

Application of Combined Coagulation-Ultrafiltration Membrane Process for Water Treatment

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Abstract—The objectives of this research are to identify the membrane fouling potential due to different fractions of NOM and correlate the physicochemical properties of NOM and membranes with the adsorption of humic substances on membrane and investigate the mechanism of coagulation affecting UF, and find the optimum conditions of the combined of coagulation with UF membrane filtration for NOM removal. For Nakdong river water, the humic acid fraction was the most reactive precursor fraction for the formation of the ratio of THMFP/DOC (STHMFP) and TOXFP/DOC (STOXFP). The result of adsorption kinetics tests showed that hydrophobic organics adsorbed much more quickly than hydrophilic organics on both membranes. Thus, hydrophobic compounds exhibited a preferential adsorption onto membrane. In case of the effect of membrane properties on the adsorption of organic fractions, the adsorption ratio (C_i/C_e) was greater for the hydrophobic membrane than for the hydrophilic membrane regardless of the kind of organic fractions. For combined coagulation with membrane process, flux reduction rate showed lower than the UF process alone. Also, the rate of flux decline for the hydrophobic membrane was considerably greater than for the hydrophilic membrane. Applying the coagulation process before membrane filtration showed not only reduced membrane fouling, but also improved removal of dissolved organic materials that might otherwise not be removed by the membrane. That is, during the mixing period, substantial changes in particle size distribution occurred under rapid and slow mixing conditions due to the simultaneous formation of microflocs and NOM precipitates. Therefore, combined pretreatment using coagulation (both rapid mixing and slow mixing) improved not only dissolved organic removal efficiency but also DBP (Disinfection By-Product) precursor's removal efficiency.

Key words: NOM, Coagulation, Ultrafiltration, DBPFP, Flux, Adsorption

INTRODUCTION

Aquatic natural organic matter (NOM) which occurs ubiquitously in surface waters consists of both humic (i.e., humic and fulvic acids) and nonhumic components. NOM alone does not pose a threat to human health. But, NOM in general as well as certain constituents are problematic in water treatment. For instance, some of the NOM form carcinogenic byproducts during disinfection process. The Disinfection By-Products (DBPs) are formed during the reaction between a chemical disinfectant (e.g., chlorine) and NOM in water.

As a result, the removal of NOM in general and DBPs precursors in particular has become significantly important and has been a primary target in the water treatment industry. Therefore, characterization of NOM can provide insight into water treatment process selection and applicability.

Recently, membrane technologies have been extensively investigated as a water treatment process for removal of NOM and DBPs precursors. In addition to removing virtually all particles larger than the nominal pore size, some membrane processes (e.g., Nanofiltration and Reverse osmosis) reject significant amounts of soluble species and are therefore prospective technologies for removing NOM. However, these membranes are also more easily fouled by NOM than other membranes, such as Ultrafiltration (UF) and Microfiltration (MF) which are low-pressure membranes primarily used for the removal of particulates and pathogens. Typically, the capa-

bility of UF in removing NOM depends on the membrane materials as well as the molecular weight cut-off (MWCO) of the membrane used, the interaction of NOM with membrane surface, and operating conditions. Unfortunately, the UF system, due to its high MWCO, may not be effective for removal of DBP precursors [Laine et al., 1989, 1990; Jacangelo et al., 1995], although it is efficient in reducing turbidity and particles. For example, Laine et al. [1990] evaluated the capability of UF (MWCO 100 kD) for removal of DBP precursors by using some different surface waters. Their results indicated that UF was ineffective for controlling the formation of DBPs, whereas NF with an MWCO of 400-800 Dalton effectively controlled DBP formation [Fu et al., 1994; Agbekodo et al., 1996]. Also, the concentration and type of NOM present in a raw water influence the interaction of the NOM with a particular membrane. Furthermore, Membrane fouling by NOM decreases the effective capacity of the process and causes irreversible losses in membrane permeate flux and requires more frequent replacement of the membrane. Yuan and Zydney [1999] found that humic substances, despite their small size, can cause a significant flux decline of MF membranes. Therefore, the use of pretreatment of NOM in conjunction with UF system is expected to decrease the amount of irreversible fouling and increase the permeate flux. Applying the coagulation process before membrane filtration has been suggested as means of reducing membrane fouling and improving the removal of dissolved organic materials that might otherwise not be removed by the membrane. [Lahoussine-Turcaud et al., 1990; Laine et al., 1990; Jacangelo et al., 1995]. However, little has been studied for the mechanism and conditions of the beneficial pretreatment process.

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Table 1. Characteristics of raw water taken from Nakdong river

Item	Unit	Concentration
Temperature	°C	18-22
pH	-	7.4-7.8
Turbidity	NTU	1.3-1.5
UV ₂₅₄	cm ⁻¹	0.051-0.056
DOC	mg/L	3.6-4.0
SUVA (UV ₂₅₄ /DOC)	l/m/mg/L	1.21-1.4
Alkalinity	mg/L as CaCO ₃	45-50
Conductivity	μmho/cm	194

The objectives of this research are to (1) identify the membrane fouling potential due to different fractions of NOM, including hydrophobic and hydrophilic fractions, and apparent molecular weight (AMW) fractions, (2) correlate the physicochemical properties of NOM and membranes with the adsorption of humic substances on membrane, (3) investigate the mechanism of coagulation affecting UF, and (4) find out the optimum conditions of the combined coagulation with UF membrane filtration for NOM removal.

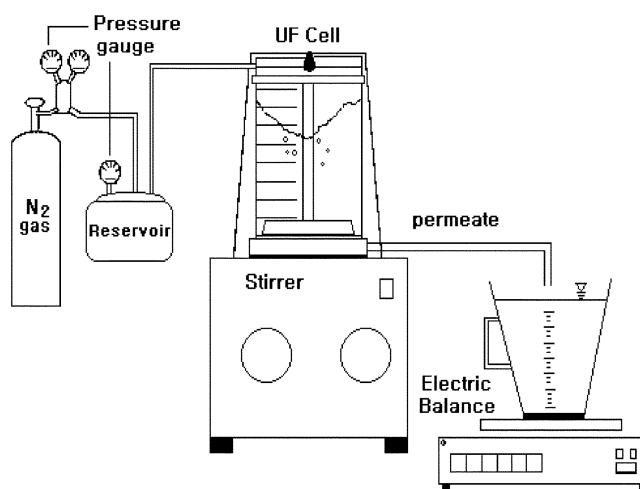
MATERIAL AND METHODS

1. Raw Water

The raw water used in this test was obtained from Nakdong river located near Busan city. The physical and chemical properties of the raw water are shown in Table 1. The raw water was prefiltered with a large-pore filter (1 μm) to remove large particles and algal clusters, and stored in a 4 °C room until use. Prefiltration, which decreased the raw water turbidity by about 80%, was done to see the genuine effect of dissolved organics on UF.

2. UF Membrane

Four different membranes, YM30, PM30, YM100, PM100 supplied by Millipore, were tested for comparison. YM membranes are made of regenerated cellulose and PM membranes are made of polysulfone. These membranes had a diameter of 76 mm with a geometric area of 0.00454 m². The characteristics of YM series membranes are more hydrophilic than PM- and XM-series membranes [Amicon, Inc., 1984]. The nominal MWCOs of the membranes were provided by manufacturers. Some of membrane characteristics have been recently reported [Cheryan, 1986; Lee and Hong, 1998]. To prepare the membrane for experiments, the membrane was first rinsed by floating it skin-side down in deionized water for 2 hr; the water was changed four times during this time period. This procedure was recommended by the manufacturer to remove trace quantities of chemicals. The washed membranes were placed in a stirred batch filtration cell illustrated in Fig. 1. The transmembrane pressure was

**Fig. 1. Schematics of membrane filtration assembly.**

regulated using nitrogen gas, and the permeate flow rate was determined by weighing permeate on an electronic top-loading balance.

3. Coagulation

Coagulation tests were conducted in an 18 L square batch reactor system which was equipped with a two-blade turbine impeller, electric motor, and speed controller. Aliquots (18 L) of each water were dosed with alum [Al₂(SO₄)₃·16H₂O] or PACl coagulant under ambient pH conditions. The solution was rapidly mixed at 250 rpm ($G=550 \text{ sec}^{-1}$) for 1 min after coagulant addition followed by slow mixing at 30 rpm ($G=22 \text{ sec}^{-1}$) for 30 min. After 30 min of settling, the supernatant was analyzed for TOC, DOC, UV₂₅₄ absorbance by following Standard Methods [AWWA, 1998]. Table 2 describes experimental conditions used in this experiment.

4. NOM Fractionation

An aliquot of NOM was fractionated into humic and nonhumic fractions by employing the technique based on the adsorption of humic substances onto XAD-8 resin under acidic conditions (pH 2). The organics adsorbed on resin was subsequently eluted with NaOH (pH 12) [Collins et al., 1986; Thurman and Malcolm, 1981].

5. AMW

AMW distribution of the NOM samples was determined with UF fractionation method suggested by the Logan-Jiang [1990]. NOM samples were fractionated by using a series of UF membranes with MWCOs of 0.5, 3, 10, and 30 kDa which are of identical material. The membrane material is classified as hydrophilic.

6. Disinfection By-Product Formation Potential (DBPFP)

DBPFP test was carried out in accordance with Uniform formation condition (UFC) [Summers et al., 1996]. The chlorination conditions selected for the UFC test are as follows: incubation time=

Table 2. Experimental conditions for four different types of processes

Process	Conditions
UF alone	Only UF process
Rapid mixing+UF	Applying UF process after rapid mixing ($G=550 \text{ sec}^{-1}$, 1 min)
Slow mixing+UF	Applying UF process after rapid (1 min) and slow mixing ($G=22 \text{ sec}^{-1}$, 30 min)
Coagulation	Rapid mixing (1 min), slow mixing (30 min), and settling (30 min)

24 h, incubation temperature=20 °C, incubation pH=8, and free chlorine residual after 24 hr=10±0.4 mg/L.

RESULTS AND DISCUSSION

1. Characteristics of NOM Fraction of Raw Water

Fig. 2 shows the fractions of NOM in Nakdong river water. The raw water DOC contains 44% of hydrophilics, 35% of fulvic acid, and 21% of humic acid. Fig. 3 shows the AMW distribution of DOC in raw water. As shown Fig. 3, 88% of DOC has a molecular weight smaller than MWCO of 10 kDa, which suggests that without sig-

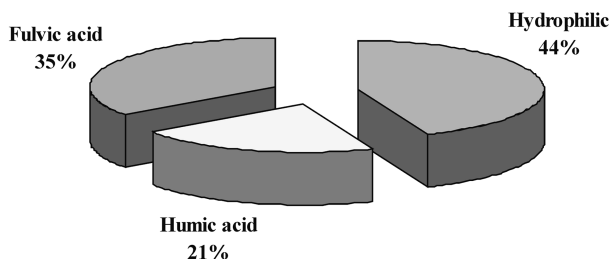


Fig. 2. Composition of NOM in Nakdong river water.

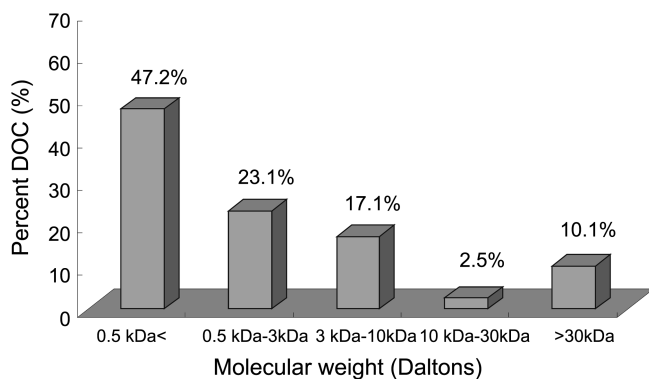


Fig. 3. Apparent molecular weight distribution for DOC of Nakdong river water.

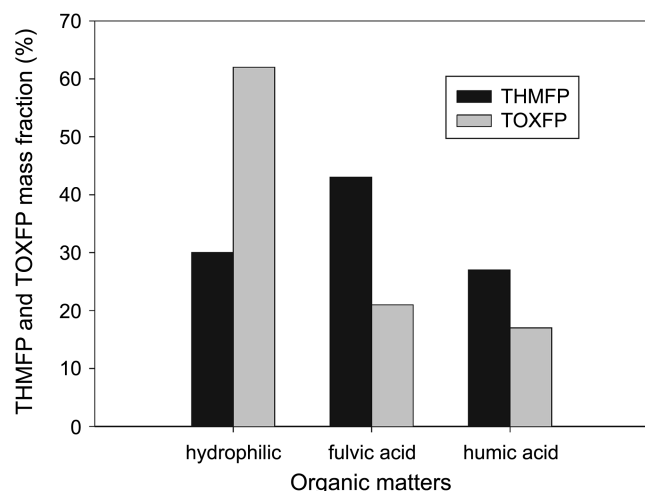


Fig. 4. THMFP and TOXFP of organic fractions in Nakdong river water.

nificant pretreatment, most organics in raw water should pass through UF membranes having a pore size of MWCO 100 kDa and 30 kDa.

2. Effect of NOM Fraction on DBPFP

Chlorinated reactivity of DOC fractions was evaluated by measuring DBPFP. In this study, trihalomethane (THM) and total organic halogen (TOX) as the DBPs were measured to find the relationship between the different fractions of NOM and DBPFPs. Fig. 4 shows THMFP and TOXFP per unit mass DOC of the different fractions of DOC. The THMFPs for hydrophobic fraction (fulvic+humic acid) and hydrophilic fraction were 69% and 31%, respectively, but the TOXFPs for those were 37% and 63%, respectively. However, as shown in Fig. 5, the humic acid fraction was the most reactive precursor fraction for the formation of the ratio of THMFP/DOC (STHMFP) and TOXFP/DOC (STOXFP). These results are in agreement with those reported by others for surface waters [Croue et al., 1996]. The experimental result shown in Figs. 3-5 suggests that more hydrophilic substances need to be removed to prevent more DBPs formation, when considering it is the major organic component in raw water.

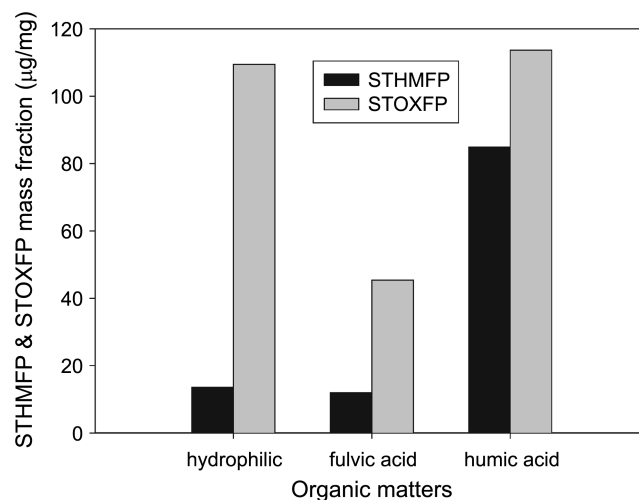


Fig. 5. STHMFP and STOXFP of organic fractions in Nakdong river water.

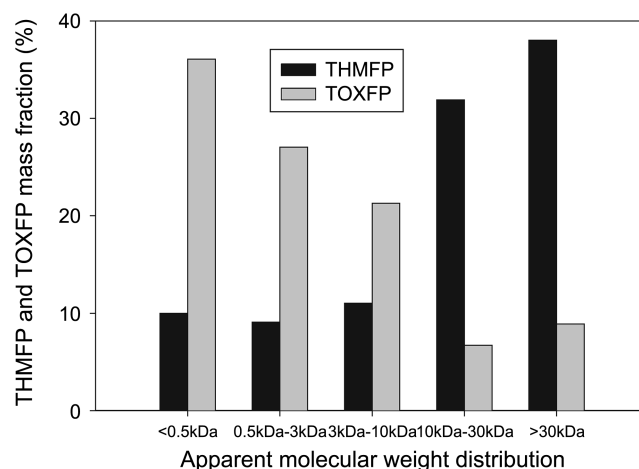


Fig. 6 THMFP and TOXFP of AMW distributions in Nakdong river water.

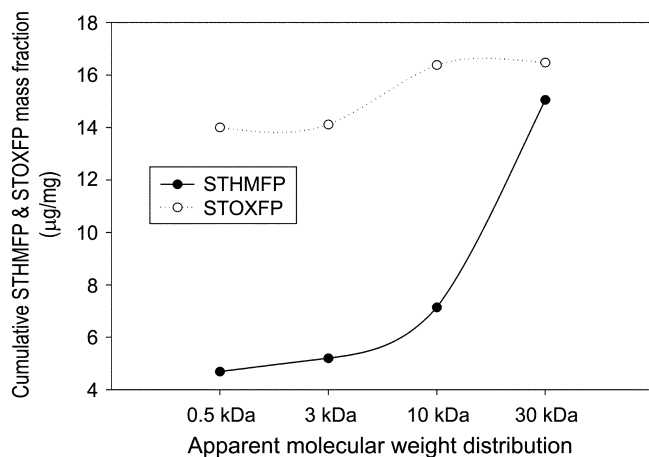


Fig. 7. Effect of AMW fraction on the cumulative STHMFP and STOXFP.

Fig. 6 and Fig. 7 show DBPFP mass and cumulative STHMFP and STOXFP with respect to the AMW fraction of NOM. As shown in Fig. 6 and Fig. 7, smaller AMW fractions (<3 kDa) exhibit the most reactive precursor for the TOXFP and STOXFP, whereas larger AMW fraction (>10 kDa) was the most reactive precursor for the THMFP and STHMFP. According to the previous study, the hydrophilic substance mainly consisted of lower (<10 kDa) AMW organics [Jung et al., 2002]. Therefore, it is necessary that the hydrophilic substance should be the most reactive precursor of the formation of TOX.

3. Adsorption Kinetics of Organic Substances on UF Membrane

A static adsorption kinetic was tested to find the physico-chemical interaction between the fraction of organic matter and the membrane materials. For adsorption kinetics experiments, adsorption isotherms of different organic matters (hydrophilic and hydrophobic) were carried out for 4 days contact time by using different membrane materials. Adsorption kinetics of organic fraction on different membrane materials (hydrophobic and hydrophilic) are shown in Figs. 8 and 9, where $C(t)$ is the amount of organic adsorbed at time, t and $C(e)$ is the amount which can adsorb on the membrane

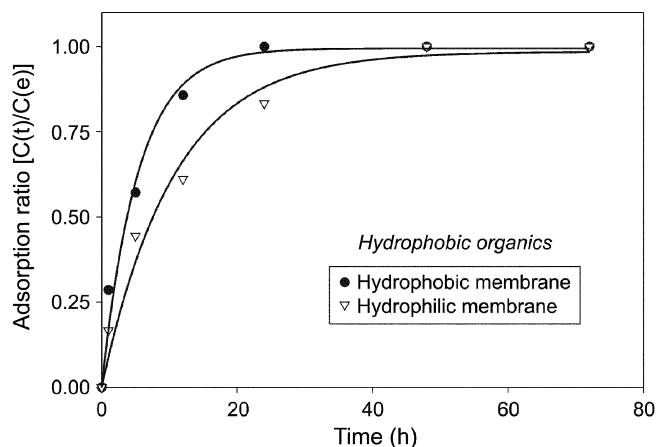


Fig. 8. Adsorption isotherms of hydrophobic organics for UF membranes.

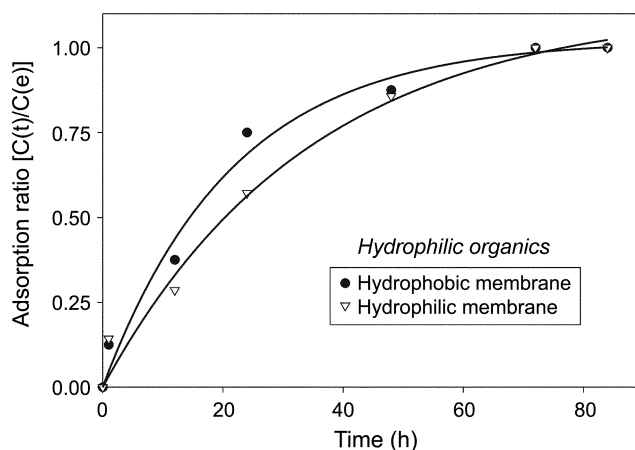


Fig. 9. Adsorption isotherms of hydrophilic organics for UF membranes.

surface at equilibrium with organic matters. As shown in Figs. 8 and 9, adsorption equilibrium was reached after 1 to 3 days depending on organic fractions. The results of adsorption kinetics tests showed that hydrophobic organics adsorbed much more quickly than hydrophilic organics on both membranes. Thus, hydrophobic compounds exhibited a preferential adsorption onto membrane as showing higher $C(t)/C(e)$ values. Based on the AMW fractions of NOM, the hydrophobic organics are composed of larger AMW fraction than the hydrophilic organics. And hence, the hydrophobic molecules might have more attachment sites, resulting in the enhanced binding to the membrane surface [Tipping and Ohnstad, 1984]. In case of the effect of membrane properties on the adsorption of organic fractions, the adsorption ratio $[C(t)/C(e)]$ was greater for the hydrophobic membrane than for the hydrophilic membrane regardless of the kind of organic fractions. Thus, the choice of a hydrophilic membrane can reduce flux decline by decreasing adsorption of organics. This is due to a smaller number of accessible surface area (external surface area+ pore wall surface area) on the hydrophilic membrane than hydrophobic membrane [Cuperus and Smolders, 1991]. Therefore, this result suggests that the hydrophilic membrane is good for

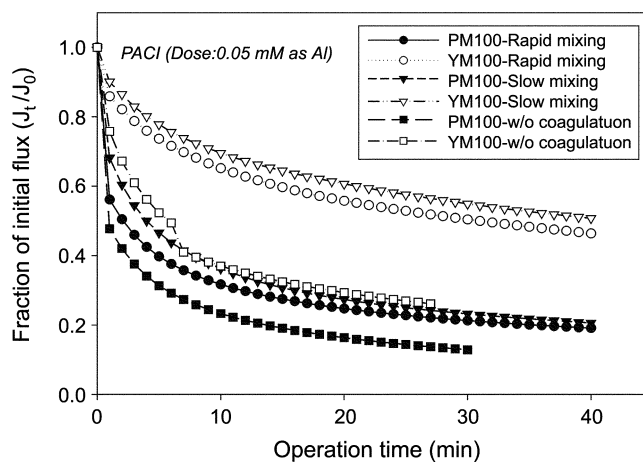


Fig. 10. Changes in flux for UF membrane as a function of coagulation pretreatment condition (YM: Hydrophilic, PM: Hydrophobic, MWCO: 100 kDa, Dose: 0.05 mM as Al).

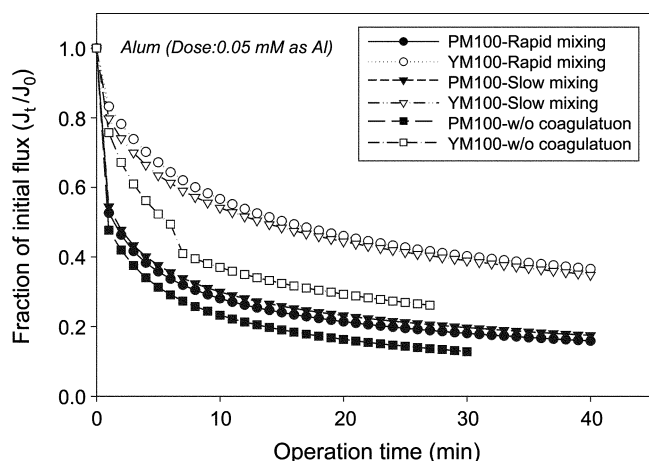


Fig. 11. Changes in flux for UF membrane as a function of coagulation pretreatment condition (YM: Hydrophilic, PM: Hydrophobic, MWCO: 100 kDa, Dose: 0.05 mM as Al).

less membrane fouling rate because hydrophilic membrane have a reduced adsorption capacity towards both hydrophobic and hydrophilic organics.

4. Effect of Pretreatments on Membrane Flux Change

Figs. 10 and 11 show the impact of pretreatment conditions on the fraction of initial flux which was estimated by the ratio of the permeate flux to the initial pure water flux. The UF membrane with MWCO 100 kDa was used, and two different coagulants (alum and PACl) were used for coagulation as a pretreatment. As different coagulation conditions as a pretreatment, the samples were fed into the UF after either rapid mixing alone or rapid mixing followed by slow mixing using alum or PACl as a coagulant. As shown Figs. 10 and 11, after 30 min of UF operation, the permeate flux significantly declined with UF alone process. However, either rapid mixing+UF or slow mixing+UF process caused much less flux decline. For PACl coagulant, the rate of flux decline was reduced for both hydrophilic and hydrophobic membrane than alum due to higher formation of flocs. In addition, the rate of flux decline for the hydrophobic membrane was significantly greater than for the hydrophilic membrane, regardless of pretreatment conditions. In general, Figs. 10 and 11 show that coagulation pretreatment significantly reduced the fouling of the hydrophilic membrane, but did little decrease the flux reduction of the hydrophobic membrane. This experimental result pointed out that particle deposition rather than organics adsorption on membrane surface is a major factor affecting flux decline for the hydrophilic membrane. But, for the hydrophobic membrane, flux decline was strongly affected by fouling due to the adsorptions of organics and microflocs onto the membrane pore. The fouling mechanism on the membrane surface and into its porous structure was analyzed in terms of several kinetic models [Hermia, 1982; McCabe, 1985]. The results showed that the fouling mechanism for the hydrophilic membrane occurred at membrane surface, for the hydrophobic membrane it occurred at membrane pore [Jung, 2002].

Also, less flux decline caused by coagulation is considered to be due to the transformation of dissolved organics into particulates which is easily removed by the size exclusion mechanism of UF. In all pretreatment cases, applying coagulation process before UF filtration

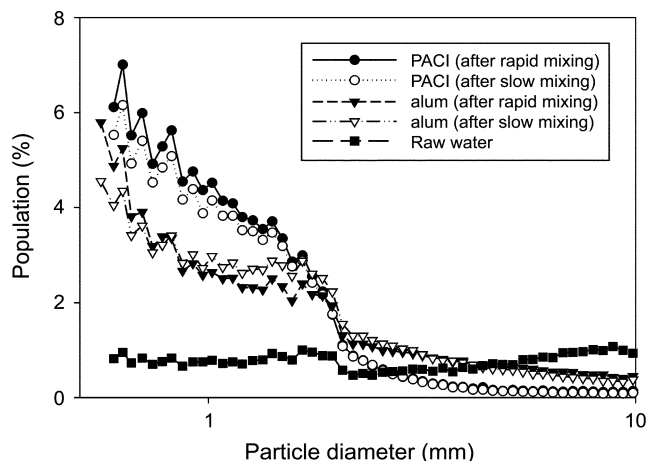


Fig. 12. Change in particle size distribution after different coagulation conditions.

showed not only increasing particle size, but also improving the flux. That is, during the coagulation, substantial changes in dissolved organics must be occurred by coagulation due to the simultaneous formation of microflocs and NOM precipitates. Wisner et al. [1989] also reported that cakes formed from humic acid destabilized with an aluminum coagulant have been found to present minimal specific resistance when humic acid is coagulated under conditions of precipitation/charge neutralization. Therefore, aggregation of small colloids and dissolved organic matter by coagulation may lead to a larger effective particle size, which may result in less specific resistance.

In order to find out the origin of different flux changes caused by the coagulation pretreatment as shown in Figs. 10 and 11, a series of tests were carried out to measure the particle size distribution after coagulation pretreatment. Fig. 12 describes the effects of mixing type and coagulant dosage on particle size distribution, showing substantial changes in particle size distribution occurred under different mixing and coagulant dosage condition. The significant change in particle size distribution after coagulation was caused by the phase change of dissolved organics into microflocs. Consequently, applying coagulation process before membrane filtration was found to be very effective in better DOC removal as well as critical flux increase due to the increase in particle size. Therefore, flux variation shown in Fig. 10 and Fig. 11 can be concluded to be caused by the different particle size distribution due to different coagulation conditions. Kim et al. [2001] also reported that significant changes in particle size distribution occurred after rapid mixing under different mixing and pH conditions.

5. Effect of Pretreatment on Organic Matter Removal

Fig. 13 shows TOC and UV_{254} removal efficiency when operated with UF alone and coagulation+UF under the experimental conditions of coagulant dose of 0.05 mM as Al (alum) with MWCO 100 kDa hydrophilic membrane. As shown in Fig. 13, UF only removed a very small amount of organics, 1% of TOC and 4% of UV_{254} , which indicates that most of the organics pass through the UF membrane of MWCO 100 kDa. Thus, effective organic matter removal should not be expected when using UF membrane only. When applying coagulation alone, TOC and UV_{254} removal efficiency was 9.8% and 46% respectively, which indicates that as low as 0.05 mM as Al dose was not enough to form easily settleable flocs that are sup-

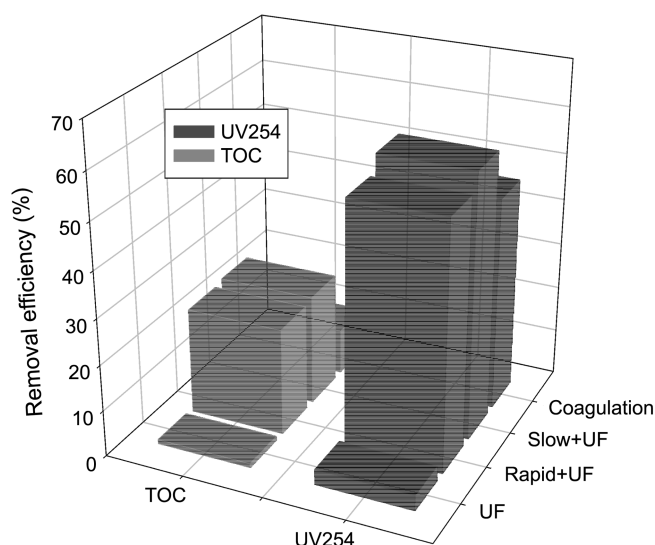


Fig. 13. Effect of the coagulation pretreatment condition on the removal of TOC and UV₂₅₄ (YM 100, coagulant: alum).

posed to be removed by sedimentation following coagulation. However, when applying coagulation (rapid)+UF and coagulation (slow)+UF, TOC removal efficiency was improved to 23.3% and 24.0% and UV₂₅₄ 53.8% and 57.5%, respectively. Increase in the organic removal efficiency should be mainly caused by the removal of microflocs formed at low alum dose. That is, during the mixing period, substantial amounts of dissolved organics were transformed into microflocs due to the simultaneous formation of microflocs and NOM precipitates. Kim et al. [2001] also reported that 40 to 50% of dissolved organic matter was converted into particulate material after rapid mixing process of coagulation. Thus, it can be concluded that another advantage of the combination of coagulation with UF is a lower coagulant dose with which particulates will be removed by the membrane and no settleable flocs are required.

Similarly, Fig. 14 also shows TOC and UV₂₅₄ removal efficien-

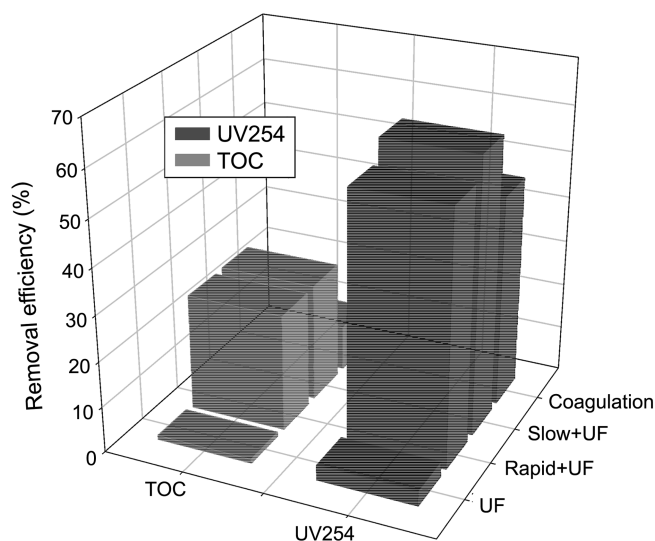


Fig. 14. Effect of the coagulation pretreatment condition on the removal of TOC and UV₂₅₄ (YM 100, coagulant: PACI).

Table 3. Removal efficiency of organic fraction after coagulation and coagulation+UF for each coagulant

Process	Coagulation only		Coagulation (slow mixing)+UF	
	Coagulant		Alum	PACI
NOM	Alum	PACI	Alum	PACI
Humic acid	22%	30%	30%	37%
Fulvic acid	14%	16%	25%	29%
Hydrophilics	7%	26%	14%	30%
Bulk	13%	23%	25%	30%

cies under different experimental conditions using 0.05 mM (as Al) of PACI coagulant dose with MWCO 100 kDa hydrophilic membrane. For coagulation (rapid)+UF and coagulation (slow)+UF, TOC removal efficiency was 25.6% and 25.8% and UV₂₅₄ 55.5% and 60.3%, respectively. In the result of Fig. 13 and Fig. 14, when only UF with 100 kDa membrane, organic removal efficiency shows low, but applying coagulation process before UF filtration could show higher organic removal. Therefore, applying a combination of coagulation and UF for river water treatment improved not only TOC removal efficiency but also DBPFP removal efficiency.

Changes in organic fraction after alum or PACI coagulation are summarized in Table 3. For both alum and PACI, the combined pretreatment with UF achieved higher organic removal. Especially, the PACI coagulant shows higher humic, fulvic, and hydrophilic removal than alum coagulant due to its higher amount of polymeric Al species contained in the PACI. Kang et al. [2001] also reported that PACI coagulant showed the most efficient TOC and turbidity removal among the aluminum coagulants used due to its highest amount of polymeric Al species contained in the PACI. In addition, the humic acid fraction was preferentially removed with both alum and PACI. The humic acid fraction was the most reactive precursor fraction for the formation of STHMFP and STOXFP. Therefore, combined process of coagulation and UF membrane performed much better for the removal of organic material and DBP precursors regardless of the coagulants used.

CONCLUSIONS

The raw water DOC contains 44% of hydrophilics, 35% of fulvic acid, and 21% of humic acid. The THMFPs for hydrophobic fraction (fulvic+humic acid) and hydrophilic fraction were 69% and 31%, respectively, but the TOXFPs for those were 37% and 63%, respectively. However, the humic acid fraction was the most reactive precursor fraction for the formation of STHMFP and STOXFP. Also, 88% of DOC has a molecular weight smaller than MWCO of 10 kDa, which suggests that without significant pretreatment, most organics in raw water should pass through UF membranes having the pore size of MWCO 100 kDa and 30 kDa. The smaller AMW fractions (<3 kDa) exhibit the most reactive precursor for the TOXFP and STOXFP, whereas larger AMW fraction (>10 kDa) was the most reactive precursor for the THMFP and STHMFP.

The result of adsorption kinetics tests showed that hydrophobic organics adsorbed much more quickly than hydrophilic organics on both membranes. Thus, hydrophobic compounds exhibited a

preferential adsorption onto membrane. In case of the effect of membrane properties on the adsorption of organic fractions, the adsorption ratio $[C(t)/C(e)]$ was greater for the hydrophobic membrane than for the hydrophilic membrane regardless of the kind of organic fractions.

The rate of flux decline for the hydrophobic membrane was significantly greater than for the hydrophilic membrane, regardless of pretreatment conditions. The pretreatment of the raw water significantly reduced the fouling of the hydrophilic membrane but did little decrease the flux reduction of the hydrophobic membrane. Also, combined pretreatment using coagulation improved not only dissolved organic removal efficiency but also DBP precursor's removal efficiency. Applying coagulation process before membrane filtration showed not only reduced membrane fouling, but also improved the removal of dissolved organic materials that might otherwise not be removed by the membrane. Especially, the PACl coagulant showed higher DBP precursors removal than alum due to its high amount of polymeric Al species contained in the PACl.

NOMENCLATURE

DBPFP	: Disinfection By Product Formation Potential
STHMFP	: Specific Trihalomethane Formation Potential ($\mu\text{g THMFP}/\text{mg DOC}$)
STOXFP	: Specific Total Organic Halogen Formation Potential ($\mu\text{g TOXFP}/\text{mg DOC}$)
MWCO	: Molecular Weight Cut Off
AMW	: Apparent Molecular Weight
SUVA	: Specific UV Absorbance ($\text{UV}_{254}/\text{DOC}$)
$C(t)$: the amount of organics adsorbed at time $[t]$
$C(e)$: the amount which can adsorb on the membrane surface at equilibrium
J_0	: permeate flux of initial purewater
J_t	: permeate flux at each time $[t]$
G	: velocity gradient
UFC	: Uniform Formation Condition

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