of 79.3% at 3% concentration with humic acid as foulant. Using sodium alginate, concentration at 5% $C_2H_2O_4$ obtained 75.3% recovery rate whereas $C_6H_8O_7$ got 49.7% recovery rate at 0.1% concentration. In the case of sodium alginate, $C_2H_2O_4$ 5% gained the highest recovery rate for all chemical agents with 75.3%, while $C_6H_8O_7$ recorded 25.0% at 3% concentration. Therefore, $C_2H_2O_4$ performed better than $C_6H_8O_7$. Generally, higher concentration of chemical agent gives higher recovery rate. However, in the case of $C_6H_8O_7$, it was recorded that as the chemical agent concentration increases, the recovery rate decreases. This claim is supported by scanning electron microscopy (SEM) images.

Table 3
Recovery rate (%) at various chemical cleaning agents

Concentration	NaOH		NaOCl		H_2SO_4		HNO_3		$C_2H_2O_4$		$C_6H_8O_7$	
	H.A	S.A	H.A	S.A	H.A	S.A	H.A	S.A	H.A	S.A	H.A	S.A
0.1%	19.3	17.6	7.3	30.3	12.3	8.4	24.5	44.3	72.4	68.8	49.7	25.0
1%	10.8	14.6	27.9	33.9	3.5	13.6	17.5	46.8	65.3	69.1	33.0	17.3
3%	17.1	24.0	28.8	38.1	21.5	20.8	27.4	52.4	79.3	73.2	24.9	16.6
5%	17.4	28.5	45.0	48.4	19.2	38.1	22.2	71.3	56.8	75.3	18.4	9.4

At high concentration of sodium alginate, high recovery rate was recorded, while for humic acid, no trend was observed. Foulants made from sodium alginate have higher viscosity than humic acid due to the formation of brown algae, which is easier to remove.

3.2. Results of combination chemical cleaning

From the results obtained from single chemical cleaning with humic acid and sodium alginate as foulants, NaOCl and $C_2H_2O_4$ were chosen for chemical cleaning in series experiments. In the case of humic acid, the experiment was conducted at 1% NaOCl and 3% $C_2H_2O_4$. For sodium alginate, same concentration of NaOCl and $C_2H_2O_4$ was used, which served as a basis for comparison, even though the highest percent recovery rates were achieved at other concentration. In addition, NaOH and NaOCl exceeded pH 12, which is considered to be harmful for PVDF membranes, thus 1% NaOCl (humic acid as foulant) and 0.1% NaOCl (sodium alginate as foulant) were used. For $C_2H_2O_4$, purification plants and desalination pilot plant usually employ $C_2H_2O_4$ at concentration of 0.1–3%, and based on the results, in case of humic acid as foulant, 3% $C_2H_2O_4$ was best among other concentrations and using sodium alginate as foulant, percent recovery was recorded from 68.8 to 75.3%, which shows insignificant differences in value. Therefore, using 0.1% concentration appeared to give more stable performance. Using humic acid as foulant, the most efficient cleaning sequence was $C_2H_2O_4$–NaOCl–$C_2H_2O_4$ at both operating temperatures of 2 and 23˚C with cleaning duration of 2 h–4 h–2 h, respectively, as shown in Fig. 3. The highest percent recovery rate was recorded at 97.6% at 23˚C.

In case of 2˚C, the recovery rate was 86.2% for 4 h duration time, $C_2H_2O_4$–NaOCl–$C_2H_2O_4$ series type recorded 93.9% recovery rate.

However, for sodium alginate as foulant, NaOCl–$C_2H_2O_4$–NaOCl at both operating temperatures of 2 and 23˚C with cleaning duration of 2 h–4 h–2 h, respectively, obtained are shown in Fig. 3. The highest recovery rate was gained at 98.0% at 23˚C. In the case of 2˚C, the same tendency was observed at 23˚C. In case of 4 h duration, NaOCl–$C_2H_2O_4$–NaOCl series type recorded 95.7% recovery rate.

In the case of sodium alginate, viscosity was observed to increase as the temperature decreases. Thus, sodium alginate foulants have lower tendency to be removed than humic acid foulants at lower temperature. For the combination chemical cleaning, sodium alginate recorded lower recovery rate than humic acid.

Operation in series mode was more efficient that single chemical cleaning, however, direct comparison between two modes could not be elucidated due to the difference on the total cleaning time. It should be noted that for a single chemical cleaning, the total cleaning time was 4 h, which is half of the total cleaning time for the operation series mode. Thus, single chemical cleaning needs longer duration time. Chemical cleaning is usually done in elevated temperature to assure high flux recovery rate. To investigate the effect of temperature on membrane cleaning, the system was operated at lower temperature. Fig. 3(a) and (b) shows the comparison between two different operating temperatures (2 and 23˚C). At 2˚C, the recovery rate was lower than at 23˚C. It is possible to predict that lower temperature cannot activate chemical reaction due to the increased density and viscosity from 2 to 23˚C. If chemical cleaning is to be performed during winter season, heat exchanger is needed to control the temperature of the chemical agents. Thus, the system should be operated at elevated temperature to obtain an efficient performance.

3.3. Membrane morphology by SEM analysis

SEM analysis was conducted after cleaning the fouled membrane. Figs. 4 and 5 show the surface of the membrane using $C_6H_8O_7$ agents at the end of the experiment. Recovery rate at 0.1% concentration was higher than 1, 3, and 5%. High recovery rate was recorded at lower concentration. SEM images revealed that severe fouling is observed at high concentration. It was predicted that components of artificial seawater as well as other fouling factors accumulated on hollow fiber membrane and reacted with $C_6H_8O_7$ agent.

Fig. 6, shows the comparison of the cleaning methods for membrane fouled with humic acid [33–35]. Using 1% NaOCl seemed to be cleaner than fouled membrane, and using 3% $C_2H_2O_4$ too. Membrane surface was still covered with foulants. However, 3% $C_2H_2O_4$ flux recovery is 79.3%, because the foulants on the membrane pores were removed by chemical cleaning. SEM image of the series type is shown in Fig. 6(l). Combination chemical cleaning method efficiently removed the foulants on the surface of the hollow fiber membranes as well as the membrane pores.

In case of sodium alginate, SEM image of the membranes after chemical cleaning is shown in Fig. 7. Using 0.1% NaOCl, foulants still remain on the membrane surface obtaining a very low recovery rate at 30.3%, which indicates that membrane pores were not efficiently cleaned. For combination chemical cleaning as shown in Fig. 7(p), the hollow fiber surface was

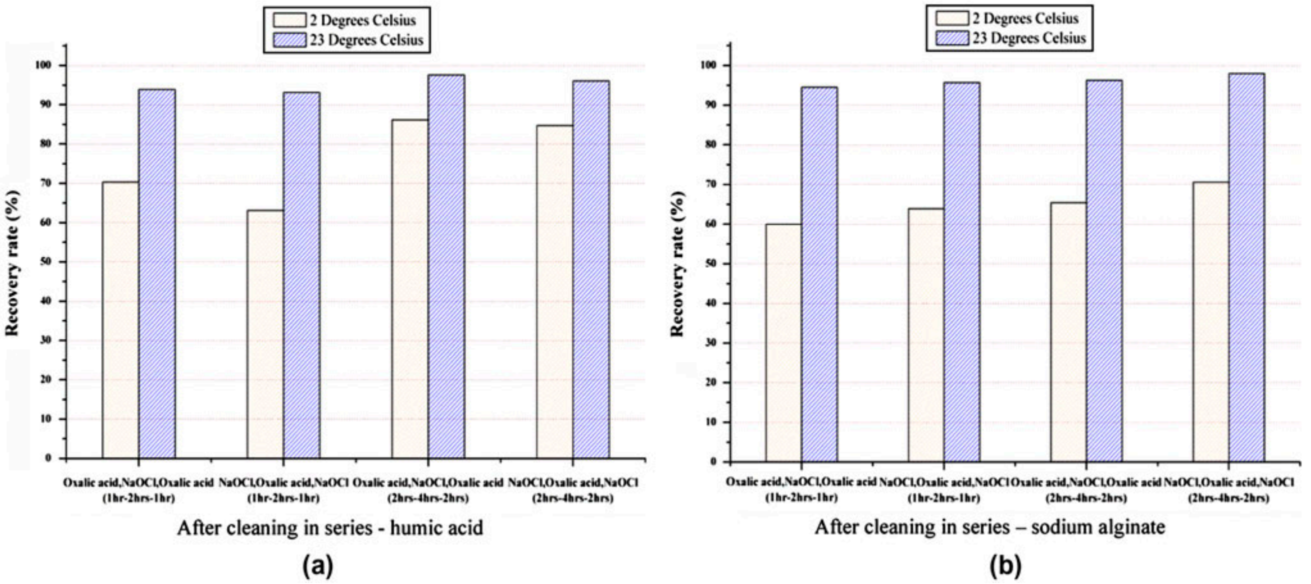


Fig. 3. Combination chemical cleaning recovery rate at 23 and 2℃.

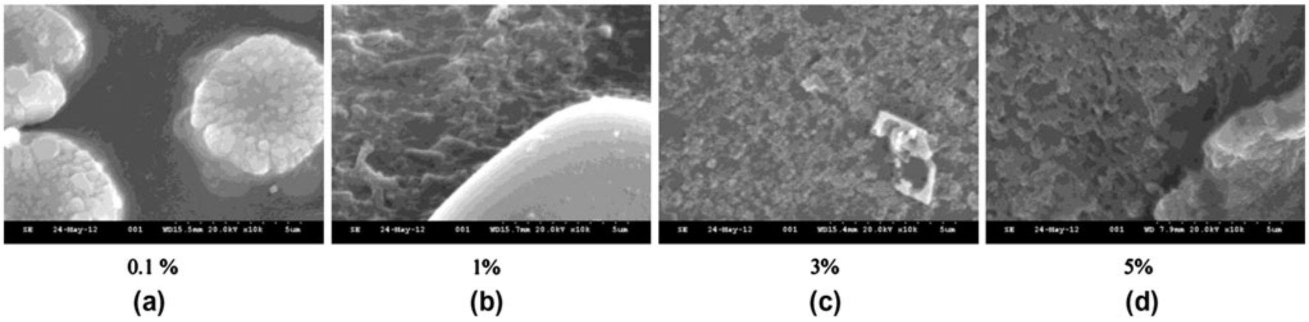


Fig. 4. SEM images of membrane with humic acid as foulant after chemical cleaning ($C_6H_8O_7$).

covered with foulants (white) as observed in SEM images, which were removed using combination chemical cleaning. The combination method recovered the highest recovery rate at 98.0%.

3.4. Measurement of tensile strength

Tensile strength was evaluated by analyzing the values of tensile strength (N/mm^2) of the virgin

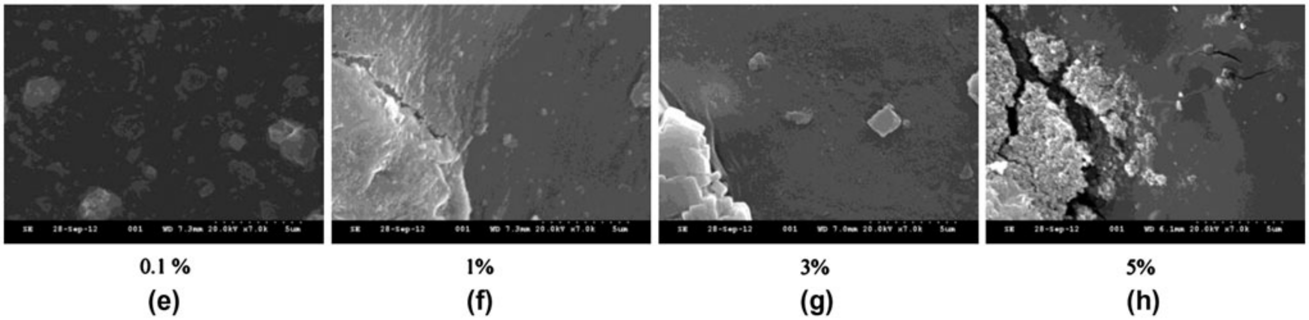
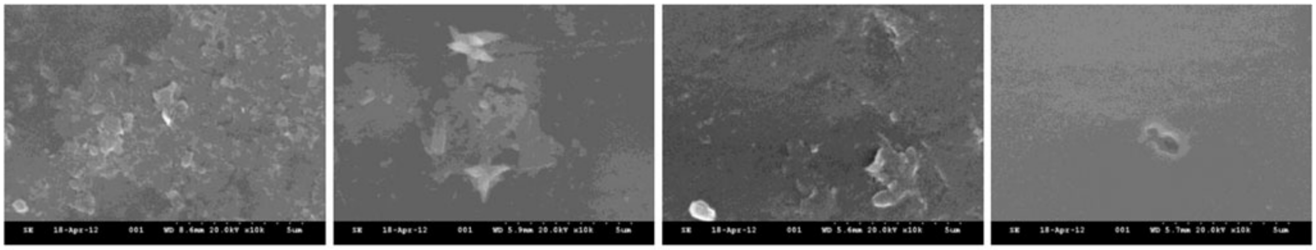
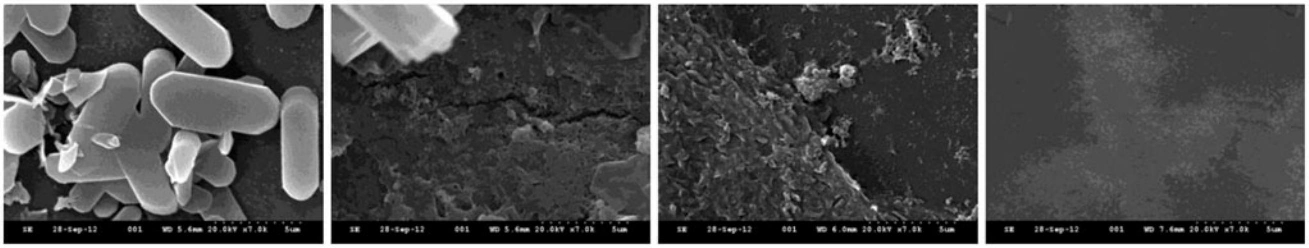


Fig. 5. SEM images of membrane with sodium alginate as foulant after chemical cleaning ($C_6H_8O_7$).



Fouled membrane (i) NaOCl 1% (j) Oxalic acid 3% (k) Oxalic acid-NaOCl-Oxalic acid (l)

Fig 6. SEM images of membrane with humic acid as foulant before and after chemical cleaning.



Fouled membrane (m) NaOCl 0.1% (n) Oxalic acid 0.1% (o) NaOCl-Oxalic acid-NaOCl (p)

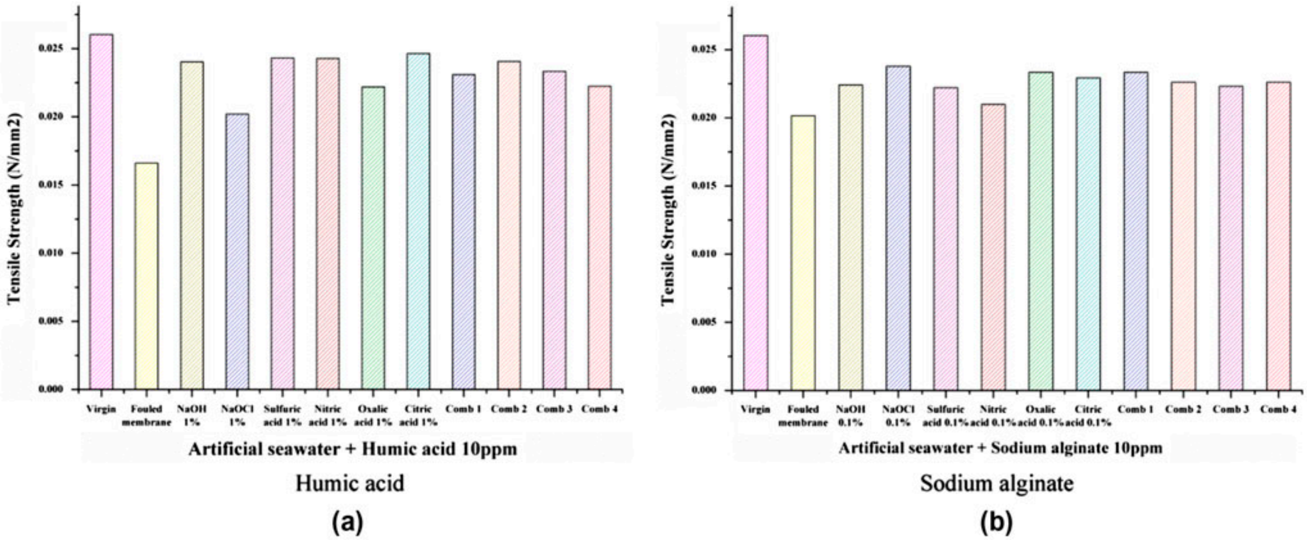
Fig. 7. SEM images of membrane with sodium alginate as foulant before and after chemical cleaning.

membrane, fouled membrane, and cleaned membrane. Tensile strength was calculated using following equation:

$$\sigma_{\beta} = l_{\beta} \times A_T \tag{3}$$

where σ_{β} is the tensile strength (N/mm^2), l_{β} is the maximum load (N), and A_T is membrane area (mm^2) [36,37].

Tensile strength is commonly used in the structural material for stress and strain linear relationship. The tensile strength was measured by pulling the



Humic acid (a) Sodium alginate (b)

Fig. 8. Tensile strength of membrane after chemical cleaning with humic acid and sodium alginate as foulant.

hollow fiber strings 5 cm up and down at a rate of 10 mm/min. Three trials were performed and the values were averaged.

Tensile strength of the virgin membrane and fouled membranes (humic acid and sodium alginate) were 0.02604, 0.01661, and 0.02016 N/mm^2, respectively. In the case of humic acid, after conducting the chemical cleaning at 1% concentration, the highest tensile strength was recorded using 1% $C_6H_8O_7$ as the cleaning solution with 0.02464 N/mm^2, while the lowest was observed using 1% NaOCl with 0.02021 N/mm^2. As shown in Fig. 8, in addition, alkaline agents and organic acid agents performed better than inorganic acid. Inorganic acid agents have low recovery rate than other agents. Foulants on membrane pores were not efficiently removed obtaining low value of tensile strength. Using sodium alginate, after conducting the chemical cleaning at 0.1% concentration, the lowest tensile strength was recorded using HNO_3 with 0.02099 N/mm^2, whereas 1% NaOCl recorded 0.02379 N/mm^2. For sodium alginate, all agents obtained similar values for tensile strength. However, in the case of humic acid single chemical cleaning, higher flux recovery rate recorded lower tensile strength value while lower recovery rate agent has higher tensile strength value.

Using combination chemical cleaning, higher flux recovery agents show better performance and similar value of tensile strength.

4. Conclusion

From the results, it was clear that flux recovery rate was affected by the kind of chemical cleaning agent used. In the case of humic acid, NaOCl cleaned the membrane more efficiently than NaOH. At 5% NaOCl, the best recovery rate in alkaline was obtained. However, all concentrations of NaOH as well as 3 and 5% NaOCl recorded over pH 12. PVDF membrane could be brittle when expose to pH higher than 12 for long time. Thus, 1% NaOCl was selected as the cleaning agent to be used in a combination chemical cleaning. For the acid chemical cleaning agents, 3% $C_2H_2O_4$ was the most effective, with a recovery rate of 79.3%. In the case of NaOH, HNO_3, and H_2SO_4 all concentrations have similar recovery rates. Thus, lower concentration is more efficient than higher concentration. For combination chemical cleaning, $C_2H_2O_4$–NaOCl–$C_2H_2O_4$ was observed to be the most effective at 8 h cleaning time with recovery rate of 97.6%.

Using sodium alginate as foulant, 5% NaOCl obtained the best performance recovery rate of 48.4% but still unstable due to high pH. Also, 0.1 and 1% NaOCl recorded 30.3 and 33.9%, respectively. Flux recovery rates were similar, thus 0.1% NaOCl was selected for use in series type chemical cleaning. For the acid chemical cleaning, all $C_2H_2O_4$ concentrations gained the highest flux recovery rate among other agents. The $C_2H_2O_4$ concentrations of 0.1, 1, 3, and 5% recorded 68.8, 69.1, 73.2, and 75.3% flux recovery rate, respectively. Nevertheless, 0.1% $C_2H_2O_4$ was used for combination chemical cleaning because a more stable performance is obtained at lower concentration. For combination chemical cleaning, NaOCl–$C_2H_2O_4$–NaOCl was observed to be the most effective at 8 h cleaning time, with recovery rate of 98.0%.

Cleaning efficiency was directly affected by cleaning temperature. Higher efficiency was achieved at higher cleaning temperature and lower efficiency was achieved at lower temperature. Result of tensile strength was different for humic acid and sodium alginate due to fouling factors.

This experiment was conducted in batch mode, thus further research is needed to study the effect of chemical cleaning in a continuous mode. Experiments could also be done using real seawater.

Nomenclature

Q	—	filtration flowrate, L/h
A	—	effective membrane area, m^2
ω_T	—	viscosity at actual temperature, μ
ω_{25}	—	viscosity at 25°C, μ
Flux_A	—	flux after chemical cleaning, LMH
Flux_F	—	flux of the fouled membrane, LMH
Flux_i	—	initial pure water flux, LMH
σ_β	—	tensile strength, N/mm^2
l_β	—	maximum load, N
A_T	—	membrane area, mm^2

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