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Characteristics of flocs formed by polyaluminum chloride during flocculation after floc recycling and breakage

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ABSTRACT

The formation and the size distribution of the flocs were studied using a floc imaging system, and the relationship between breakage/regrowth of flocs from coagulation and flocculation with polyaluminum chloride and flocculation by floc recycling was investigated, from which it was found that both the processes exhibited similar performance in adsorption/charge neutralization zones; however, there were significant differences in restabilization and sweep coagulation zones. In adsorption/charge neutralization zones, the size distribution of flocs shifted toward flocs growth during the two processes. At the coagulant dosage of less than 0.010 mmol L^{-1}, the residual turbidity and the particle number frequency of small flocs decreased, and both increased at the coagulant dosage of more than 0.010 mmol L^{-1}. In restabilization and sweep coagulation zones, the floc size distribution shifted toward floc withering. Residual turbidity deterioration occurred after the breakage/regrowth of flocs, which was reduced significantly in the sweep coagulation zone than in restabilization zone. For floc recycling, the residual turbidity showed a decreasing trend, and the optimization effect was obtained at the coagulant dosage of more than 0.080 mmol L^{-1}. Moreover, for the entire coagulation zone, the range of flocs distribution reduced during breakage/regrowth, while it expanded during floc recycling.

Keywords: Breakage/regrowth; Floc recycling; Adsorption/charge neutralization; Restabilization; Sweep coagulation

1. Introduction

Coagulation/flocculation is one of the most widely applied solid–liquid separation techniques in water treatment works. Colloidal particles generally aggregate to flocs by means of coagulant dosing with substances such as ferric and aluminum salts. The coagulant dosage of coagulant influences cationic species hydrolyzed in water which leads to the flocculation mechanism. Once the coagulant dose is applied, the cationic species attach to the colloidal surfaces, resulting in the increase in charge potential. At low dosages, charge neutralization is a possible mechanism for colloids aggregation due to the breakage of the potential barrier, and the charge potential approaches to 0 mV [1]. If it exceeds the isoelectric point, the electrical potential changes to positive

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hindering the colloid collision. This is called restabilization [2]. When the coagulant dosage is high enough to create amounts of amorphous metal hydroxide precipitate that could produce the enmeshment effect on colloids, the flocs are formed mainly by sweep flocculation [3].

Recirculation of chemical sludge to facilitate coagulation and flocculation was first reported as a water treatment technique in 1873 [4], and was generally used for water softening [5], domestic and industrial wastewater treatment [6,7], and drinking water treatment [8]. However, the feasibility of its application was debated greatly, and the studies on the floc recycling technique for water treatment were gradually tappered off for historical reasons and the immaturity of detection methods.

Recently, numerous studies have focused on the recycling of flocculated sludge, indicating a reduction in coagulation dosage [9–13]. Moreover, some research studies have demonstrated the mechanisms of breakage/regrowth of flocs. Yu et al. illustrated that residual turbidity was reduced significantly by floc breakage and regrowth when the flocculation mechanism was dominated by adsorption/charge neutralization [14].

The key step in sludge recycling methods involved reimporting the flocs from sediment to raw water. This raises the question: does sludge recycling with flocs formed by different flocculation mechanisms significantly influence water treatment mechanisms? We assumed that there is a relationship between flocs breakage/regrowth and floc recycling methods. However, few studies address the recycling of broken flocs.

Therefore, the objective of this study is to systematically examine the process of flocs breakage and regrowth, along with flocs recirculation. A comprehensive understanding of the two processes would facilitate the use of flocculated sludge recycling in engineering practice.

2. Materials and methods

2.1. Materials

The test was conducted using a floc imaging system consisting of a rectangular reactor of 20 cm × 20 cm × 30 cm with an effective volume of 12 L, a stirring propeller with a 100 mm × 80 mm × 2 mm sized blade (IKA, Eurostar100, Germany), and a high-resolution camera with 14 million pixels and a high-resolution lens of 10 million pixels. The visual capacity of the imaging system is 20 μm in theory. Fig. 1 shows a schematic diagram of the experimental system.

The Kaolin stock suspension was prepared by dispersing kaolin stock (100 g) into deionized water (1 L) and stirring the mix for 2 h at high speed (1,000 r min^{-1}). To maximize dispersion, the pH of the suspension was set at 8.5 by adding NaOH of 0.5 mol L^{-1} solution. The supernatant was used as the kaolin stock suspension after resting overnight. The test water used in the experiment was prepared by diluting stock suspension with the tap water (temperature of 22℃, pH of 8.3, and alkalinity of 8.4 ~ 15.9 mg L^{-1} as $CaCO_3$). The alkalinity was adjusted by adding $NaHCO_3$ solution of 0.5 mmol L^{-1}. The pH was adjusted to 7.90 ± 0.05 by adding HCl of 2 mol L^{-1} or NaOH solution. The water samples employed in the coagulation experiments had a turbidity between 40 and 43 NTU (Hach, 2100Q, USA).

Commercially available polyaluminum chloride (PACl) with 30% Al_2O_3 content and 70–75% basicity was used to prepare the coagulant solution of 0.24 mol L^{-1} as Al.

2.2. Jar test and image capture

The reactor was filled with 12 L of test water. Image acquisition during propeller stirring consisted of two photographs min^{-1} for breaking and one photograph min^{-1} for slow stirring.

For the floc breakage and regrowth experiment, the suspension was rapidly stirred for 1 min at 300 r min^{-1} and then slowly stirred for 15 min at 70 r min^{-1}. After allowing the suspension to stand for 10 min, the turbidities of the water samples were measured. We followed this step by conducting breaking actions for 1 min at 300 r min^{-1} and then slow stirring for regrowth at 70 r min^{-1} for 15 min. The contents were left to settle for 10 min and then water sample turbidities were measured again.

The floc recycling is the process of returning the floc sediments into raw water and conducting a new flocculation cycle after rapid mixing. The flocculated sludge employed for the floc recycling experiment was prepared using a parallel experiment to that described above, except breakage and regrowth step was not conducted after stirring for 1 h (turbidity was tested at 10 min). Then, the settled flocculated sludge was poured into the test water. After rapid stirring for 1 min, the same dosage of coagulant was dosed. Following another 1 min of rapid stirring, suspension was subjected to 15 min of slow stirring, followed by settlement for 10 min. Water sample turbidities were then measured again.

Zeta potential (Brookhaven, Zetaplus, Germany) was monitored 1 min after the coagulant was dosed.

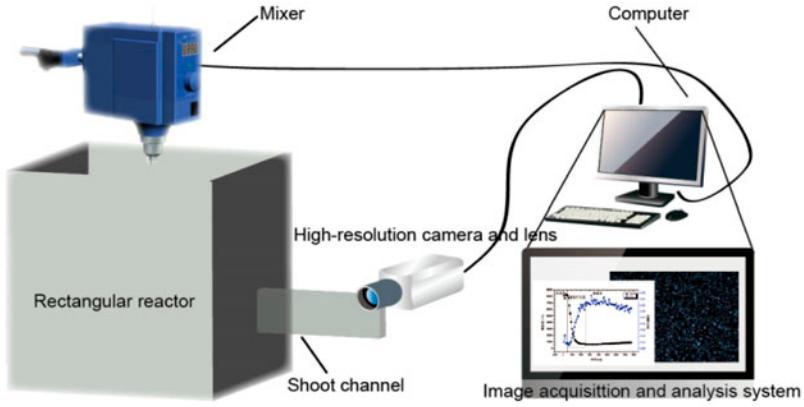

Fig. 1. Schematic diagram of the floc imaging system.

All the water samples were taken from 3 cm below the water surface.

2.3. Measurement and analysis

The average size and the size distribution of flocs were obtained by means of background subtraction and binarization using image-pro plus software after image acquisition. Flocs characteristics were investigated by breakage (B), recovery (R), and strength (S) factors which are calculated as follows [15–18]:

$$B = \frac{d_a - d_b}{d_a} \times 100\% \quad (1)$$

$$R = \frac{d_c - d_b}{d_a - d_b} \times 100\% \quad (2)$$

$$S = \frac{d_b}{d_a} \times 100\% \quad (3)$$

where d_a is the steady-state floc size before breakage, d_b is the floc size after breakage, and d_c is the steady-state floc size after regrowth period.

3. Results

3.1. Effect of coagulant dosage on zeta potential and residual turbidity change

Zeta potential is a physical property exhibited by a charged floc at the shear plane. Its value indicates the dominant flocculation mechanism at different coagulant dosages. Fig. 2 shows the variation of zeta potential of flocs with different coagulant dosages. The absolute zeta potential decreased rapidly with increasing coagulant dosage. At a PACl dosage of

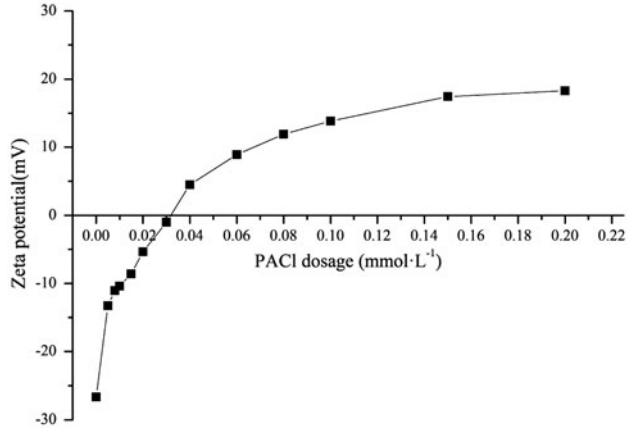

Fig. 2. Effect of coagulant dosage on zeta potential of flocs.

0.030 mmol L^{-1} (as Al), the flocs approached isoelectrical point. At PACl dosage of more than 0.030 mmol L^{-1}, the flocs potential shifted from negative to positive, and increased continuously from there. This could reduce the outlet quality.

When considering residual turbidity variability before flocs breakage (Fig. 3(a)) and without floc recycling (Fig. 4(b)) combined with zeta potential variability (Fig. 2), it is clearly that, at the coagulant dosage less than 0.030 mmol L^{-1}, the residual turbidity decreased rapidly. The zones with negative zeta potential indicated that adsorption/charge neutralization dominated the flocculant mechanism. When the coagulant was more than 0.030 mmol L^{-1}, the increase in zeta potential shifted the flocculation to restabilization zones with positive zeta potential. The residual turbidity increased gradually, finally reaching the maximum lever at a coagulant dosage of 0.100 mmol L^{-1}. According to the reduction of residual turbidity at coagulant dosage of

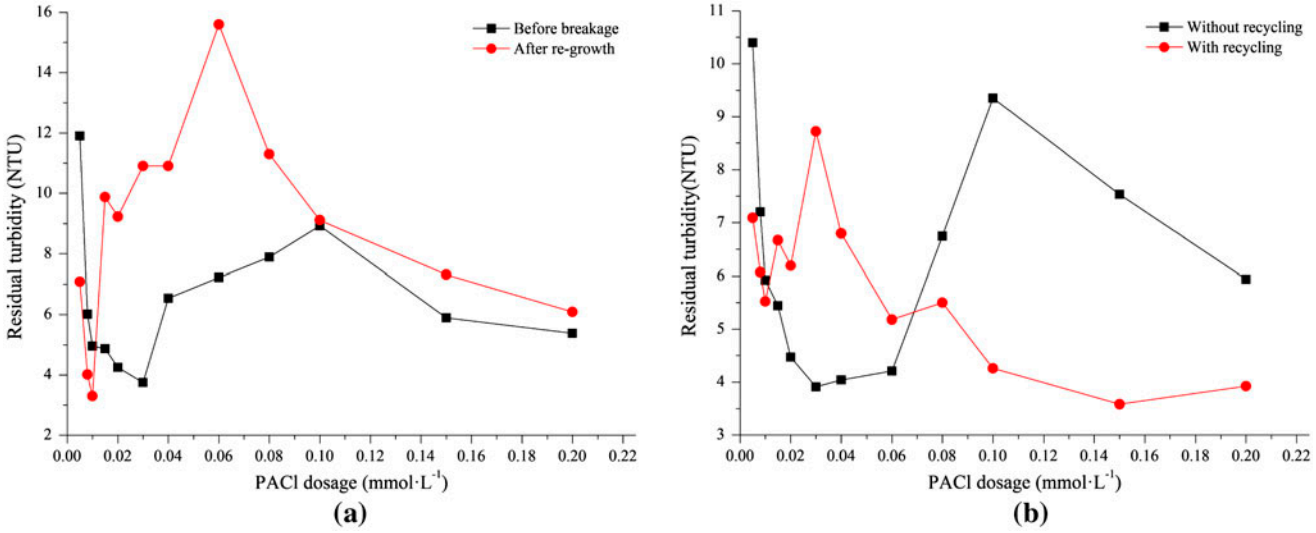

Fig. 3. Effect of coagulant dosages (0.005–0.200 mmol L^{-1}) on residual turbidity (a) before flocs breakage and after flocs regrowth and (b) with and without floc recycling. At the coagulant dosage less than 0.010 mmol L^{-1}, the breakage and regrowth of flocs achieved optimal effect. At the coagulant dosage less than 0.010 mmol L^{-1} or more than 0.080 mmol L^{-1}, the floc recycling achieved optimal effects.

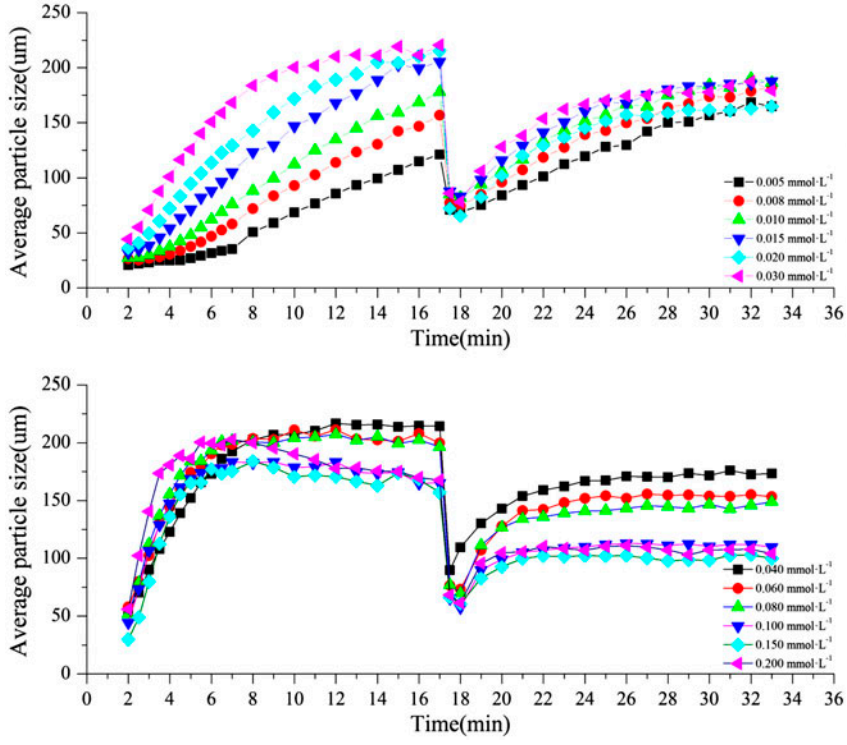

Fig. 4. Breakage and regrowth of flocs at different coagulant dosages from 0.005 to 0.200 mmol L^{-1}.

more than 0.100 mmol L^{-1}, flocculation enter the sweep coagulation zones.

The differences in the residual turbidity before flocs breakage and after flocs regrowth is shown in Fig. 3(a). The results indicated that in the adsorption/

charge neutralization zones at a PACl dosage less than 0.010 mmol L^{-1}, the residual turbidity decreased with floc regrowth. No optimization effects were observed in the residual turbidity as zeta potential approached zero at PACl dosage more than 0.010 mmol L^{-1}. In restabilization and sweep coagulation zones, instead of optimization, residual turbidity deterioration occurred. The turbidity in the floc regrowth process increased, reaching the maximum at a dosage of 0.060 mmol L^{-1}, and then decreasing continuously. Therefore, the difference in residual turbidity before breakage and after floc regrowth was much lesser in sweep coagulation zones compared to restabilization zones. This indicates that the sweep coagulation had minor effects after floc breakage and regrowth.

The difference in the residual turbidity with and without floc recycling is shown in Fig. 3(b). In the adsorption/charge neutralization zones, the trend was similar to that in the breakage and regrowth process. Floc recycling lowered the residual turbidity at a PACl dosage less than 0.010 mmol L^{-1}, while a deterioration effect was observed at a dosage of more than 0.010 mmol L^{-1}. Compared to the breakage and regrowth process, the floc recycling lowered the residual turbidity in the restabilization and sweep coagulation zones. Moreover, the optimization effect occurred only during floc recycling when the sweep coagulation zones were not reached at a PACl dosage less than 0.080 mmol L^{-1}. In the sweep coagulation zones, significant reduction in the residual turbidity was observed.

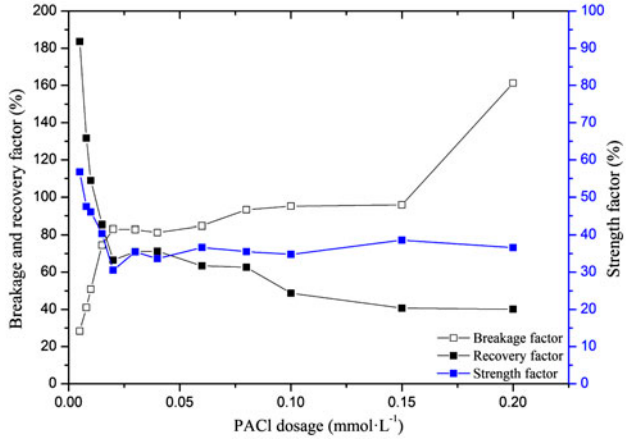

Fig. 5. Effect of coagulant dosages from 0.005 to 0.200 mmol L^{-1} on the floc factors. At a coagulant dosage less than 0.010 mmol L^{-1}, the flocs were completely reversible.

3.2. Breakage/regrowth of flocs and floc recycling

Floc breakage and regrowth of flocs at different PACl dosages is shown in Fig. 4; Fig. 5 shows the corresponding breakage, recovery, and strength factors. The results reveal that floc growth rate was accelerated with the increase in coagulant dosage. PACl flocs were reversible after breakage. An increase of breakage factor led to a decrease of the recovery factor, and high strength factors were obtained when the coagulant dosage was in the adsorption/charge neutralization zones. The complete reversibility of flocs was observed with residual turbidity optimization in breakage and regrowth, as well as in floc recycling. The flocs recovery factor was 108.92% at a PACl dosage of 0.010 mmol L^{-1}. However, the flocs were partially reversible and the complete reversibility was lost at a dosage exceeding 0.010 mmol L^{-1}, thus increasing the flocculation turbidity. In the restabilization zones, the zeta potential of flocs shifted from negative to positive, the repulsion between flocs increased, and flocs relying on precipitate formation by amorphous metal hydroxide or Van Der Waals force before the complete coverage of colloids by precipitate became loose and difficult to settle down. The broken flocs were not able to obtain the same particle size as before breakage due to the repulsion force between the tiny particles deposited by precipitate. Therefore, the residual turbidity increased and the flocs had poor reversibility in restabilization zones. At higher coagulant dosages (0.100, 0.150, and 0.200 mmol L^{-1}), the sweep coagulant dominated the flocculation mechanism, and the long resident time in flocculation led to the reduction in floc size. Although floc sizes were large, the breakage factor was more than 90% and the flocs were easy to break.

The growth of flocs with and without recycling is shown in Fig. 6. The average floc size increased after floc recycling regardless of the coagulation mechanisms. Moreover, the flocculation rate increased due to the rise of floc concentration in the adsorption/charge neutralization and restabilization zones. The floc particles consisted of the initial particles in raw water and broken flocs in the reactor because the recycling flocs returned to the raw water. The rapid mixing process did not completely break the recycling flocs into the same size as initial particle size in raw water. Thus, the recycling enhanced the flocculation and changed the ways flocs form.

3.3. Flocs size distribution

The flocculation–sedimentation was significantly affected by floc size distribution. Usually, small flocs

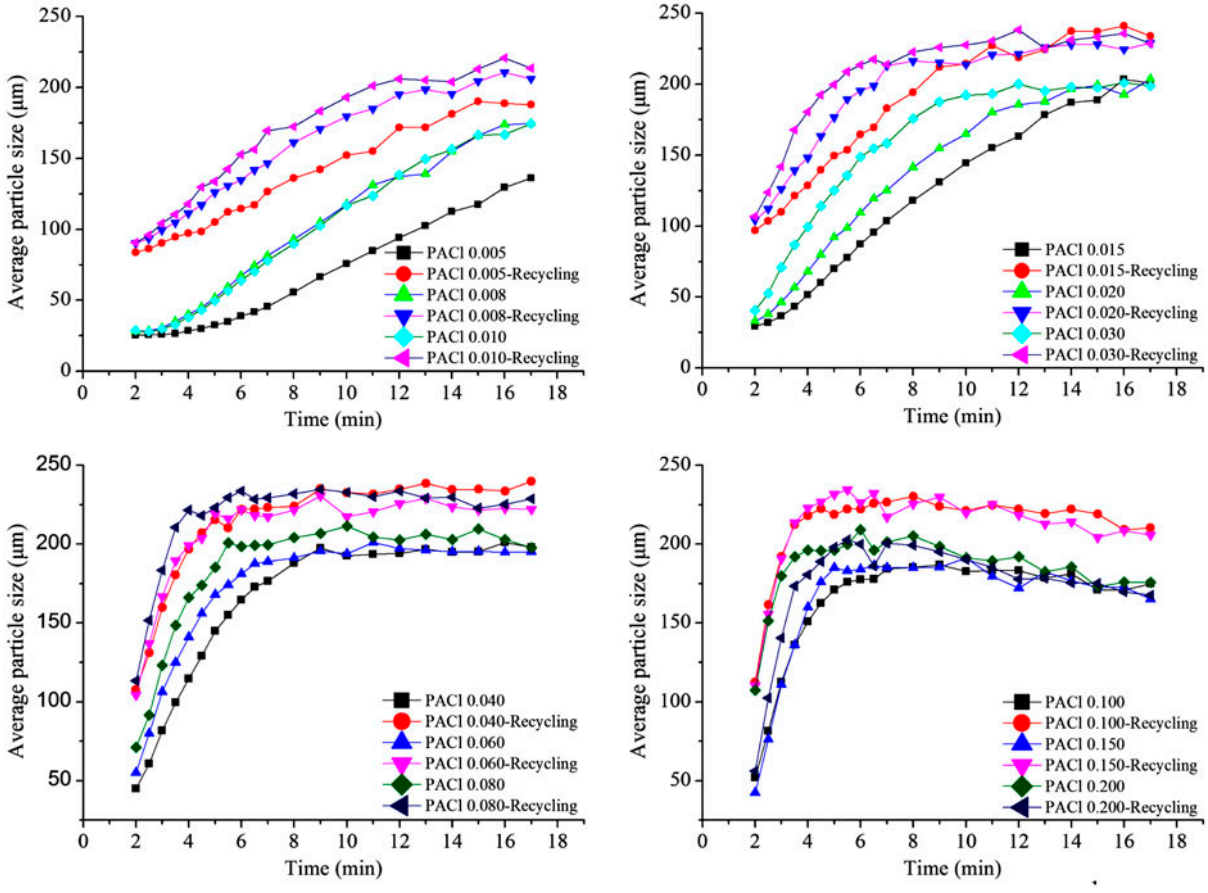

Fig. 6. Effect of coagulant dosages from 0.005 to 0.200 mmol L^{-1} on the growth of flocs with and without floc recycling. Average floc size increased with floc recycling.

had poor removal efficiency. For the flocs with similar density, the sedimentation was slower for the smaller flocs [19,20]. Therefore, large amounts of initial particles without flocculation and small flocs were the main reasons for the high effluent turbidity.

Fig. 7 compares the floc size distribution before breakage and after regrowth. It shows that the range of floc size distribution was diminished after breakage and regrowth of flocs. In adsorption/charge neutralization zones, the size distribution of flocs shifted toward floc growth, thus reducing the particle number frequency of small flocs ranging from 0 to 50 μm. It is these sizes that are capable of optimizing the residual turbidity; big flocs of 100–400 μm dominated the particle composition. In the zone, whose potential was approaching zero (0.015–0.030 mmol L^{-1}), the total particle number frequency of flocs increased dramatically after breakage. The particle number frequency of small flocs was more than that before breakage, resulting in poorer performance in the residual turbidity during regrowth. In restabilization and sweep coagulation zones, the size distribution of flocs shifted toward floc withering.

The comparison of floc size distribution with and without recycling was shown in Fig. 8. The charts demonstrate a wider size distribution of flocs after recycling. A significant increase in particle number frequency of flocs was not observed under different flocculation mechanisms. In adsorption/charge neutralization zones, the particle number frequency of large flocs (more than 100 μm) increased after recycling. Similar to the process of floc breakage/regrowth, the small flocs in the zone, where residual turbidity could be optimized, decreased as well. Conversely, the small flocs in the zone whose potential approached zero increased. In restabilization and sweep coagulation zones, although the floc size distribution was enlarged, and many big flocs (more than 400 μm) were formed, the dominant flocs did not increase and the particle number frequency of small flocs increased. This did not increase the residual turbidity. Instead, the residual turbidity in two zones continuously decreased after

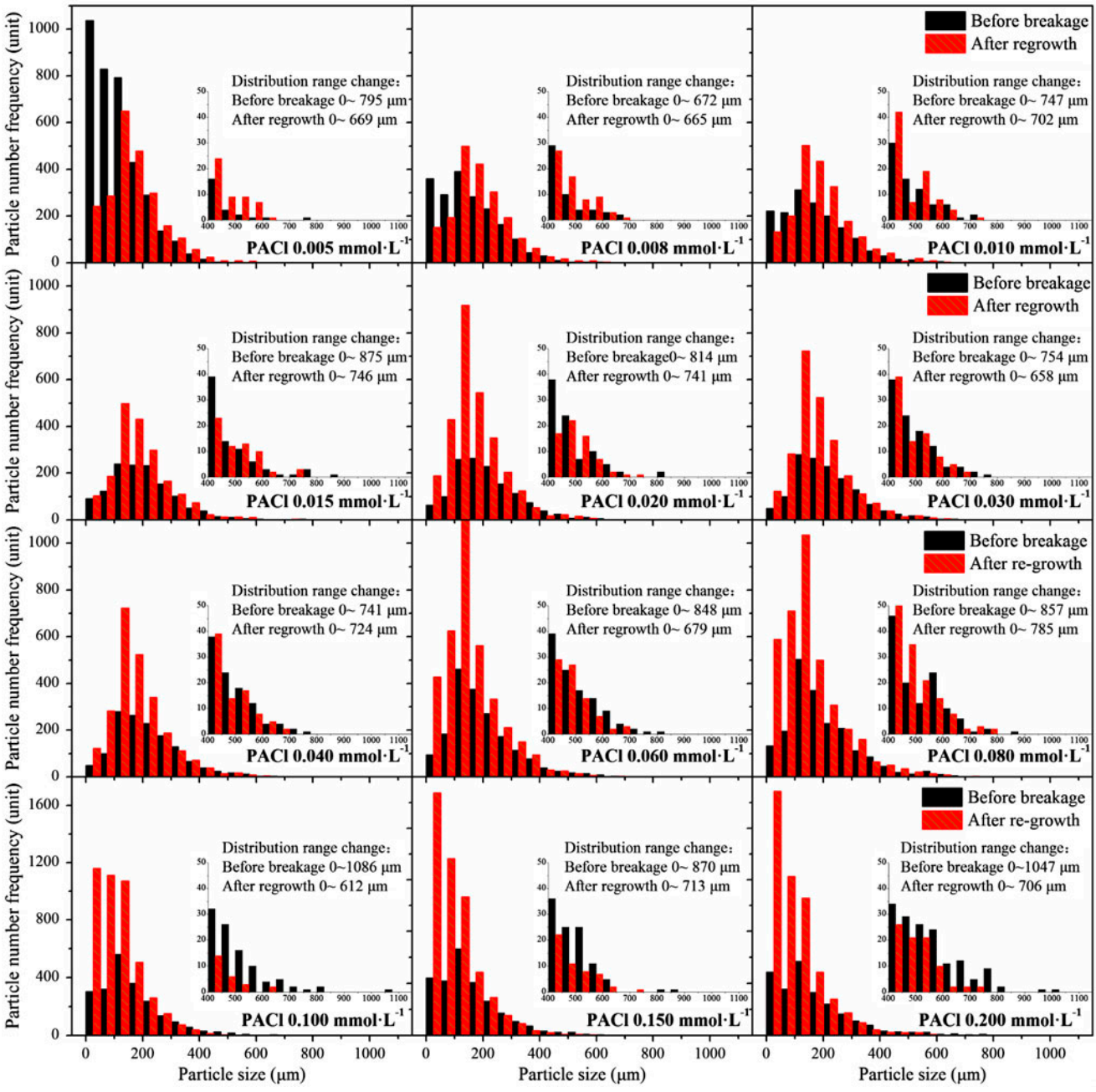

Fig. 7. The size distribution of flocs before breakage and after regrowth at different coagulant dosages from 0.005 to 0.200 mmol L^{-1}. At the coagulant dosage less than 0.010 mmol L^{-1}, the particle number frequency of small flocs decreased during regrowth.

recycling, and the optimization effects were enhanced in sweep coagulation zones.

4. Discussion

The flocs formed by different flocculation mechanisms showed special patterns during recycling. The results indicated the similarity and differences between the floc breakage and regrowth process, and the floc recycling process.

Similar results were obtained for both the floc breakage/regrowth process and floc recycling process in adsorption/charge neutralization zones. The optimization of the residual turbidity occurred at low

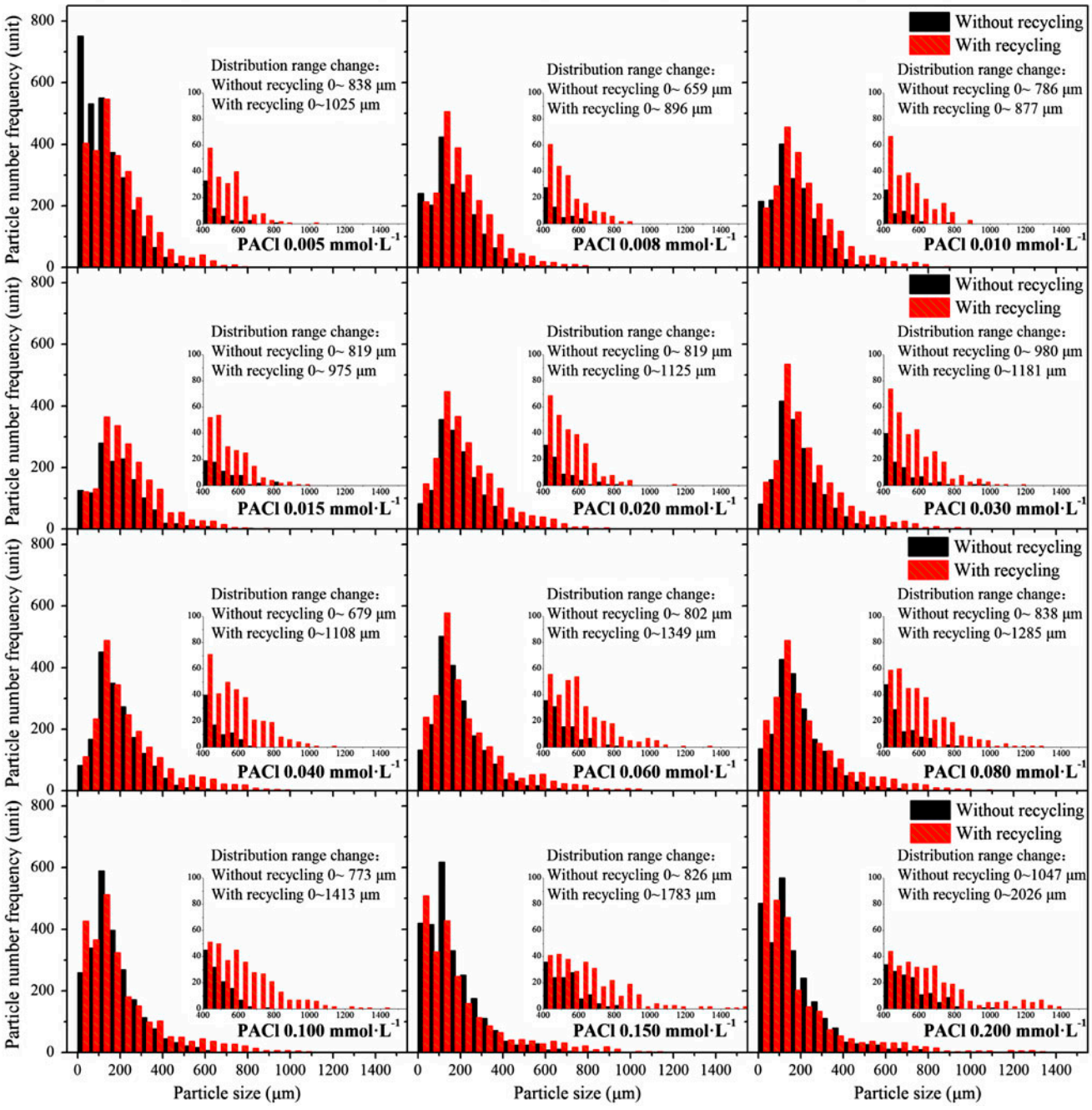

Fig. 8. The size distribution of flocs with and without recycling at different coagulant dosages of 0.005 ~ 0.200 mmol L^{-1}. At a coagulant dosage less than 0.010 mmol L^{-1}, the particle number frequency of small flocs decreased with recycling.

coagulant dosage, the number of small flocs decreased, and the size distribution of flocs shifted significantly toward flocs growth. Additionally, the flocs had complete reversibility under the above-mentioned conditions, and the particle number frequency of small particles increased and approached the zero potential, thus contributing to an increase in the residual turbidity. Yu et al. demonstrated the similar changes in the size distribution of flocs and turbidity [14]. However, the residual turbidity was not optimized in the entire adsorption/charge neutralization zones. This may be due to the different test reagent used. The Al species classified as Al_a (monomeric species), Al_b (medium polymer species) and Al_c (colloidal

or solid species), defined by ferron-complexation timed spectrophotometric method are relatively stable in a PACl solution, but the different proportions of components, especially Al_c, have deep effects on turbidity removal [21].

In the restabilization and sweep coagulation zones, the floc size distribution shifted toward floc withering. The same phenomenon was observed during floc recycling. Nevertheless, several differences were found between the two processes. First, the deterioration of the residual turbidity occurred in restabilization zones, but mitigated in sweep coagulation zones. In particular, the residual turbidity showed a declining trend in the latter zones, and its optimization effects were obtained during recycling before reaching sweep coagulation zones. Second, the range of size distribution of flocs was diminished after floc breakage and regrowth, and expanded after recycling. Therefore, the floc operation methods led to the differences between the two processes.

Floc formation mechanisms of the two processes with different flocculation mechanisms were investigated to explain their differences and similarities. The position exhibiting adsorption ability on the particle surface was named as "active site." The positive charge was able to generate active sites in the negatively charged system and vice versa. The adsorption/charge neutralization was the dominant flocculation mechanism, while the broken flocs were reversible at a low coagulant dosage (Fig. 9). The breakage and regrowth of flocs utilized the active sites that were not used before the process, thus reducing the number of small flocs, and increasing the average particle size of the flocs group. The recycling of the flocs back into the raw water led to the mixing of the broken flocs with the initial particles, partly attaching the initial particles to the surface of the broken flocs. The addition of the coagulant at this point resulted in new active sites on the surface of the broken flocs, leading to adsorption of the initial particles. Flocs were also formed by the aggregation of initial particles and small flocs with each other. In addition, the concentration of the active sites should be controlled within a certain range, because exceeding the specific concentration, i.e. approaching the zero potential, decreased the collision efficiency between the particles. This led to the ineffective flocculation of the smaller flocs and an increase in residual turbidity.

In the restabilization and sweep coagulation zones, the active sites in the adsorption/charge neutralization zones impeded the flocculation of the entire system, because the flocs were positively charged and the particles inside the flocs were completely charged after breakage. This resulted in difficult floc regrowth, making the number of small flocs and the residual turbidity increase and effluent water quality worse. For floc recycling, the rapid stirring offered complete contact between broken flocs and the initial negatively charged raw water colloids, thus adhering negatively charged particles to the broken flocs. After rapid stirring to break flocs, adding coagulant increased the positive charge on the negatively charged particles, causing adherence to the broken flocs. Although particle size increased, it impeded the flocculation.

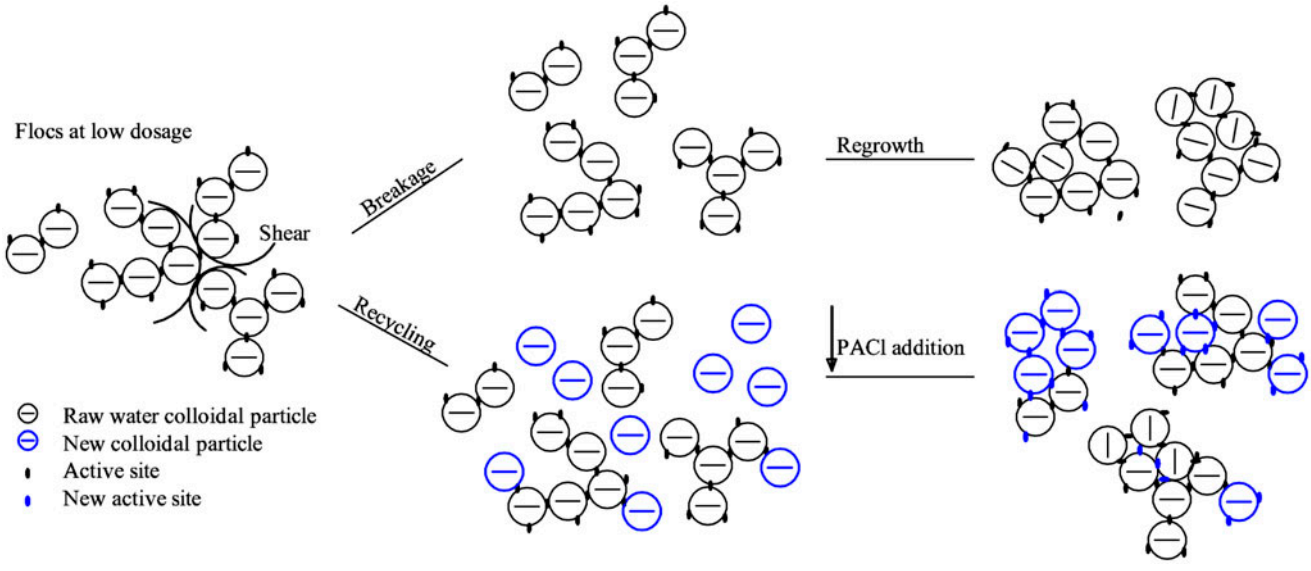

Fig. 9. The breakage/regrowth and recycling of flocs at low coagulant dosage.

However, the further increase in coagulant dosage resulted in the increase in number of flocs, increasing the collision probability and decreasing the residual turbidity. When the coagulant dosage was high enough to create sweep flocculation, the effluent water quality was improved.

5. Conclusions

The main conclusions of this study are described as follows:

(1) During the flocs breakage and regrowth processes, flocs were completely reversible at coagulant dosage less than 0.010 mmol L^{-1}. The flocs were significantly irreversible at dosage of more than 0.010 mmol L^{-1}, thus resulting in a decrease in recovery factor and an increase in breakage factor. Regardless of the characteristic of reversibility of the broken flocs, the average size of flocs after recycling was larger than those without recycling.

(2) In adsorption/charge neutralization zones, the number of small flocs decreased in the two processes, and the optimization effects in the residual turbidity were achieved at coagulant dosage of less than 0.010 mmol L^{-1}. In contrast, the number of small flocs increased in the two processes and the residual turbidity deterioration occurred at coagulant dosage of more than 0.010 mmol L^{-1}.

(3) In restabilization and sweep coagulation zones, the particle number frequency of flocs increased dramatically, and the residual turbidity deterioration occurred during breakage and regrowth process. In particular, at coagulant dosage of 0.060 mmol L^{-1}, the residual turbidity achieved the maximum. In sweep coagulant zones, the deterioration effects on the residual turbidity were reduced. The floc recycling effectively lowered the residual turbidity, and showed optimization effects at the coagulant dosage of more than 0.080 mmol L^{-1}.The optimization effects were remarkably enhanced in sweep coagulation zones.

(4) The breakage and regrowth process of flocs diminished the size distribution range, while the recycling of flocs shifted toward flocs growth during the two processes. Besides, in stabilization and sweep coagulation zones, the size distribution of flocs shifted toward flocs withering, and the particle number frequency of small flocs increased during the two processes.

This study demonstrates the relationship between the breakage/regrowth and flocculation of floc recycling. The results of this paper suggest that a low dosage of coagulant in floc recycling could facilitate the improvement of flocculation sludge reuse in water treatment works. To confirm this method's applicability, more studies are needed to assess floc properties under different flocculation mechanism.

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