Desalination and Water Treatment
www.deswater.com

doi: 10.5004/dwt.2017.11413

64 (2017) 419–424
February

Sizes of iron hydroxide particles formed during ferric coagulation processes

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Received 3 April 2016; Accepted 9 August 2016

ABSTRACT

The aim of the study was to apply laser granulometer for measuring sizes of particles formed in iron-based coagulation process in water, which did not contain significant amounts of organic compounds. The research was conducted using tap water, obtained from underground sources. Iron sulfate in doses of 2.0–10.0 mgFe L^{-1} was added to water. During initial stage of flocculation, iron hydroxide particles reached dimensions between 0.03 and 0.5 μm (at coagulant dose of 2.0 mg Fe L^{-1}) and between 0.1 and 0.5 μm (for higher doses). Maximum dimensions of flocks amounted to 130–500 μm and at doses greater/equal to 4.0 mgFe L^{-1} did not depend on coagulant doses. Merely, for a dose of 2.0 mgFe L^{-1} and after 40 min of flocculation, the flocks reached dimensions of 85–320 μm and continued to enlarge. With increasing dose of coagulant, flocks faster enlarged their dimensions and faster reached maximum sizes. Initially, the smallest particles with dimensions smaller than 0.1 μm still remained in the solution. Persistence of the smallest particles ranged up to 7 min.

Keywords: Ferric coagulants; Iron hydroxide; Flocculation process; Size of particles; Laser granulometer

1. Introduction

Coagulants are commonly used in water treatment to remove colloidal substances (organic and inorganic) and finely dispersed suspensions (causing mostly color and turbidity), mainly from surface water [1,2]. Aluminum and iron-based coagulants are also used to remove heavy metals from water. The mechanism of coagulation/co-precipitation with aluminum and iron salts relies on adsorption of sparingly soluble heavy metal compounds on the surface of $Al(OH)_3$ or $Fe(OH)_3$ flocks. The effectiveness of this process depends on type of metal, its form of occurrence in water, type and dose of coagulant, temperature and chemical composition of water (mainly alkalinity and pH). Furthermore, the effectiveness of metals removal is the highest in particular pH range at which the solubility of $Al(OH)_3$ and $Fe(OH)_3$ is the smallest, and formation of sparingly soluble metal complexes is also possible. It is considered that for most heavy metals, if there are no organic ligands, better coagulants are iron-based compounds (as usually happens in case of ground water treatment), which can be used at higher pH values than aluminum salts. Moreover, iron compounds are less sensitive to low water temperature [1].

The removal of arsenic in coagulation/co-precipitation with iron ions is an example of iron coagulants application to heavy metals removal from surface and groundwater intended for human consumption [3–9]. In this method, iron (III) compounds are most commonly used, especially $FeCl_3$ and $Fe_2(SO_4)_3$. The method involves addition of iron compounds into the water, mixing and then the passage of the water through the conventional filters with granular filter media or through membrane filters. The most important factors affecting the efficiency of the process are iron salt doses, pH, form of arsenic occurrence and contact time. Research conducted in the pilot plant scale using natural groundwater indicated that ferric chloride coagulation integrated with microfiltration (coagulant dosing, aeration/mixing, filtration)

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Presented at the Conference on Membranes and Membrane Processes in Environmental Protection (MEMPEP 2016), Zakopane, Poland, 15–19 June 2016.

enabled effective removal of arsenic to the level complying with quality of water intended for human consumption [8,9]. Good results of arsenic removal and reduction of its concentration from approximately 36–40 μg L^{-1} in raw water to less than 10 μg L^{-1} were obtained, even at relatively low doses amounting to 2.1 mg L^{-1} of ferric coagulant. Together with arsenic, naturally occurred in raw water iron was also removed, as well as the dispensed in the form of iron (III) sulfate. It is also important that those results were achieved at slightly alkaline pH value of water (pH = 7.74) and without pH correction.

As in the discussed example, in current technological sequences after the coagulation process, it is more often suggested to replace conventional filters by low-pressure membrane technologies: microfiltration or ultrafiltration. These solutions combine traditional treatment methods such as coagulation, adsorption, ion exchange or chemical oxidation with membrane techniques in so-called hybrid or integrated systems [7,10,11]. In the coagulation integrated with membrane filtration, method often resigns from slow stirring process. After coagulant dosing and fast mixing, water is directed to the membrane module. The advantage of using membranes in place of the conventional filters is creation a more effective barrier to microorganisms, ability to remove smaller sized flocs (obtained at lower doses of coagulants and short stirring time), improvement of overall system efficiency and avoidance of overloading the filter bed [7,12,13].

Coagulation process involves two stages. During first stage (directly after coagulant dosing) physical and chemical reactions proceed, what leads to destabilization of colloidal particles. Whereas during second phase, known as flocculation, flocks are formed as a result of transport and collisions of destabilized particles. Flocks gradually increase their sizes as a consequence of particles merging. In case of ferric coagulation application for heavy metals removal from water, which does not contain organic substances (the most common situation in ground water), flocks are formed almost solely from precipitated iron (III) hydroxides. The effects of flocculation (above all: formed flocks sizes) are determined by factors such as type and amount of removed contaminants, color and turbidity of water, type and dose of coagulant, physical and chemical properties of water (mainly pH, alkalinity, hardness and temperature). Technological parameters also have significant role – especially stirring intensity and duration of flocculation process [14–19].

After flocculation process, formed suspension must be removed from the water. In conventional systems, it is removed by sedimentation and rapid filtration, while in hybrid systems – by membrane filtration. With regard to ultrafiltration and microfiltration, fundamental mechanism of separation particles present in water is sieve mechanism. In this case, separation of substances relies on physical sieving, whereas the efficiency of the process depends on membrane porosity and sizes of removed particles. Therefore, to achieve rewarding efficiency of separation process, it is necessary to fulfill the condition that sizes of particles removed from water need to be larger than diameters of membrane pores. Furthermore, presence of particles with dimensions comparable with the dimensions of the membrane pores may result in irreversible membrane blocking. This undesirable process is one of the major factors limiting application of membrane techniques into practice. Therefore, knowledge of particles sizes formed during coagulation/flocculation process should be one of the basic criteria for selection of separation technique (e.g., type and sizes of membrane pores as well as duration of process). The final result of treatment is not only connected to the amount of large agglomerates formed during flocculation process but also to the presence of the finest particles in water, which have not been merged into agglomerates.

The aim of the study was to apply laser granulometer for measuring sizes of particles formed in iron-based coagulation process in water, which did not contain significant amounts of organic compounds.

2. Materials and methods

The research was conducted using tap water. The supplied water was obtained from underground sources and all its components fulfilled the quality standards of water intended for human consumption [20]. Especially iron concentration was in trace amounts. The pH of water was slightly alkaline, in the range of 7.66–7.90. Other parameters such as alkalinity was about 5.2–5.7 mval L^{-1}, acidity about 0.23–0.37 mval L^{-1}, conductivity ranged from 586 to 597 μS cm^{-1} and the hardness of water was 180 $mgCaCO_3$ L^{-1}. The water was also unchlorinated. In order to degas (especially to remove small gas bubbles that could impede the interpretation of results), the sampled water was stored for at least 20 h at a temperature of 3°C–8°C. Nevertheless, measurements were carried out at a temperature of 8.5°C–9.5°C to reflect natural conditions of groundwater intake.

In the study, iron-based coagulant PIX 112 (produced by Kemipol in Police), that is, an aqueous solution of iron (III) sulfate $Fe_2(SO_4)_3$, was applied [21]. The coagulant was diluted to the concentration of 6.4 and 24.0 mgFe L^{-1}. Solution was prepared immediately before the beginning of tests. Measurements were conducted for the following coagulant doses: 2.0, 4.0, 7.0 and 10.0 mgFe L^{-1}.

Coagulation process was carried out in cylindrical vessel (reactor) of working volume of 6.0 L, equipped with a paddle stirrer. The reactor was placed in the water bath and filled with degassed, tap water. When the temperature became constant, exact dose of coagulant was added to the water. The fast stirring lasted 30 min at a rate of 50 rpm. Subsequently, revolutions were reduced to 17 rpm and flocculation process was conducted for 40 min. Particles sizes' measurements were carried out online every 16 s during first 5 min. Next, they were measured every 5 min.

To measure sizes of particles formed during flocculation process, the laser diffraction particle size analyzer Mastersizer Hydro 2000MU (Malvern Instruments Ltd.) was used [22–25]. The measuring range of the analyzer is between 0.02 and 2,000 μm. The instrument provides results as the particle size distribution, which illustrates percentage share of particles with equivalent diameter in relation to the total volume of all particles in the sample (by volume).

The measurements were taken with following settings: Particle = Default, Refractive index = 1.520, Absorption index = 0.1; Dispersant = Water, Refractive index = 1.330; Analysis model = General purpose; Sensitivity = Normal.

Measurement time for each laser light equaled 5 s, whereas single measurement took in total approximately 16 s.

Therefore, the time measurement accuracy of flocculation process was 16 s. For the purposes of graphical presentation of the results, it was assumed that the result was obtained for 10 s after the beginning of the particular measurement.

The standard measurement methodology using the laser granulometer for simultaneous mixing and sampling requires the use of integrated set, which consists of analyzer and mixing system. The mixing system includes beaker with a capacity of 1.0 L and automatic stirrer (with adjustable stirring speed) connected with centrifugal pump, which delivers a sample to the analyzer. The sample, after passing the measuring cell, goes back to the beaker (water with suspended solids flows in the closed circuit). However, all attempts to measure post-coagulation particles sizes revealed that even at the lowest stirring speed (600 rpm), larger flocks were broken up. During such stirring, the maximum flocks sizes reached approximately 30 μm (d50). Therefore, they were over eight times smaller than formed with the use of separate slow-speed stirrer. For this reason, the mixing system integrated with analyzer was only used to measurements within the first minute of coagulant dosing (mainly during fast stirring).

In order to carry out particles sizes measurement, it was necessary to modify the test bench. Flocculation process was carried out in a separate vessel (reactor) with a working capacity of 6.0 L equipped with a paddle stirrer. Moreover, the reactor was placed in the water bath to maintain constant, low temperature during the test. Analyzed water was collected by peristaltic pump to the granulometer measuring cell. The pump was installed behind the cell, so that its working did not influence on flocks sizes. Pump capacity was 91 cm^3 min^{-1} and the maximum water flow rate in supplying conduits to the measuring cell did not exceeded 0.085 m s^{-1}. After passing through the cell, the sample of water with solid suspend was not recirculated (except for the first 5 min of stirring). It was assumed that larger flocks were susceptible to disintegration, while flowed through peristaltic pump. Moreover, if such small particles got to the reactor again, they could disrupt subsequent measurements. Total volume of water sampled during measurement was, on average, 0.5–0.6 L. Thus, it did not exceed 10% of the total reactor volume.

The strength of the signal from the particles formed in the coagulation process (especially in the initial phase of the flocculation and at lower doses of coagulant) was weak. Thereby, apart from data directly connected with tested particles, data from background (background means the raw water) often appeared in measurements. Therefore, for processing data from analyzer, special prepared Excel spreadsheet was used. Preliminary analysis of the data relied on manual removal of data from the background. Subsequently, for the "cleaned" data particle size distribution graph was made. In addition, the characteristic values (such as d10, d50, d90) and the smallest as well as the largest observed particles were determined. The d10 diameter is defined as equivalent diameter of particles which represent 10% of the total volume of all measured particles. Values d50 and d90 can be similarly interpreted.

It should be noted that each time the term dimension or size of particles/flocks is mentioned in this study, it means the equivalent diameter of the particles, which value is the average of the measurement interval with the range of approximately ±7% of given value.

3. Results and discussion

Obtained results show that the basic factor, which determined the flocculation process and sizes of formed flocks was coagulant dose and thereby, the initial concentration of iron (III). Rate at which flocks enlarged their sizes progressively raised with increasing dose of coagulant.

The particle size increase in time, from the first moment of coagulant dosing, is presented in Figs. 1 and 2. Already in the first seconds of dosing, the smallest particles of flocks appeared in the solution, what confirmed conversion of iron into insoluble form. Regardless of the coagulant dose in the range of 4.0 to 10.0 mgFe L^{-1}, dimensions of those particles were similar and generally reached from 0.1 μm to almost 0.5 μm (accordingly 0.11–0.13 μm (d10) and 0.33–0.38 μm (d90)) (Figs. 1 and 2). For lower doses of coagulant (2.0 mgFe L^{-1}), particles had dimensions much smaller than 0.1 μm and starting from 0.03 to 0.04 μm (Figs. 3–5). In successive seconds (17–33 s after dosing of coagulant) flocks of dimensions >1.0 μm began to form in the water for all coagulant doses, while smaller flocks (of a size smaller than 0.5–0.6 μm) still remained in the solution. Then, a steady growth of flocks followed and for doses ≥4.0 mgFe L^{-1} flocks reached sizes of 250–260 μm (d50) (Fig. 1). As the time process reached a steady state, at which disintegration of flocks equaled their agglomeration, speed of flocks enlargement and time after which they achieved maximum sizes depended on iron (III)

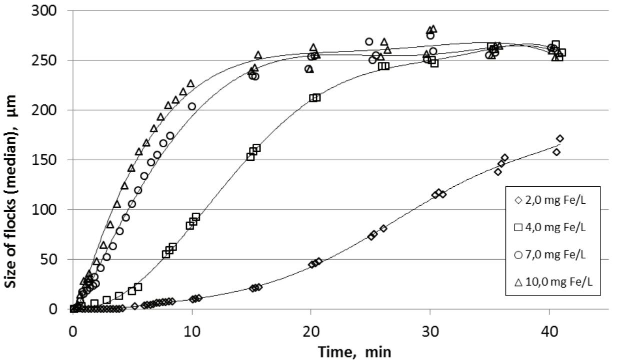

Fig. 1. The average sizes of iron hydroxide flocks (median) during flocculation for different doses of iron (III) sulfate ($Fe_2(SO_4)_3$).

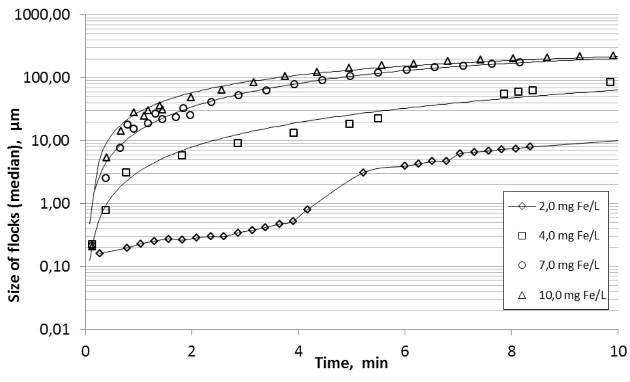

Fig. 2. The average size of iron hydroxide flocks (median) during first minutes of flocculation for different doses iron (III) sulfate ($Fe_2(SO_4)_3$).

sulfate dose. With increasing dose of coagulant, flocks faster enlarged their dimensions and faster reached maximum sizes. At the dose of 4.0 mgFe L^{-1} the time was about 30 min, whereas at the dose of 10 mgFe L^{-1} it was about 15 min. Only for the dose of 2.0 mgFe L^{-1} despite over 40 min mixing, flocks achieved sizes about 160–170 μm (d50) and their growth was still observed. In this case, however, also after ~35 min of mixing first signs of slowdown in growth of flocks sizes were observed. Probably in this situation the reason was not the dimensions of flocks (and their disintegration as a result of mixing), but decrease in number of particles caused by lower amount of iron compounds in water. Since d50 value of flocks exceeded 10 μm, the ratio between d10 and d50 value as well as between d90 and d50 was almost constant for all coagulant doses. For the majority of measurements, particles with d10 diameter were 2.0–2.1 times smaller than particles with d50 diameter. On the other hand, particles with d90 diameter were 1.8–1.9 times larger than particles with d50 diameter. Simplifying, we can say that particles with d10 diameter were twice smaller, whereas particles with d90 diameter were twice larger than particles with d50 diameter.

The important observation during granulometric studies in the initial phase of flocculation was persistence of double particle size distribution, which is shown in Fig. 3. Except for curve which illustrated particle size distribution of larger dimensions, for a certain time there was maintained a separate curve that depicted the particle size distribution of very small dimensions. Mentioned sizes were similar to those which occurred in the first few seconds after coagulant dosing. These data were verified by filtering small volumes of solution sampled at about 20 s and sampled at 1.5 min after dosing of coagulant (at a dose of 2.0 mgFe L^{-1}). Filtration was carried out by membrane filters with a pore size of 1.2 μm. Obtained proportions of iron concentration in permeate in relation to the total concentration of iron in water before filtration were similar to the percentage share of particles smaller than 1.0 μm measured with granulometer.

Based on the obtained results, the course of flocculation process during the initial phase of the process can be deduced. Only a few seconds after dosing of coagulant small particles with dimensions from 0.1 μm (for dose 2.0 mgFe L^{-1} from 0.03 μm) to 0.5–0.6 μm occurred in water. In the following few seconds, the majority of particles aggregated into larger flocks usually exceeding size of 1.0 μm (from about 0.8 μm up to 1.0 μm) which progressively increased their dimensions. Despite regular growth of flocks during initial phase, the smallest particles still remained in the solution and they were consequently grouped into larger agglomerations. This process lasted from few seconds to few minutes and depended on coagulant dose. Consequently, the smaller was the coagulant dose, the longer was the time of smallest particles permanence.

Fig. 4 presents percentage share (by weight) of particles with sizes smaller than 1.0 μm, depending on the time of coagulant dosing and different dosages of coagulant. For higher doses (7.0 and 10.0 mgFe L^{-1}) particles smaller than 1.0 μm remained no longer than 1 min. However, for smaller doses of coagulant this time elongated significantly. For the dose of 4.0 mgFe L^{-1} it was about 2.5 min. Whereas for the dose of 2.0 mg L^{-1}, 4.0 min after dosing of coagulant, half of the weight of dosed iron still remained in the form of particles smaller than 1.0 μm (particles smaller than 1.0 μm were half of the weight of all particles). They completely disappeared after 7.0 min. It must be also observed that at this time the smallest particles could have sizes smaller than 0.1 μm (Fig. 3. and 5(b)).

The obtained results of particles sizes measurements could be useful to optimize flocculation process and design reaction chambers as well as slow stirring conditions. Slow stirring time should be dependent on coagulant doses which were applied. For the smallest doses, approximately 2 mgFe L^{-1}, even after 40 min of slow stirring flocks did not reach maximum sizes. However, for larger doses of the coagulant it was favorable to shorten the slow stirring time. For doses 4.0 mgFe L^{-1} and 7.0 mgFe L^{-1} the time was appropriately about 30 and 20 min. For high coagulant doses (10 mgFe L^{-1}) flocks reached maximum sizes after about 15 min of slow stirring. In this case, further extension to the mixing time did not result in an increase in flocks sizes. Moreover, it did not cause reduction in the amount of the smallest flocks or increase in their dimensions. However, it should be noticed that after achieving steady state, further extension of flocculation time did not cause the decline in effects of flocculation or decreasing in flocks dimensions. Furthermore, too short stirring time might have significant influence on effects of post-coagulant particles retention, also in case of membrane processes such as microfiltration and ultrafiltration. For coagulant doses lower than 4.0 mg Fe L^{-1} slow stirring time should not be shorter than 6–7 min, because then small particles may still remain in water. These particles

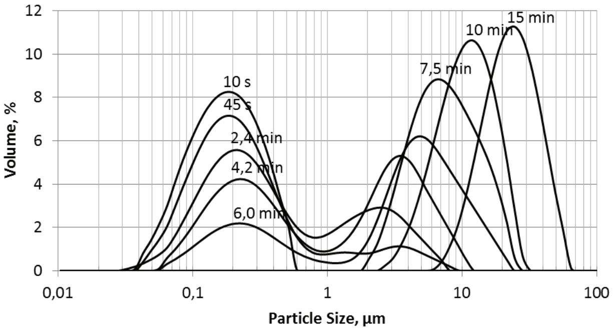

Fig. 3. Particle size distribution (by volume) of iron (III) hydroxide for coagulant dose 2.0 mgFe L^{-1} at different flocculation times.

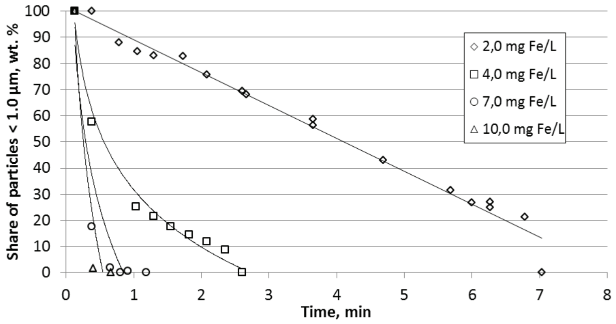

Fig. 4. Percentage share of particles (by weight) of a size smaller than 1.0 μm, depending on the time of coagulant dosing and different dosages of coagulant ($Fe_2(SO_4)_3$).

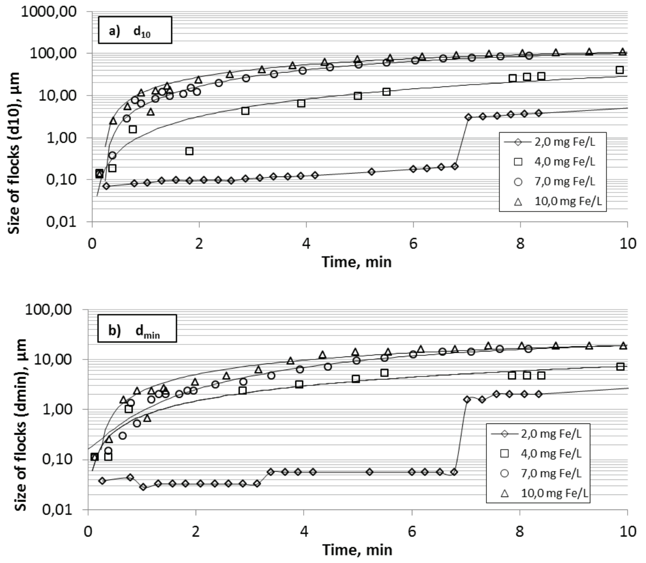

Fig. 5. The smallest dimension of iron hydroxide flocks (d_{10} and $d_{\min}$) during first minutes of flocculation for different doses of iron (III) sulfate ($Fe_2(SO_4)_3$): (a) d_{10} (b) $d_{\min}$.

may pass into the permeate and thus cause an increase of iron concentration in permeate. It may also lead to irreversible blocking of membrane pores.

From the perspective of particles separation in the filtration processes (both, conventional and membrane filtration) the most important are not only large particles, but also the smallest ones – especially their quantity. Obtained results showed that during flocculation process (regardless of the large agglomerates formation) significantly smaller particles could remain in water for a long time too (Figs. 4, 5(a) and (b)). They corresponded to the size of particles formed in the first few seconds after coagulant dosing (with dimensions smaller than 1.0 µm and often ranging approximately 0.1 µm and less). In case of small doses of coagulant, this time might be as long as several minutes (Figs. 3, 4, 5(a) and (b)). Although the percentage share of the smallest particles was not high (depending on the flocculation time and coagulant doses, for particles with 0.1 µm sizes and smaller ones it accounted less than 10% by weight of all particles), their presence could lead to irreversible blocking of membranes (microfiltration and as well as ultrafiltration membranes with the largest pore diameters).

4. Conclusions

Particles sizes measurements conducted with the laser granulometer enable observation of flocculation process. Only a few seconds after dosing of coagulant small particles with dimensions from 0.1 µm to approximately 0.5 µm (for dose 2.0 mgFe L^{-1} – from 0.03 µm) occurred in water. Then a steady growth of flocks to the size of 130–500 µm followed. At the time, process reached a steady state at which disintegration of flocks equaled their agglomeration. Speed of flocks enlargement and time after which they achieved maximum sizes depended on iron (III) sulfate dose. With increasing dose of coagulant, flocks faster enlarged their dimensions and faster reached maximum sizes. Despite regular growth of flocks during initial phase, smallest particles still remained in the solution. This process lasted from few seconds to few minutes and was dependent on coagulant dose. Consequently, the smaller was the coagulant dose, the longer was the time of smallest particles permanence.

Flocculation time might have significant influence on effects of post-coagulation particles retention, for both, microfiltration and ultrafiltration with larger pore diameters. In terms of coagulant doses below 4.0 mgFe L^{-1}, slow stirring time should not be shorter than 6–7 min. Otherwise, small particles, which are more likely to pass to the permeate (which may cause undesirable increase of iron concentration in the permeate), would remain in water. This situation may also intensify irreversible pore blocking effect.

Particles sizes analyses carried out using the laser granulometer could be useful to optimize flocculation process and design reaction chambers/slow stirring. During this research, it was observed that the lower the dose of coagulant was, the longer the retention time in such systems was recommended.

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