Flocculation Kinetics Using Fe(Ⅲ) Coagulant in Advanced Water Treatment: The Effect of Sulfate Ion

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Abstract

The study of flocculation kinetics is of fundamental interest in the field of water treatment, because rational study of the factors affecting the coagulation process should be based on the rate of particle growth. The effect of sulfate on flocculation kinetics were examined using ferric nitrate as a coagulant to coagulate kaolin clay in water under several experimental conditions. Both the particle size distribution data obtained from the AIA and the on-line measurement of turbidity fluctuation by the PDA were used to measure flocculation kinetics. Results show that sulfate ion added to the kaolin suspension played an important role in the flocculation process, not only improving flocculation kinetics at more acidic pH levels but also changing surface charge of particles. The kinetics of flocculation were improved mainly by the enhanced rate and extent of Fe(Ⅲ) precipitation attributed to the addition of sulfate, and thereby, better interparticle collision frequency, but little by the charge reductions resulting from the sulfate addition. The increase in sulfate concentration beyond 3×10^{-4}M (up to 2×10^{-3}M) did not induce further improvement in flocculation kinetics, although the higher concentrations of sulfate ion substantially increased the negative ZP value of particles.

Key Words : Flocculation Kinetics, Fe(Ⅲ) Coagulant, Sulfate ion, Turbidity Fluctuation.

1. INTRODUCTION

The study of flocculation kinetics is of fundamental interest in the field of water treatment, because rational study of the factors affecting the coagulation process should be based on the rate of particle growth. Therefore, the study of flocculation kinetics can provide a better insight into the flocculation mechanism, the various parameters involved, their impact on the performance of the process, and then improving the design and performance in subsequent separation steps. Much attention has been given to the two mechanisms of coagulation in recent years, adsorption-destabilization(A/D) and sweep flocculation. However, there has been little work done concerning the differences on flocculation kinetics covering the full range of coagulation domains encompassing both the A/D and sweep floc mechanisms. In addition, while some studies have been conducted on the effect of sulfate ion on flocculation efficiency, little work has been performed using flocculation kinetics as a tool to study those effects.

This research focuses on the use of Fe(Ⅲ) salt to make quantitative studies of the rate at which flocculation processes occur, the factors on which these rates depend, and the mechanisms

involved. Fe(Ⅲ) salts are receiving attention as alternative coagulants to alum for several important reasons; (1) concerns about residual aluminum concentration in treated waters and post-precipitation of aluminum in distribution systems, (2) possible cost savings comparing to other alternative coagulants, and (3) better coagulation efficiency at low water temperature and more efficient removal of natural organic matters than alum. Sulfate ion is an interesting model ion in coagulation for at least three reasons; (1) it is one of the most common anions in natural waters and is known to affect coagulation with metal salt coagulants, (2) it is also the associated anion with commonly used metal salt coagulants, such as alum and ferric sulfate, and (3) its use causes the formation of complexes with metal salt coagulants, and hence affects the coagulation process.

2. BACKGROUND

The presence of certain ions, particularly anions, has been shown to alter the pH range of optimum coagulation, the reaction period for efficient flocculation, the optimum coagulant dose, and the residual coagulant in the effluent (AWWA, 1971). Among the ions commonly found in natural waters, sulfate ion is different in the nature of the interaction between simple ions like K^+, Na^+, Cl^-, and NO_3^- and the particle surface. Flynn(1984) noted that the sulfate ion complexes Fe(Ⅲ), and thus, affects hydrolysis kinetics, but, this is not the case for Fe(Ⅲ) salts of nitrate and perchlorate which produce essentially indifferent electrolyte solutions. Stumm and Morgan(1962) presented alkalimetric titration data of Al(Ⅲ) and Fe(Ⅲ) in the presence of anions, and demonstrated that the

quantity of OH^- ion required was less than the stoichiometric requirements for the neutralization of any excess acid plus precipitation of aluminum hydroxide. Since less OH^- ions have to enter into the lattice if other coordinating anions are present, the pH of optimum coagulation will frequently be lower than it is in the absence of complex forming ligands. Since the adsorption of anions is coupled with a release of OH^- ions, adsorption is favored by lower pH values. Dzombak and Morel(1990) also showed that more SO_4^{2-} adsorption on ferric (hydr)oxide surface was accomplished at more acidic solution.

The role of sulfate ion on the hydrolysis-precipitation of Al(Ⅲ) solutions was studied by de Hek et al.(1978). They noted that the divalent sulfate ion is known to affect the course of the hydrolysis-precipitation process as well as the composition, texture, and structure of the resultant precipitates. They concluded that the observed behavior of the sulfate ion is that expected of a catalyst which accelerates the overall process by lowering the kinetic barriers(Energetic and/or entrophic). Dousma et al.(1979) reported that the major difference between sulfate system compared with chloride or nitrate system is the catalytic effect of SO_4^{2-} in promoting nucleation and growth of Fe(Ⅲ) precipitates at lower supersaturation. Several authors(Flynn, 1984; Dzombak and Morel, 1990; Thompson and Tahir, 1991) also reported that the presence of SO_4^{2-} resulted in precipitation at a greater rate and at lower pH values or $[OH^-]/[Fe]$ mole ratios than solutions with only uninegative anions. There are also reports that sulfate ion produced a dramatic effect of significantly widening optimum coagulation pH range, by eliminating restabilization and widening the sweep zone to the acidic side (Hanna and Rubin, 1970; Letterman and

Table 1. Summary of Experimental Conditions

Parameters	Description
Primary particle	Kaolin clay (1.8 um avg. dia.)
Particle concentration	25, 50, and 100 mg/ℓ of Kaolin clay
	(Turbidity: 24, 53, and 106 NTU)
Dilution water	distilled water with 0.005 M $NaNO_3$
Coagulant	stock solution: 0.25 M as $Fe(NO_3)_3.9H_2O$
	Dosing solution of 10 mg/mℓ as $Fe(NO_3)_3.9H_2O$
Rapid mixing	250 rpm (G=455 s^{-1} at 23 °C) for 1 min
Slow mixing	30, 45, 60rpm (G=20, 37, 56 s^{-1} at 23 °C)
Suspension pH	6.0, 6.5, 6.8, and 8.0
Sulfate concentration	0 to 0.002 M using 1 M Na_2SO_4 solution

Vanderbrook, 1983).

3. EXPERIMENTAL MATERIALS AND METHODS

3.1 Experimental Materials

These experiments were conducted using the tightly controlled experimental conditions deduced from conventional jar test results. The experimental conditions were chosen for the comparison of flocculation kinetics spanning the full range of coagulation domains normally encountered in water treatment practices. The experimental parameters used in this work are summarized in Table 1. Kaolin clay, with a log mean diameter of 1.8 μm, was used as the primary particulate system. A stock clay suspension of 800 mg/ℓ was prepared in a 60 L plastic tank equipped with a mixing device, and brought to pH 7 with 0.1 N NaOH. This large volume of stock clay suspension stored at neutral pH provided a reproducible means of adding particles to water. The turbidity was checked on a Model 18900 ratio turbidimeter (Hach Chemical Co., Loveland, CO), and the zeta potential (ZP) was measured using a Model 104 Lazer Zee Meter (Pen-Kem Inc., Hudson, NY).

The dilution water used to make a desirable concentration of clay suspension from the stock clay solution was distilled water. The distilled water was stored in a 300 ℓ polyethylene tank before use, while it was being brought to equilibrium with atmospheric CO_2 and temperature. When diluting clay suspension, a desired amount of 1 M $NaNO_3$ was added to maintain 0.005 M of $NaNO_3$, which gives 0.005 M ionic strength. The choice of 0.005 M $NaNO_3$ was based on the results obtained from preliminary tests, in which the 0.005 M ionic strength used did not give any sign of aggregation of clay particles due to destabilization and facilitated consistent measurements of the ZP.

Ferric nitrate was used as a coagulant to allow sulfate concentration to be controlled as an independent variable. The stock solution contained 0.25 M $Fe(NO_3)_3.9H_2O$ was stored at room temperature. The dosing solution of 10 mg/mℓ as $Fe(NO_3)_3.9H_2O$ was prepared volumetrically from the stock solution the day before it was used to ensure consistent speciation in the coagulant dosing solution. The pH of stock and dosing solutions were checked periodically to check for potential aging of Fe (III) in the solution. The suspension pH was

adjusted to the desired target pH by using 0.02 to 0.05 N NaOH or 0.01 N HNO_3. As shown in Table 1, a minimum pH of 6.0 was selected for one pH value on the basis of preliminary jar tests because coagulation at this pH can be expected to be mainly caused by the A/D mechanism. A pH of 8.0 was selected for the highest pH that showed coagulation due to rapid precipitate formation. A stock solution of 1 M Na_2SO_4 solution was used as a source of sulfate for the study of sulfate ion effect.

3.2 Flocculation Kinetics Instrumentation

The quantitative measure of change in aggregate size or, alternatively, the change in number of primary particles due to the growth of aggregates can be used to assess the kinetics of flocculation. In this research, two methods were used to assess flocculation kinetics. The first method was the direct quantitative measure of the changes in the particle size distribution by use of automatic image analysis system (AIA) (Lemont, OASYS). The AIA can measure and analyze the number of particles in several discrete sizes, ranging from 0.5 to $400 \mu m$, depending on a magnification power selection for the microscope used. A schematic of the image analysis system is shown in Fig. 1. The AIA system was coupled directly to an Olympus BH-2S upright, transmitted light microscope via a video camera. The image to be analyzed by the AIA was input to the system through a TV/video camera mounted on the microscope and digitized.

The second method was the rather indirect qualitative measurement by use of an Photometric Dispersion Analyzer (PDA) (Rank Brother LTD, Cambridge, UK) in which the degree of turbidity fluctuation due to the continuous changes in the number concentration and sizes
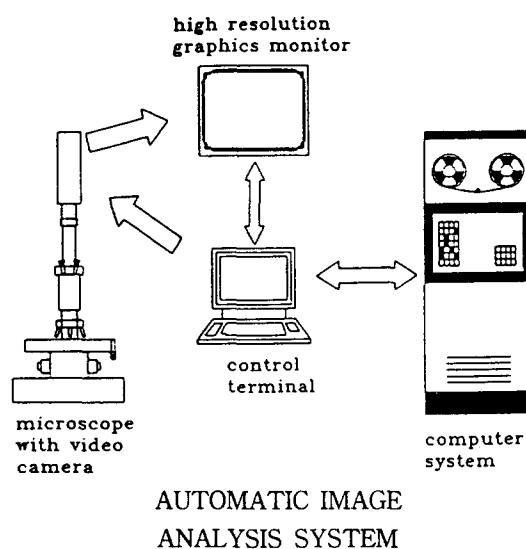

AUTOMATIC IMAGE
ANALYSIS SYSTEM

Fig. 1. Schematic of Automatic Image Analysis system (AIA).

of particles can be obtained and related to the rate of flocculation. It has been shown that the root mean square(rms) value of the fluctuating signal varies with the square root of the particle concentration and with the size of the suspended particles in a rather complex manner(Gregory, 1985). Assuming that variation in particle number follows Poisson distribution, Gregory(1985) derived formulas as expressed in Eq. (1) for the relationship between the PDA output, and concentration and size of suspended particles using a modified Lambert-Beer law

$$V_{rms}/V = (L/A)^{1/2} (\sum N_i C_i^2)^{1/2} \qquad (1)$$

where, V is the voltage corresponding to the intensity of light after passing through a suspension and V_{rms} is the standard deviation of the voltage fluctuation around the mean value (V). L and A are optical path length and effective cross-sectional area of the light beam. N_i and C_i represent the number concentration and scattering cross-section of particles of type i, respectively. The nature of the sum in Eq.(1)
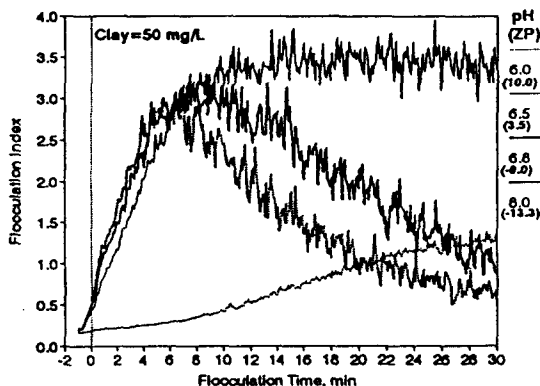
Fig. 2. Effect of pH on the rate of flocculation(clay:
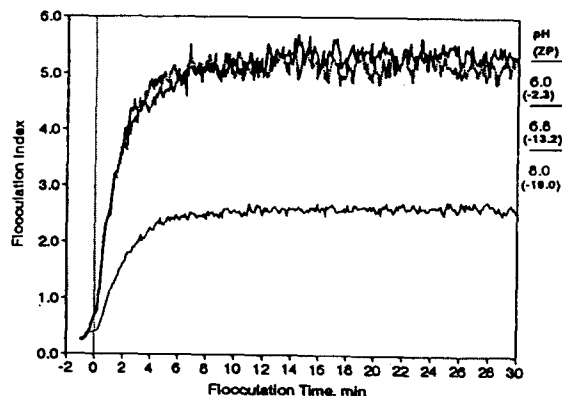50 mg/ℓ, dose: 5 mg/ℓ, 30 rpm).

Fig. 3. Effect of pH on the rate of flocculation (clay:
100 mg/ℓ, dose: 5 mg/ℓ, 45 rpm).

3.3 Experimental Methods

Bench scale flocculation tests were carried out in an 18 ℓ square batch reactor. The 18 ℓ reactor was equipped with a two-flat blade impeller, electric motor with speed controller, and a tachometer. The detailed experimental setup and methods used in this study are presented in the author's earlier work(1995). All these experiments were conducted at room temperature (23±0.5 ℃).

4. RESULTS AND DISCUSSION

4.1 Kinetics of Flocculation

In the earlier work presented by Kang(1995), a comparison of particle count measurements by the AIA and the growth of aggregates expressed as the singnals of the PDA was made to assess the reliability of both kinetics measuring techniques. The flocculation kinetics data, as presented in Kang(1995), demonstrated that both the particle size distribution data obtained from the AIA and the on-line measurement of trubidity flutuation by the PDA

provided reliable and sensitive indications of flocculation kinetics, as evidenced by the rate of disappearance of primary particles and the rate of aggregation of particles.

Figs. 2 and 3 present the rate of flocculation over the pH range 6.0 to 8.0 for 50 and 100 mg/ℓ of clay conentration under the same experimental conditions. In the X-axis, the time zero means the time at which the flocculatin was initiated immediately after rapid mixing, thus the negative time represents the rapid mix cycle. Generally, Figs. 2 and 3 shows the PDA monitor responses collected at every 5 seconds. The ratio value was referred as flocculation index for the sake of illustration and discussion. Fig. 2 and 3 indicates that the ratio, V_{rms}/V, always increases in the course of aggregation until a limiting value is reached.

Figs. 2 and 3 demonstrate the dramatic impact of suspension pH on the rate of flocculation. Overall, under the same coagulant dose, the curves in Figs. 2 and 3 show marked increases in the rate of flocculation as the suspension pH increases from pH 6.0 to 8.0. These results show that flocculation occurred rapidly during the first few minutes of the process. As the particles became larger and the
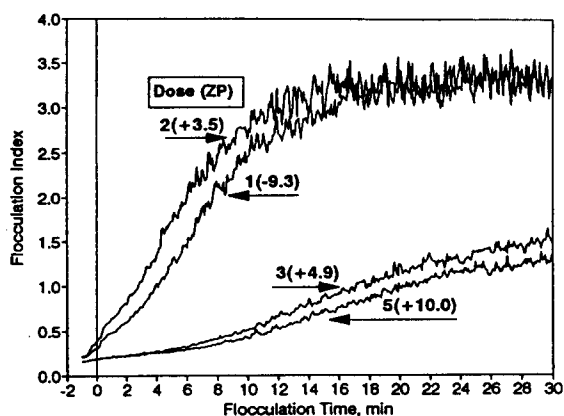
Fig. 4. Effect of coagulnt dose on the rate of flocculation (pH 6.0, clay: 50 mg/ℓ, 30 rpm).

concentrations of particles in suspension was reduced, the kinetics of flocculation was slowed

down. It was noted for 50 mg/ℓ clay concentrations that the rate of flocculation at pH 6.0 was substantially slower than at higher pH. For 100 mg/ℓ of clay concentration as shown in Fig. 3, the ZP values indicate that complete charge neutralization was not achieved, nor necessary, at 5 mg/ℓ of coagulant dose. Thus, the improvement of flocculation kinetics with increasing suspension pH was evident in spite of a more negative charged particles obtained at the higher pH. Comparison of the results shown in Figs. 2 and 3 suggests that destabilization by complete charge reduction is not sufficient to cause rapid particle aggregation. That is, the aggregation of clay suspension by Fe(Ⅲ) coagulant required additional particles due to the
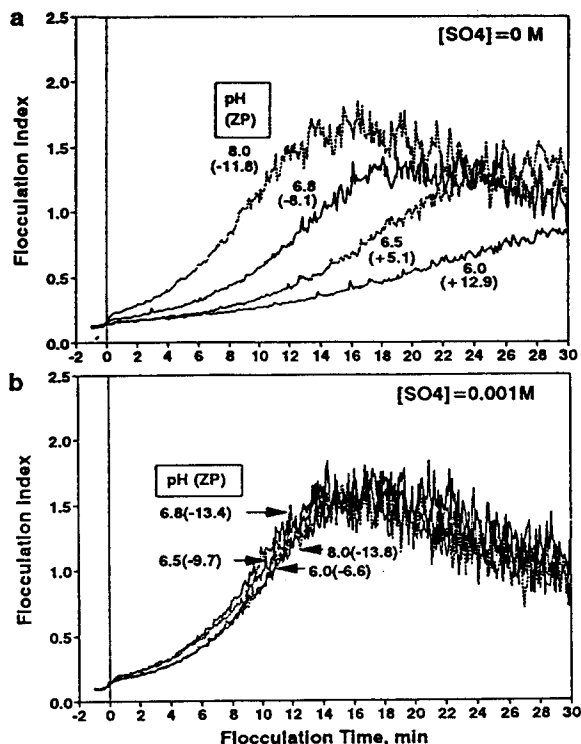

Fig. 5. Effect of sulfate on the rate of flocculation (clay: 25 mg/ℓ, dose: 5 mg/ℓ, 30 rpm): (a) without using sulfate; (b) using 0.001 M SO_4^{2-}.
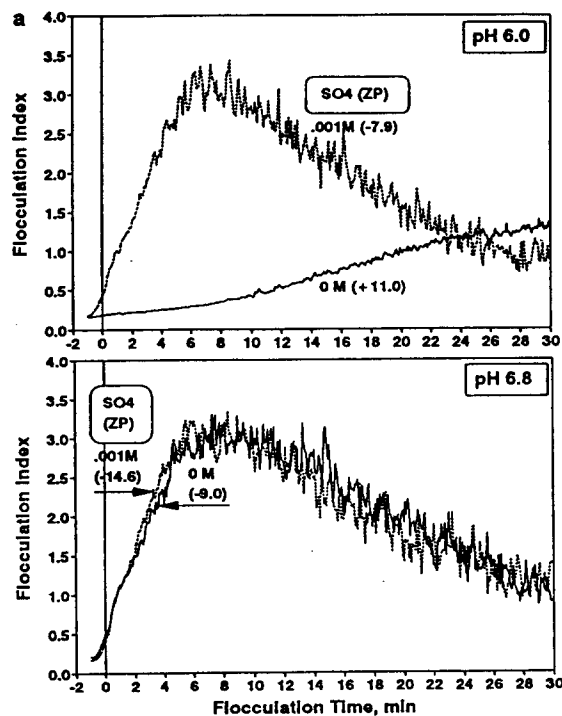

Fig. 6. Effect of sulfate on the rate of flocculation (clay: 50 mg/ℓ, dose: 5 mg/ℓ, 30 rpm): (a) pH 6.0; (b) pH 6.8.

precipitation of iron hydroxide solids to enhance flocculation kinetics, even at the high clay concentration of 100 mg/ℓ.

The effect of the coagulant dose on the flocculation kinetics for 50 mg/L clay concentration are presented in Fig. 4 at pH 6.0 to elucidate the mechanism of coagulation. Fig. 4 indicates that particle aggregation was more rapid slightly at 2 mg/ℓ than 1 mg/ℓ. At or above 3 mg/ℓ dose, the rate of flocculation was significantly reduced, probably attributed to charge restabilization of particles in suspension. This result confirmed that the overall process of particle aggregation at pH 6.0 was caused mainly by adsorption-destabilization of clay suspension by the addition of a hydrolyzed Fe(III) species.

4.2 Effects of Sulfate

Figs. 5(a) and (b) represent the rate of flocculation expressed by the flocculation index against mixing time without and with the addition of 10^{-3}M SO_4^{-2} using 25 mg/ℓ clay concentration at different pH levels. Again, Fig. 5(a) demonstrates the dramatic impact of suspension pH on the rate of flocculation. It is noteworthy that in the presence of 10^{-3}M SO_4^{2-}, the impact of suspension pH on the rate of flocculation is substantially reduced as shown in Fig. 5(b). As noted in the figures, the sulfate eliminates the positive ZP at lower pH and slightly increases the negative ZP at higher pH levels. The plots in Fig. 5(b) indicate that the kinetics at all pH levels converge on the best kinetics observed in Fig. 5(a). It is also noted that the negative ZP values resulting from the sulfate addition did not impair flocculation kinetics.

Plots of flocculation index as a function of flocculation time for 50 mg/ℓ kaolin are presented in Figs. 6(a) and (b) at pH 6.0 and
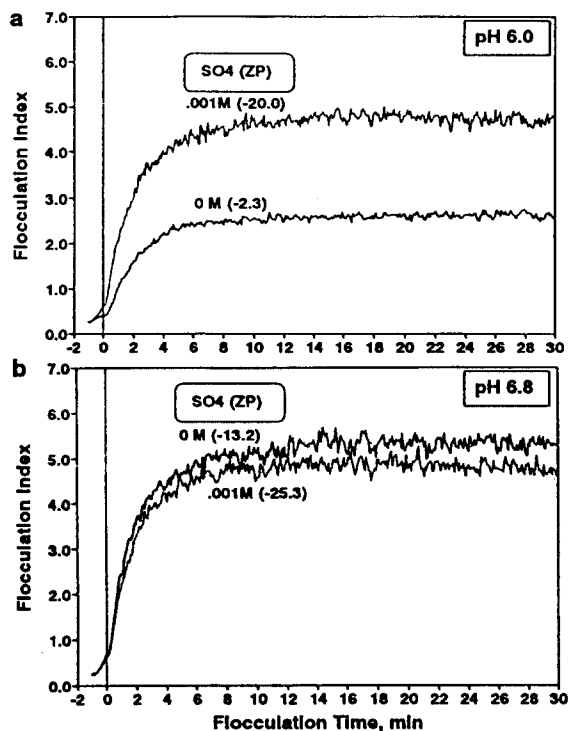

Fig. 7. Effect of sulfate on the rate of flocculation (clay: 100 mg/ℓ, dose: 5 mg/ℓ, 45 rpm): (a) pH 6.0; (b) pH 6.8.

6.8, respectively. The plots in Figs. 6(a) and (b) show, once again, the pronounced effect of sulfate on the rate of flocculation. At pH 6.0 in Fig. 6(a), the presence of 10^{-3} M sulfate greatly speeds up the rate of flocculation; whereas, at pH 6.8 in Fig. 6(b), it has little impact. It is noted in Fig. 6(a) that in the absence of sulfate, the flocculation index continued to increase over the full 30 min flocculation period. But, in the presence of sulfate, the flocculation index reached its maximum at about 7 min of flocculation time followed by a substantial decrease caused by the settling of bigger sized flocs during the flocculation process. Comparison of Figs. 6(a) and (b) shows that the flocculation kinetics under acidic pH conditions are greatly improved if an appropriate amount of sulfate ion is present.

The effect of sulfate on the flocculation rate

for higher clay concentration, 100 mg/ℓ at pH 6.0 and 6.8, is shown in Figs. 7(a) and (b), respectively. Again, these results clearly demonstrate the influence of pH on the degree of sulfate ion adsorption and the extent of changes in flocculation kinetics caused by sulfate ion. As shown in these figures, the dramatic impact of sulfate on the rate of flocculation observed at pH 6.0 is not evident at pH 6.8, even though it still affected surface charges of particles. Once again, the ZP values in Fig. 7(a) indicate that the flocculation kinetics is much faster at far more negative ZP values under the same applied coagulant dose. It is noteworthy in Fig. 7(a) that under high clay concentration, which may provide better particle collision frequency, the kinetics of flocculation was still improved by enhanced formation of precipitates attributed to the addition of sulfate, but not by favorable surface charges of particles (i.e., the ZP is close to zero without sulfate) for flocculation. This result suggests that the interaction between clay particles and iron precipitates appears important for rapid flocculation kinetics under all clay concentrations investigated herein.

Figs. 8(a) and (b) show the experimental results for flocculation kinetics under different sulfate concentration at pH 6.0 and 8.0 using coagulant dose 1 and 5 mg/ℓ, respectively. For these experimental results, the coagulation dosage of 1 mg/ℓ was applied at pH 6.0 to bring about optimum destabilization conditions under the A/D mechanism, i.e., ZP of particles was maintained close to zero. At pH 6.0, as shown in Fig. 8(a), the presence of sulfate ion greatly speeds up the rate of flocculation. The increase in sulfate concentration beyond $3 \times 10^{-4}M$ did not induce further improvement in flocculation kinetics, even though the concentration of sulfate ion affected the particle
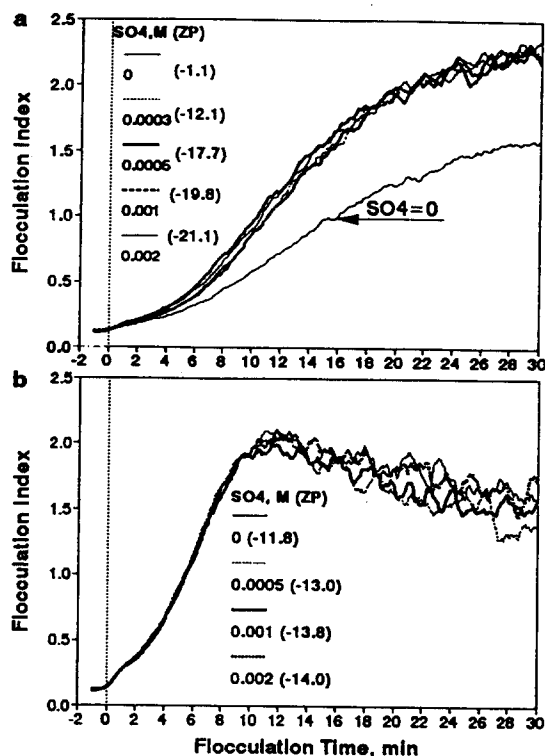

Fig. 8. Effect of sulfate concentration on the rate of flocculation (clay: 25 mg/ℓ, 45 rpm): (a) pH 6.0, dose 1 mg/ℓ; (b) pH 8.0, dose 5 mg/ℓ.

ZP values significantly. At pH 6.0, it is noteworthy that the flocculation kinetics is much better at higher negative charges caused by the addition of SO_4^{2-}. In contrast, as shown in Fig. 8(b), the flocculation data at pH 8.0 indicate that the influence of the sulfate on flocculation process is essentially negligible at higher pH. This observation may be explained by a decreased adsorption of sulfate ion on particle surfaces at higher pH. The ZP measurements confirmed this conclusion. Thus, the changes in surface charges of particles by addition of sulfate at pH 8.0 were minimal compared to those at pH 6.0. The decrease in the amount of adsorbed sulfate at the higher [OH⁻]/[Al] ratio was also noted by de Hek et al.(1978) and Dzombak and Morel(1990).

To evaluate the relative importance of the role of sulfate on flocculation, the net negative charge of 50 mg/ℓ kaolin was brought to near neutral ZP or slightly negative ZP using 2 mg/L of coagulant dose at pH 6.0 and 6.5 either with or without the addition of 10^{-3} M sulfate into the clay suspension. As shown in Fig. 9(a), at pH 6.0, the use of sulfate in the flocculation process results in more negative charges of particles but results in a faster rate of flocculation. This result, again, suggests that the kinetics of flocculation are improved most likely attributed to the addition of sulfate, but not by favorable surface charges for flocculation. However, the pronounced influence of sulfate on flocculation process disappeared with just 0.5 unit increase in pH, as evident in Fig. 9(b), even though the sulfate still significantly

affected surface charges of particles. It can be concluded that the the sulfate effect is likely to be insignificant at the higher pH levels typically used in sweep flocculation in which the interaction of clay particles with iron precipitates is dominant.
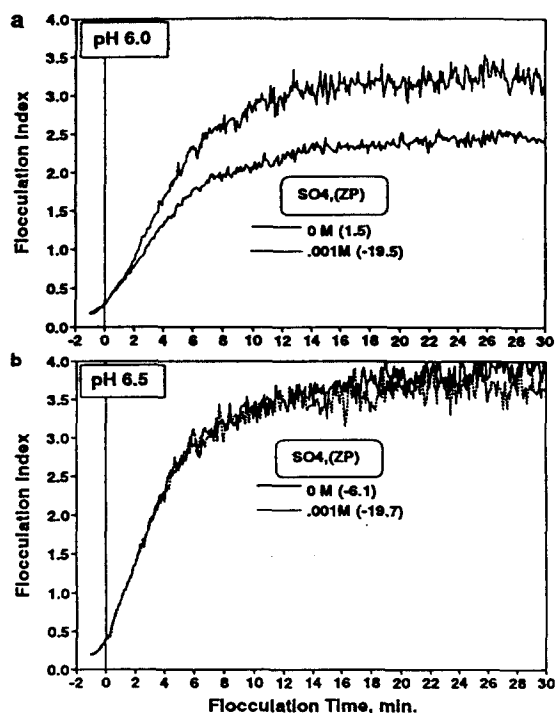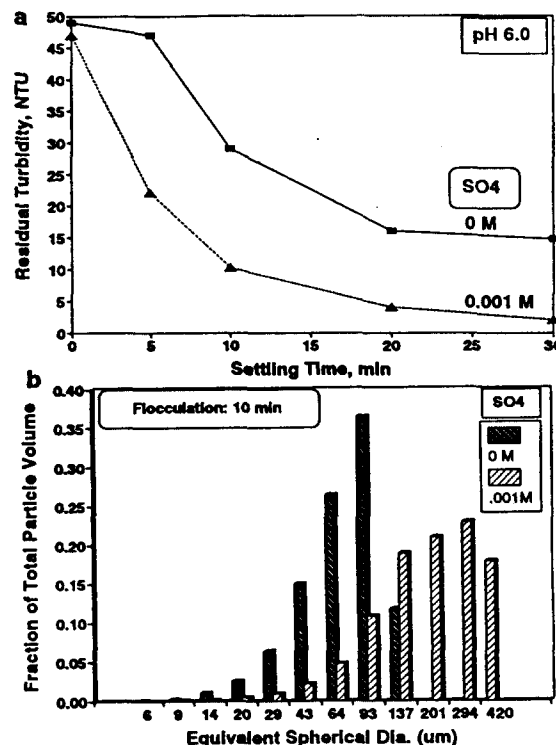

Fig. 9. Effect of sulfate (a) on the changes in residual turbidity; (b) on the floc volume distributions of 10 min flocculated suspensions (pH 6.0, clay: 50 mg/ℓ, dose: 2 mg/ℓ, 45 rpm).

Fig. 9. Effect of sulfate on the rate of flocculation (clay: 50 mg/ℓ, dose: 2 mg/ℓ, 45 rpm): (a) pH 6.0; (b) pH 6.5.

Figs. 10(a) and (b) present the effect of sulfate addition on supernatant turbidity during sedimentation after 30 min of flocculation and on the floc volume distributions after 10 min of flocculation. These experimental results were obtained during the same experiment presented in Fig. 9(a) to illustrate the clear difference of flocculation kinetics and the reliability of

flocculation index used in this study. The floc volume distribution were attained from the AIA using a low microscope magnification (36X). Fig. 10(a) indicates that the flocs formed under the conditions of better flocculation kinetics settled much faster, and resulted in lower residual turbidity. This result shows that the sulfate ion markedly improves the performance of flocculation under these experimental conditions. The floc volume distributions at the different experiment conditions is shown in Fig. 10(b). In general, Fig. 10(b) shows that the presence of sulfate during the progress of flocculation resulted in a decrease in the volume of smaller particles and an increase in the volume of larger particles. This shift in the floc volume distribution indicates the substantial changes in floc growth caused by the presence of sulfate. The results shown in Figs. 10(a) and (b) again demonstrate the reliable use of flocculation index as a means to study flocculation kinetics.

5. CONCLUSIONS

1. There was marked increases in the speed of flocculation with pH increase in experiments between pH 6.0 and 8.0 in the absence of sulfate ion.

2. Sulfate ion added to the kaolin suspension played an important role in the flocculation process, not only improving flocculation kinetics at more acidic pH levels but also changing surface charge of particles.

3. Considering the entire range of clay concentrations investigated, the impact of sulfate on flocculation kinetics and reduction of ZP of particles were more dramatically beneficial in the more acidic pH range and at the lower clay

concentrations.

4. The kinetics of flocculation were improved mainly by the enhanced rate and extent of Fe (III) precipitation attributed to the addition of sulfate, and thereby, better interparticle collision frequency, but little by the charge reductions resulting from the sulfate addition.

5. The increase in sulfate concentration beyond 3×10^{-4}M (up to 2×10^{-3}M) did not induce further improvement in flocculation kinetics, although the higher concentrations of sulfate ion substantially increased the negative ZP value of particles.

ACKNOWLEDGMENTS

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상수처리시 Fe(Ⅲ) 응집제를 이용한 응집동력학에 관한 연구 : 황산이온의 영향

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(1994년 11월 17일 접수)

상수처리시 응집동역학에 관한 연구를 수행하기 위하여 Fe(NO₃)₃·9H₂O을 응집제로 사용하여 Kaolin현탁액을 응집시키는 실험을 행하였다. 응집 동력학은 응집과정중 입자크기의 증가율을 측정하는 것으로서 응집의 mechanism에 대한 이해와 응집시 사용되는 최적 응집제의 량과 종류 그리고 최적 pH를 선정하는데 효과적으로 사용될 수 있다. 본 실험에서는 기본적 응집 동력학에 대한 연구를 토대로 수중의 황산이온이 응집동력학에 미치는 영향에 대하여 고찰하였다.

본 실험에 사용된 각 실험조건들은 일련의 Jar Tests를 통하여 선정되었으며 상수처리시 제기될 수 있는 광범위한 탁도와 pH를 포함하고자 하였다. 본 연구에서는 응집 동력학을 측정하기 위하여 '자동영상분석계(AIA)' 를 이용하여 응집과정중의 입자의 크기분포와 '광산란분석계(PDA)' 를 이용한 응집과정중의 탁도변화에 관한 자료를 상호 비교하였다.

본 실험에서 도출된 결과에 의하면, Kaolin현탁물에 가해진 10^{-3}M의 황산이온은 응집과정에 상당히 큰 영향을 초래하였으며 응집된 입자표면의 전위(zeta potential)변화에 큰 영향을 주는 것으로 밝혀졌다. 응집과정에 가해진 황산이온은 약산성(≤ pH 6.8)에서의 응집과정을 크게 향상시키는 것으로 나타났다. 응집 동력학의 효율증진은 황산이온이 첨가됨으로 인하여 Fe(Ⅲ) 침전물 형성율의 촉진에 기인한 수중입자의 충돌빈도의 증가에 의한 것으로 규명 되어졌다.