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Insights into the role of mixing conditions in coagulation–flocculation process: evaluation from performance, flocs formation and density perspectives

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ABSTRACT

Appropriate mixing conditions are always favorable during enhanced coagulation for the desirable performance. However, up to now, there are little studies systematically investigated the effects of mixing duration and intensity on coagulation–flocculation efficiency based on a comprehensive consideration. Different mixing intensity and duration were applied in coagulation and flocculation, respectively, using aluminum sulfate as the coagulant at a predetermined dosage where significant hydroxide precipitate occurs. Flocs formation, breakage and regrowth were monitored using a 'turbidity fluctuation' technique, photometric dispersion analyzer (PDA) measurement. In addition, the flocs images were in situ captured using a charged-coupled device camera to further the knowledge of flocs characteristics, such as fractal dimension and settleability. The results indicated that a slightly prolonged rapid mixing time (120 s) improved floc density with better settleability when the mixing intensity in coagulation stage was maintained at a central level. In contrast, a little higher mixing intensity (40.2 s^{-1}) in flocculation stage induced more compact flocs, however, with smaller homogeneous size. The compensatory interaction of mixing conditions between coagulation and flocculation stages was verified. Particularly, this work was expected to provide a useful suggestion on optimizing coagulation and flocculation separately, and thus the desirable performance can be obtained by the optimized operational design.

Keywords: Coagulation; Flocculation; Flocs characteristics; Fractal dimension; Settleability

1. Introduction

In most cases, coagulation–flocculation (CF) is an indispensable processing unit aiming at colloids/particles and dissolved organic matter removal during the drinking water treatment [1,2]. The CF process is known as determined by coagulant type and dosage, particle concentration, solution pH, mixing intensity and duration [3]. In particular, the adjustment of pH is previously recommended as an effective method of enhanced coagulation to improve organics removal [4].

In addition, mixing conditions (intensity and duration) greatly influence the general coagulation–flocculation performance and even the following membrane fouling when the coagulant type and dosage have been predetermined [5–7]. The relative literatures can be dated from 1980s during which several researchers suggested that the most important factors during coagulation–flocculation process are the rapid mixing parameters [8–10]. By contrast, slow mixing conditions in flocculation are not as critical as the former [11]. However, there are also conflictual views concerning the importance of rapid mixing speed and duration [12,13]. Some researchers [14,15] claimed that instantaneous rapid mixing should be applied for achieving the best chemicals hydrolyzing and particles destabilization, while others recommended a prolonged rapid mixing duration [13]. Consequently, these apparent conflicting opinions about rapid mixing intensity and duration design are still remained and a further study is required.

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In recent years, certain progress was achieved from aspect of the mixing conditions in coagulation–flocculation process [5,14,16]. Zhang et al. [16] paid attention to the effects of slow mixing intensity and duration on turbidity removal and floc size under different coagulation mechanisms using polyaluminum chloride. The results indicated that slow mixing intensity exerted a more marked positive effect on charge neutralization coagulation than on sweep flocculation. Yu et al. [5] further evaluated the effects of rapid mixing time and slow mixing speed on coagulation–flocculation process. The results demonstrated that prolonged rapid mixing time decreased the final floc size, and a suitable slow mixing intensity should be definitely chosen for floc growth despite the employed different rapid mixing parameters. Previous studies mainly obtained the optimal rapid and slow mixing parameters by conducting single-factor experiments while other factors were fixed. However, the interaction between each rapid and slow mixing parameters should be better comprehensively elucidated, and the integrated design and operational diagram could be expected to be suggestive for real coagulation–flocculation application.

Therefore, the aim of this work is to clarify the requirements of rapid and slow mixing during coagulation–flocculation process, respectively, and to systematically investigate the interactions between these factors as well. Some specific techniques including a continuous monitor technique (PDA measurement) and online charged-coupled device (CCD) photography were employed to give a plausible explanation on the experimental phenomenon. In this regard, floc characteristics were derived from PDA measurement and CCD images, and floc settleability were also evaluated.

Particularly for a clear definition in this paper, 'coagulation' is only used for the stage when coagulants are hydrolyzed and particles are destabilized; 'flocculation' refers to the period of flocs formation.

2. Materials and methods

2.1. Suspensions and coagulant

Kaolinite clay (Fuchen Chemical Reagent Factory, Tianjin, China) was used in this work to prepare the model suspensions. Kaolin clay of 300 g was added in 1 L Milli-Q deionized (DI) water, and then mixed at high speed of 1,000 rpm for 0.5 h using a magnetic stirrer. In particular, the pH of this suspension is suggested to be initially adjusted to 7.5 to obtain full dispersion before intense agitation [17]. After quiescent settlement for 16 h, the supernatant about 700 mL was decanted as the stock suspensions. Its solid content was measured gravimetrically to be 76.8 g/L, and the average diameter was determined to be ~1.53 μm using particle size analyzer (DelsaNano, Beckman Coulter, USA).

One of the most widely used coagulants, aluminum sulfate ($Al_2(SO_4)_3 \cdot 18H_2O$, Aladdin Chemical Corporation, Shanghai, China), was employed throughout this study. The stock solution was prepared using DI water at the concentration of 0.2 M (calculated as Al). It should be renewed every 2 weeks to avoid ageing phenomenon and to improve reproducibility of this work. In addition, the adjustment of pH was conducted using 0.1 M NaOH or HCl solution.

2.2. Jar test

Before coagulation experiments, stock solution of kaolin was diluted into local tap water (Beijing) giving the concentration of test solutions of 50 mg/L (corresponding to 38.9 ± 1.3 NTU). To offset the unexpected effect of hardness (divalent metal ions, Ca^{2+} and Mg^{2+}) in tap water, a small amount of humic acid (Aldrich, USA) was added simultaneously [18], and the final pH of the test solution was adjusted to be 7.0.

In the preliminary coagulation experiments (section 3.1), the effect of certain commonly used mixing conditions on coagulation performance was evaluated using a programmable six-paddle blender (1 L) with 50 mm × 40 mm flat paddle impeller (ZR4-6, Zhongrun Instrument Company, Shenzhen, China).

In order to obtain a further understanding of the role of mixing in coagulation and flocculation process, respectively, a large-scale rectangular stirred tank (length × width × height: 150 × 150 × 250 mm) was self-designed and made by quartz. Water sample of 4 L was filled before experiment. The mixing apparatus is comprised of an agitator (Eurostar 60 control, IKA, Germany) and an impeller (R1342, IKA, Germany) shown in Fig. 1, which can blend 40 L water and be set up to 3,000 rpm, was used in the following tests (sections 3.2–3.4). A predetermined amount of coagulant was dosed after the initial rapid mixing for 30 s to make the test solution well distributed. More details about mixing speed (intensity) and duration applied during the coagulation–flocculation stage will be presented in section 3.2.

2.3. Floc monitoring

Kinetics of flocs formation, breakage and subsequent regrowth was measured using iPDA 2000 (Rank Brothers, UK) by the 'turbidity fluctuation' method. The operational procedure was similar to that of Yu et al. [19]. Two indexes including the average transmitted light intensity (dc value) and the root mean square (rms) value of the fluctuating component of the intensity were simultaneously monitored. The ratio (rms/dc) was outputted and defined as the flocculation index (FI) which gives a sensitive measurement of particles aggregation [20]. The value of FI is strongly correlated with

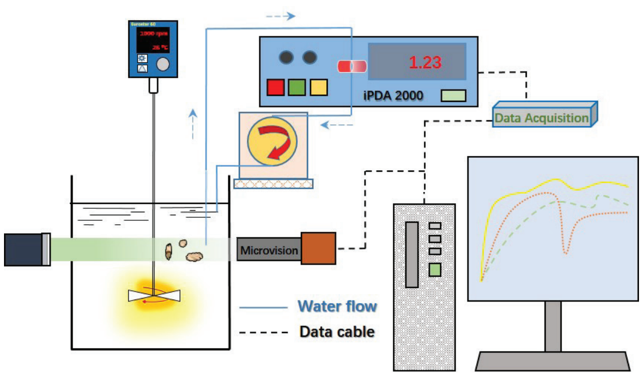

Fig. 1. A schematic diagram of online floc formation monitor system.

floc size, and increases as flocs grow according to Yu et al. [17]. The data were recorded using a data acquisition system (Pico ADC-11, Pico Technology, UK) every 2 s after the initial FI value kept at a steady rate.

In order to take a further examination of flocs characteristics, such as floc size and its fractal dimension, an in situ photography system was employed in this work. This system (Fig. 1) is composed of a digital CCD camera (MV-EM510C/M, Microvision, China) and a light source (BT-TCL16/G, Microvision, China). The CCD camera has a sensor matrix consisting of 2,456 (horizontal) × 2,058 (vertical) pixels and provides pictures with a resolution of 178 pixels/μm. Besides, coaxial parallel light equipped ensures the brightness of images captured at a relatively high speed. This process was computer-controlled and the images were analyzed using an open-source software (Image-Pro, 7.0).

2.4. Floc characteristics

Coagulation performance is significantly determined by floc characteristics, such as floc size, fractal dimension and density. Specifically, the two-dimensional fractal dimension is defined by a power law relationship between the projected area (As) and the characteristic length of the flocs, denoted as 'l'. It can be calculated from:

$$As \quad l^{D_2} \qquad (1)$$

where D_2 is the two-dimensional fractal dimension.

The value of D_2 ranges from 1 to 2. A smaller value 1 represents a linear shape, while a larger value 2 indicates a spherical floc [21].

In addition, floc density is a crucial factor determining the settleability of flocs. According to He [22], floc settleability can be evaluated by the rate of variation of residual turbidity with the prolonged settlement duration. A steeper slope of the variation of residual turbidity indicates better settleability of flocs. A good correlation was also confirmed in this study (Fig. 2), as the fit coefficient R^2 was up to 0.9472.

3. Results and discussion

3.1. Coagulation under common mixing conditions

Previous studies on coagulation paid little attention on the influence of applied mixing conditions, such as mixing speed and duration. In most cases, parameters during coagulation and the following flocculation stage were directly employed according to the operational experience, as shown in Table 1. However, some other researchers pointed out that the applied mixing conditions exerted a significant influence on flocs formation and of course the treatment efficiency [5,15]. It was not difficult to find that a rapid mixing speed at 200 rpm (106.4 s^{-1}) lasting for 1 min in the coagulation period was commonly adopted regardless of the pollutants and coagulants, as summarized in Table 1. Similarly, nearly the same mixing speed at 40 rpm (12.2 s^{-1}) of 15 min was always applied in the flocculation stage to allow floc growth. To identify the influence of mixing conditions, different agitation intensity and time were performed in the following experiment. A predetermined amount of 0.12 mM $Al_2(SO_4)_3$ was added throughout this study where hydroxide precipitate occurs, and the relative coagulation performance and zeta potential can be found in Fig. 3. To investigate the effect of

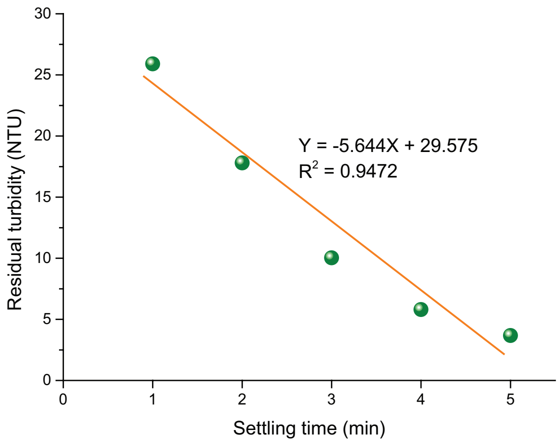

Fig. 2. Correlation between residual turbidity and settling time.

Table 1
A summary of common used mixing parameters in coagulation–flocculation process

Water sample	Coagulant	Mixing speed	G value	Mixing duration	Apparatus	Reference
HA-kaolin water	$AlCl_3$	R: 200 rpm; S: 40 rpm	NG	R: 1 min; S: 15 min	ZR4-6, Zhongrun, China	Wang et al. [23]
Algae-rich water	$Al_2(SO_4)_3$	R: 200 rpm; S: 30 rpm	R: 350 s^{-1} S: 25 s^{-1}	R: 1 min; S: 15 min	Lighter, Taiwan	Lin et al. [24]
Source water in a Beijing WTP, China	$FeCl_3$	R: 300 rpm; S: 50 rpm	R: 367 s^{-1} S: 33 s^{-1}	R: 1 min; S: 15 min	ZR4-6, Zhongrun, China	Zhou et al. [25]
RW from Parramatta, Sydney, Australia	PTS	R: 200 rpm; S: 40 rpm	NG	R: 1 min; S: 15 min	PB-900TM, Phipps and Bird, USA	Zhao et al. [26]
Simulated micro-polluted surface water	PACl	R: 200 rpm; S: 50 rpm	NG	R: 1 min; S: 15 min	ZR4-6, Zhongrun, China	Su et al. [27]

Note: HA, humic acid; WTP, water treatment plant; RW, river water; PTS, polytitanium sulfate; R, rapid mixing; S, slow mixing; NG, not given.

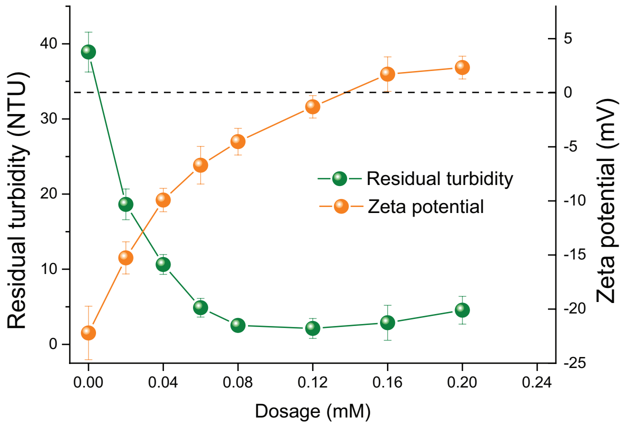

Fig. 3. Influence of coagulant dosage on residual turbidity and zeta potential.

Table 2
Typical hydrodynamic values during coagulation–flocculation

N (rpm)	*G* (s^{-1})
20	4.8
40	12.2
60	21.1
80	31.0
100	41.9
200	106.4
250	143.7
300	183.6
350	225.9
400	270.4

Apparatus: a programmable six-paddle blender (ZR4-6, Zhongrun Instrument Company, Shenzhen, China); $G = 0.08554 \times N^{1.345}$.

mixing conditions comparatively, the same apparatus (ZR4-6, Zhongrun, China) as reported in Table 1 was employed, and the relative information of mixing speed and gradient (*G*) value was also provided in Table 2.

Series of single-factor experiment were carried out (Fig. 4) when the other three factors were maintained at the constant values of the control group (rapid mixing: 350 rpm (225.9 s^{-1}) for 1 min; and slow mixing: 60 rpm (21.1 s^{-1}) for 15 min. As displayed in Fig. 4(a), the result evidently indicated that a rapid mixing speed of 200 rpm in coagulation stage was insufficient to obtain the best turbidity removal efficiency, since the residual turbidity decreased continuously with the increasing mixing speed up to 360 rpm (234.6 s^{-1}). As to the rapid mixing time (Fig. 4(b)), the variation of residual turbidity exerted an obvious distinct trend. The result suggested that a prolonged mixing time (>76 s) in coagulation stage showed a negative influence on turbidity removal, and this phenomenon was consistent to the findings of other researchers [5,15].

In flocculation period, micro-flocs formed by destabilized particles grow to large flocs during which suitable mixing conditions are indispensable. As shown in Figs. 4(c) and (d), turbidity removal performance was greatly improved by the increase of mixing speed (from 30 rpm (8.3 s^{-1}) to 60 rpm (21.1 s^{-1})) and the prolonged mixing duration (from 5 to 15 min), respectively. Additionally, longer flocculation duration up to 25 min showed slight further improvement on turbidity removal compared with that of slow mixing of 15 min.

3.2. Interaction between mixing intensity and duration in coagulation/flocculation

Although many researches [5,18,28] on the mixing conditions in coagulation–flocculation have been carried out, there were little literature reported the interactive influence of mixing intensity and duration on coagulation–flocculation process, respectively. Due to this reason, a large-scale trial system was constructed as described in section 2.2. This system can provide a broader range of agitation intensity and can be also used for online capture of flocs images. The selected *G* value of this agitator was listed in Table 3.

As reported by Amirtharajah and Mills [29], coagulants are normally hydrolyzed in only a few seconds after dosing at the beginning of rapid mixing. And particles destabilization and micro-flocs formation proceed in the coagulation stage, and their performance is significantly determined by the applied mixing conditions. As shown in Fig. 5(a), rapid mixing speed ranging from 200 rpm (61.9 s^{-1}) to 1,000 rpm (691.7 s^{-1}) was applied within a duration from 5 to 120 s. Generally, the prolonged rapid mixing duration exerted a positive influence on the turbidity removal, with an exception when the speed beyond 750 rpm (449.3 s^{-1}). This result was consistent with the former phenomenon in section 3.1 and also with other findings [5,15]. Francois [30] considered that a further increase in rapid mixing time gives rise to micro-flocs breakage and then many smaller flocs are generated which may be responsible for the poorer turbidity removal efficiency. In particular, there was an interesting finding that the zeta potential of flocs apparently decreased with the prolonged rapid mixing time (Fig. 5(c)). This reduction of zeta potential might be primarily attributed to the release of negatively charged particles back into suspensions caused by the breakage of micro-flocs during the prolonged rapid mixing time up to 120 s. In comparison, the rapid mixing speed in the coagulation stage caused a relatively little influence on the zeta potential of flocs when the speed was beyond 350 rpm (143.3 s^{-1}), as shown in Fig. 5(d). Consequently, it can be concluded that rapid mixing time is more important when the corresponding speed was kept a central level.

Fig. 5(b) shows the influence of slow agitation intensity on coagulation performance within a duration up to 30 min. In this period, small flocs aggregate together to form large flocs. Particularly, flocs with good properties always contribute to the desirable treatment performance. In order to obtain the lowest residual turbidity, a proper mixing speed should be controlled, slower or faster speed both showed a negative effect (Fig. 5(b)). It can be seen clearly that the similar value of optimal residual turbidity was obtained when the speed of 100 rpm (21.8 s^{-1}) was applied. However, higher speed (>150 rpm) significantly exerted an adverse effect on the particles removal, mainly because of the breakage of flocs as suggested by other researchers [5,16]. Consequently, it can be concluded that a proper slow mixing intensity is more important for the treatment performance when the mixing time is relatively enough.

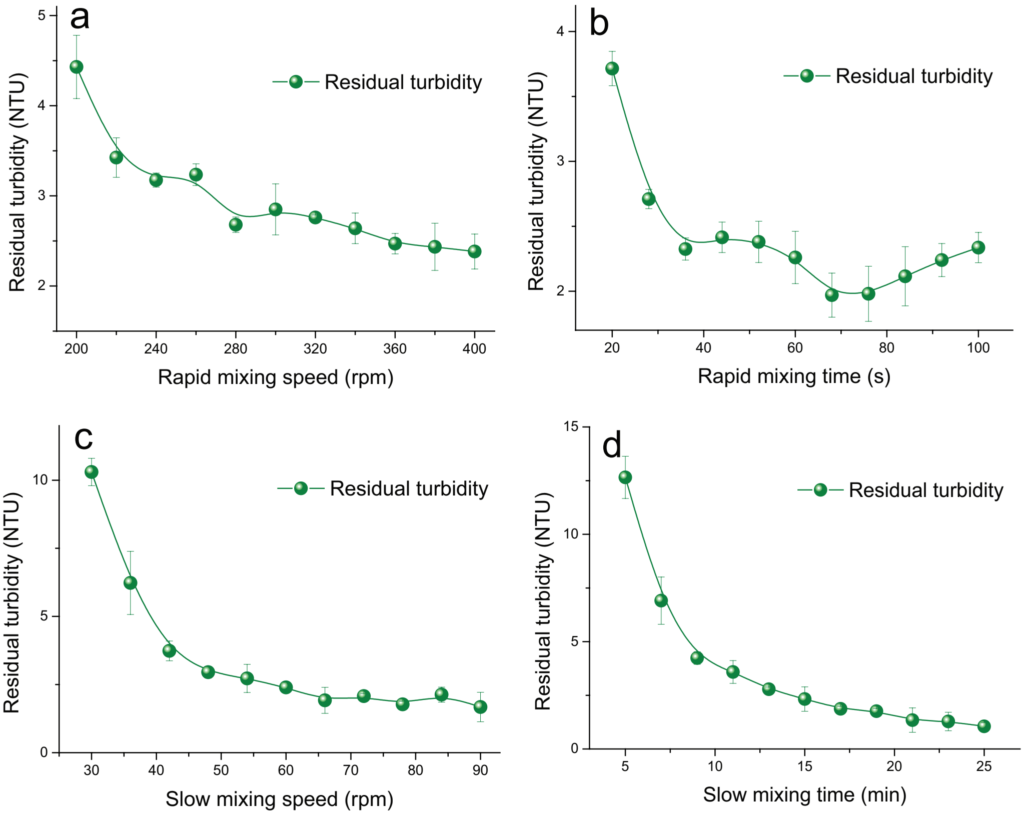

Fig. 4. Influence of single factor on turbidity removal: rapid mixing speed (a); rapid mixing time (b); slow mixing speed (c) and slow mixing time (d).

Table 3
Selected hydrodynamic values during coagulation–flocculation process

N (rpm)	G (s^{-1})
50	7.7
100	21.8
150	40.2
200	61.9
300	113.6
450	208.8
550	281.2
650	362.5
750	449.3
1,000	691.7

Flocculator: Eurostar 60 control, IKA, Germany; Impeller: R1342, IKA, Germany. G value was calculated according to He [22].

3.3. Interaction between coagulation and flocculation

Zhang et al. [16] indicated that desirable mixing conditions in the flocculation stage can effectively limit the negative influence caused by the improper mixing conditions in the coagulation stage, and thus defined it as a 'compensatory effect'. Based on the above single-factor experiments, it can be found that rapid mixing duration and slow mixing speed significantly dominated the general coagulation–flocculation efficiency when the other factors were kept at the central level. However, little researches systematically evaluated the synergetic effect of mixing parameters in the coagulation and flocculation process, respectively. Therefore, it was supposed to clarify the interaction and eventually optimize the operational domain in terms of practical application (Fig. 6).

Coagulation–flocculation tests were carried out under different rapid mixing duration up to 120 s and within a range of slow mixing speed from 50 rpm (7.7 s^{-1}) to 300 rpm (113.6 s^{-1}), respectively. The results (Fig. 6(a)) demonstrated that an undesirable turbidity removal efficiency can be resulted from higher intensity of slow mixing (300 rpm) despite of the rapid mixing time. In contrast, the better performance with lower residual turbidity (<4 NTU) was obtained when the two main factors were both maintained at the central level, except for the condition of 10 s rapid mixing with 50 rpm (7.7 s^{-1}) slow mixing and 120 s rapid mixing with 250 rpm (86.4 s^{-1}) slow mixing. For clearer realization of the performance, the selected mixing conditions were presented by the various shapes and colors, as shown in Fig. 6(b). The desirable performance can be found in the 'orange domain', which suggested that the requirement for slow mixing speed decreased with the prolonged rapid mixing duration. This phenomenon 'compensatory effect' was also confirmed previously by Zhang et al. [16]. Especially, the best turbidity removal performance at every slow mixing speed was presented by 'star' in Fig. 6(b), and this demonstrated that 30 s was the minimum demand for rapid mixing time to obtain the optimal performance regardless of the slow mixing speed

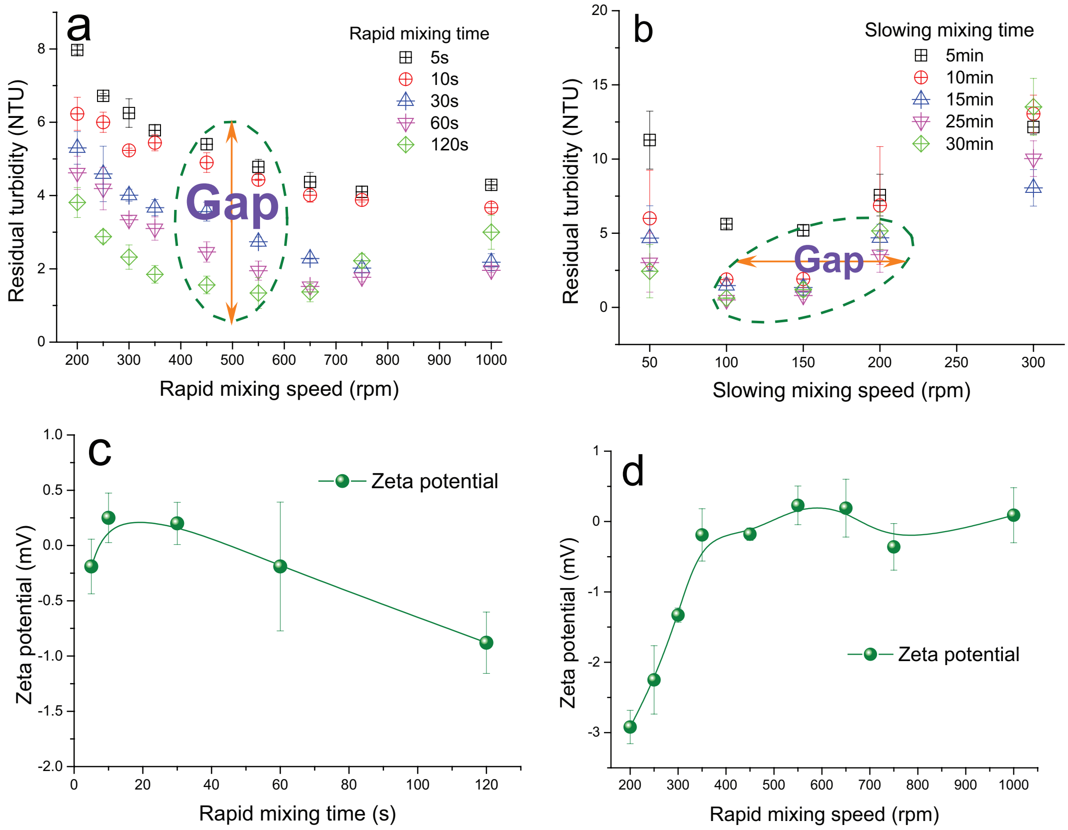

Fig. 5. Influence of mixing conditions in coagulation (a) and flocculation (b) on turbidity removal. Influence of rapid mixing time (c) and speed (d) on zeta potential.

applied. Hence, a relatively enough rapid mixing duration is almost compulsory for the particles destabilization in coagulation to achieve the optimal performance.

3.4. Mechanism

To explain the underlying reason of the above results, we employed a 'turbidity technique' (PDA measurement) and online CCD photography to monitor flocs formation and capture their images, respectively.

Fig. 7(a) shows the effect of rapid mixing duration, at the speed of 550 rpm (282.1 s^{-1}), on the flocs formation. According to Gregory and Chung [20], FI value keeps a proportional relationship with floc size. It can be seen that the floc size formed under 10 and 30 s rapid mixing were nearly the same. However, floc size became smaller with the prolonged duration, especially when the rapid mixing time was selected at 120 s. Moreover, the corresponding residual turbidity continually decreased in many cases when the rapid mixing duration ranged from 10 to 120 s, as shown in Fig. 5(a). And this result was contrary to the literatures [15,28]. In previous study [15], an extended rapid mixing time up to 60 s exerted an obvious negative influence on turbidity removal when $Al_2(SO_4)_3$, PAX-XL9 or polyDADMAC was employed. In particular, the similar finding was also confirmed in the current work only when a stronger rapid agitation intensity (>750 rpm, 449 s^{-1}) was applied. Therefore, it can be concluded that to extend rapid mixing duration properly can be effective for the improvement of turbidity removal when the rapid mixing intensity was controlled under a certain value of 750 rpm (449.3 s^{-1}).

Normally, floc size (FI value) can be kept stable under a certain slow stirring speed. The roughness of FI curve also gives an indication of general floc size distribution since a specific value is theoretically related to a certain floc size [31]. In this regard, a flat curve represents the similar size of flocs. As shown in Fig. 7(a), it can be therefore deduced that floc size gradually became more homogeneous and smaller when the rapid mixing time was prolonged from 10 to 120 s. Besides, this speculation was also verified by the images captured by a CCD camera (Fig. 8(a)). Specially, there was an interesting finding that the settling velocity, reflected by the ratio ('*k*') of turbidity reduction, improved with the decrease of floc size. This phenomenon suggested that flocs settleability was prone to keep a strong relationship with their density, however, not with their sizes. Besides, as shown in Fig. 8(a), the flocs with a circular shape can settle faster than that of linear shape, and this result was same to the others' finding [26,32]. Consequently, it can be concluded that particulates removal efficiency can be improved by properly prolonging the rapid mixing duration when the stirring intensity was kept at a suitable level.

As to the influence of slow mixing speed on the floc formation, as shown in Fig. 7(b), a higher speed significantly reduced the floc size in the steady state. Besides, it can be obviously observed that FI values fluctuated more slightly when the slow mixing speed increased. This phenomenon

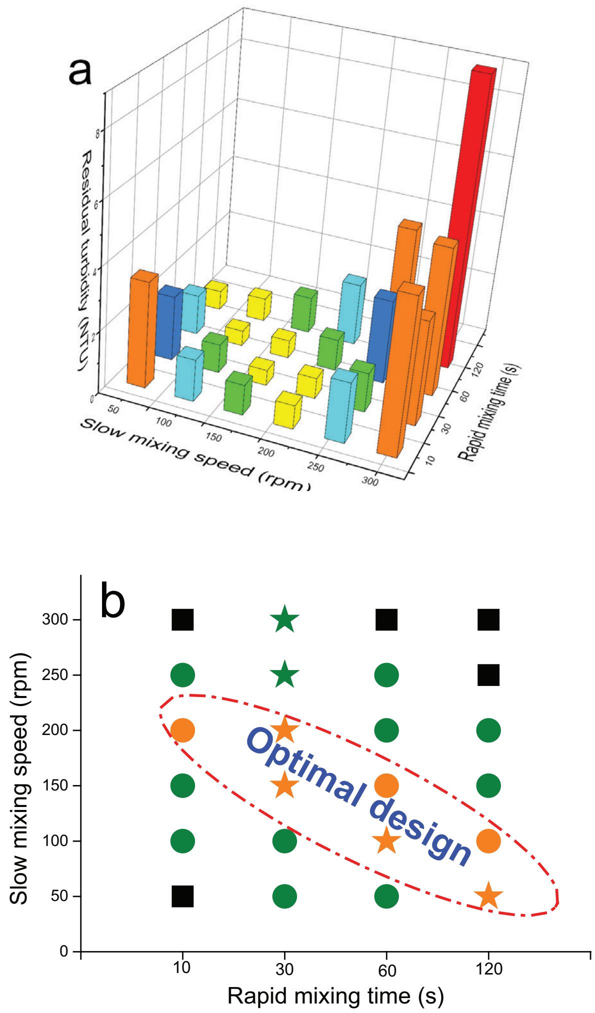

Fig. 6. Interaction between coagulation and flocculation on turbidity removal (a); optimal operational design of coagulation–flocculation process (b). Residual turbidity level: orange < 0.8 NTU, 0.8 NTU < green < 3.0 NTU and black > 3.0 NTU.

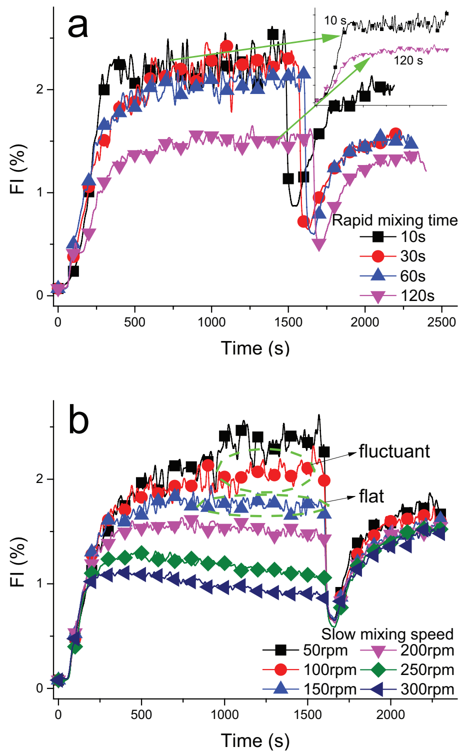

Fig. 7. Flocs growth, breakage and regrowth. Influence of rapid mixing duration (a) and slow mixing speed (b).

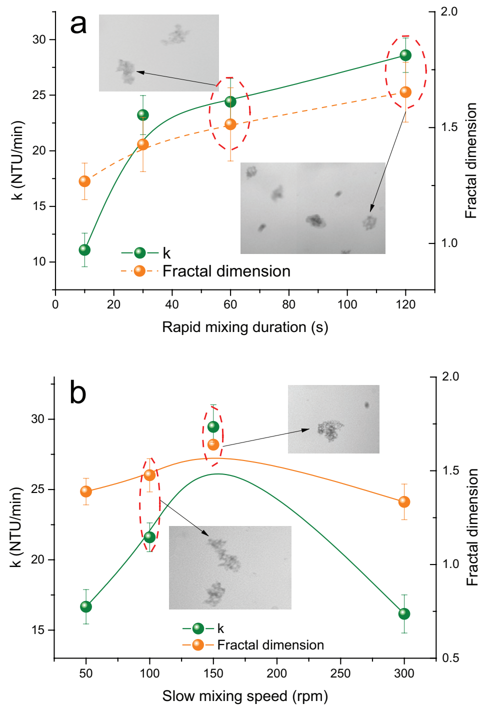

Fig. 8. Relationship between flocs settleability and fractal dimension. Influence of rapid mixing duration (a) and slow mixing speed (b). '*k*', calculated as the variation ratio of residual turbidity, was selected as the representative for floc settleability. Representative flocs images were selected from images of online capture using a CCD camera.

indicated that – flocs (similar and smaller size) formed under an increased mixing speed (Fig. 7(b)), in contrast, flocs formed under the speed of 50 rpm (7.7 s^{-1}) achieved the largest size. Also the images (Fig. 8(b)) confirmed this result, and the flocs formed under a speed of 150 rpm (40.2 s^{-1}) in flocculation period obtained the highest density with a circular shape. Hence, that floc size does not dominantly determine the flocs settleability has been further confirmed.

Yu et al. [33] indicated that flocs structure changes continually since the flocs internal bonds break under shear and reform at the more favorable points. To obtain an optimal design in coagulation–flocculation process (Fig. 6(b)), a proper increase of the slow mixing speed is an effective approach to improve particulates removal efficiency when the rapid mixing duration was relatively short (10–30 s). This phenomenon can be attributed to the breakage caused by the increased shear force which provides more opportunities for flocs to connect at more favorable sites. And thus turbidity can be further reduced and flocs were more compact and dense, although became smaller.

4. Conclusions

This paper systematically investigated the requirement of mixing conditions for coagulation–flocculation process, respectively. Flocs characteristics including fractal dimension and density determined by various mixing conditions were also evaluated. The main conclusions were as follows:

- A slightly prolonged rapid mixing time under proper mixing intensity improved particles removal performance and flocs settleability, although with smaller size.
- Compact flocs with good settleability were obtained by increasing the agitation intensity in the flocculation stage which could provide additional opportunities for flocs to connect at more favorable sites.
- The compensatory effect of mixing conditions between coagulation and flocculation process was verified by the optimal operational design.
- To optimize the coagulation and flocculation process separately was expected be suggestive in the drinking water treatment, in terms of flocs properties and of course water quality.

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