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The effects of physical cleaning and chemical backwashing on foulant formation in a microfiltration membrane intended for the reuse of wastewater

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ABSTRACT

Biologically treated water contains a sufficient amount of organic matter and microorganisms to cause a variety of problems with microfiltration membranes. Membrane fouling caused by these organic materials is difficult to control with a single physical cleaning procedure. In this study, the effect of aeration conditions on chemical backwashing and foulant removal during chemical backwashing was analyzed. The cleaning efficiency improved as the chemical concentration and contact time increased. The chemical backwashing in the aeration condition showed outstanding cleaning efficiency. This indicated that physical cleaning was required during chemical backwashing because it initiated flow inside the submerged membrane tank. The foulant removal analysis indicated that the particle removal rate increased under the aeration condition, based on the turbidity and total organic carbon results, and that the amount of effluent dissolved matter increased as the chemical concentration and contact time increased, based on dissolved organic carbon, and UV radiation at 254 nm (UV_{254}) results. The Fourier transform infrared spectroscopy analysis indicated that chemical backwashing under the aeration condition controlled polysaccharides, a major foulant.

Keywords: Chemical backwashing; Organic fouling; Physical cleaning; Microfiltration; Wastewater reuse

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1. Introduction

Due to rapid industrial development and population growth, contemporary society has a rapidly increasing demand for water resources, and the concomitant increase in wastewater has led to the deterioration of water quality. Thus, it is urgent to develop efficient water treatment and reuse techniques. Reuse of treated wastewater has already been implemented for a long time in many countries such as the United States, the United Kingdom, and Japan. In the United States, each state has its own guidelines for water reuse [1]. In particular, in Orange County, California, wastewater treatment plant effluent has been reused as agricultural water and groundwater cultivation water using a high-level treatment system, Water Factory 21, since the late 1970s [2]. The development of stable and advanced water treatment techniques for water reuse has drawn much attention, and the application of membrane techniques such as microfiltration (MF) and ultrafiltration has increased in recent wastewater treatment processes [3–6]. Water treatment and wastewater reuse techniques using membranes have the advantages of superior pollutant removal efficiency, a wide application range, mobility of processes, and devices and high energy efficiency, and thus, have been widely used in various fields [7–10].

Despite the advantages of water treatment processes using membranes, an inevitable problem of these techniques is membrane fouling. Therefore, the membrane requires cleaning at regular intervals. It has been pointed out that increased filtration resistance from membrane fouling could reduce the membrane permeate flux and affect process efficiency [11–13].

It is also been reported that the actual reuse of treated water using membranes and membrane fouling is caused by organic matter such as particulates and microbial metabolites and treated wastewater and membrane fouling are greatly affected by colloidal materials with 0.1–0.45 μm size or by macromolecular organic matter with a 0.01–0.1 μm size composed mostly of extracellular polymeric substances (EPS) [14–19].

Membrane fouling can be divided into reversible membrane fouling and irreversible membrane fouling. It is generally known that the clogging of membrane micropores is irreversible, and the resistance caused by the deposition of cake layers that are formed on the membrane surface with time is reversible [20].

Among them, reversible membrane fouling forms gel or cake layers by the adsorption of particles on the membrane surface and can be removed by aeration. In addition, it applies greater air shear forces to the membrane surface or performs backwashing to remove materials accumulated and precipitated on the membrane surface.

However, irreversible membrane fouling, which is the clogging of pores within the membrane due to colloidal materials and dissolved matter, can be removed via strong chemical cleaning [21–25]. Irreversible membrane fouling is difficult to be prevented with only physical cleaning.

Therefore, in this study, to prevent the membrane fouling that occurs during MF, idle and aeration conditions were used for the physical cleaning method, and chemical backwashing was applied to a plate-type membrane, which has a problem with backwashing, using a non-pressure flow method.

The effects of the chemical concentration and cleaning time on the reduction ratios of the irreversible filtration resistance were compared and evaluated. After the experiment, the membrane surface was analyzed, and run-off water quality (TOC, DOC, UV254, turbidity) was analyzed.

2. Materials and methods

2.1. Membrane and raw water

The membrane used in this experiment was a submerged PTFE (polytetrafluoroethylene) plate-type MF membrane (Pure-EnviTech Co., Korea) with a surface area of 0.3 m^2 and a pore size of 0.1 μm. The membrane was fouled up to a certain degree after the filtering of raw water, which was the supernatant of a secondary settling pond treated at the wastewater treatment plant of Sungkyunkwan University (Korea). Table 1 lists the water quality of the raw water.

2.2. Experimental device and operation conditions

Fig. 1 shows the experimental device used in this study. It consisted of a submerged membrane tank with a 32 L of effective volume and a filtration/backwashing device. The filtration device consisted of a

Table 1
Properties of the raw water used in this study

Parameters	Secondary effluent
Turbidity (NTU)	2.8–4.6
SS (mg/L)	6–8
COD_{cr} (mg/L)	5–40
$SCOD_{cr}$ (mg/L)	0–8
TN (mg/L)	15–20
TP (mg/L)	2–3.5
NO_3-N (mg/L)	15–20
NH_3-N (mg/L)	0–2

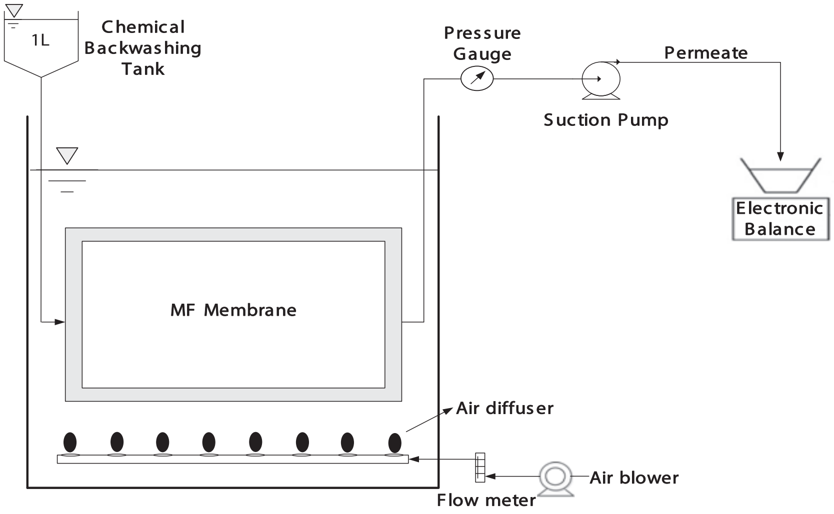

Fig. 1. Schematic of the lab-scale experiment device.

peristaltic pump (Cole-Parmer, USA) whose revolutions per minute can be controlled, and a digital pressure gauge that can measure suction pressure. One liter of chemical backwashing water was injected in the fouled membrane in the distilled water tank by gravity driven because the plate-type membrane surface could swell and be damaged if pressure is applied during the chemical backwashing. The experiment was performed at various concentrations of chemical backwashing water (0, 300, 600, and 1,200 mg/L NaOCl). The chemical contact time after the chemical backwashing was set at 30, 60, 90, and 120 min. During the chemical contact time, to evaluate the reduction ratio of the filtration resistance based on the physical cleaning, idle conditions and aeration conditions were applied. The idle condition was a stationary state without air supply in the tank; in the aeration condition, air was supplied to the blower installed at the bottom of the reaction tank at a flow rate of 30 L/min. To measure the filtration resistance before and after the cleaning experiment, the filtration was performed using a peristaltic pump, the filtration pressure was measured using an electronic pressure meter and the permeate flux was measured using an electronic balance. Table 2 lists the operation conditions.

2.3. Analysis method

The flux (J) is calculated by the flow rate through the unit membrane area per unit time. The pure water flux is obtained by Darcy's law. The inherent membrane resistance (R_m) is calculated by Eq. (1). When membrane fouling occurs, not only inherent membrane resistance (R_m), but also the irreversible resistance (R_i) and reversible resistance (R_r) should be considered to calculate the total filtration resistance (R_T). The total filtration resistance of the fouled membrane is calculated using Eq. (2). The removable filtration resistance in physical cleaning and chemical backwashing is calculated as shown in Eq. (3). Subsequently, the reduction ratios of the irreversible resistance (R_i/R_t) were calculated by calculating the irreversible resistance after a certain period of time as shown in Eq. (4). The cleaning efficiency can be calculated using Eq. (5).

Moreover, the foulants removed from the membrane surface were analyzed by collecting the samples within the submerged membrane tank at regular time intervals. Total organic carbon (TOC), dissolved organic carbon (DOC), UV_{254}, and the turbidity were analyzed.

Maruyama et al. (2001) reported that it was possible to estimate the major functional groups of organic matter using attenuated total reflectance Fourier transform infrared spectroscopy (ATR-FTIR) analysis. Therefore, the composition of membrane surface foulants and the relative reduction amount of the foulants after physical cleaning and chemical backwashing were examined using ATR-FTIR (Nicolett 5700, Thermo Electron Corporation, USA) equipped with diamond crystal ATR unit. All spectra were collected with 32 scans at 1 cm^{-1} resolution and analyzed with OMNIC 7.3 software after the experiment was completed:

Table 2
Operation conditions

Parameters	NaOCl concentration (mg/L)	Physical methods	Chemical contact time (min)
Operating conditions	300	W/O aeration/W aeration	30, 60, 90, 120
	600	W/O aeration/W aeration	30, 60, 90, 120
	1,200	W/O aeration/W aeration	30, 60, 90, 120

(1) First, the inherent membrane resistance (R_m) is calculated.

$$J = \frac{dV}{Adt} = \frac{\Delta P}{\mu\ R_m} \tag{1}$$

(2) Second, the total filtration resistance (R_T) is calculated.

$$J = \frac{\Delta P}{\mu\ R_T} = \frac{\Delta P}{\mu(R_m + R_i + R_r)} \tag{2}$$

(3) Third, the reversible resistance which can be removed via physical cleaning and chemical backwashing is calculated.

$$R_r = R_T - (R_m + R_i) \tag{3}$$

Fourth, irreversible resistance that cannot be removed via physical cleaning and chemical backwashing (R_i) is calculated.

$$R_i = R_T - (R_m + R_r) \tag{4}$$

$$\text{Cleaning efficiency}(\%) = \frac{R_T - R_t}{R_T - R_m} \times 100 \tag{5}$$

where J: flux ($m^3/m^2/s$), v: filtration volume (m^3), t: filtration time (s), ΔP: transmembrane pressure (Pa), μ: viscosity (Pa s), R_T: total filtration resistance (1/m), R_t: after cleaning resistance (1/m), R_m: inherent membrane resistance (1/m), R_i: irreversible resistance that cannot be removed via physical cleaning and chemical backwashing (1/m), R_r: reversible resistance that can be removed via physical cleaning and chemical backwashing (1/m).

3. Results and discussion

3.1. Evaluation of the physical cleaning based on the chemical backwashing

To investigate the reduction of irreversible membrane fouling based on the chemical backwashing and physical cleaning method, the total filtration resistance of the membrane fouled with the treated wastewater was measured, and the irreversible resistance depending on the chemical concentration and contact time was measured during chemical backwashing. The graph in Fig. 2(a) shows the reduction ratios of the irreversible resistance (R_i/R_T) depending on the chemical backwashing concentration for the idle condition. In the idle condition, for the 30, 60, and 120 min contact times after chemical backwashing, the filtration resistance reduction ratios were 0.93–0.87 at 0 mg/L NaOCl, 0.94–0.76 at 300 mg/L NaOCl, 0.85–0.68 at 600 mg/L NaOCl, and 0.83–0.6 at 1,200 mg/L NaOCl. The reduction ratios of the irreversible resistance increased as the chemical concentration and cleaning time increased, except at 0 mg/L NaOCl, which was used for the water backwashing.

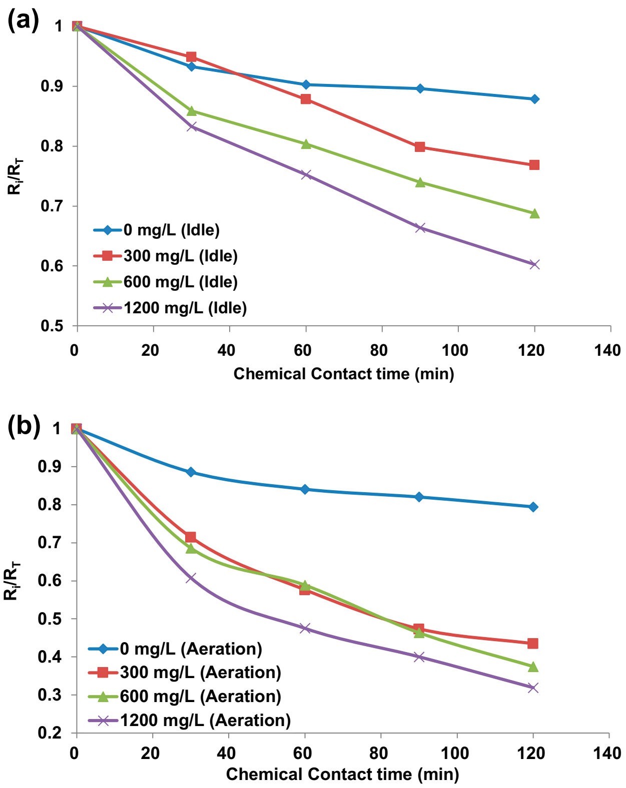

Fig. 2. R_i/R_T of (a) idle and (b) aeration conditions, depending on the chemical backwashing concentration.

The graph in Fig. 2(b) shows the R_i/R_T values based on the NaOCl concentration for the aeration condition during chemical backwashing. In the aeration condition, for the 30, 60, and 120 min contact times after the chemical backwashing, the reduction ratios of the irreversible resistance were 0.88–0.79 at 0 mg/L NaOCl, 0.71–0.43 at 300 mg/L NaOCl, 0.68–0.37 at 600 mg/L NaOCl, and 0.6–0.31 at 1,200 mg/L NaOCl. It was found that in the aeration condition, the reduction ratios of the irreversible resistance for NaOCl chemical backwashing was more than twice that for non-NaOCl backwashing at each contact time. Also, the irreversible resistance decreased as the NaOCl concentration and contact time increased, but the 300–600 mg/L NaOCl concentrations showed similar irreversible resistance reduction ratios. It is considered that aeration is more effective than chemical concentration. Fig. 3 shows the cleaning efficiency depending on the physical cleaning and chemical backwashing concentration. As described earlier, the cleaning efficiency improved as the chemical concentration and contact time increased, except in the 30 min idle condition.

3.2. *Evaluation of the membrane foulant removal characteristics according to the chemical backwashing and physical cleaning*

In this experiment, chemical backwashing was performed at various NaOCl concentrations (300, 600, and 1,200 mg/L) for 10, 30, 60, 90, and 120 min chemical contact times according to the physical cleaning method, and samples of the effluent organic matter in the tank were collected and analyzed at each contact time. As shown in Fig. 4(a), for the idle condition, the amount of effluent organic matter was found to be smaller than the amount of other foulants based on the turbidity, which represents particulate materials.

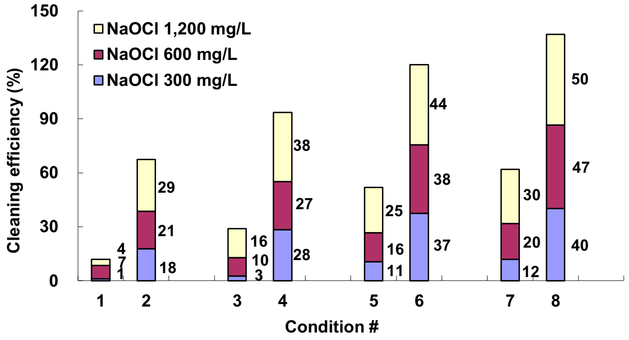

Fig. 3. Cleaning efficiency depending on the physical cleaning and chemical backwashing concentration; #1: Idle 30 min, #2: Aeration 30 min, #3: Idle 60 min, #4: Aeration 60 min, #5: Idle 90 min, #6: Aeration 90 min, #7: Idle 120 min, #8: Aeration 120 min.

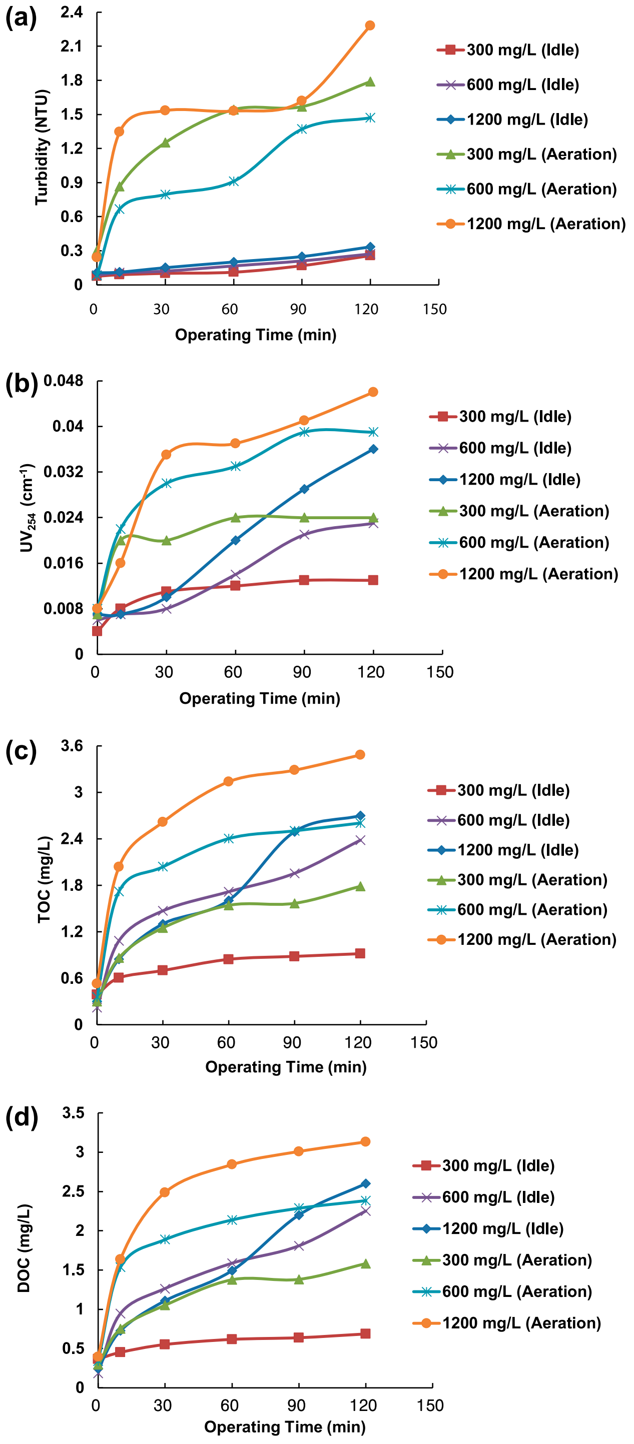

Fig. 4. Amount of effluent foulants according to the time and (a) turbidity, (b) UV_{254}, (c) TOC, and (d) DOC.

On the other hand, as shown in Fig. 4(b)–(d), the amount of effluent organic matter increased as the concentration and time increased, based on the TOC, DOC, and UV_{254} results. The rate of increase in the amount of effluent organic matter in the tank with the passage of time was higher in the aeration condition than under the idle condition, based on the TOC, DOC, and UV_{254} results. This is because the air bubbles of the upflow aeration generated from the blower at the bottom of the membrane applied shear force to the materials deposited on the membrane surface, and thereby detached the materials from the membrane surface.

Therefore, it is thought that the filtration resistance for the membrane filtration process could be reduced by removing the foulants that are clogging the micropores using chemical backwashing and aeration, because the flow speed and condition of the air bubbles are important in controlling the fouling.

3.3. FTIR analysis of the membrane surface foulants with chemical backwashing and physical cleaning

In the spectrum of the fouled membrane, a wide peak at 3,000–3,600 cm^{-1} centered around the peak at 3,286 cm^{-1} represented stretching of the O–H bond of hydroxyl functional groups, and the peak at 2,924 cm^{-1} represented stretching of the C–H bond [26,27]. Moreover, the peak at 2,363 cm^{-1} is affected by the stretching of the C–O bond [28]. The two peaks at 1,636 and 1,541 cm^{-1} represent the protein secondary structures called amides I and amides II, respectively [29]. These proteins are among the major components of bound EPS [30]. The peak at 1,374 cm^{-1} represents a carboxylate or inorganic ion [31]. The strong peak at 1,052 cm^{-1} represents polysaccharides, a major component of bound EPS materials, along with protein [30,31]. Based on the FTIR analysis, it is thought that EPS materials such as protein and polysaccharides from the biofilm were the major membrane foulants [32].

Ivnitsky et al. reported that the change in the FTIR spectrum of organic matter could be observed by calculating the major peak ratios depending on the intensity [32,33]. In other words, the change in the relative concentration of the foulants could be examined by comparing the intensity ratios of the major peaks for each sample.

Therefore, in this study, the removal characteristics of the membrane surface foulants depending on the physical cleaning method were evaluated using major peak ratios at 3,286, 2,924, 2,363, 1,636, 1,541, 1,374, and 1,052 cm^{-1}. In addition, it is possible to examine changes in pollutant concentrations relatively by comparing the intensity ratios of the major peaks by C–O bond stretching as the peak at 2,363, 3,286, 2,924, 2,363, 1,636, 1,541, 1,374, and 1,052 cm^{-1}.

Figs. 5 and 6 show the results of the FTIR analysis of the surface foulants from the membranes for the 120 min contact time, to which each physical cleaning method was applied according to the chemical backwashing concentration. For each membrane to which the idle or aeration condition was applied, similar peaks were observed regardless of the change in chemical concentration.

Fig. 7 shows the results of the analysis of the relative comparison of the membrane foulants based on the major FTIR spectrum peak ratios. The relative

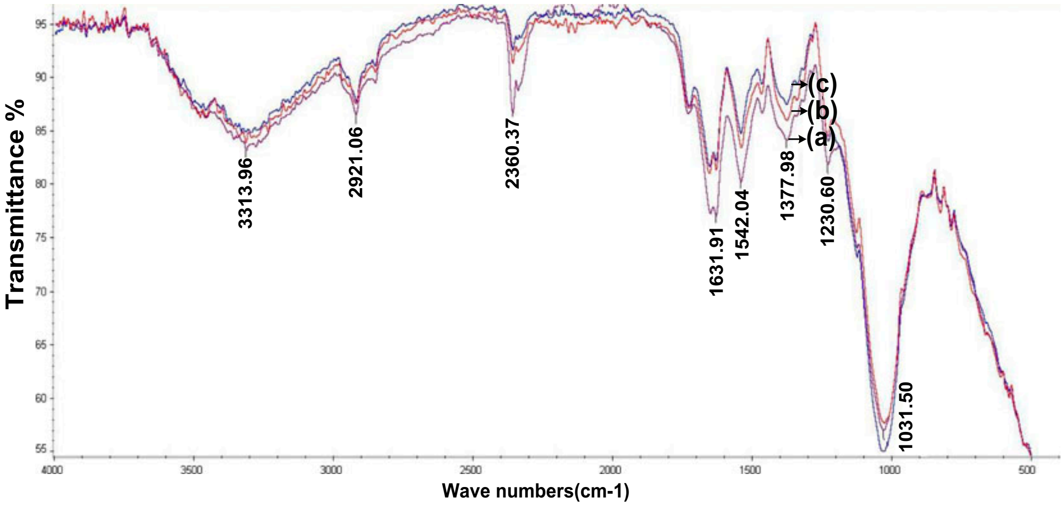

Fig. 5. Membrane foulant FTIR comparison analysis under the idle condition for (a) 300 mg/L NaOCl, (b) 600 mg/L NaOCl, and (c) 1,200 mg/L NaOCl.

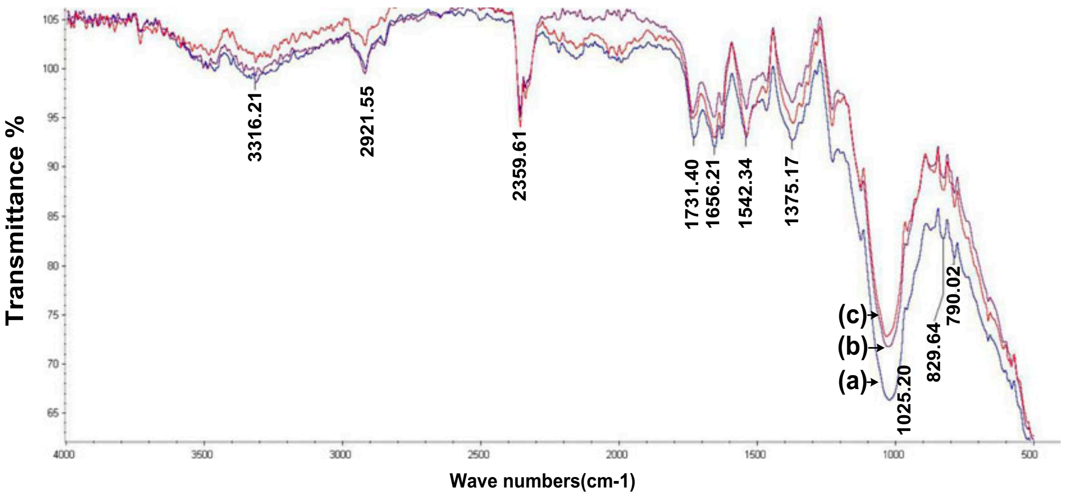

Fig. 6. Membrane foulant FTIR comparison analysis under the aeration condition for 300 mg/L NaOCl, (b) 600 mg/L NaOCl, and (c) 1,200 mg/L NaOCl.

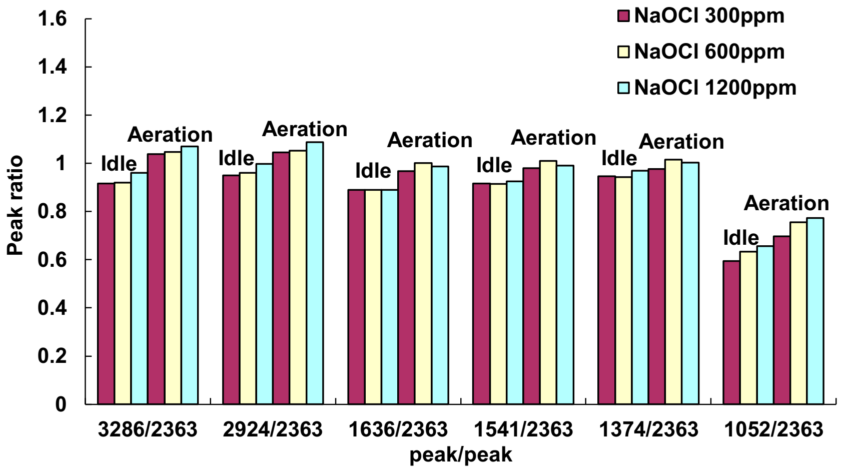

Fig. 7. Membrane foulant FTIR spectrum peak ratio analysis depending on the physical cleaning.

peak ratios of the idle or aeration condition increased as the chemical concentration increased, but there was no significant change. However, the relative peak ratios showed significant changes based on the physical cleaning method. The peak ratios were the lowest under the idle condition, which also showed the lowest cleaning efficiency, and the peak ratios were high under the aeration condition. Also, the peak ratios of the polysaccharides increased as the chemical concentration increased, and the polysaccharide peak ratios were high in the aeration condition, which also showed high cleaning efficiency. Based on these results, it was found that cleaning efficiency and polysaccharides were closely related.

4. Conclusion

In this study, chemical backwashing to control membrane fouling was applied to a wastewater reuse membrane process, and the cleaning efficiency and membrane foulant removal characteristics depending on the physical cleaning method were compared and evaluated.

For the cleaning efficiency of chemical backwashing and physical cleaning, cleaning efficiency improved as chemical concentration and contact time increased. Moreover, considering that the cleaning efficiency was much higher in the aeration condition than in the idle condition, it was found that physical cleaning, which could form a water flow, is essential for chemical backwashing during contact time. The aeration condition applied during the chemical contact time was found to have superior cleaning efficiency for each condition including the chemical concentration and contact time.

The results of the analysis of foulants that chemical backwashing removed indicated that for the aeration condition, high concentrations of particulate materials were detected based on the turbidity and TOC results, and that the amount of the effluent dissolved matter increased as the concentration and time increased, based on the DOC and UV_{254} results. These findings are thought to be because the aeration condition applied shear force to the particulate foulants that were deposited on the membrane surface, and thereby detached the foulants from the membrane surface. The analysis of the FTIR relative peak ratios of the foulants indicated that the relative peak ratios increased as the chemical concentration increased, but insignificantly. However, the peak ratios showed significant changes according to the physical cleaning method. In particular, the polysaccharide peak ratios were high in the aeration condition, which also showed high cleaning efficiency. Therefore, it was found that the cleaning efficiency and polysaccharides were closely related.

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