Environ. Eng. Res. Vol. 9, No. 2, pp. 58~65, 2004
Korean Society of Environmental Engineers

POLYAMINE FLOCCULANTS FOR THE ENHANCEMENT OF FLOCCULATION EFFICIENCY IN DRINKING WATER TREATMENT

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(received April 2003, accepted April 2004)

Abstract : Laboratory studies were conducted to evaluate the flocculation efficiency of synthesized polyamine flocculants used in combination with PAC (polyaluminum chloride) to treat water samples from the Nak-dong River in Korea. The use of polyamine flocculants in combination with PAC improved removal efficiency of turbidity, TOC and UV_{254}. The result of jar test indicated that addition of polyamine flocculant significantly reduced PAC consumption. The optimal polyamine flocculant concentrations were found to be 0.4 - 0.5 mg/L at lower turbidity (~16.0 NTU) and 0.5 - 1.0 mg/L at high turbidity (~740 NTU), respectively. Results indicated that the combination of PAC and polyamines was effective in reducing PAC dosage, lowering residual aluminum ion concentration in the treated water and improving turbidity removal.

Key Words : Polyamine, flocculant, PDA, turbidity, drinking water

INTRODUCTION

Flocculation has been used to treat suspended particles and dissolved organic matter from water. Flocculating agents are generally classified into two groups: metal salt inorganic flocculants and organic polymer flocculants.[1) Metal salt flocculants destabilize the suspension by compressing of the electrical double layer surrounding the particles. Polymer flocculants are water-soluble organic polymers containing functional groups such as amino or carboxyl in their polymer backbone. Their molecular weight ranges from a few thousands to millions (g/mol). According to their electric charge in the aqueous medium, the polymer flocculants are classified as cationic, anionic, and non-ionic.[2)] Destabilization of a suspension of particles by organic polymer flocculants occurs via adsorption of the long chain organic polymer on the particle and subsequent formation of particle-polymer-particle bridges.[3)]

Metal salt flocculants such as aluminum sulfate, PAC (polyaluminum chloride) and PACS (polyaluminum chloride silicate) have been used in drinking water treatment. These compounds may produce residual aluminum ion in the finished water that exceeds World Health Organization (WHO) recommended limits. In Korea, the acceptable residual aluminum ion

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level in drinking water must be below 0.2 mg/L since 1995.

The ability of water treatment plants to respond to fluctuations in raw water quality is essential. In some cases, like heavy rainy season, the use of inorganic flocculants is not sufficient to meet drinking water quality because fluctuations in raw water quality lead to failure of water treatment with metal salts. In recent years, the use of organic polymer flocculants has been considered for the removal of suspended particles and dissolved organic matter.[2,4] The different destabilization mechanism of these flocculants is thought to be a key factor in determining the effectiveness in the treatment of raw water with high turbidity.

In this paper, polyamine flocculants were synthesized in our laboratory and were used in combination with varying concentrations of PAC to treat samples from Nak-dong River. Jar tests were conducted to examine their efficiency in reducing turbidity, TOC and UV_{254}. The effect of polyamine addition was evaluated by comparing two treatments: (i) PAC alone and (ii) PAC/polyamine in combination. Pilot plant tests were also conducted to investigate the effect of polyamine addition on the residual aluminum and epichlorohydrin concentration.

Materials and Methods

Polyamine Synthesis

Polyamine flocculants were synthesized by the polycondensation of dimethylamine (DMA) and epichlorohydrin (EPI) using a two-step polycondensation reaction.[5~6] In the first step, dimethylamine was reacted with epichlorohydrin by adding drop wise through a dropping funnel for 3-7 hours at 30-40°C. In the next step, the reaction mixture was heated for 2-5 hours at 90-95°C and deionized water was added to reaction mixture. Polyamine with 50% solid content was obtained. Four polyamine solutions were produced, each with a different molar mass. The molecular weight of each polyamine polymer was determined by intrinsic viscosity.

The intrinsic viscosity of each polyamine solution was determined by extrapolation of η_{sp}/c or $\ln(\eta/\eta_0)/c$ values to the zero concentration according to the Huggins' (Eq. 1) and Kramer's (Eq. 2) equation, respectively[7]:

$$\eta_{red} = \frac{\eta_{sp}}{c} = [\eta] + k'[\eta]^2 c \quad \text{(i)}$$

where η_{red} is the reduced viscosity (dL/g), η_{sp} is specific viscosity (dimensionless), c is concentration of polymer (g/dL), η is the intrinsic viscosity (dL/g), and k' is a constant.

$$\eta_{inh} = \frac{\ln(\eta/\eta_o)}{c} = [\eta] + k''[\eta]^2 c \quad \text{(ii)}$$

where η_{inh} is the inherent viscosity (dL/g), η is the viscosity at the flow time of polymer solution, t, η_o is the viscosity at the flow time of solvent (1.0% NaCl solution), t_o in the Ubbelohde viscometer, respectively, and k'' is a constant. The specific viscosity is often represented in terms of flow time:

$$\eta_{sp} = \frac{\eta}{\eta_o} - 1 = \frac{t}{t_o} - 1 \quad \text{(iii)}$$

The intrinsic viscosity is used as a measure of the molecular weight of the polymer. The intrinsic viscosities, $[\eta]$, of the polyamine flocculants were 0.055 dL/g (Kufloc 101A), 0.115 dL/g (Kufloc 102A), 0.263 dL/g (Kufloc 201A) and 0.445 dL/g (Kufloc 301A) which correspond to molar mass of approximately 10,000, 12,000, 47,000 and 100,000 g/mol, respectively. PAC (10 wt% Al_2O_3, Gyung-Gi Chemical, Korea) and Kufloc 101A and 102A were used for the flocculation experiments. Kufloc 201A and 301A were used for the aggregation experiments.

Particle Aggregation Studies

A photometric dispersion analyzer (PDA, Rank Brothers Ltd., UK., Model PDA 2000) was used to determine the kinetics of particle aggregation of the synthesized polyamines following the procedure and using the apparatus of Pang and Englezos.[8~9)] The PDA monitors the fluctuations in intensity of light transmitted through the suspensions. The changes in the degree of aggregation were reflected by changes in the root mean square of the fluctuations in voltage (V_{rms}). The ratio of V_{rms} to the mean transmitted light intensity (DC) was used as an indicator of the relative aggregate size. The theoretical background and the technical details of the PDA are well discussed elsewhere.[10~11)]

The effect of polyamine concentration on particle aggregation was determined using aqueous Na_2SO_4-clay systems. A clay-Na_2SO_4 suspension was prepared by dissolving 34.09 g Na_2SO_4 in 352 mL water and adding 8 mL of 1% (w/w %) clay stock suspension. 40 mL of polyamine solution (0.1 wt.% Kufloc 201A) was prepared separately and kept at constant temperature (30°C) using a water bath. After equilibrium, the clay-Na_2SO_4 suspension was continuously stirred with a magnetic stirrer and circulated through the PDA cell using a peristatic pump at a rate of 50 mL/min. The V_{rms}/DC ratio of the suspension was measured for initial 20 seconds. Then the polyamine solution was added into the suspension and the degree of particle aggregation was monitored for further 13-20 minutes.

Water Quality

A six-cube jar test apparatus (Phipps & Bird Stirrer, Model 7790-400, VA, USA) of each jar containing 1 L of water was used to evaluate the effectiveness of PAC and PAC/polyamine in removing turbidity, TOC and UV_{254}. Rapid mixing was performed for 1 minute at a paddle speed of 180 rpm. Flocculation was conducted for 10 minutes at 50 rpm and sedimentation was allowed for 20 minutes without stirring. After settling, 50 mL of the supernatant was sampled and turbidity (HACH 9200 Turbidimeter, CO, USA) was measured immediately. Total organic carbon (Shimadzu, TOC-5000A, Tokyo, Japan) and UV_{254} (Hewlett Packard, UV Spectrophotometer 8452A) were measured to determine the removal efficiency of dissolved organic matter in the water by PAC and/or polyamine flocculants.

Analysis of Residual Aluminum and Epichlorohydrin Concentration

Pilot test was also performed on water samples with low and high turbidity obtained from Nak-dong River. The effect of polyamine addition on residual aluminum and epichlorohydrin concentration was determined by comparing PAC alone and PAC/polyamine treatment. The operating condition of the pilot plant is listed in Table 1. The residual aluminum concentrations in the raw and treated water samples were determined by the Inductively Coupled Plasma (Perkin Elmer, Optima 300 DV ICP, CT, USA).

The sub-samples of the polyamine were analyzed for residual toxic materials such as epichlorohydrin, 1,3-dichloro-2-propanol, 2,3-dichloro-

Table 1. Operating conditions and specifications of the pilot-scale water treatment plant

Parameter		Operation condition
Flow rate		20 ton/day
Retention time	Mixing tank	2 min
	Flocculation tank I	12 min
	Flocculation tank II	12 min
	Sedimentation tank	240 min
Impellor speed	Mixing tank	120 rpm
	Flocculation tank I	25 rpm
	Flocculation tank II	20 rpm
Linear velocity in sand filter		210 m/d

1-propanol, and 3-chloro-1,2-propanediol using gas chromatography (Hewlett Packard 5890 Series II) equipped with flame ionization detector. The column used in this study was a fused silica capillary column purchased from Superco Co. (Supercowax™ 10, Seoul, Korea). Nitrogen was used as a carrier gas and the flow-rate was 1.63 mL/min. The split ratio was 20:1 and injection port was set at 230°C. The oven temperature was programmed to be 130°C for 1 minute and then ramped at 10°C min/L up to 220°C. The residual material was extracted using an isopropanol-chloroform mixture (50:50, v/v %). The solvent extraction was conducted by shaking in a shake bath for 1 hour. The extractant was centrifuged at 2,000 rpm for 20 minutes. The extractant was analyzed to determine the residual concentration of epichlorohydrin, 1,3-dichloro-2-propanol, 2,3-dichloro-1-propanol, and 3-chloro-1,2-propanediol.

Results and Discussion

Particle Aggregation Studies

The effect of polyamine (Kufloc 201A) addition on flocculation was examined by comparing against treatment without polyamine (Fig. 1). As shown in the figure, addition of small amount (0.5 mg/L) of polyamine resulted in flocculation of the clay to the equilibrium particle size after 600 seconds. A high dose of polyamine (2.5 mg/L) created greater equilibrium particle size than small dose (0.5 mg/L) as indicated by relative particle size. Addition of cationic polyamine (Kufloc 201A) reduces the surface charge of clay and enhances the flocculation of negatively charged clay particles.[8~9) Addition of 0.5-2.5 mg/L of polyamine was successful in improving flocculation efficiency. An implication of this result is that only limited amount of polyamine should be used for optimal flocculation. It should be noted that a high dose of polyamine flocculant can decrease overall flocculation efficiency due to particle repulsion. Therefore, optimal dose of polyamine flocculant should be determined for economic feasibility.

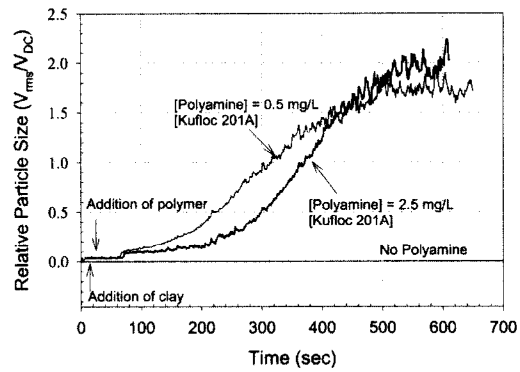

Fig. 1. Flocculation efficiency of polyamine.

Fig. 2 shows the effect of polyamine molecular weight on the flocculation. Kufloc 301A 100,000 g/mol and 201A 47,000 g/mol solutions were used (0.001 wt.%). Both polyamines reached equilibrium particle size after approximately 1,200 seconds. However, Kulfoc 301A generated much larger particle size than Kufloc 201A due to its higher molecular weight (MW = 100,000 g/mol) and intrinsic viscosity ([η] = 0.445) than Kufloc 201A (MW = 47,000 g/mol; [η] = 0.263). The use of polyamine with higher molecular weight and intrinsic viscosity may facilitate flocculation and subsequently reduce the time to reach equilibrium particle size (Fig. 2).

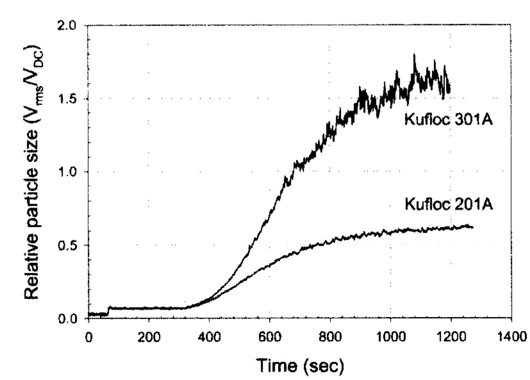

Fig. 2. Effect of Kufloc 201A concentration on clay aggregation.

Water Quality Studies: Effect of Polyamine Flocculant

Effect of polyamine flocculants in combination with PAC on the removal of turbidity, TOC and UV_{254} were examined. Water samples

Table 2. Effect of polyamine flocculants on the removal of turbidity, TOC, and UV_{254} at average turbidity

Raw Water Quality				Added Flocculants [mg/L]			Final Water Quality		
Turbidity [NTU]	pH	TOC [mg/L]	UV_{254} [cm^{-1}]	PAC	Kufloc 101A	Kufloc 102A	Turbidity [NTU]	TOC [mg/L]	UV_{254} [cm^{-1}]
23.0	7.89	3.39	0.108	30	0	0	2.0	2.88	0.038
				15	0	0	3.3	3.01	0.048
				15	0.5	0	1.7	2.51	0.027
				15	0	0.5	1.7	2.78	0.033
34.3	7.32	3.02	0.150	30	0	0	2.0	2.11	0.030
				15	0	0	2.7	2.52	0.037
				15	0.5	0	1.4	2.39	0.035
				15	0	0.5	1.4	2.17	0.031

Table 3. Effect of polyamine flocculants on the removal of turbidity, TOC, and UV_{254} at high turbidity

Raw Water Quality				Added Flocculants [mg/L]			Final Water Quality		
Turbidity [NTU]	pH	TOC [mg/L]	UV_{254} [cm^{-1}]	PAC	Kufloc 101A	Kufloc 102A	Turbidity [NTU]	TOC [mg/L]	UV_{254} [cm^{-1}]
646	6.56	5.62	2.895	40	0	0	4.9	3.23	0.058
				20	0	0	30.3	4.44	0.228
				20	1.0	0	4.3	3.40	0.072
				20	0	1.0	4.2	3.33	0.069
2,138	6.86	8.63	3.931	80	0	0	6.7	2.68	0.058
				40	0	0	84.3	4.19	0.227
				40	1.0	0	5.9	3.00	0.042
				40	0	1.0	5.2	2.86	0.034

having average turbidity (Table 2) and high turbidity (Table 3) were tested and compared. In Table 2, two raw water samples at different turbidity (23.0 NTU and 34.3 NTU) were obtained from the Nak-dong River. 15 mg/L of PAC was not able to reduce turbidity below 2.0 NTU. Application of 30 mg/L of PAC lowered the turbidity to 2.0 NTU. This is probably due to insufficient floc formation at lower PAC dose. At the lower concentration of PAC (15 mg/L), turbidity, TOC and UV_{254} removal were improved by adding 0.5 mg/L of polyamine flocculants. Both Kufloc 101A and 102A (0.5 mg/L) lowered turbidity below 2.0 NTU. This suggests that the use of polyamine flocculants can reduce PAC dose and may result in lower amount of sludge production.[12)]

The polyamine flocculants were also tested for the treatment of water samples of high turbidity (Table 3). High fluctuation in turbidity is often observed in Nak-dong River water due to heavy rains in summer. For the treatment of raw water with a lower turbidity (646 NTU), the addition of 20 mg/L of PAC lowered the turbidity to 30.3 NTU. A concentration of 40 mg/L PAC reduced the turbidity from 646 to 4.9 NTU. For raw water with a higher turbidity (2,138 NTU), 80 mg/L PAC reduced the turbidity to 6.7 NTU. After addition of 40 mg/L of PAC, the turbidity was decreased to 84.3 NTU. Higher PAC dosages were needed to improve water quality, but may produce more residual aluminum ion in the treated water.[13)] At fixed amounts of PAC (20 mg/L for 646 NTU and 40 mg/L for 2,138 NTU), addition of 1.0 mg/L of polyamines lowered turbidity, TOC and UV_{254} (Table 2). Kufloc 102A was more efficient than Kufloc 101A due to higher molecular weight and intrinsic viscosity.

Water Quality Studies: Effect of Polyamine Concentration.

Fig. 3 shows the effect of the polyamine dosage on turbidity, TOC and UV_{254} removal at raw turbidity of 16.0 NTU. The polyamine (Kufloc 101A) concentration was varied to

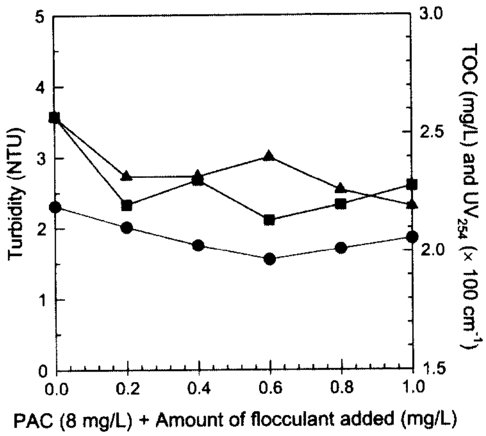

Fig. 3. Effect of Kufloc 101A concentration with PAC 8 mg/L at average turbidity.
(Raw water quality: Turbidity = 16.0 NTU, TOC = 3.04 mg/L and UV_{254} = 0.061 cm^{-1})
Symbols: ▲ = TOC, ■ = UV_{254} and ● = turbidity.

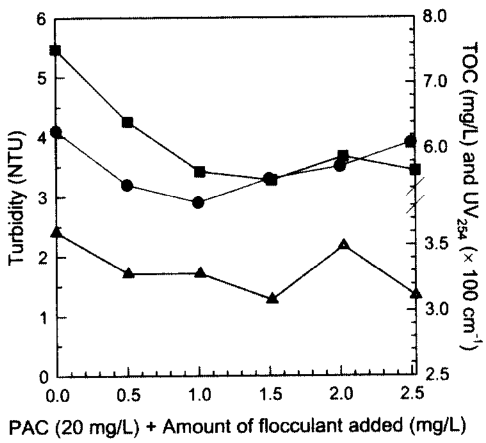

Fig. 4. Effect of Kufloc 102A concentration with PAC 20 mg/L at high turbidity.
(Raw water quality: Turbidity = 740 NTU, TOC = 4.61 mg/L and UV_{254} = 2.362 cm^{-1})
Symbols: ▲ = TOC, ■ = UV_{254} and ● = turbidity.

determine optimal polyamine concentration at constant amount of PAC (8 mg/L). The optimal polyamine flocculant concentration was obtained at the range of 0.4-0.8 mg/L at average turbidity. However, the patterns of TOC and UV_{254} values were not consistent with turbidity.

The effect of polyamine flocculant dosage on turbidity, TOC and UV_{254} removal was also examined for 740 NTU turbidity. As depicted in Fig. 4, the PAC concentration was fixed at 20 mg/L while polyamine flocculant (Kufloc 102A) concentration was varied to determine optimal dosage. The optimal polyamine dose was observed at the range of 0.5-1.5 mg/L for the treatment of water sample with high turbidity. Similar pattern was also observed in TOC and UV_{254} confirming the optimal dosage of Kufloc 102A. Turbidity, TOC and UV_{254} values increased at high polyamine dosage (above 1.0 mg/L). This is probably due to re-charging of the surface of the clay particles and subsequent electrostatic repulsion between particles.[2)]

Residual Aluminum and Epichlorohydrin Concentration

The residual aluminum concentration in the treated water samples was determined and summarized in Table 4 (low turbidity) and Table 5 (high turbidity), respectively. For the low turbidity water (Table 4), the residual aluminum concentration was 0.20 ppm in PAC alone treatment (12 mg/L of PAC) after sedimentation. In contrast, addition of 1 mg/L of polyamine in addition to 6 mg/L of PAC lowered residual aluminum concentration to 0.04-0.10 mg/L after sedimentation. After filtration, the residual aluminum concentration decreased to 0.1 mg/L in PAC alone treatment and 0.02-0.03 mg/L in PAC/polyamine treatment, respectively. A similar pattern was also observed in high turbidity water treatment. In PAC alone treatment (0.1 mg/L of PAC), the residual aluminum concentration was 0.25 mg/L after sedimentation. In contrast, the addition of 1 mg/L of polyamine flocculants (Kufloc 101A and 201A) in addition to 30 mg/L of PAC lowered residual aluminum concentration to 0.07-0.12 mg/L after sedimentation. After filtration, the residual aluminum concentration was 0.15 mg/L in PAC alone treatment while addition of polyamine lowered aluminum concentration below 0.04 mg/L. This is presumably because less amount of PAC was used in PAC/polyamine treatment. In both cases of low and high turbidity, PAC alone treatment exhibited consistently higher residual aluminum

Table 4. Residual aluminum concentration in raw and treated water in pilot plant (low turbidity)

Treatment	Intact Water [mg/L]	After Sedimentation [mg/L]	After Filtration [mg/L]
PAC [a)]	0.52	0.20	0.10
PAC + Kufloc 101A [b)]	0.31	0.04	0.03
PAC + Kufloc 201A [c)]	0.29	0.10	0.02
PAC + Kufloc 301A [d)]	0.23	0.10	0.02

a) = PAC alone (12 mg/L), *b)* = PAC (6 mg/L) + Kufloc 101A (1 mg/L), *c)* = PAC (6 mg/L) + Kufloc 201A (1 mg/L), *d)* = PAC (6 mg/L) + Kufloc 301A (1 mg/L). Raw water quality: turbidity = 13-15 NTU, pH = 7.32 and T = 20.4-23.5 °C

Table 5. Residual aluminum concentration in raw and treated water in pilot plant (high turbidity)

Treatment	Intact Water [ppm]	After Sedimentation [ppm]	After Filtration [ppm]
PAC [a)]	3.30	0.25	0.15
PAC + Kufloc 101A [b)]	1.69	0.07	0.04
PAC + Kufloc 201A [c)]	2.00	0.11	0.04
PAC + Kufloc 301A [d)]	2.19	0.12	0.02

a) = PAC alone (60 mg/L), *b)* = PAC (30 mg/L) + Kufloc 101A (1 mg/L), *c)* = PAC (30 mg/L) + Kufloc 201A (1 mg/L), *d)* = PAC (30 mg/L) + Kufloc 301A (1 mg/L). Raw water quality: turbidity = 210-530 NTU, pH = 6.43 and T = 23.5-28.4 °C

Table 6. Analysis of residual chemicals in water treatment facility and pilot plant

	Water Treatment Facility		Pilot Plant	
Chemicals	Sedimentation	Filtration	Sedimentation	Filtration
Epichlorohydrine	<0.1	<0.1	0.3	0.3
1,3-Dichloro-2-propanol	0.20	0.01	0.23	<0.01
2,3-Dichloro-1-propanol	0.06	0.01	0.11	<0.01
3-Chloro-1,2-propandiol	<1.0	<1.0	<1.0	<1.0

All units: μg/L. NPDWR limit: < 2 μg/L (i.e., 0.01% dosed at 20 mg/L).

concentrations than PAC/polyamine treatment. The addition of polyamine flocculants was highly effective in reducing residual aluminum concentration.

Several byproducts such as epichlorohydrin, 1,3-dichloro-2-propanol, 2,3-dichloro-1-propanol, and 3-chloro-1,2-propandiol are often observed in the polyamine synthesis.[14] Epichlorohydrin is regulated by National Primary Drinking Water Regulations (NPDWR) due to possible human health risks and exposure.[15] The residual concentrations of the byproducts in a water treatment facility and a pilot plant study were analyzed using a GC-MS[16] and shown in Table 6. After both sedimentation and filtration steps, the residual chemical concentrations were below NPDWR limit (< 2 μg/L, i.e., 0.01% dosed at 20 mg/L).

CONCLUSIONS

Polyamine flocculants synthesized for potable water treatment were evaluated as a means of reducing PAC dosage to remove turbidity, TOC and UV_{254} in Nak-dong river raw water in Korea. A higher aggregation rate of particulate matter was obtained as the molecular weight of the polymer was increased. The polyamine flocculants in combination with PAC efficiently removed turbidity, TOC and UV_{254}. The result of jar test showed that addition of polyamine flocculant significantly reduced PAC consumption. The optimal polyamine flocculant concentrations were found to be 0.4-1.0 mg/L at average turbidity (~16.0 NTU) and 0.5-1.5 mg/L at high turbidity (~740 NTU), respectively. Results indicated that the combination of PAC and polyamines was effective in reducing PAC dosage, lowering residual aluminum ion concentration in the treated water and improving turbidity removal.

ACKNOWLEDGEMENTS

The authors would like to thank the G7 project from the Ministry of Environment of Korea and Brain Korea 21 project from Ministry of Education of Korea for the financial support of this work.

NOMENCLATURE

η_{red} = the reduced viscosity (dL/g),
η_{sp} = specific viscosity (dimensionless),
c = the concentration of polymer (g/dL),
η = the intrinsic viscosity (dL/g),
k' = a constant
η_{inh} = the inherent viscosity (dL/g)
$\eta(t)$ = the viscosity at the flow time, t of polymer solution,
η_o = the viscosity at the flow time of solvent, to in the Ubbelohde viscometer
k'' = a constant.

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