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Optimizing the coagulant dose to control membrane fouling in combined coagulation/ultrafiltration systems for textile wastewater reclamation

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ABSTRACT

Optimal coagulation conditions need to be re-examined when coagulation is coupled to membrane filtration for wastewater treatment. This work focused on the optimization of coagulant dosing in order to control membrane fouling in ultrafiltration (UF), following coagulation for the reclamation of textile wastewater. The effects of pore size and coagulant types and dosages on flux decline were investigated using a stirred-cell UF unit. The flux was greatly enhanced for the UF membrane when a coagulant was added, whereas for the microfiltration (MF) membrane the flux decreased. This could be attributed to changes in the size of coagulated particles and their interaction with membrane pores. At a low dosage (e.g., 0.0371 mM as Al), the polyaluminum chloride (PACl) coagulant was found to control the flux decline most effectively for low ionic-strength wastewater. The optimal dose minimized the fouling and cake layer resistances, although it was sharp and dependent on influent composition. The cake layer protected the membrane from fouling, but it provided additional resistance to permeation. Analyses of turbidity, particle size, and membrane surface exhibited the characteristics of coagulated particles and their cake structures that are closely associated with flux behavior.

1. Introduction

Due to the shortage of water and more stringent regulations for wastewater treatment, water recycling is not optional, but mandatory in many countries including Korea. Of the various advanced treatment systems that have demonstrated wastewater reclamation, membrane separation is one of the most attractive systems regarding the removal of particles and dissolved contaminants in wastewater. In particular, loose membrane processes such as microfiltration (MF) or ultrafiltration (UF) can replace conventional clarification methods such as settling and sand filtration, since they can be operated at a high flux and/or a low pressure [1,2]. When a membrane is used alone, however, the flux normally decreases rapidly due to membrane fouling caused by the accumulation of colloids and organics at the membrane surface. Thus, there has been a need for the pretreatment of feed water to control membrane fouling [3,4].

Several methods prior to MF or UF have been tested with regard to minimizing membrane fouling, and to enhance membrane performance, they include coagulation, adsorption, and ozonation [5,6]. When ozonation is used before membrane treatment, it helped to reduce membrane fouling caused by organic matter, since ozone degraded organics that can foul the membranes [7–9]. The residual ozone, however, can damage the polymeric membranes used and thereby, should be destroyed otherwise ozone-resistant ceramic membranes are employed. Powdered activated carbon (PAC) adsorption improved the permeate flux in the hybrid UF or MF membrane processes regarding wastewater reuse [10–12]. PAC is effective in removing organic foulants present in feedwater, but the spent carbons that should be disposed are generated. Some studies regarding the combination of MF or UF with coagulation have also shown that coagulation was effective in reducing membrane fouling caused by fine particles, as well as in the removal of particulate contaminants [13]. The pretreatment of coagulation is thus preferred if the wastewater contains a large amount of fine particles. It has been reported that the type and dosage of coagulants had an effect on the removal of particulate contaminants from wastewater [14,15]. Also, the characteristics of coagulated particles, such as surface charges and size, were affected by the doses of coagulants. This is because the cake layer formed at the membrane surface had different permeability [16]. In our previous study, the addition of coagulants to textile wastewater reduced the fouling of UF membranes substantially [14]. The interaction of coagulated particles with membranes, however, has not been fully understood and the optimization of coagulant doses has not been established yet in hybrid membrane systems.

Therefore, the aim of this study was to optimize the coagulant dose in combined coagulation/UF processes for the reclamation

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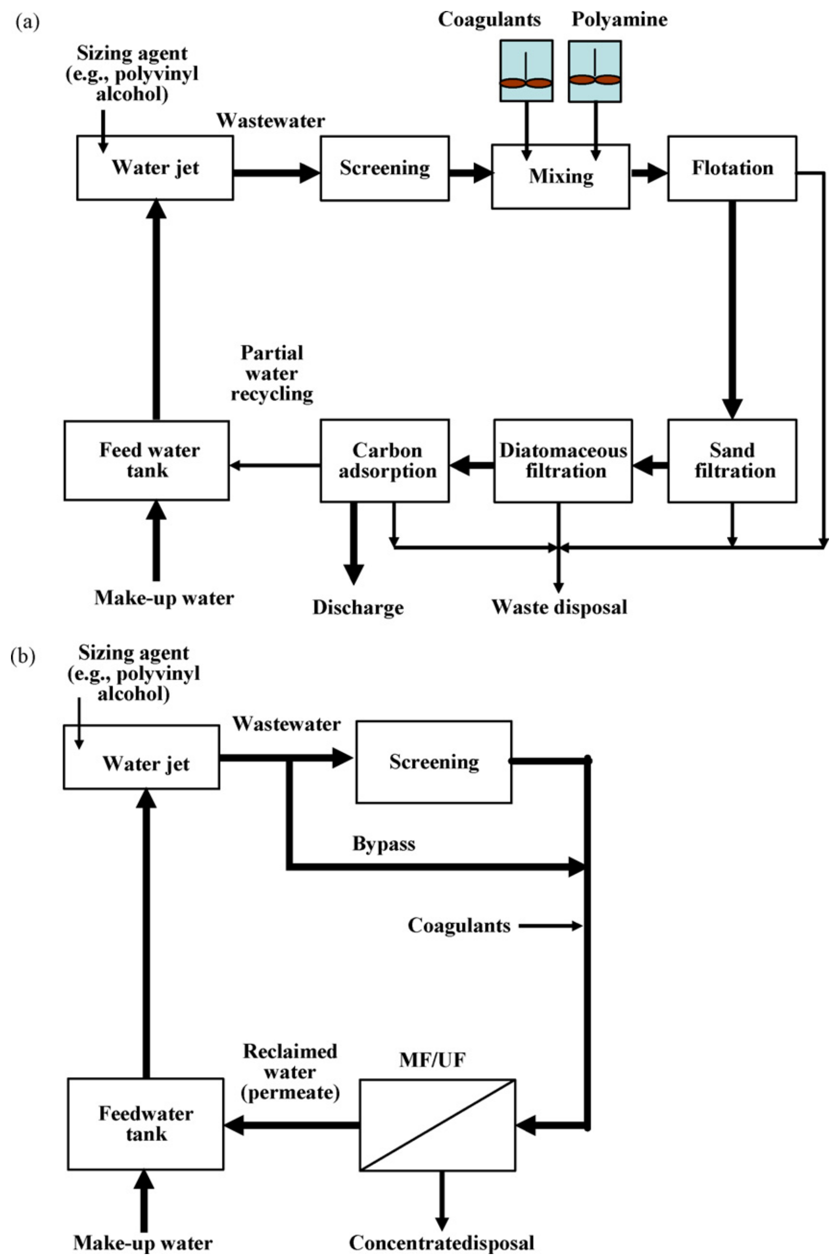

Fig. 1. Flow chart of (a) an existing textile wastewater treatment facility and (b) a proposed membrane-based wastewater reclamation system.

of textile wastewater from a water jet loom. The ultimate goal is to be able to substitute for the existing treatment facility that is composed of a series of complicated processes (e.g., flotation, sand filtration, carbon filtration, and diatomaceous filtration) (Fig. 1). The effects of coagulant types and membrane pore sizes on fouling control and turbidity removal were also evaluated by monitoring the change of the particle size distributions of wastewater at different coagulant dosages. The optimal dosage of polyaluminum chloride (PACl) and the mechanism of fouling control were explored.

2. Materials and methods

2.1. Wastewater and coagulants

Textile wastewater samples were collected from a water jet loom in a textile company in Gyeongsan, Korea and were shipped to our laboratory and stored at 4 °C before use. The wastewater samples for each experimental run were then equilibrated at room temperature for a couple of hours. The key composition of the wastewater is as follows: pH, 7.2–7.4; turbidity, 20.1–31.0 NTU; conductivity, 546–2930 μS/cm; and total organic carbon (TOC), 56 mg/L. The wastewater that had a low ionic strength of 546 μS/cm was used if otherwise mentioned. Two different coagulants, such as ferric chloride (Samchun Chemical, Korea) and PACl (Komax Chemical, Korea), were used.

2.2. Coagulation tests

Jar tests were carried out to evaluate the coagulation efficiency of PACl. 500 mL of textile wastewater was filled in each of six 1-L beakers, respectively. PACl was added to each beaker in the wide dosing range of 0.005–5 mM and the solution pH was adjusted to 7.0 using Na_2CO_3. The coagulant dose was based on the coagulation diagram reported in the literature [17]. The wastewater samples were first agitated vigorously at 170 rpm for 1 min and subsequently mixed at a reduced speed of 60 and 30 rpm for 15 and 5 min, respectively. After coagulation, the samples were settled for 90 min and then an aliquot of 50 mL (supernatant) was sampled from the clean top zone of the beaker for the measurement of residual turbidity.

2.3. Membranes and membrane operation

The mixed cellulose ester MF and regenerated cellulose UF membranes (Millipore, USA) used in this study had pore sizes of 0.45 μm, 0.1 μm, and 100,000 Da. An Amicon stirred-cell UF unit (Model 8200, Millipore, USA) was used for the MF and UF of coagulated wastewater. A predetermined volume of coagulant solutions was added into the stirred cell containing 180 mL of wastewater, the mix was agitated vigorously at 200 rpm (Reynolds number = 2.08×10^4) for 5 min (which corresponds to G and Gt values of 7120 s^{-1} and 2.14×10^6, respectively and meets the criteria required for rapid mixing), and then, membrane experiments began under 0.5 bar while mixing the solution at 200 rpm and 20 °C. During MF or UF, the permeate masses were monitored using an electronic balance in order to check membrane flux and the data were recorded on a personal computer connected to the balance.

2.4. Determination of hydraulic filtration resistance during MF/UF

The addition of coagulants, with respect to different levels of membrane flux decline, was assessed using the resistance-in-series model [18]. The model is provided in the following equation:

$$J = \frac{\Delta P}{\eta(R_m + R_f + R_c)} \quad (1)$$

where J is the flux for pure water and wastewater during UF (L/m^2 h), ΔP is the transmembrane pressure (bar), η is the dynamic viscosity of the permeate (Pa s), R_m is the intrinsic membrane resistance (m^{-1}), R_c is the cake layer resistance caused by particle deposition (m^{-1}), and R_f is the fouling layer resistance caused by internal pore clogging (m^{-1}). The initial water flux of a virgin membrane was used to calculate the R_m value, whereas the final water flux of the membrane was used to calculate the R_f value. Before the measurement of final water fluxes, the membrane cell was emptied, the used membrane was placed upside down and then backwashed at 1.0 bar with 180 mL of deionized water to remove the cake layer. The physical membrane cleaning was completed with further water flushing as follows. The membrane was returned to its proper position and washed with deionized water for 30 s while being agitated at 400 rpm. The R_c value was calculated using the flux with raw or coagulated wastewater obtained at the end of membrane filtration. The extrinsic resistance of R_c plus R_f, which is a function of time or permeation volume, was calculated using the membrane flux with wastewater samples and the initial water flux. The extrinsic resistance ($R_c + R_f$) can be calculated by subtracting the intrinsic membrane resistance (R_m) from the total filtration resistance ($R_m + R_c + R_f$).

2.5. Analytical methods

The turbidity level was measured using a Hach 2100N turbidimeter, whereas the TOC concentration was determined using an Ionics Sievers 800 TOC analyzer. Particle size distribution measurements were taken on a Beckman Coulter LS 13 320 particle size analyzer. Membrane surfaces were visualized using a filed emission scanning electron microscope (S-430, Hitachi, Japan).

3. Results and discussion

3.1. The effects of membrane pore size on fouling with the addition of coagulant

In order to choose a suitable membrane for textile wastewater treatment with coagulation, the effects of membrane pore size on relative flux (RF) decline and extrinsic filtration resistance were evaluated and are shown in Fig. 2. When no coagulant was added to raw wastewater, a sharp flux decline occurred with the 0.1-μm MF and UF membranes, but a relatively small flux reduction took place with the 0.45-μm membrane (Fig. 2a). Accordingly, the extrinsic filtration resistance ($R_c + R_f$) increased during MF and UF runs. When 0.0371 mM of PACl as Al was dosed to wastewater, the flux profile changed significantly and the added coagulant was not always helpful in improving the flux (Fig. 2b). A significant improvement in flux was observed with the addition of PACl for the UF membrane, whereas for the MF membranes, the flux showed a severe decline. The filtration resistance profiles varied correspondingly. In particular, the 0.45-μm membrane showed a further flux decline of more than 25%. This can be explained by the pore blocking theory as follows [19]. The fine colloids, which can clog the UF membrane, increase in size with coagulation, so they may not foul the membrane any more. Some of the coagulated particles, however, are still

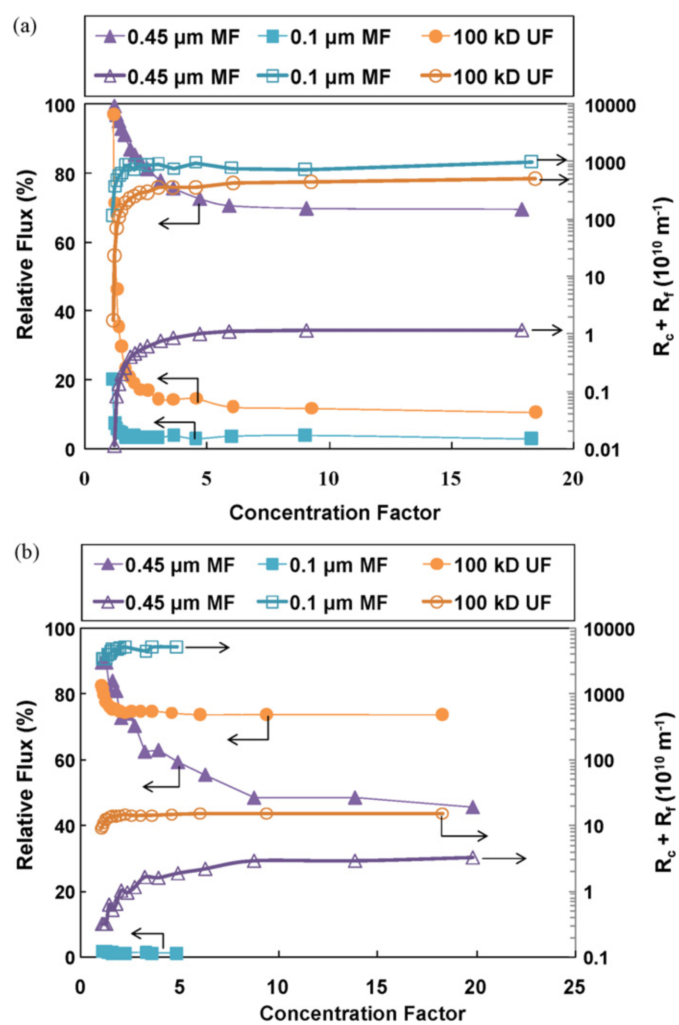

Fig. 2. The variation of relative flux and extrinsic filtration resistance for MF and UF membranes having different pore sizes: (a) without and (b) with dosing PACl (0.0371 mM as Al). Concentration factor is defined as the ratio of retentate volume to initial volume.

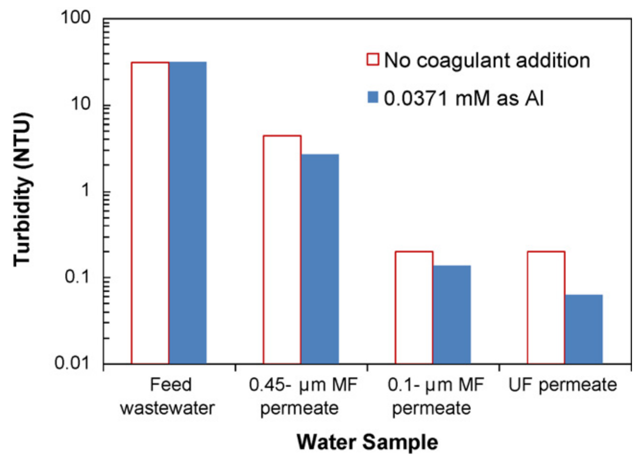

Fig. 3. The variations of influent wastewater and permeate turbidity with and without coagulation during MF and UF.

within the range of particle sizes that can plug the pores of the MF membrane. Fig. 3 supports the above scenario. It can be primarily assumed that when no coagulant was added to wastewater, the turbidity removed was attributable to the rejection of particles only by the membrane. It was revealed that some fine particles were still being rejected with a membrane pore size of 0.1 μm or smaller. The coagulation process helped achieve further turbidity removals. Especially, the greater turbidity removal by the UF membrane occurred with coagulation than that of the 0.1-μm MF membrane, although there was no difference in permeate turbidity between them without coagulation. The results implied that some particles that had been even smaller than UF pores grew bigger and rejected. Thus, the 0.1-μm MF membrane should have had a possibility of pore plugging by such coagulated particles leading to further flux reduction. As a result, of the membranes tested, the UF membrane was found to be the most improved in terms of fouling control, along with the addition of coagulants for the hybrid membrane treatment of textile wastewater. Therefore, the UF membrane was used for the remainder of this study.

3.2. The effects of coagulant types and dosages

To begin with, jar tests were conducted with a range of PACl doses (0.00927–0.371 mM as Al) to identify the optimum dose for coagulation (Fig. 4). The turbidity removal efficiency increased with higher PACl doses and nearly leveled off at a PACl dosage of

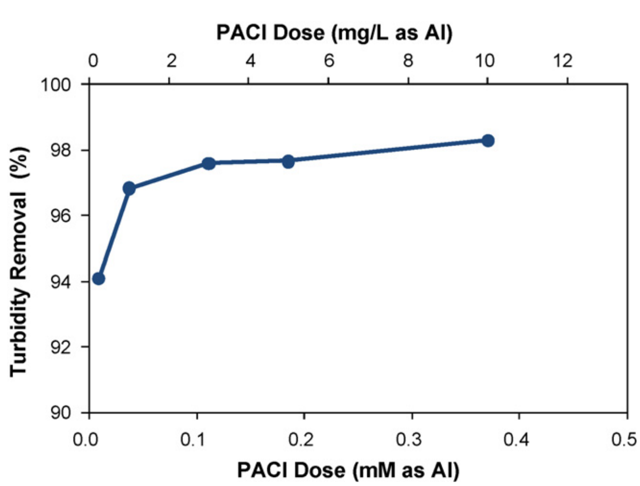

Fig. 4. Turbidity removal efficiency as a function of PACl dosage in coagulation tests.

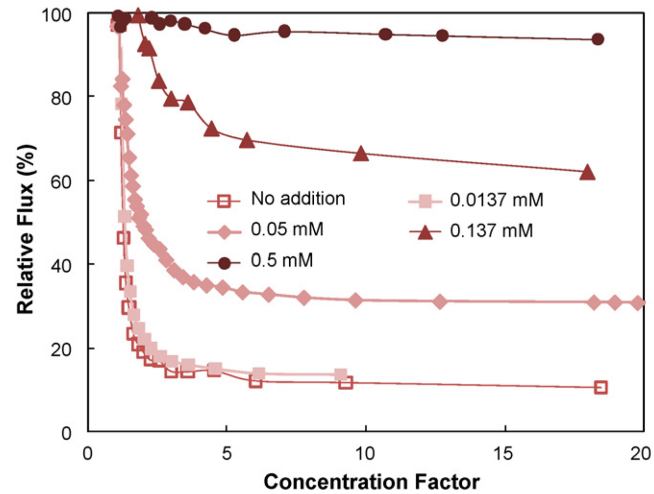

Fig. 5. The effects of the addition of ferric chloride on permeate flux during UF of textile wastewater.

approximately 0.1 mM as Al. Based on these data, therefore, UF tests were conducted with different metal coagulant doses, such as <0.1, 0.1–0.2, and >0.2 mM. Figs. 5 and 6 show the effects of the types and doses of coagulants on the flux for the UF of textile wastewater. The coagulants used affected the behavior of flux decline during UF treatment. Without any addition of coagulants, the fluxes declined sharply by more than 85%. The flux, however, increased by approximately 20, 60 and 80% when 0.05, 0.179 and 0.5 mM of ferric chloride as Fe were added, respectively, whereas there was no improvement in the flux with a very small dose of ferric chloride (0.0137 mM). Trends in flux decline changed significantly with the addition of PACl, as shown in Fig. 6. Although a substantial increase in flux was achieved with 0.0371 mM of PACl as Al, the flux dropped with a larger dose of PACl; the addition of 0.185 and 0.371 mM of PACl as Al had a negative effect on flux enhancement, possibly due to the deposition of precipitated coagulants. In summary, PACl controlled membrane fouling more effectively at a minimal dose to neutralize charge and/or form bridges between particles. In contrast, a substantial dose of iron, which belongs to a dose to generate a sweep floc, was needed with respect to fouling control. The degree of fouling control, with the dosing of coagulants to wastewater, should be associated with the combined effects of

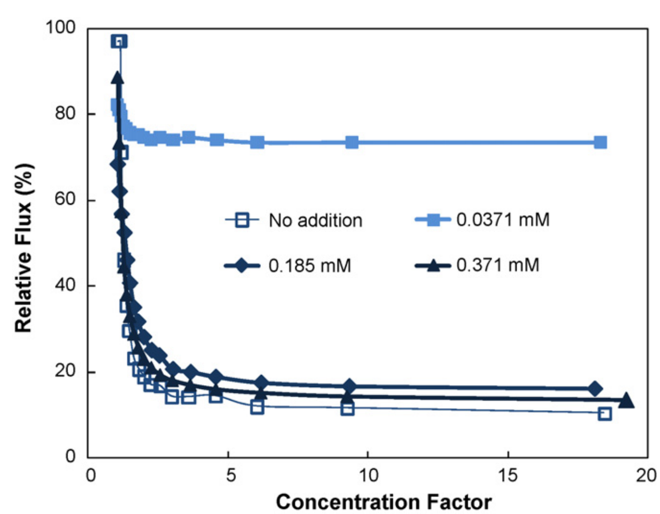

Fig. 6. The effects of the addition of PACl on membrane flux during UF of textile wastewater.

Table 1
The effects of wastewater particles and coagulants on hydraulic filtration resistance.

Feed	Coagulant	Dose (mM as Fe or Al)	R_m (10^{10} m^{-1})	R_c (10^{10} m^{-1})	R_f (10^{10} m^{-1})
Raw wastewater	–	–	42.4	39.5	11.0
Raw wastewater	PACl	0.0371	42.6	11.9	1.94
Raw wastewater	PACl	0.371	43.5	33.0	9.15
Raw wastewater	$FeCl_3$	0.0179	43.3	37.8	10.5
Raw wastewater	$FeCl_3$	0.179	42.8	15.7	1.40
Deionized water	PACl	0.0371	43.1	0.324	0.288
Deionized water	PACl	0.371	43.3	1.87	7.27
Deionized water	$FeCl_3$	0.0179	42.8	1.92	6.95
Deionized water	$FeCl_3$	0.179	43.2	1.30	0.180

coagulated wastewater particles and precipitated coagulants. As a result, the dosage of a coagulant should be optimized in order to minimize membrane fouling, rather than having this achieved by adding a sufficient amount of a certain coagulant.

The hydraulic filtration resistances were calculated using Eq. (1) and the values are given in Table 1. All R_m values were of the same level, confirming that the membranes tested for each run showed almost the same permeability. For wastewater, R_c was always higher than R_f. This was likely due to the formation of cake layers with particles present in wastewater. A decrease in R_c occurred with the dosing of coagulants to wastewater (most substantially with 0.0371 mM of PACl as Al). The decrease in R_f was also significant at a lower dose of PACl and a higher dose of ferric chloride. Overall, the two coagulants tested showed different effects on flux decline, depending on their dosages. The larger PACl dosage aggravated the flux decline thus leading to a further increase in R_c. A small dose of ferric salt, however, did not seem to be enough to coagulate wastewater colloids. Therefore, a larger dose was needed.

To check for any influence of the coagulants themselves, PACl and ferric chloride were added to the deionized water and then, the solution pH was adjusted to 7. For deionized water with a larger PACl dose of 0.371 mM as Al and a smaller ferric chloride dose of 0.0179 mM as Fe, the R_f contribution was more significant for the total resistance than that of R_c. This indicates that the self-precipitated PACl or ferric hydroxide particles themselves contribute to the clogging of UF membrane pores. This supports the fact that the fouling control with the dosing of coagulants to wastewater is linked to the combined consequence of coagulated wastewater particles and precipitated coagulants. Thus, it was found that a small dose of PACl (e.g., 0.0371 mM as Al) was most effective in controlling R_c and R_f, as well as minimizing chemical sludge production, and further investigation regarding the optimal PACl dosage was needed (which will be discussed in a later section).

3.3. The optimization of PACl doses in terms of fouling control

To determine the optimum dosage of PACl in terms of fouling control, the PACl dosage varied in the range of 0–5.0 mM as Al and its effects on flux decline were examined, along with the analyses of the turbidity levels of supernatant and UF permeate (Figs. 7 and 8). The supernatant turbidity decreased substantially with higher PACl dosages, while the UF permeate turbidity remained stable within a range of 0.07–0.25 NTU not being influenced significantly by the coagulant dosage. This justifies dosing a small quantity of the coagulant where membrane fouling can be minimized as well. As expected, there existed an optimal PACl dosage with respect to fouling minimization, but depending on the characteristics of feed wastewater. For wastewater with a low ionic strength (546 μS/cm), the highest flux was obtained at approximately 0.0371 mM of PACl as Al, whereas for wastewater with a high ionic strength (2390 μS/cm), the flux was maximal at a PACl dose of approximately 0.5 mM as Al. The optimal PACl dose was shifted to a higher level with the increased ionic strength. This is possible because particles that exist in a high ionic-strength environment are more stable and thereby their coagulation occurs with larger coagulant dosages. A previous finding demonstrated a similar phenomenon that high alkalinity increased the coagulant dosage for charge neutralization of particles [20]. Since the optimal range was sharp and varied in response to changes in influent composition, it might be hard to achieve the optimum in a practical application. Nevertheless, the above results have important implications that an excessive amount of coagulant should not be added with regard to fouling control as

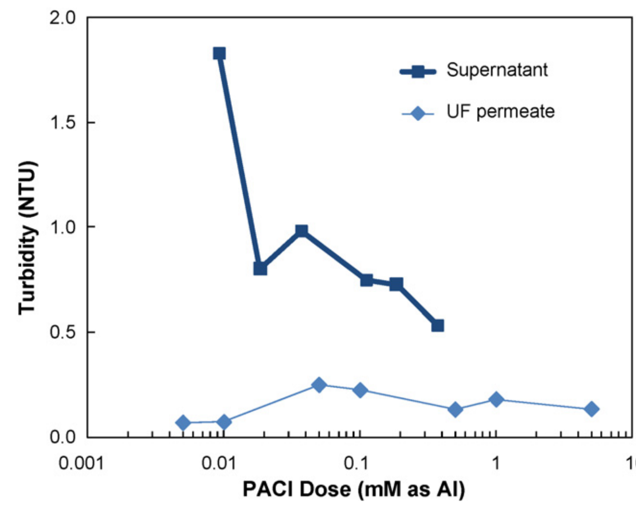

Fig. 7. The variations of the turbidity of supernatant and UF permeate during combined coagulation/UF treatment of textile wastewater. The supernatant was obtained after 90-min settling with the addition of PACl.

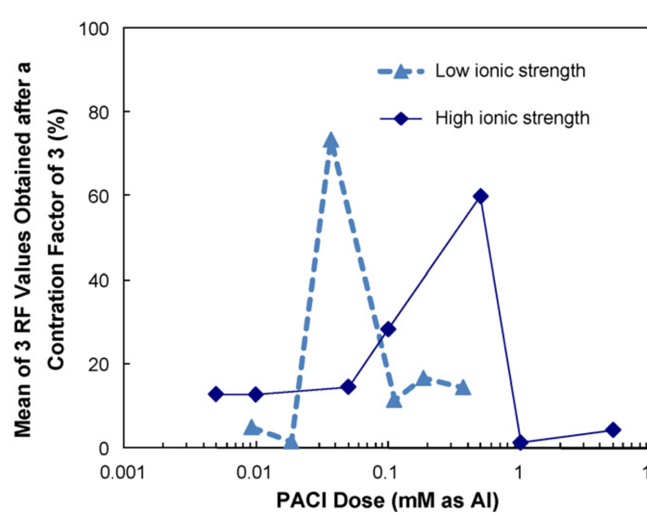

Fig. 8. The effects of PACl dosage on flux during UF of textile wastewater with different ionic strengths.

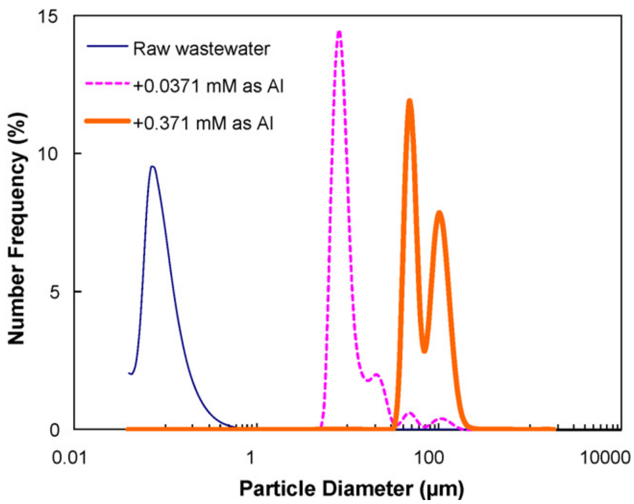

Fig. 9. The size distributions of particles in wastewater with and without the addition of PACl.

well as turbidity removal. Further studies on the sensitivity and control of the optimal dose are needed.

It appeared that the size of coagulated particles kept increasing with higher PACl dosages (Fig. 9). As discussed above, however, some small particles that were not detectable with the particle size analyses still existed in coagulated wastewater and thereby can cause membrane fouling during UF (refer to Figs. 2 and 3). Thus, the coagulation conditions in the combined coagulation/UF system should not be determined solely based on particle size, as this has been often used as an indictor in determining optimal coagulation conditions.

3.4. An analysis of the cake layer formation during the UF of coagulated wastewater

The UF membrane surface was visualized using a SEM in order to examine the changes of cake layer structures with coagulation (Fig. 10). The open pores of the virgin membrane were observed with SEM (Fig. 10a). When 0.00927 mM of PACl as Al was added, a large portion of the membrane surface was covered with the cake layer. The degree of membrane surface coverage became severer, with higher PACl dosages, as shown in Fig. 10b–d. In particular, a thick cake layer that grew on top of the membrane surface was observed with a large PACl dose of 0.371 mM as Al (Fig. 10d). A portion of the top layer of the cake even had shown open pores. This was possible because the size of the coagulated particles became bigger with the addition of more PACl (see Fig. 9), but it did not mean that the cake layer resistance would decrease. As discussed above, the cake layer resistance increased with larger PACl doses, due to the formation of thick cake layers. Thus, it was thought that the cake layer must have had dual functions: it served to protect the membrane from pore plugging (internal fouling), but it

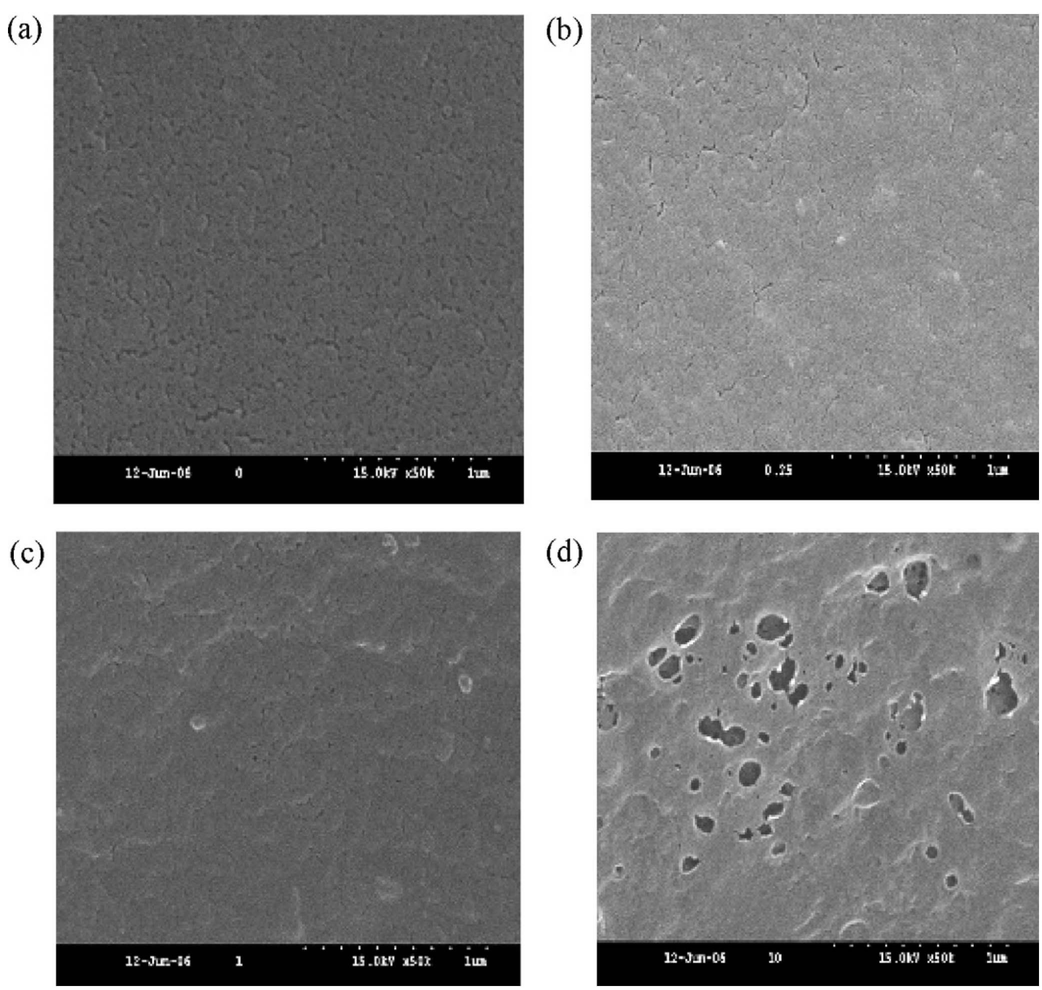

Fig. 10. SEM pictures of virgin and used membrane surfaces during the UF of wastewater with different PACl dosages: (a) virgin membrane; (b) 0.00927 mM as Al; (c) 0.0371 mM as Al; and (d) 0.371 mM as Al.

also offered additional resistance to permeation. Consequently, this phenomenon should affect the optimization of coagulant doses in the combined coagulation/UF system.

4. Conclusions

The addition of ferric and polyaluminum coagulants was investigated with regard to controlling membrane fouling during the UF of textile wastewater generated from the water jet loom process. The effects of membrane pore sizes and coagulant types and dosages on membrane permeability were evaluated using a stirred-cell membrane filtration unit. The examination of hydraulic filtration resistances, particle size, and cake layer structures was conducted based on the resistance-in-series model, particle size analysis, and SEM. The following conclusions can be drawn.

The dosing of coagulants to textile wastewater exhibited the best control of fouling for the UF membrane, with a MWCO of 10 kDa, among the MF and UF membranes tested. The types and dosages of coagulants applied affected the flux decline drastically during the UF treatment of textile wastewater. Flux decline was mitigated when the dosage of ferric chloride coagulant increased up to 0.5 mM as Fe, whereas PACl exacerbated fouling at a larger dosage (e.g., >0.1 mM as Al for wastewater with a low ionic strength). The optimal PACl dosage needed to minimize the flux decline during UF was sharp and dependent on the characteristics of influent wastewater. The reduction in UF permeability, with the larger PACl dose, was mainly attributed to the increase of the cake layer resistance. The growth of cake layers was observed with SEM pictures. The cake layer, however, must have exhibited dual functions while acting as a protective barrier for membrane fouling in that it provided additional resistance to permeation.

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