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Evaluation of the image analysis method using statistics for determining the floc size and settling velocity in ballasted flocculation

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ABSTRACT

The global climate changes lead to more severe floods and droughts recently. Since the increase of nonpoint water contaminants, it is highly required the technology for the treatment of the high turbidity and the increased water volume in the water such as ballasted flocculation. The purpose of this study is to determine the floc size and settling velocity among the properties of the ballasted flocculation process. The appropriate number of floc samples which can be reliable in the use of the image analysis method was determined by comparing the statistical margin of error with the maximum experimental and measurement error. It was confirmed that a certain degree of reliability can be obtained at a confidence level of 95% when 40 samples are measured whereas it is identified that when the confidence level is raised to 99%, the number of samples must be 80 or more. On the other hand, it was confirmed that even if the number of samples is changed through the normal distribution density function curve using the average value calculated according to the number of samples, there was not much difference in the average value and standard deviation. In the ballast flocculation experiments of 25 and 120 μm using the number of samples set based on the reliability analysis, the floc size, and the settling velocity increased with the increase of the injection amount of the 25 μm ballast. In the case of the 120 μm ballast, the floc size did not increase significantly, but the average settling velocity increased as much as that of 25 μm ballast. This result implies the probability of the change of floc density.

Keywords: Water treatment; Ballasted flocculation; Image analysis; Statistical analysis

1. Introduction

The global climate changes lead to more severe floods and droughts recently. Since the increase of nonpoint water contaminants of the flood season causes the turbidity soaring and the occurrence of the green algae, it is highly required the technology for the treatment of the high turbidity and the increased water volume in the water [1].

Among coagulation technologies, which are a technique to remove colloidal particles in water by increasing the size of the flocs, the ballasted flocculation usage has advantage of increasing the treatment efficiency of the pollutants through the reduction of the area and the increase of the floc settling velocity [2–4]. The main purpose of the ballasted flocculation is to increase the settling velocity by increasing the diameter and/or density of the floc by injecting a ballast such as microsand, sludge, and magnetite [5]. It is necessary to analyze the relationship between the change in the floc size and the settling velocity, since the growth of the aggregates during the floc-forming process could involves a reduction of the floc density [6]. Since the formed flocs generally have different sizes, the distribution of the floc sizes is very important for understanding the settling characteristics of the flocs.

Compared with laser-based analysis of nanosized particles using conventional static and dynamic light scattering [7], and particle analyzer with high equipment cost and difficult to field measurement [4,8], the newest measurement

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techniques for the floc size distribution is image-based analysis using optical devices [9], which has advantages that it is easy to measure particles with a size of several micrometers, the price of the equipment is reasonable, and field measurement is acceptable. The microscopic image-based method has the advantage that the individual particles can be seen at a high magnification, and the characteristics of the floc shape and internal structure can be analyzed. However, flocs must be fixed on a microscope slide to obtain floc images [10].

Although the method of analyzing images using a charge coupled device (CCD) camera is limited by the concentration of the particles in the suspension and observed only in a two-dimensional form, the external influence can be ignored and it is measured in the wide range (several μm to several mm) [11]. In addition, it is very effective in simultaneously analyzing the diameter and settling velocity of the floc since a CCD camera enables continuous image acquisition [12–14]. However, since this method observes only a part of the whole floc, it is difficult to accurately identify the size distribution of the floc, so it is important to ensure the representativeness of the observed samples [15].

Therefore, in this study, the floc characteristics of ballasted flocculation were analyzed by image-based method using CCD camera. To ensure a representative of the distribution of diameter and settling velocity, the appropriate number of floc samples was determined statistically in the 90^, 95%, and 99% of confidence interval.

2. Materials and methods

2.1. Laboratory experiments of ballasted flocculation

2.1.1. Properties of ballasts

Among the commercial ballasts, magnetite has been reported to be hydrophobic, strong against wear, high specific gravity and reusable [16]. It is also known that the floc settling velocity is high due to the high density. In this study, the two types of magnetite-based ballast (Bioneer Co., Daejeon, Korea) were the silica coated and negative charged 25 μm (–39.7 mV, density 2.89 g/mL) manufactured by the reactions of Eqs. (1) and (2), and the no charged 120 μm (density 4.87 g/mL) manufactured by the reaction of Eq. (1), respectively. Table 1 and Fig. 1 show the properties and the scanning electron microscope (SEM) images of 25 and 120 μm ballasts.

$$2FeCl_3{\cdot}6H_2O + FeCl_2{\cdot}4H_2O + 8NH_4OH \rightarrow Fe_3O_4(\text{magnetite}) + 8NH_4Cl + 6H_2O \quad (1)$$

$$\text{Magnetite} + \text{TEOS} + CH_3CH_2COOH \rightarrow \text{Silica magnetite} \quad (2)$$

In the aspect of usability in water treatment, a risk by the Fe dissolution was tested by sampling the solution every week from the stirring tank applying a bare ballast for 1 month.

Table 1
Characteristics of ballast (25, 120 μm)

Items	Ballast #1	Ballast #2
Diameter (μm)	25	120
Density (g/mL)	2.89	4.87
Formula	Fe_3O_4/SiO_2	Fe_3O_4
Surface charge (mV)	–39.7	No charge

2.1.2. Formation of ballasted flocs in the static mixer

We used a static mixer known to be capable of complete mixing and shortening of residence time [17,18] (Fig. 2). Table 2 shows that the condition that the mixing efficiency with the ballast and the use of the chemical can be reduced through the increase of the mixing strength. The linear velocity was 0.6–1 m/s, the velocity gradient (*G* value) and the GT value were more than 1,000 s^{-1} and 1,000, respectively, and the flow rate was 0.7 m^3/h.

The reason for applying the static mixer is to increase the *G* value that the 300 s^{-1} of *G* value in the conventional mixing method is not enough to completely mix the ballast having a large specific gravity. The static mixer is to increase the *G* value, because the 300 s^{-1} of *G* value in the conventional mixing method is not enough to completely mix the ballast having a high specific gravity. In addition, Francois [19] showed that a mixing rate of 1,000 s^{-1} was the maximum *G* value for the kaolin synthetic water using aluminum-based coagulants.

The raw water was synthesized with a kaolin and adjusted to 50 mg/L. The turbidity was maintained at about 50 NTU to satisfy the criterion of high turbidity (30 NTU) or more, based on the standard for the water treatment facility in Korea. pH was adjusted to 7 using the 1 N sulfuric acid, the polyaluminium

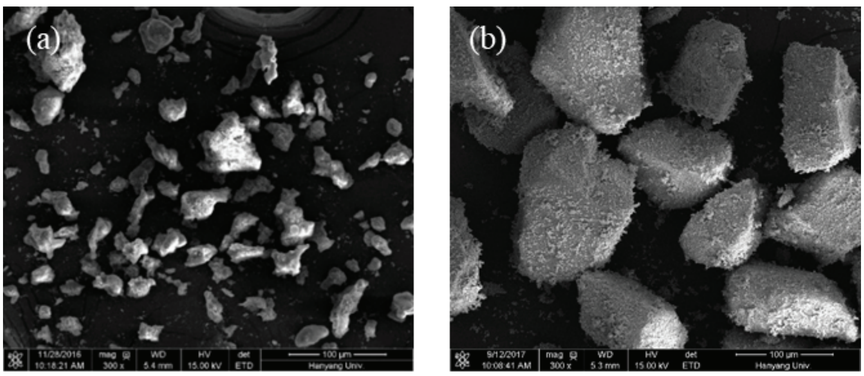

Fig. 1. Results of SEM image: 25 μm ballast (a) and 120 μm ballast (b).

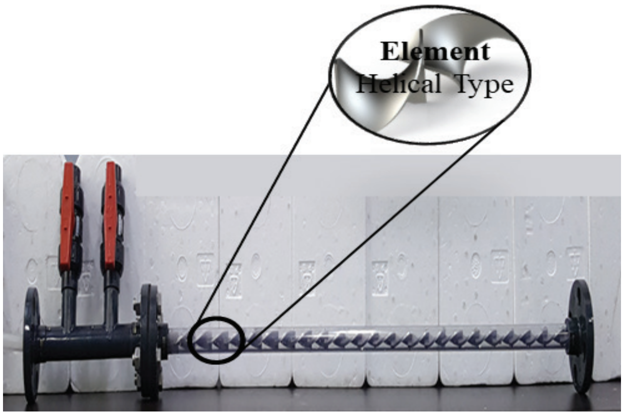

Fig. 2. Mixing device (static mixer).

Table 2
Mixing condition and specification of static mixer

Division	Division	Static mixer
Specification	Q (m^3/h)	0.7
	Temperature (°C)	25
	D (m)	0.016
	L (m)	0.96
	V (m^3)	1.95×10^{-4}
Mixing condition	Linear velocity (m/s)	0.97
	G value (s)	1,280
	GT value	1,280
	Retention time (s)	1.00

chloride coagulant (Al_2O_3 10%) was injected to be 30 mg/L estimated by the preliminary jar test (data not shown), and the ballast was injected at 25, 50, 75, and 100 mg/L with a 25 μm negative charged ballast (zeta potential –39.7 mV, specific gravity 2.89) and 120 μm no charge ballast (specific gravity 4.87), after obtaining the appropriate number of samples from statistics analysis. The diameter and settling velocity of the floc formed after a rapid agitation (3 s) with a static mixer and a settling (3 min) at a small basin were evaluated.

2.2. Image analysis device and methods

2.2.1. Image analysis devices

Images were acquired by the CCD camera having 23.7 frames per second (Mako G-507C PoE, NeXber, Anyang, Korea), and the recording software (STP-6-S-STD). The lens can be zoomed from a minimum of 0.7 times to a maximum of 4.50 times, and the white back light was provided by a light emitting diode lamp. Fig. 3 shows the schematics of the image analysis devices which are composed with three sections. The column section induces the floc settling, the CCD camera section captures the image of the floc, and the computer section records and analyses the images.

The image analysis procedure is shown in Fig. 4. In the image analysis devices, the floc images passing through the column are recorded as image or movie files in the computer, and then the area of the pixel per floc is calculated using a

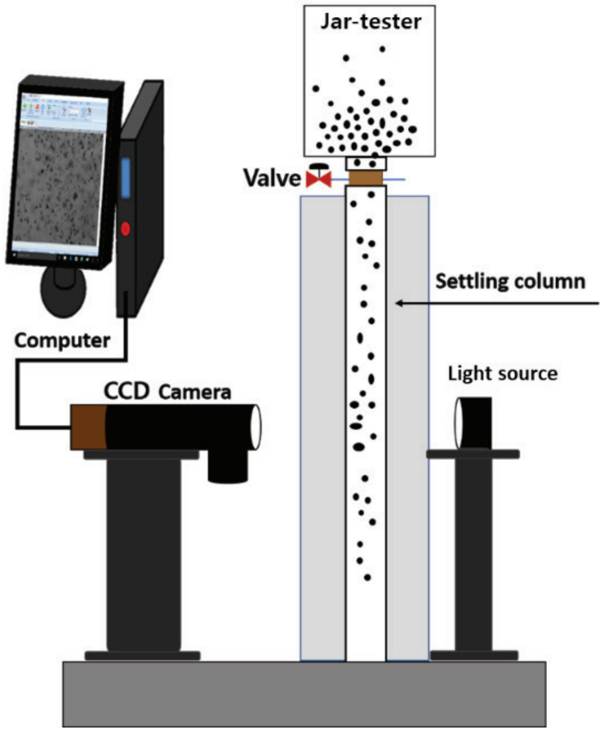

Fig. 3. Schematic of image analysis devices.

public domain software (ImageJ). The number of pixels in the projection area of floc was multiplied by the factor converting to the actual area. In this experiment, the factor was 22 times per pixel.

Finally, the diameter of the floc was approximated by the square root of the spherical equivalent area [13,20] as shown in Eq. (3).

$$d = \sqrt{\frac{4A}{\pi}} \tag{3}$$

where d is the particle diameter and A is the actual area calculated from the projection area.

2.2.2. Identification of floc

The boundary of the floc image captured by the CCD camera becomes unclear, because of the influence of the length between the camera and the light source, and the position of the floc in the settling column. Particularly, since the actual area of the floc is calculated using the image, it is necessary to control the focus of the floc interface. In this study, the floc interface was controlled by using the function of noise remover and threshold process in ImageJ software. As shown in Fig. 4(a), the noise reduction function clarified the interface of the floc by switching the background of the images to white. When the settling process occurs, the floc image distortion generated by the position difference in the column is first improved to reduce the error when calculating the area of the floc.

The threshold process was used to improve the nonfocused images of the flocs once again after controlling the interface of the floc through the noise reduction function [11].

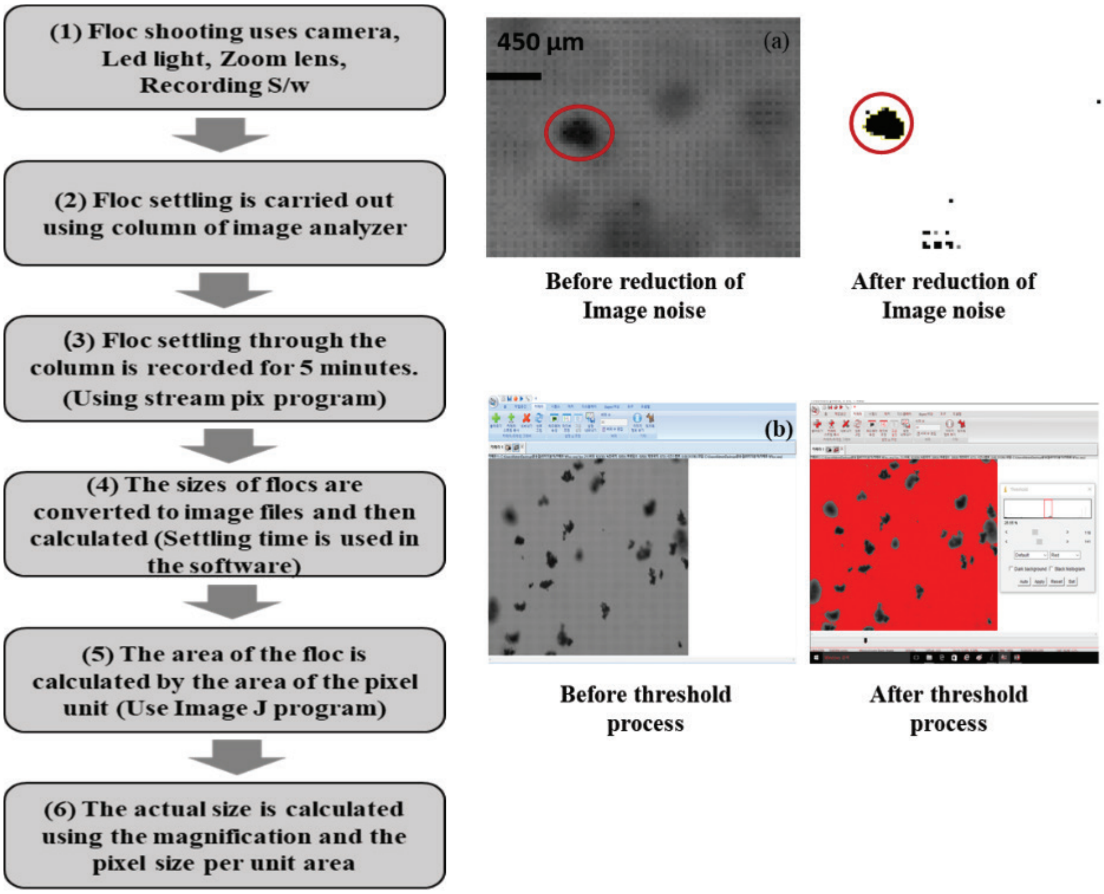

Fig. 4. Procedure of image analysis: image noise process (a) and threshold process (b).

Using the threshold function, almost all the particles in the image can be identified, regardless of the degree of focus, except for very blurry flocs, as shown in Fig. 4(b).

2.3. Image sample size determination using statistics

2.3.1. Comparing the margin of error with the number of samples

We evaluated the appropriate number of samples that can offer the reliability of the floc size distribution for the floc image analysis at the 90% (1.645), 95% (1.96), and 99% (2.58) of the confidence level (interval). As discussed in section 1, because the image analysis has the disadvantage of observing only a part of the whole floc, it is difficult to accurately identify the size distribution of the floc.

The reliability of the results of a sample is evaluated using a sample error value, which refers to the difference between the estimate obtained from the sample and the true value of the population, but only partially investigating the sample without examining the entire population. This is called the tolerance or the margin of error as shown in Eq. (4).

$$\text{Margin of error} = Z_{\alpha/2}\sqrt{\frac{\delta^2}{n}} \tag{4}$$

where $Z_{\alpha/2}$, δ, and n represent the confidential interval, the standard deviation, and the sample number, respectively.

In this study, the number of samples for reliability analysis was changed with 30, 40, 80, 120, 160, 200, and 240. Since experimental and measurement errors in our experiment were, respectively, estimated to be below 10%, the maximum 20% error was considered as the minimum requirement of reliability analysis and compared with the margin of error.

2.3.2. Evaluating the image analysis results with the normal distribution curve

As another reliability test, we evaluated whether the floc diameter and settling velocity according to the number of samples sufficiently express the normal distribution.

Since the normal distribution is a distribution determined by the mean μ and the standard deviation σ, which means scatter, the shape of the probability density function from the observations about diameter and velocity is plotted as a function of the bell shown in Eq. (5).

$$\text{Probability density function, } f(x) = \frac{1}{\sigma\sqrt{2\pi}}\exp\left(-\frac{(x-\mu)^2}{2\delta^2}\right) \tag{5}$$

where μ and x represent the mean value and the value of sample, respectively.

The important nature of the normal distribution lies in the central limit theorem. The central limit theorem explains that the sample mean follows the normal distribution, whatever the data is from. This is important to know where the center of the data is located. In order to know the center of the data, the sample mean is obtained. When the size of the data increases due to the central limit theory, it follows the normal distribution. Therefore, various statistical processes for grasping the center of the data are easy. According to the central limit theory, since the distribution of the sample mean approaches the normal distribution as the sample size increases, the mean and standard deviation of this normal distribution are equal to the sample mean (population mean) and sample standard deviation (population standard deviation). Therefore, it is possible to grasp the population mean and standard deviation by analyzing the sample mean and standard deviation according to the change in the number of samples.

3. Results and discussion

3.1. Laboratory tests of ballasted flocculation process

Fig. 5 shows the changes in the average diameter and settling velocity by the injecting amount of 25 and 120 μm ballast. It was found that the average diameter and settling velocity of the floc increased in proportion to the amount of the ballast increased. By the result of comparing the floc diameters, the diameter of a typical floc without a ballast was about 600 μm, whereas the floc diameter with 25 and 120 μm ballasts injected at a dosage of 100 mg/L was 1,200 and 900 μm, respectively. At the 100 mg/L of injection, the settling velocity was about 18 m/h without ballast, whereas it was the 43.2 m/h for 25 μm and the 43.0 m/h for 100 μm with ballasts, respectively. By the result of the Fe dissolution test, the concentration of Fe ion in the solution was about 0.01 mg-Fe/L, which satisfied the Korean water standard of 0.3 mg/L or less for both ballasts (data not shown).

3.2. Statistics analysis of flocs

3.2.1. Reliability analysis of floc according to the number of floc samples

Fig. 6 shows the results of evaluating the reliability of the floc size and settling velocity by comparing the margin of error with the number of samples on 25 μm ballast.

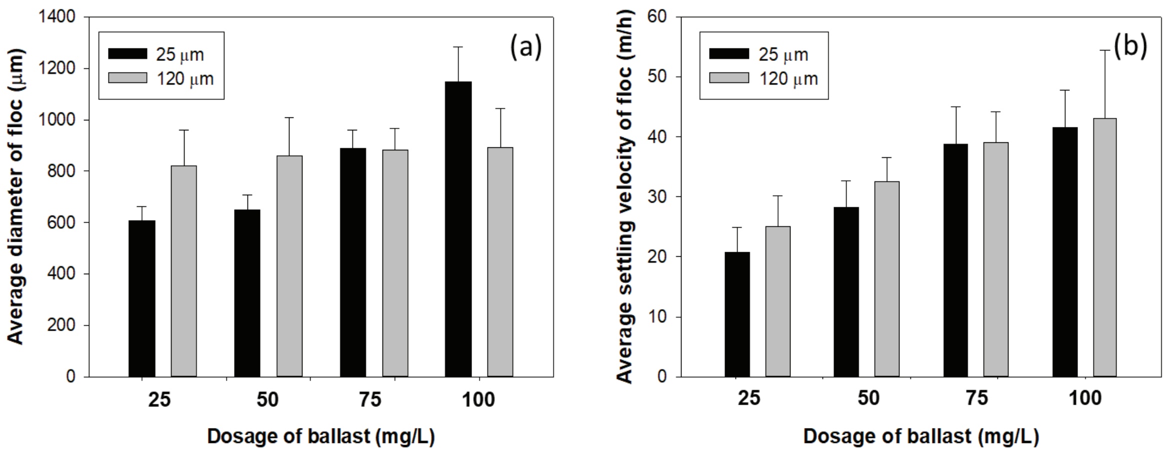

Fig. 5. Results for ballasted flocculation floc diameter (a) and settling velocity (b).

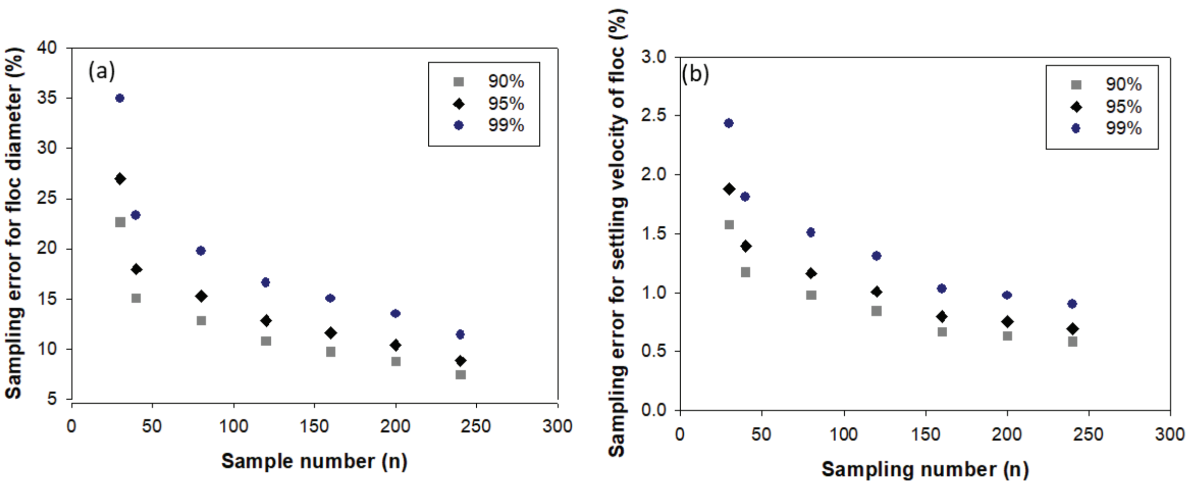

Fig. 6. Margin of error (%) according to the number of samples on 25 μm ballast: floc size (a) and settling velocity (b).

The margin of errors of the seven-different number of samples was analyzed using a 25 μm ballast at 90%, 95%, and 99% confidence level. The results show that the margin of error for the floc diameter and settling velocity is gradually decreased from 40 samples at all confidence levels. However, the error values of the settling velocity were lower than that of the floc size because the margin of error is importantly affected by the standard deviation. Therefore, the reliability analysis was compared with the margin of errors for the floc size due to the larger error. When the margin of error is below 20% at the 90% and 95% confidence level, the minimum number for testing the floc distribution is more than 40 samples, because the experimental and measurement errors are assumed to be below 20%. However, when the confidence level is increased to 99%, it is necessary to analyze more than 80 samples to satisfy below the 20% of the experimental and measurement errors. Since the 95% of confidence level is often used in the field [5], the 40 samples are enough for the image analysis.

3.2.2. Correlation between floc properties and normal distribution

Table 3 shows the average value and the standard deviation of the floc size and settling velocity. The floc size and settling velocity of the sample were 647.87 μm (±3.75 μm) and 28.35 m/h (±0.82 m/h), respectively. All values of each parameter give no huge differences according to the number of samples.

However, the normal distribution curves using probability density function (Eq. (5)) show an important difference in Fig. 7. In both cases of Fig. 7, the normal distribution curves at 30 and 40 samples are somewhat higher or lower than the average curves, and it tends to converge with increasing number of samples. In Table 3, the mean values are very similar, but for a small number of samples, we can know that there are many samples collected near the average value. This tendency can be explained by the central limit theorem, regardless of the number of samples for image analysis, showing similar average value and dispersion value.

3.3. Property changes with the amount of ballast injection

Table 4 shows the average value and the standard deviation of the floc size and settling velocity. The floc size and settling velocity of the sample were dramatically increased up to two times with the increase of the ballast dosage comparing with the result without ballast.

Fig. 8 shows normal distribution curves of the floc size and settling velocity according to the injection amount of 25 μm ballast. The average diameter and settling velocity increase with the increase of the injection amount of the ballast. As the amount of the ballasted flocculation increases, the distributions of curves were widened. After 50 mg/L of injection, the average floc size and settling velocity were dramatically increased.

Table 5 shows the average value and the standard deviation of the floc size and settling velocity. The settling velocity and floc size of the sample were increased up to 2.5 times with the increase of the ballast dosage comparing with the result without ballast. Similar to the result of using 25 μm ballast, the standard deviation value became larger as the

Table 3
Measured values according to the number of floc samples (n)

Division		Values according to the number of floc samples (n)						
		30	40	80	120	160	200	240
Diameter (μm)	Mean	644.1	648.3	648.6	648.3	648.9	649.1	648.1
	Standard deviation	75.4	60.0	69.5	71.7	74.8	75.0	69.7
Settling velocity (m/h)	Mean	27.8	28.2	28.3	28.5	28.7	28.6	28.5
	Standard deviation	5.3	4.5	5.3	5.6	5.1	5.4	5.5

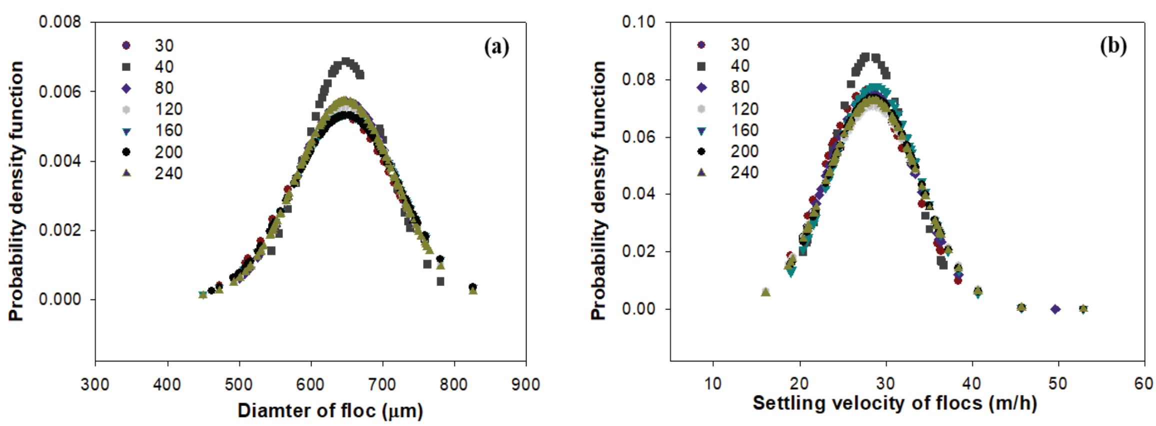

Fig. 7. Results of normal distribution of 25 μm ballast with the number of samples: floc diameter (a) and settling velocity (b).

Table 4
Values according to the amount of 25 μm ballast injection (mg/L)

Division		Values according to dosage of ballast (mg/L, 25 μm ballast)				
		0	25	50	75	100
Diameter (μm)	Mean	587.7	606.2	648.3	888.8	1,147.7
	Standard deviation	79.7	54.9	60.0	70.1	137.1
Settling velocity (m/h)	Mean	17.3	20.7	28.2	38.8	41.5
	Standard deviation	5.0	4.2	4.5	6.2	6.3

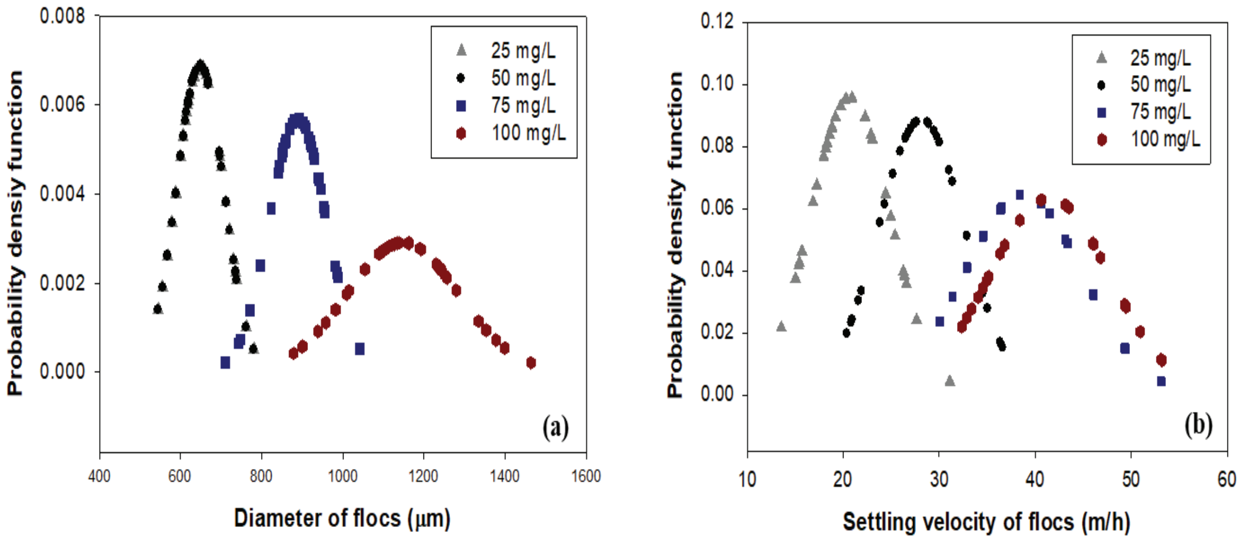

Fig. 8. Results of normal distribution of 25 μm ballast with the injection amount: floc diameter (a) and settling velocity (b).

Table 5
Values according to the amount of 120 μm ballast injection (mg/L)

Division		Values according to dosage of ballast (mg/L, 120 μm ballast)				
		0	25	50	75	100
Diameter (μm)	Mean	587.7	820.7	856.7	862.5	890.3
	Standard deviation	79.7	139.8	148.3	151.3	153.2
Settling velocity (m/h)	Mean	17.3	25.0	32.8	36.1	42.6
	Standard deviation	5.0	5.2	5.1	5.1	8.9

amount of injection increased and it was confirmed that it spreads.

Fig. 9 shows the variation of the diameter and settling velocity of the floc according to the amount of injection of 120 μm ballast. The change in the average diameter of the flocs increased with the increase of the amount of injection, but the change width was not large compared with the result using the 25 μm ballast. The shape of the normal distribution curve for the floc size is also similar with each other. On the other hand, the variation of settling velocity with 120 μm ballast was increased with increasing the amount of injection. Almost same floc sizes and the increased settling velocities imply the probability of the change of floc density.

4. Conclusion

The purpose of this study is to determine the floc size and settling velocity among the properties of the ballasted flocculation process. The appropriate number of floc samples which can be reliable in the use of the image analysis method was determined by comparing the statistical margin of error with the maximum experimental and measurement error. It was confirmed that a certain degree of reliability can be obtained at a confidence level of 95% when 40 samples are measured whereas it is identified that when the confidence level is raised to 99%, the number of samples must be 80 or more. On the other hand, it was confirmed that even if the number of samples is changed through the normal

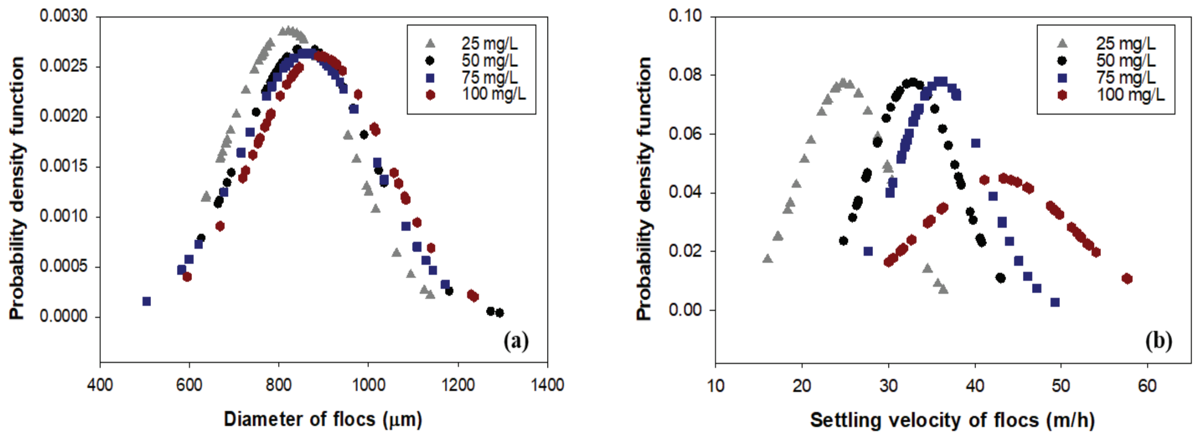

Fig. 9. Results of normal distribution of 120 µm ballast with the injection amount: floc diameter (a) and settling velocity (b).

distribution density function curve using the average value calculated according to the number of samples, there was not much difference in the average value and standard deviation.

In the ballast flocculation experiments of 25 and 120 µm using the number of samples set based on the reliability analysis, the floc size and the settling velocity increased with the increase of the injection amount of the 25 µm ballast. In the case of the 120 µm ballast, the floc size did not increase significantly, but the average settling velocity increased as much as that of 25 µm ballast. This result implies the probability of the change of floc density.

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