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Combined coagulation/ceramic membrane ultrafiltration system for reclamation of degreasing washing water

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ABSTRACT

Currently, there is growing emphasis on wastewater reclamation and reuse around the world. In this study, a combined coagulation/ceramic membrane ultrafiltration system was used to treat degreasing washing water under high pH/alkalinity conditions containing surfactants, organic matter and other materials resulting from rinsing processes in the plating industry. Coagulant exists as sol or gel species that was not suitable for complexation with smaller surfactants and organic matter under high pH/alkalinity conditions. A pH neutralization process was introduced to improve the removal efficiency of the coagulant prior to coagulation. An optimal coagulant (ferric chloride), optimal coagulation pH 5, and optimal coagulant dosage (700 mg Fe/L) were proposed as a result of coagulation tests. Under slightly acidic conditions, species distribution of ferric chloride consisted of monomers and medium polymers that were favorable for the formation of surfactant–coagulant or organic matter–coagulant complexes. In coagulation/ultrafiltration tests, filtration with in-line coagulation enhanced flux and also reduced resistance caused by internal pore blocking. This phenomenon is due to the rapid formation of a cake layer on the ceramic membrane and this cake layer lessens the adsorption of small foulants in feed water on the membrane. Moreover, filtration with in-line coagulation provided water quality similar to that of filtration of the supernatant. It is concluded that filtration with ferric chloride and in-line coagulation are valuable processes for treating degreasing washing water with reduced capital costs by excluding the need for flocculation and sedimentation zones in a water treatment plant.

Keywords: Surfactant; Coagulation; Coagulant; Ceramic membrane; Fouling

1. Introduction

The plating process is generally divided into pretreatment with a degreasing agent, plating using various heavy metals, and post treatment, and there are rinsing processes after each step. While effluent containing high concentrations of contaminants is generated in each main process, the effluent of the rinsing process has relatively low contaminants, but the quantities are huge. That is, metal plating

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industries generate large volumes of wastewater from spent rinses. Indeed, data published by the Ministry of Environment (Republic of Korea) in 2013 showed that about 58,000 m^3/d of wastewater was produced. Also, water consumption is about 400 L/m^2 in metal surface treatment processes, while the best reclamation that can be achieved is less than 10 L/m^2, according to surveys of South African metal finishing companies [1]. The contaminants may include surfactants, organic compounds, heavy metals, such as chromium, copper, zinc, lead, nickel and iron, and other cations and anions, depending on the cleaning or plating process. So, various reutilization technologies such as coagulation, sedimentation, electrodialysis, and membrane filtration have been studied and many engineers have sought to decrease water consumption [2–4].

Large amounts of surfactants are used as synthetic washing agents to clean a target for plating. It is difficult to develop a single and effective treatment method for surfactants in plating wastewater due to the diversity of surfactants used. However, it is necessary to remove surfactants successfully for improving the efficiency of the final treatment processes, such as RO filtration and ion exchange processes at thestage of removalof heavy metals. There are many techniques, such as biodegradation, coagulation, foaming, oxidation, adsorption, and membrane processes, to remove surfactants [5–8]. Among the diverse methods, membrane technology is one of the leading techniques in the reclamation of wastewater from rinsing waters containing surfactants and detergents. Although high-pressure membrane processes, such as RO and NF, are attractive separation techniques due to their excellent removal capacity, there are restrictions that arise because of the low permeate flux and high capital and exploitation costs. On the other hand, low-pressure membrane separation processes (MF and UF) are not always sufficiently effective to achieve the target water quality. Especially, the micellar-enhanced ultrafiltration (MEUF) method has been investigated extensively for the removal of heavy metals and organic pollutants, but the size of surfactant monomers is too small, so they pass through the membrane [9–12]. In case of rinsing wastewater, it is also problematic to remove surfactants by MEUF because of the relatively low surfactant concentrations.

Thus, we attempted to remove organic matter, including surfactants, at low concentrations from plating wastewater through ceramic ultrafiltration membranes after coagulation and flocculation. Although the ceramic membrane is more expensive than organic membrane, the ceramic membrane is resistant to organic solvents, acid–alkali liquids, high temperatures, and pressures [13–15]. While there are some studies about surfactant removal by ceramic membrane [16–18], all of them were also operated by the MEUF process. Consequently, we tried to eliminate surfactants through the ceramic membrane after the size of surfactants was sufficiently increased via coagulation. Thus, the objective of this study is to find conditions for the coagulation precipitation process for the treatment of plating wastewaters with surfactant content, especially in terms of organic matter and surfactant removals. Optimal coagulation conditions were determined by organic matter removability, membrane flux, and resistance.

2. Materials and methods

2.1. Characteristics of raw wastewater and analytical tools

Raw wastewater was collected from a local electrostatic plating plant, located in Incheon, Korea, and delivered to our laboratory and stored at 4°C before use. The raw wastewater was equilibrated at room temperature and filtered using a 1-μm cartridge filter before each experiment with the feed water in the coagulation test. The characteristics of the feed water are shown in Table 1. The feed water had a high pH and alkalinity, which resulted in high consumption of coagulant and pH adjustment solution. pH and alkalinity were measured with a pH meter (Professional Plus, YSI) calibrated daily using pH buffer solutions. Chemical oxygen demand (COD) and total organic carbon (TOC) analyses were performed as an indirect measurement of surfactants in the feed water. COD was measured with a water testing kit (HACH, USA) and spectrophotometer (DR 6000, HACH). TOC was analyzed using a TOC analyzer (TOC-V CPH, Shimadzu).

2.2. Coagulation test

In this study, jar tests were conducted to identify an optimal coagulation condition using coagulants, such as ferric chloride and PACl, and when pH adjustment was needed, predetermined sulfuric acid or sodium hydroxide solutions were added for 30 s with rapid mixing. To determine optimal coagulation conditions, evaluations of treatment efficiencies according to coagulant types, optimum pH, and

Table 1
Characteristics of the raw wastewater

pH	11.97
COD (mg/L)	422
TOC (mg/L)	245
Alkalinity (mg/L as $CaCO_3$)	3,183

coagulant dose were required. The flow chart for finding an optimal coagulation condition is shown in Fig. 1. pH of feed water was adjusted to 7 to check whether a pH neutralization process prior to coagulation was effective. An optimal coagulation pH was determined at different final pHs with a fixed dose of coagulant; then an optimal coagulant dose was determined at different doses of coagulant with a constant final pH. Mixing and precipitation schemes for the jar test were as follows. Feed water was placed in a beaker and mixed for 2 min rapidly at 250 rpm, followed by slow mixing at 30 rpm for 13 min using an agitator that allowed the feed water to be mixed appropriately. Coagulated matters in the feed water were settled using a centrifugal separator for 10 min at 4,000 rpm. At the end of the test, treated feed water was taken and analyzed for water quality measurements.

2.3. Combined coagulation/ceramic membrane ultrafiltration test

A schematic of the combined coagulation/ceramic membrane ultrafiltration test is shown in Fig. 2. The different types of solutions were placed in pressure vessel 1 for filtration and distilled water was placed in pressure vessel 2 for backwashing in each experiment. Prior to filtration, the new ceramic membrane was cleaned through backwashing at 2 bar for 3 min, which was twice as high as the operating pressure to eliminate residual contaminants on the ceramic membrane. Nitrogen gas, supplied from a gas cylinder, was controlled by a digital pressure regulator to keep the pressure constant. The permeate was collected in a beaker (500 mL) under which a digital balance was placed to calculate the flux. TAMI ceramic membrane (INSIDE DisRAM, TAMI Industries, France) was used for this laboratory-scale experiment. The ceramic membrane is a flat-sheet membrane and the operating condition and specification of the membrane are summarized in Table 2.

In the case of filtration with in-line coagulation, the feed water was placed in a beaker (1 L). Rapid mixing was provided with a jar-tester (C-JT, Chang Shin Scientific Corporation, Korea) at 250 rpm during coagulant addition for 2 min. A predetermined pH adjustment solution was added 30 s after rapid mixing where no slow mixing was applied. In another way, the mixing scheme of supernatant filtration was conducted with rapid mixing at 250 rpm and slow mixing at 30 rpm, followed by sedimentation with a centrifugal separator for 10 min at 4,000 rpm.

2.4. Filtration resistance fraction

The resistance-in-series model was used to identify the fouling characteristics. The model is shown in Eq. (1). R_m can be calculated using the initial water flux of a new membrane and resistance at the end of the filtration of the membrane is R_t. After membrane filtration for 30 min, the cake layer on the membrane surface was treated and backwashed. The pure water flux of the treated membrane was used to calculate R_f. R_c can be calculated by subtracting $R_f + R_m$ from R_t.

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

where J—the flux of a membrane (L/m^2/h), ΔP—transmembrane pressure (Pa), η—dynamic viscosity of the feed (Pa·s), R_t—total resistance (1/m), R_m—membrane-inherent resistance (1/m), R_c—cake resistance (1/m), R_f—resistance by internal pore blocking (1/m).

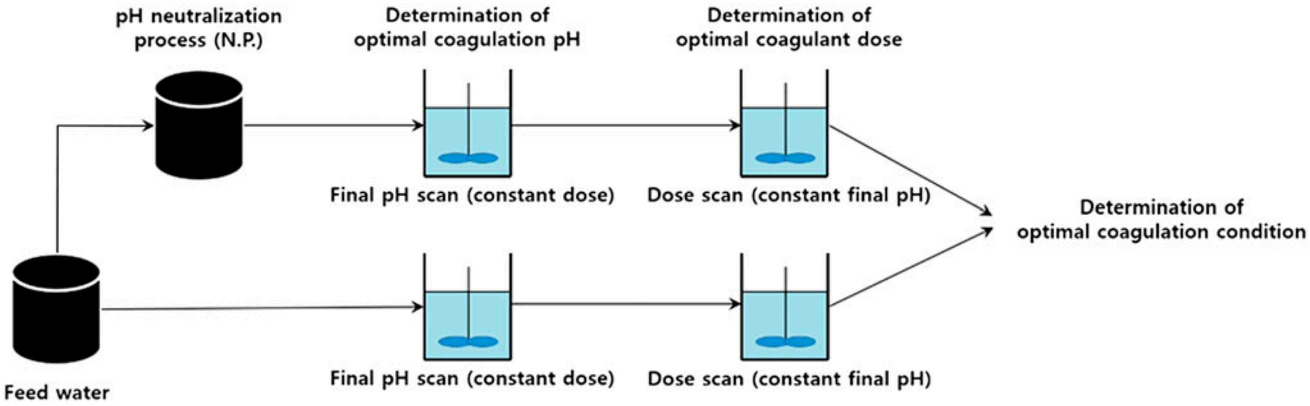

Fig. 1. Experimental stages to find an optimal coagulation condition.

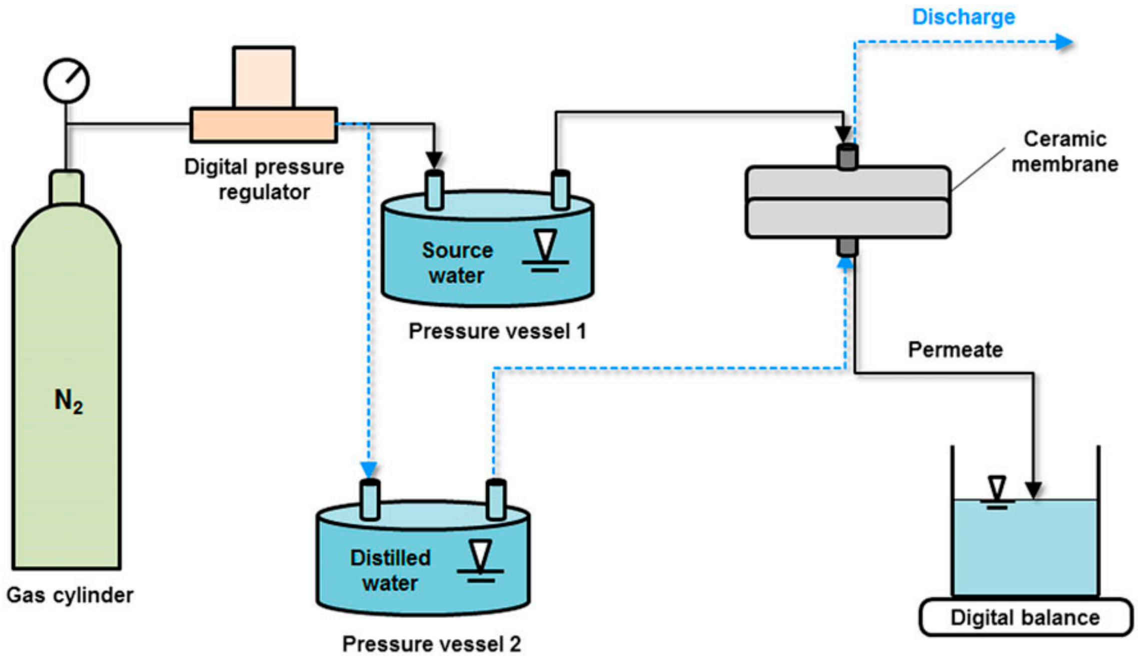

Fig. 2. Schematic diagram of the experimental setup.

Table 2
Operating conditions and specification of the membrane used for laboratory-scale experiments

Ceramic membrane	
Support	TiO_2
Membrane	ZrO_2—TiO_2
Molecular weight cut off, kDa	150
Thickness, mm	2.2
Effective area, m^2	0.00131
Operating pressure, bar	1
Operation mode	Dead-end filtration
Disk holders	
Material	Stainless steel 316 L
Operating temperature (℃)	<130

3. Results and discussion

3.1. Effects of neutralization on coagulation sedimentation

In this test, prior to coagulation, the feed water pH was adjusted to 7 to identify the effect of a pH neutralization process on coagulation efficacy. Figs. 3 and 4 show the effects of neutralization on COD and TOC removal efficiencies in the jar test using ferric chloride and PACl as coagulants.

High removal efficiencies of both COD and TOC were obtained at pH 5 using ferric chloride and PACl as shown in Figs. 3(a) and 4(a). These results may be explained by comparing the removal of natural organic matter (NOM) having a more soluble fraction. Charge neutralization and chemical interaction are the dominant mechanisms, not entrapment (sweep coagulation), and these mechanisms occur under slightly acidic conditions (pH 5) in the coagulation system for the removal from low-turbidity water [19]. Another researcher also said that the conventional coagulation process for the treatment of high-alkalinity water was not suitable and maximum NOM removal could be obtained under slightly acidic conditions (pH < 6) [20]. It should be considered that surfactants are organic compounds with low molecular weights containing hydrophobic groups and hydrophilic groups (anionic in this case), meaning there are possibilities that a mechanism for the removal of surfactants follows one of soluble NOM. This idea can be found in a previous report [8]. It is said that the correlation coefficient (R^2) between surfactants and COD removal efficiencies was 0.996 and it means that mechanism for the removal of surfactants is analogous to one for the removal of organic matter. From this point of view, pH control is one of the major factors and should be controlled for the efficiency of coagulation for the removal of surfactants as well as NOM.

The removal efficiencies of COD and TOC were 87 and 89% in each using ferric chloride at a dose of 800 mg Fe/L as shown in Fig. 3(b). However, in a jar test without a pH neutralization process, on the other hand, the removal efficiencies of COD and TOC were

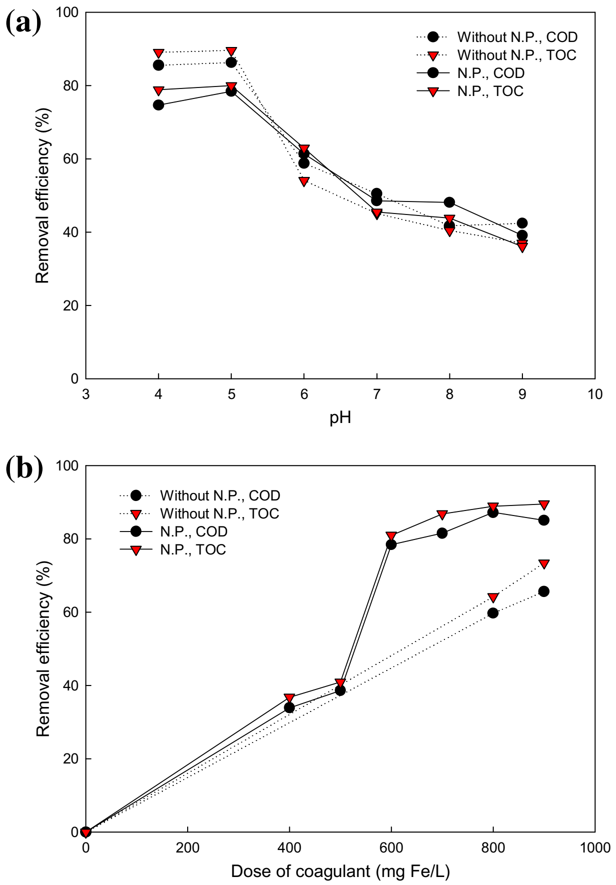

Fig. 3. Removal efficiencies of COD and TOC (a) at different final pHs with a fixed ferric chloride dose of 565 mg Fe/L with a pH neutralization process and 1,065 mg Fe/L without a pH neutralization process and (b) at different doses of ferric chloride with a final pH 5 (N.P. = pH neutralization process).

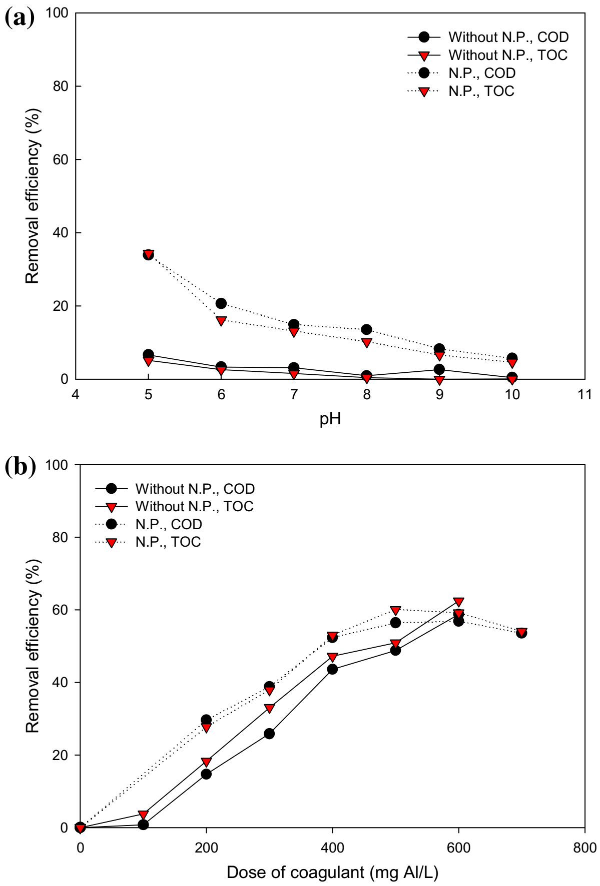

Fig. 4. Removal efficiencies of COD and TOC (a) at different final pHs with a PACl dose of 250 mg Fe/L with a pH neutralization process and 100 mg Fe/L without a pH neutralization process and (b) at different doses of PACl with a final pH 5.

60 and 64% at a dose of 800 mg Fe/L. Likewise, using PACl, higher removal efficiencies of both COD and TOC were obtained with a pH neutralization process. However, no further effect of neutralization occurred at a dose of 600 mg Al/L (Fig. 4(b)).

3.2. *Effect of type of chemicals (coagulants) on removal efficiency*

Fig. 5 shows the difference in removal efficiencies of COD and TOC using ferric chloride and PACl with a pH neutralization process. As shown in Fig. 5, in terms of removal efficiencies of COD and TOC, PACl was a more efficient coagulant than ferric chloride at a low dosage of coagulant (below 500 mg Fe or Al/L). However, at a dose of 600 mg Fe or Al/L and above, the removal efficiency with ferric chloride exceeded that with PACl for COD and TOC. A previous study indicated that PACl (prehydrolysis of $AlCl_3$) with high neutralization ability could enhance the removal efficiency of DOC at a low dosage of coagulant in high pH/alkalinity water, but improvement of removal efficiency of DOC using $AlCl_3$ occurred at higher dosages [19]. This was because a significant drop in water pH occurred when using $AlCl_3$, while the water pH changed only slightly when using PACl with a high OH/Al ratio [19]. Thus, an inorganic coagulant that exists as a monomer and medium polymer species under acidic conditions can maximize complexing reactions with surfactants. On the other hand, PACl, an inorganic polymer coagulant, has a high OH/Al ratio having high stability when the pH of the water changes, so the amount of speciation transformation is lower than with the inorganic coagulant [21]. A dosage of ferric chloride of 700 mg Fe/L was required to obtain over 81% COD and 86% TOC removal efficiencies and further increases in dosage of

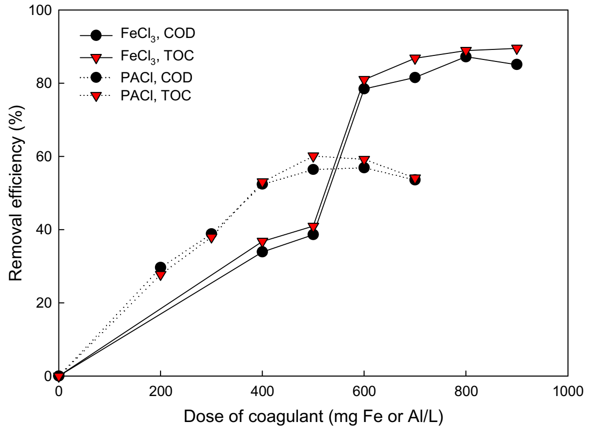

Fig. 5. Removal efficiencies of COD and TOC at different doses of coagulants, ferric chloride and PACl, with a pH neutralization process.

ferric chloride did not make significant differences to removal efficiencies of COD or TOC.

3.3. *Effects of in-line coagulation on flux decline and membrane fouling*

3.3.1. *Effects of in-line coagulation on flux decline*

The optimal coagulation conditions (type of coagulants: ferric chloride, optimal pH: 5, and coagulant dose: 700 mg Fe/L) obtained from the jar tests were applied to precoagulate the feed water and different fractions of feed water were prepared to compare the effects between filtration with in-line coagulation and filtration of the supernatant on flux decline. It has been reported that good coagulation conditions for coagulation/settling treatment should lead to good performance in water quality and fouling control for an in-line coagulation/ultrafiltration process with dead-end filtration [22].

As shown in Fig. 6, in-line coagulation can enhance normalized flux compared with filtration of the supernatant. The normalized flux with in-line coagulation after 30 min operation was 0.46. On the other hand, in the case of the filtration of supernatant, the normalized flux was 0.26, around half of that with in-line coagulation. These results mean that small foulants remained in feed water and unsettled coagulated matters in the supernatant contributed to the sharp drop in flux.

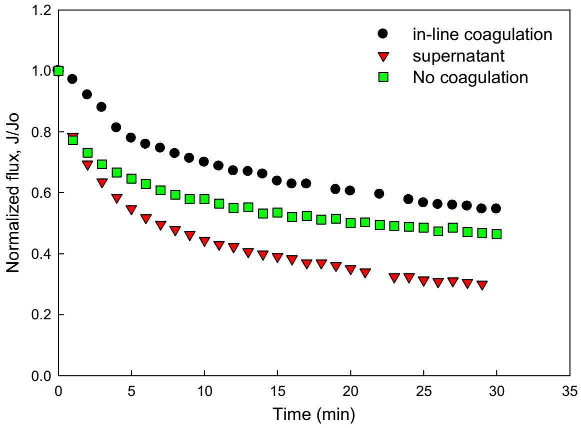

Fig. 6. Variation of normalized flux of filtration with in-line coagulation, filtration of supernatant, and filtration without coagulation.

Table 3
Removal efficiency of combined coagulation/ceramic membrane ultrafiltration system with in-line coagulation, filtration of supernatant, filtration without coagulation, and supernatant

Contents	COD (%)	TOC (%)
Filtration with in-line coagulation	82.0	84.7
Filtration of supernatant	83.5	85.8
Filtration without coagulation	6.9	0.8
Supernatant	81.3	83.5

COD and TOC removal efficiencies in each experiment are shown in Table 3. COD and TOC removal efficiencies of filtration with no coagulation solution (pH 5) were only 6.9 and 0.8%, respectively. In contrast, 82.0 and 84.7% removal efficiencies of COD and TOC were obtained in filtration with in-line coagulation, and 83.5 and 85.8% removal efficiencies of COD and TOC were obtained with filtration of the supernatant. These differences in removal efficiencies of COD and TOC between filtration with in-line coagulation and filtration of the supernatant were less than 2%, meaning that capital costs could be reduced by eliminating the flocculation and sedimentation zones and also filtration with in-line coagulation could secure water quality similar to filtration of the supernatant. Additionally, the reason why removal efficiency of combined coagulation/membrane filtration was lower than one of coagulation sedimentation processes was attributed to organic compound concentration differences in the raw wastewater.

3.3.2. *Membrane fouling*

The hydraulic filtration resistances using a resistance-in-series model were calculated with Eq. (1) and the values are given in Table 4. The highest extrinsic, cake layer, and internal pore blocking resistances were developed with supernatant filtration. In the filtration of supernatant, internal pore blocking resistance was

Table 4
Effects of filtration type on hydraulic filtration resistance

Resistance	Filtration with in-line coagulation	Filtration of supernatant
R_t	3.56×10^{12}	7.33×10^{12}
R_c	1.41×10^{12}	3.66×10^{12}
R_f	5.04×10^{11}	1.75×10^{12}

dominant, compared with the filtration with in-line coagulation. For the in-line coagulation system, the ratio of R_f to R_c was 0.36, while the ratio of R_f to R_c was 0.48 for supernatant filtration. This was likely due to the rapid formation of a cake layer on the membrane when filtering the feed water containing flocs and the cake layer on the ceramic membrane retained the noncoagulated matters and surfactants in feed water by reducing adsorption onto it. That is, noncoagulated organic matter and surfactants in the feed water caused internal pore blocking resistance, but when a cake layer was formed, internal pore blocking resistance was reduced. Compared with in-line coagulation for the removal of NOM, several studies have reported that precoagulation in microfiltration/ultrafiltration improved membrane performance and alleviated membrane fouling caused by reduced pore blockage [23–25]. It may be that the floc characteristics of NOM are similar to those of surfactants. It should be considered that the coagulation aggregate produced under complexation and charge neutralization mechanisms at pH 5 is looser than the one produced under sweep coagulation mechanisms at pH 7 and these loose flocs make the cake layer's porosity higher [26].

4. Conclusions

In this paper, a study was conducted on a combined coagulation/ceramic membrane ultrafiltration system for the reclamation of degreasing washing water having high pH/alkalinity. The optimal pHs for coagulation are in the acidic region (pH 5) for both ferric chloride and PACl as coagulants. When coagulation tests with a pH neutralization process were conducted, around 25% improvements of removal efficiencies of both COD and TOC were obtained using the same dosage of ferric chloride and PACl. This means that a pH neutralization process prior to coagulation could save the expenses for coagulant and also for handling of sludge because the demand for coagulant was decreased. Traditional coagulant, ferric chloride, was more efficient than prehydrolyzed coagulant, PACl, at high doses. This is because monomer and medium polymer species were suitable for treating surfactants having low molecular weights, while sol and gel species were not.

A combined coagulation/ceramic membrane ultrafiltration test was conducted for a range of conditions including filtration with in-line coagulation, filtration of supernatant and filtration with no coagulation solution. The highest flux occurred for filtration with in-line coagulation rather than filtration of supernatant, while there were not great gaps in the removal efficiencies of COD and TOC. The ratio of internal pore blocking resistance (R_f) to cake layer resistance (R_c) was decreased with in-line coagulation by making cake layer on the membrane in a short period of time.

In conclusion, a combined coagulation/ceramic membrane ultrafiltration can be considered as a suitable system for reclamation of degreasing washing water under high pH/alkalinity conditions, applying controlling membrane fouling and removing organic matter and surfactants in degreasing washing water.

Acknowledgments

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