Floc morphology evolution in a continuous-flow flocculation and sedimentation reactor

Analysis of floc morphology in a continuous-flow flocculation and sedimentation reactor

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Abstract

The floc morphology was investigated in a continuous-flow reactor, in order to understand the evolution of flocs in practical flocculation and sedimentation processes in water utilities. Kaolin-humic acid suspension was used as the test water, and polyaluminum chloride was chosen as the coagulant. An *in-situ* recognition system was applied to analyze the floc size, boundary fractal dimension, and eccentricity ratios. Particle numbers and turbidity were also determined in the sedimentation stage. At a coagulant dose of 1 mg/L as Al, the average floc size increased from 62 to 78 μm and the boundary fractal dimension was around 1.14, suggesting that flocs were compact and continuously grew during the entire flocculation process. However, with the dose increased to 5 mg/L, the average floc size decreased and stabilized at around 65 μm , with the fractal dimension of 1.20. It can be concluded that the excess coagulant doses resulted in the formation of chain-shaped, lower density, and more branched structure flocs, thereby restricting flocs' further growth in the subsequent flocculation. Floc morphology analysis suggested that charge neutralization dominated in the initial flocculation stage, then the bridge and sweep mechanisms were dominant in the subsequent flocculation. In addition, compared with the traditional inclined plate settler, a novel V-shaped plate settler introduced in this study had an advantage in small size floc (less

than 5 μm) removal. The V-shaped region could promote aggregate restructuring and re-flocculation; therefore, the V-shaped plate settler provides an alternative method for sedimentation.

Keywords: Flocculation, sedimentation, continuous-flow reactor, in-suit recognition system, V-shaped plate settler tank

Introduction

Flocculation and sedimentation are the most common processes for particle and colloid removal, which have been considered to be the first step in drinking water treatments (Jarvis et al., 2005; Wang et al., 2011). In previous studies, flocculation conditions (such as coagulant types, coagulant doses, mixing intensity, and pH value) have been extensively investigated in order to improve flocculation efficiency (Shin and Sobsey, 2015; Zhao et al., 2013; Hua et al., 2014)

It should be noted that floc characteristics also play important roles in flocculation. Flocs undergo the growth stage, breakage stage, restructuring stage, and finally achieve stability with a balance of aggregation and breakage (Gmachowski, 2009; Nan et al., 2011; Nasser, 2014). Floc characteristics (such as floc size, structure, and strength) change continuously in these flocculation stages. Previous studies have confirmed that floc structures have a significant impact on flocculation efficiency (Nan et al., 2009; Vandamme et al., 2014). For instance, aggregates that are highly porous and loose are susceptible to breakage and difficult to settle, and vice versa (Kim et al., 2013). Furthermore, analyses of floc characteristics have also been applied to investigate the effect of floc breakage in the coagulation/ultrafiltration process (Yao et al., 2015), as well as the influence of coagulant dose on the process (Yao et al., 2015).

Numerous flocculation studies have been conducted in jar tests (Li et al., 2014; Yu et al.,

2011; Yang et al., 2014). Although jar tests can predict the behavior of flocculation processes in many cases, the jar-test method can merely reflect the final status of flocs. It has been confirmed that the hydraulic conditions impose an important influence on floc characteristics in flocculation and further affect the purification efficiency (He et al., 2012). However, the hydraulic conditions of flocculation in practical water utilities cannot be effectively simulated in jar tests. In addition, flocs generated during flocculation can directly impact sedimentation efficiency (Becker et al., 2009). Therefore, it is more feasible to apply a continuous-flow reactor for an integrated understanding of the floc evolution in flocculation and sedimentation process.

Based on the fact that flocculation and sedimentation are complicated and chaotic processes, a continuous-flow reactor is necessary to simulate the practical flocculation and sedimentation process in water utilities. However, few studies have been conducted in such reactors. To the best of our knowledge, the evolution of floc characteristics in a continuous-flow flocculation and sedimentation reactor has never been reported. In order to clarify these problems, this study for the first time systematically monitors and analyzes the evolution of floc characteristics in a continuous-flow flocculation and sedimentation reactor, thereby reflecting floc status in practical flocculation and sedimentation processes in water utilities to a greater extent.

This study concerns the evolution of flocs in a continuous-flow reactor that contains four flocculation tanks and one settler tank. A synthetic kaolin-humic acid suspension was used as the test water sample. The investigation focuses on the floc characteristics, in terms of the average floc size, boundary fractal dimension, and eccentricity ratios. In addition, a novel V-shaped plate settler was designed in order to improve the sedimentation efficiency. The particle numbers and effluent turbidity were measured to evaluate and compare the efficiency of the V-shaped plate settler and a traditional inclined plate settler.

1. Material and methods

1.1 Kaolin-humic acid suspension

Kaolin solution was prepared by dissolving 1.80 g of kaolin (Tianjin, China) with 1 L tap water. In order to obtain a sufficiently dissolved kaolin solution, the pH of the solution was adjusted to 12 using 2 mol/L NaOH. The solution was mixed at 1000 r/min for 60 min by a stirrer and then placed in the dark for 24 hr. Humic acid solution was obtained by dissolving 36 mg of humic acid (Shanghai, China) in 0.1 mol/L 200 mL of NaOH. The solution was filtered by a 0.45 μm fiber filter membrane. The kaolin-humic acid suspension was a mixture of the kaolin solution and humic acid solutions. The pH of the solution was adjusted to 7.2 using 0.1 mol/L NaOH and 0.1 mol/L HCl. The preparation process had been successfully used in our previous study (He et al., 2012).

1.2 Apparatus

In order to simulate practical flocculation and sedimentation processes in drinking water utilities, a continuous-flow reactor was used in this study. As shown in **Fig. 1**, the flocculation section consisted of four equal pools, named pool A, pool B, pool C and pool D. The volume of each pool was 12.5 L ($L \times W \times H = 250 \text{ mm} \times 250 \text{ mm} \times 200 \text{ mm}$). Synthetic test water flowed through pool A to pool D and sequentially flowed into an up-flow settling tank. The total volume of the up-flow settler was 81 L ($L \times W \times H = 450 \text{ mm} \times 600 \text{ mm} \times 350 \text{ mm}$). The settling tank was divided into three parts, named unit E, unit F, and unit G, respectively. Two kinds of plates were installed in unit F, corresponding to a traditional inclined plate sedimentation tank and a novel V-shaped plate sedimentation tank.

Fig. 2 illustrates a cross-sectional view of the V-shaped plate and traditional inclined plate. Both of the plates had an inclined plate section, whereas the V-shaped plate had an additional V-shaped region. The intervals of the inclined plate section in the two kinds of plates were 5 mm, with a 60-degree angle. In the V-shaped region, the minimum interval was 5 mm with the angle of 60 degrees. The height of the inclined plate region was 100 mm, whereas, the V-shaped plate had a total height of 120 mm including 80 mm height for the inclined plate region and 40 mm height for the V-shaped region.

1.3 Procedure

The kaolin-humic acid suspension was mixed with tap water (Harbin, China). The tap water contained total alkalinity of 108 mg/L as CaCO_3 and hardness of 152 mg/L as CaCO_3 with conductivity of $492 \mu\text{S}/\text{cm}^2$. The experimental water contained around 100 mg/L of kaolin and 2 mg/L of humic acid. The pH of the experimental water was around 7.3-7.8. The initial turbidity of the test water samples was 100 ± 5 NTU, measured by an online turbidity meter (700AQ, WTW, Germany). The experimental water was directly pumped into the inflow pipe of the continual-flow reactor.

Polyaluminum chloride (PACl, 1%, W/W, Tianjin, China) was used as the coagulant in this study. According to experimental conditions, a certain amount of PACl was directly pipetted by a metering pump (D25RE2, Dosatron, France) into the inflow pipe near the inlet of the continual-flow reactor to achieve the desired PACl concentration.

The experimental water sequentially flowed through the four pools in the flocculation section with a flow rate of 2 L/min. In pool A, the experimental water and coagulants were adequately mixed, with a relatively high shear rate of 548 sec^{-1} . The shear rate was successively reduced to 69 sec^{-1} for pool B, 32 sec^{-1} for pool C, and 15 sec^{-1} for pool D, respectively. The gradual decreasing trend of shear rates was consistent with practical water utilities. Then, water samples sequentially flowed into the up-flow sedimentation tank. The hydraulic retention time in the settler was approximately 40 min.

In addition, the flocculation section was equipped with an *in-situ* recognition system as described below, in order to capture *in-situ* digital particle images. An online 2200PCX particle counter (HACH, USA) and an online turbidimeter (HF, USA) were installed in the sedimentation stage to measure the particle number and effluent turbidity.

1.4 *In-situ* recognition system

An *in-situ* recognition system was used to capture real-time digital particle images in the flocculation stage (He et al., 2012; Yao et al., 2014). The *in-situ* recognition technique could obtain the real-time status of floc morphology, avoiding changes of floc structure resulting

from sampling (Chakraborti *et al.*, 2000). This technique has been successfully used to evaluate the evolution of aggregates during the flocculation process (Chakraborti *et al.*, 2003; Xiao *et al.*, 2011). The obtained digital images were used to calculate the floc size and the fractal dimension.

A schematic of the *in-situ* recognition system is shown in **Fig. 3a**. The *in-situ* recognition technique system consisted of an automated stroboscopic lamp and a computer-controlled high-speed digital charged coupled device (CCD) video camera (SVS-VISTEK GmbH, Germany). The stroboscopic lamp was used to illuminate suspended particles in the flocculation reactor. The camera was installed opposite the lamp in order to offset the backlit shadows of particles. The camera had 992 (horizontal)×510 (vertical) pixels, and the interrogation window was 4500 μm×2310 μm. A computer was applied to control the camera, as well as store and analyze the particle images.

As shown in **Fig. 3b**, 9 sites were chosen as CCD camera locations to capture floc images in pool B, pool C, and pool D. 30 images (one image per second) were taken at each CCD camera location to calculate the average floc size and boundary fractal dimension in different experimental conditions. The number of aggregates in different captured images varied from 1000 to 150, depending on the coagulant dose and the flocculation stage. A decrease in number of aggregates implied the aggregation of small particles.

1.5 Floc size and boundary fractal dimension

Floc size is an important parameter of floc morphology. The size of irregularly shaped flocs can be expressed with an equivalent diameter, as described in Eq. (1), where D_p represents the floc's equivalent diameter; A is the projected area of a floc.

$$D_p = (4A/\pi)^{1/2} \quad (1)$$

The two-dimensional projection boundary fractal dimension (D_{pf}) is a statistical quantity to reflect the extent of fill space in fractal flocs (Zhou *et al.*, 2012). In this study, D_{pf} was used to depict the surface morphology of the flocs, which could effectively reduce the interference

of self-similarity and scale invariance of aggregates (He and Nan, 2012; Niemeyer et al., 1984; Johnson et al., 1994) . Better than the traditional mass-based fractal dimension, the boundary-based fractal dimension could reflect differences in morphology for flocs having the same mass (Mingli, 2007). Values of D_{pf} varying from 1 to 2 imply floc projected areas ranging from a sphere shape to a line shape. The boundary fractal dimension was calculated according to Eq. (2), where D_{pf} is boundary fractal dimension; P is particle perimeter (Mandelbrot et al., 1984).

$$A \propto P^{2/D_{pf}} \quad (2)$$

2 Result and discussion

2.1 Evolution of floc size

The four pools in the flocculation section separated the flocculation process into four stages. Pool A ($G=548 \text{ sec}^{-1}$) performed a coagulation stage where the test water and coagulants were mixed rapidly. Following pool A, flocculation pool B, pool C, and pool D represented changes in the flocculation stage. **Fig. 4** indicates the evolution of average floc sizes and the boundary fractal dimensions in flocculation pool B, pool C, and pool D at three different coagulant doses.

As shown in **Fig. 4a**, at coagulant dose of 1 mg/L as Al, the average size of flocs first achieved a relatively high value of 62 μm in pool B and further slightly increased to around 65 μm in pool C; then, the average floc size sharply increased to 78 μm in pool D. At coagulant dose of 3 mg/L as Al, the average size of flocs showed a steady growth trend from around 42 μm in pool B to 70 μm in pool D. However, the evolution of average floc size presented a different trend under the coagulant dose of 5 mg/L as Al. The average floc size markedly increased from 48 μm in pool B to 65 μm in pool C, but remained at 65 μm in pool D.

At relatively low coagulant doses (1 and 3 mg/L as Al), continuous flocculation occurred continuously from pool B to pool D. At these experimental conditions, charge neutralization played an important role at relatively high mixing intensity ($G=96 \text{ sec}^{-1}$ in pool B and $G=32$

sec⁻¹ in pool C). These stir rates could effectively promote floc collision, leading to a stable increase in floc size (Colomer et al., 2005). The further decrease of stirring intensity (15 sec⁻¹ in pool D) could prevent floc breakage, resulting in an apparent growth of the average flocs size via the sweep floc mechanism (Soos et al., 2008).

However, at a relatively high coagulant dose (5 mg/L as Al) a balance of aggregate collision and breakage had been achieved in pool C. The absence of relatively small size flocs and the decrease in the number of flocs reduced the particle collision probability, thereby preventing the growth of flocs in pool D (Moussa et al., 2013).

As a consequence, the evolution trend of the average size of flocs in the different flocculation stages was dependent on both the coagulant dose and the hydraulic conditions. Higher coagulant doses do not result in the formation of bigger flocs, but can achieve flocculation balance more quickly.

2.2 Evolution of boundary fractal dimension

Evolution of the boundary fractal dimensions (D_{pf}) of flocs was also investigated in the continuous-flow flocculation pool B, pool C, and pool D at three coagulant doses. The effective D_{pf} could evaluate the flocs' compaction and effective density. A previous study indicated that the boundary fractal dimension of 1 implied a sphere-shaped aggregate, whereas a fractal dimension of 2 implied a chain-shaped particle (Nan et al., 2009).

Fig. 4b indicates that the values of floc boundary fractal dimension showed slight fluctuations in the three pools. As shown in **Fig. 4b**, the boundary fractal dimension of aggregates ranged from about 1.14 to 1.20, which was in agreement with a published study applying a kaolin-humic acid solution as test water (Xiao et al., 2011).

The fractal dimensions of flocs (around $D_{pf}=1.14$) in the three pools at a coagulant dose of 1 mg/L as Al were lower than that for the coagulant dose of 5 mg/L as Al (around $D_{pf}=1.20$), suggesting that flocs formed at a relatively low coagulant dose were more compact throughout the whole flocculation process. The higher fractal dimension value represented the more irregular shape and rougher surface of the aggregates (Moussa *et al.*, 2013).

Moreover, at coagulant dosage of 3 mg/L as Al, the value of the fractal dimension decreased gradually from roughly 1.20 in pool B to 1.15 in pool D. This trend suggests a transition of floc structure from irregular shapes to regular shapes, accompanied by the formation of smoother and more compact flocs. As shown in **Fig 4a**, the floc size presented an increase trend at the coagulation dosage of 3 mg/L. Both the change in floc size and the fractal dimension at this coagulant dosage imply a good floc growth status.

Therefore, the increase of coagulant doses promoted the generation of more convoluted flocs with more branches, but having lower density. However, this kind of floc structure was too weak to resist the shear rate, leading to the breakage of flocs. These experimental results are comparable to the results shown in Fig. 4a. As a consequence, a relatively low coagulant dose (1 mg/L as Al) could generate flocs having higher density, which contributed to the formation of bigger flocs when the testing water sequentially flowed through the three pools. However, a relatively high coagulant dose (5 mg/L as Al) led to the formation of less compact and more branched flocs, resulting in the restriction of floc growth.

2.3 Flocs size distributions

Fig. 5 shows the cumulative floc size distributions in flocculation pool B, pool C, and pool D at three different coagulant doses. As presented in **Fig. 5**, the floc size distributions presented the same pattern in the three pools at coagulant dosage of 5 mg/L as Al, suggesting the floc size entered a steady stage at the beginning of flocculation in pool B. However, at 3 mg/L of coagulant, the proportion of floc size less than 100 μm declined from roughly 86% in pool B to roughly 81% in pool D, implying that the frequency of small size flocs appeared to decrease during the three sequential flocculation stages. Furthermore, a similar trend was observed at the coagulant dose of 1 mg/L as Al. These results indicated that aggregation was occurring in the entire continuous-flow flocculation stage at relatively low coagulant doses (1 mg/L and 3 mg/L as Al), supporting the earlier discussion in Section 2.1.

In addition, the cumulative percentage of small size flocs (less than 100 μm) in pool D with 1 mg/L PACl dose (76%) was less than that for the 5 mg/L PACl dose (85%), suggesting

that a relatively low coagulant dose may produce more large flocs via electrostatic and bridging mechanisms. This result was in good agreement with the evolution of floc size, as discussed in Section 2.1. Consequently, it is speculated that flocs formed under relatively low coagulant doses were able to gradually grow into large flocs through a series of flocculation processes.

2.4 Floc eccentricity ratios

The eccentricity ratio refers to the ratio of an aggregate's short axis and long axis, and can represent the irregularity of flocs (Meng et al., 2015). A value approaching 1 indicates flocs with a spherical shape, and conversely, a value close to 0 indicates a chain structure. **Fig. 6** illustrates the floc eccentricity ratios at different coagulant doses in flocculation pool B, pool C, and pool D. A pattern of normal distribution of the eccentricity ratios was observed at all three coagulant dosages. At the coagulant dose of 5 mg/L as Al, the eccentricity ratios of flocs were concentrated in the range 0.3 to 0.5, indicating that chain-shaped flocs dominated in the entire flocculation process at this relatively high coagulant dose. At coagulant dose of 3 mg/L as Al, the eccentricity ratio of 0.5 accounted for a large proportion in all the flocculation pools. In addition, the presence of eccentricity ratios close to 1 increased at the coagulant dose of 1 mg/L as Al, compared with the higher coagulants doses (3 and 5 mg/L as Al).

At coagulant doses of 1 and 3 mg/L as Al, the eccentricity ratio (ranging from 0.8 to 0.9 in pool D) was apparently higher than that in pool B and C, suggesting that flocs changed to a spherical shape and became more compact during the flocculation process at relatively low coagulant doses. These results are consistent with the former analysis on floc size evolution and distribution. It can be concluded that a relatively high coagulant dose (5 mg/L as Al) is much more inclined to generate chain-shaped and lower density flocs in pool B, resulting in the restriction of flocs' further growth in pool C and pool D.

2.5 Floc morphology

Fig. 7 shows the evolution of floc morphology captured by the in-situ recognition system at the coagulant dose of 3 mg/L as Al. The floc size gradually increased with the test water continuously flowing from pool B to pool D. These changes in floc morphology were consistent with the evolution of floc size described in Section 2.1.

In flocculation pool B, a relatively high shear rate ($G=96 \text{ sec}^{-1}$) promoted particle collision and aggregation, resulting in the formation of small and compact flocs in this flocculation stage. Based on the floc morphology, it is speculated that the flocs formed mainly via a charge neutralization mechanism in pool B. Then, the decrease of shear rate in pool C and pool D had a beneficial effect on aggregation of floc fragments, forming more branched structure flocs in pool C. Moreover, the larger particles aggregated in pool D by the bridge mechanism.

2.6 Comparison of the inclined plates and V-shaped plates

Table 1 shows the number of particles in the traditional inclined plate settler and the V-shaped plate settler at the coagulant dose of 1 mg/L as Al. In unit E, the particle numbers in the two settlers were similar because of the similar structures in this unit. The small size particles (less than 5 μm) were dominant, whereas flocs with size larger than 8 μm were scarcely observed due to rapid sedimentation of relatively large flocs.

In unit F, the application of the two kinds of plates resulted in a significant difference in the particle numbers. The total floc number in the inclined plate settler was greater than that in the V-shaped plate settler, suggesting better sedimentation efficiency for the V-shaped settler. In unit F, the V-shaped plate region had apparently fewer small size flocs (less than 5 μm), but more relatively large size flocs, compared with the inclined plate region. The number of small size flocs (less than 5 μm) in the inclined plate section was 8268 ± 322 , which was nearly twice the number in the V-shaped plate section, 4721 ± 124 .

These results indicate that the conventional inclined plates were ineffective for sedimentation of relatively small size floc, which has been reported by a previous study (Adelman et al., 2013). However, the V-shaped plate had a positive effect on the removal of

relatively small size flocs (less than 5 μm), owing to the re-aggregation that took place in the V-shaped region. As shown in **Fig. 2**, the V-shaped plate not only possessed the properties of the inclined plate, but also added a V-shaped region as a miniature folded-plate flocculation tank. Therefore, the small size flocs probably collided and restructured in this region (Jarvis et al., 2005). The re-flocculation process resulted in the formation of stronger and more compact aggregates that tend to precipitate (Parsapour et al., 2014). Furthermore, due to the effective sedimentation in the V-shaped region, the total floc amount in unit G of the V-shaped settler was obviously less than that for the inclined plate, implying a better sedimentation efficiency for the V-shaped settler.

2.7 Effluent turbidity of the continuous-flow reactor

Fig. 8 presents the effluent turbidity for the traditional inclined plates and the novel V-shaped plates. As shown in **Fig. 8**, the sedimentation efficiency was coagulant dosage dependent. An apparent reduction of effluent turbidity was observed in both settler units as coagulant dosages increased from 0.5 to 2.0 mg/L as Al. In this range of coagulant doses, the V-shaped plates presented higher turbidity removal efficiency than the inclined plates. The effluent turbidity showed the same trend at coagulant doses ranging from 3.0 to 5.0 mg/L as Al in both the V-shaped plate sedimentation and the inclined plate sedimentation. Based on the former analysis, the V-shaped plate could effectively precipitate relatively small size flocs via re-flocculation at the V-shaped structure. Therefore, the V-shaped plate settler presented a better precipitation rate than the inclined plate settler at relatively low coagulant doses.

3. Conclusions

This study investigated the evolution of floc morphology in a continuous-flow flocculation and sedimentation reactor. The results of floc size investigations indicated that the floc evolution in different flocculation pools was coagulant dose dependent. The value of boundary fractal dimension increased from 1.14 at 1 mg/L PACl dose to 1.20 at 5 mg/L PACl dose, illustrating that a linear shape floc tended to be produced at a relatively low coagulant

dose, whereas a spherical shape tended to be generated at a relatively high coagulant dose. The results for floc size distribution were in a good agreement with the average floc size results. The eccentricity ratio of aggregates mainly ranged from 0.3 to 0.6, suggesting that flocs with elliptical shapes were dominant in the entire flocculation process, which was supported by the results obtained in the fractal dimension analysis. Floc morphology measurements appeared to show a sustained growth of aggregates in the entire flocculation process at the coagulant dose of 3 mg/L as Al, which was consistent with the evolution of average floc size. It can be concluded that flocs maintained sustained growth at relatively low coagulant doses throughout the entire flocculation reactor, whereas the floc growth could be restricted when coagulant doses were excessive. Consequently, at relatively low PACl doses (1 mg/L or 3 mg/L), small and compact aggregates were formed by charge neutralization in pool B; then, flocs further grew in pool C and pool D by a sweep floc mechanism.

A traditional inclined plate settler and a V-shaped plate settler were investigated in the precipitation section. The experimental results indicated that the inclined plates were effective for the removal of relatively large size particles, whereas the V-shaped plates had advantages in the sedimentation of small size aggregates. The V-shaped plates provided a region for restructuring of aggregates and promoted the formation of more compact and larger aggregates, thereby decreasing the particle number and reducing the effluent turbidity. As a result, the V-shaped plate settler may be an alternative method to improve sedimentation efficiency.

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JES-D-16-00304-Table

Table 1 Number of particles in different unit of the inclined plate settler and the V-shape plate settler (coagulant dose 1 mg/L as Al)

Flocs size	Particle amount (number/mL)					
	Inclined plate settler			V-shape plate settler		
	unit E	unit F	unit G	unit E	unit F	unit G
<5 μm	4782±126	8268±322	1932±58	5033±199	4721±124	444±18
5-8 μm	545±22	971±59	243±25	501±29	2143±80	33±4
>8 μm	4±1	20±3	1±1	4±1	195±17	3±1

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