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論　文
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Analysis of sedimentation behavior of flocculated Na-montmorillonite in a semi-dilute regime — effects of the container height —

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Abstract: A systematic observation of the sedimentation behavior of montmorillonite suspension coagulated in 1.00 M NaCl solution was made using glass cylinders of different heights in the semi-dilute regime which starts from a very slow rate followed by gel collapse ending slow consolidation. Intensive analysis was done for the maximum interface settling rate (Q) as a function of the volume fraction of montmorillonite (ϕ) using cylinders of different heights. The velocities were plotted against the reciprocal height of the cylinder to obtain the velocity of infinite height as a function of the volume fraction of montmorillonite. On the basis of Richardson-Zaki's equation; $Q = Q_f(1-\alpha\phi)^{4.65}$, the swelling ratio of α, and the velocity of zero limit dilution (Q_f) were obtained from the slope of the plot and the intercept of the abscissa, respectively. Using the obtained values of α and Q_f, the representative diameter of the floc, d_f, is determined by the application of Stokes' Law. The obtained values of Q_f and d_f were confirmed to be consistent with the result of the measurement of the settling velocity of a single floc against diameter independently determined by the method of Two-way camera.

Key Words : flocculated suspension. Na-montmorillonite, settling velocity, container height, sedimentation turbulence

1. Introduction

Colloidal materials and soil exist throughout aquatic environments. Their sedimentation behaviors are closely related to the solid-liquid separation process such as water treatment, and the transportation of the cohesive sediment in rivers and estuaries (Adachi and Iwata, 2003; Chassagne, 2019). Although these substances are found to be aggregated flocs and they play an important role in the transport in aquatic environments, the settling behavior is hardly noticed. In paddy field agriculture, this phenomenon is seen in the plowing process (Adachi, 2019). The fine sediment discharged from the surface of the paddy field by the excess water moving to the river raises the water turbidity in the downstream region (Wakai et al., 2005; Sudo et al., 2009). On the other hand, in the process of irrigation, the cohesive sediment tends to be temporarily deposited in the stagnation of the irrigation drainage canal. At the time of the commencement, chemical substances derived from agricultural chemicals such as fertilizer were adsorbed on the surface of the clay particles constituting the aggregates, and polluted river water. Such contaminants may endanger the entire ecosystem (Winterwerp and Walther, 2004). In these natural water environments, most particles exist as flocs. Therefore, flocs are very important transport units rather than individual particles and may influence various environmental phenomena. Thus, the settling process of flocs under gravity is considered to be one of the most fundamental problems in the transport of chemicals.

In order to understand the sedimentation of flocculated suspension, it is necessary to correlate the hindered sedimentation to the single floc sedimentation. The velocity of a single floc settling under gravity can be derived by the force balance applying Stokes' Law:

$$Q_f = \frac{g(\rho_f - \rho_w)d_f^2}{18\mu} \tag{1}$$

Where Q_f, g, ρ_f, ρ_w, d_f, and μ, denotes the settling velocity of a single floc (mm s^{-1}), the gravitational constant (m s^{-2}), the density of the floc (g mm^{-2}), the density of the solution (g mm^{-2}), the floc diameter (mm), and the viscosity of the solution (Pa·s), respectively. In this derivation, a floc is assumed to be an impermeableand rigid sphere with

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equal projected area.

When the concentration of flocculated material increases, the settling velocity will be hindered due to the interference from the neighboring flocs. The presence of neighboring particles will increase apparent viscosity and reduce the difference in density. In the case of a solid sphere, Richardson and Zaki (hereinafter R-Z) obtained the following equation for the hindered sedimentation based on the intensive measurement using a fluidized bed of monodispersed particles (Richardson and Zaki, 1954):

$$Q = Q_{\mathrm{o}}(1-\phi)^{4.65} \tag{2}$$

Where Q denotes the maximum interface settling rate (mm s^{-1}), Q_{o} is the settling velocity of a single sphere (mm s^{-1}), and ϕ is the solid volume fraction. It should be noted that equation (2) is only targeted on mono-dispersed particles. In the flocculated suspension, the sedimentation behavior is more complicated. The formed flocs are obviously far from mono-diapered spheres. For this problem, Michaels and Bolger (hereinafter M-B) did a comprehensive analysis using the suspension of kaolinite flocs (Michaels and Bolger, 1962). They have demonstrated that R-Z's equation is widely applicable to the sedimentation of flocculated kaolinite, introducing the concept of effective volume fraction of solid which is expressed by the swelling ratio due to flocculation. It's worth noting that the flocculated material of kaolinite is far from monodispersed spheres, nevertheless, M-B demonstrated the validity of R-Z's equation. Regarded as a settling unit, floc holds its structure with the consist of primary clay particles and pore water. The solid volume fraction (ϕ) and the swelling ratio of the floc (α) can be expressed in the following relationship:

$$\alpha = \frac{\phi_{\mathrm{f}}}{\phi} \tag{3}$$

On the basis of equation (2), Michael and Bolger showed the relationship between the floc characteristics and the interference settling rate as,

$$Q = Q_{\mathrm{f}}(1-\alpha\phi)^{4.65} \tag{4}$$

Imai (1981) classified the sedimentation behavior of flocculated sediments into three regimes corresponding to the concentration of the suspension. They are dilute, semi-dilute, and concentrated regimes. In the first dilute regime, flocs settle freely interacting with each other very weakly. The motion of each floc seems hydrodynamically independent. The larger flocs settle faster than the smaller flocs, thus the interface between the formed supernatant and the region of settling floc present becomes unclear. In the second semi-dilute regime, on the other hand, the interaction between suspending flocs seems to influence the whole region generating a network-like structure. A clear interface between transparent supernatant and a region occupied by the suspended flocs can be recognized. It should be noted that suspended flocs are not rigidly connected. However, the duration of the first stage is reflected by the colloidal interaction (Wu and Adachi, 2016). The movement of the interface is very slow and after a while, the initially formed network-like structure will collapse due to gravity. This is the onset of the second stage results in a rapid rate of sedimentation which will end with the accumulation of settled flocs in the bottom. The formed bottom sediment will start to consolidate at a very slow rate which is regarded as the third stage. In the last concentrated regime, the initially formed flocculated sediment network structure settles very slowly from the beginning. The slow process is essentially the consolidation process that continues very long period. Among all, the semi-dilute regime is a very characteristic regime that reflect the presense of flocs which is very fragile. This domain is important in the sedimentation process in the esturaly (Wolanski and Gibbs, 1995). In this study, we mainly focused on the second stage of the semi-dilute regime.

Montmorillonite is a kind of alkaline clay. The flocculation of montmorillonite coagulated in NaCl solution is also suitable for the analysis of sedimentation experiments because of the easy generation of visibly big flocs. The cohesive force between particles and the resulting size of flocs under a certain shear rate can be simply controlled by the concentration of sodium chloride (Miyahara et al., 1998; Miyahara et al., 2001; Di et al., 2019). The resulting size of flocs is approximately ten times larger than the case of kaolinite (Kuroda et al., 2003; Kinoshita et al., 2017). In our previous study, we demonstrated the significant effect of cylinder heights on the rate of sedimentation by the generation of so-called sedimentation turbulence (Ghazali et al., 2020). Increasing the initial height will promote the rate of interface sedimentation due to the development of large flocs during the sedimentation period, which was regarded as the feed-forward mechanism. That is, once the collision between two flocs is taken place due to the difference of settling velocity, it will result in the formation of bigger floc. The generation of bigger floc will result in increase of rate of sedimentation thus the more frequent collision between flocs will be induced. This iteration process will be acccelrated during sedimentation process and more probable development of floc size will be predicted in this duration, settling velocity and rate of collision can be predicted in heigher settling container which allows longer

travel distance. It is necessary to investigate this effect to obatain the representative rate by the experiment under different initial height conditions.

Miyahara et al. (2001) tried to confirm the consistency of the rate of hindered sedimentation of the flocculated suspension of Na-montmorillonite with the rate of single floc sedimentation referring to the analytical method reported by M-B. However, their trial was not quantitatively successful due to the incomplete consideration of the cylinder heights. A similar trial was reported by Kinoshita et al. (2017) using flocculated materials of Na-kaolinite. They concluded the validity of the analytical method of M-B to evaluate the state of suspension. However, they are not able to conclude the consistency between the rate of hindering sedimentation and that of single flocs. As their research pointed, although many studies have been carried out to use microscopy combined with image analysis and photography to define the shape, porosity, and structure of the floc, the difficulty of measuring the settling velocity of single flocs with high accuracy still needs to be improved.

In the analysis of M-B, it was found that the settling rate is influenced by the height of the container. For this problem, they proposed the method to obtain the settling velocity of sedimentation by considering infinite container height (from the plot of the results vs the reciprocal value of interface height H). That is, it is necessary to carry out the experiment as a function of container height and establish the infinite container height condition though M-B's method. In addition, to prevent the accurate analysis of single floc sedimentation, we applied the method of Two-way camera in the present study, which two cameras are positioned in orthogonal directions in the horizontal plane to obtain accurate data on settling flocs. By the application of this method, the scatter of floc diameter measured for the floc settling velocity is reduced by approximately 20–30 % evaluated by the coefficient of variation (Adachi and Daitokurin, 2020). In order to verify the effectiveness of the R-Z formula on the hindered sedimentation of flocculated Na-montmorillonite and consistency with the single floc sedimentation, we conducted systematic analysis of sedimentation behavior of flocculated suspension of Na-montmorillonaite with different height of cylinder and a single floc sedimentation using Two-way camera.

2. Materials and Methods

2.1 Sample preparation

In this study, we made flocs from the suspension of the Na-montmorillonite (Miyahara et al., 1998). Montmorillonite powder (Kunipia-F) was purchased from Kunimine Industry Co. Ltd., Japan. Montmorillonite powder was dispersed in deionized water, and the suspension was left undisturbed to remove the coarse components by sedimentation. The upper part of the dispersion was siphoned into a NaCl solution of sufficiently high concentration to replace the exchangeable cation with Na^+. Dialysis procedures were done to remove sulfur and glycerol. The dispersion was dialyzed against distilled water until the electrical conductivity of the percolate decreased to 2.00 μS cm^{-1} with water renewal. Then, the gel-like dispersion was stored in a refrigerator at 4.0 °C as the stock solution. The volume fraction of the dispersion was measured gravimetrically.

2.2 Measurement of the rate of hindered sedimentation

The relationship between interface height (H) and settling time under different solid volume fractions in 1.00 M NaCl solution is measured. Na-montmorillonite suspension and NaCl solution were gently mixed by the end-over-end method using two cylindrical measuring cylinders inverted 7 to 8 times. Through this mixing procedure, floc was generated in standard size and distributed evenly (Michaels and Bolger, 1962). The suspension was left to stand for the observation of settling behavior in circular measuring cylinders of different heights from 200 mm to 500 mm as shown in Fig. 1. The change of interface settling height H was recorded as a function of elapsed time, t. The inner diameter of the settling cylinders was 50.00 mm. Solid volume fraction (ϕ) was adjusted as 1.00 $\times 10^{-4}$, 1.25 $\times 10^{-4}$, 1.50 $\times 10^{-4}$, 1.75 $\times 10^{-4}$, 2.00 $\times 10^{-4}$ and 2.50 $\times 10^{-4}$. The lower limit of volume fraction is the limit value of the interface that can be visually obtained. The ultimate NaCl concentration was 1.00 M. During the measurement, the temperature was controlled at 20.0 °C ± 0.5.

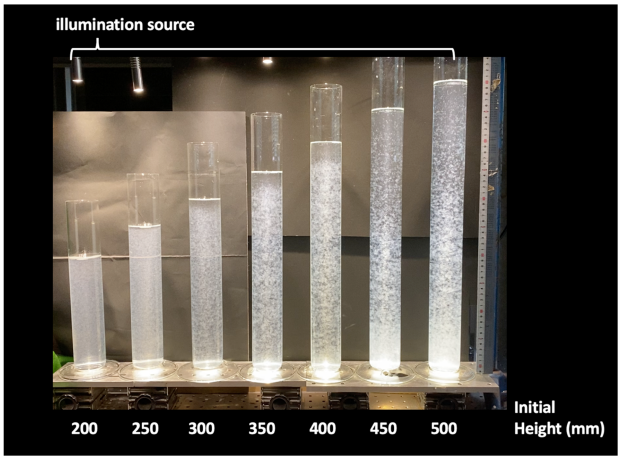


Fig. 1 Experimental setup of the hindered sedimentation measurement.

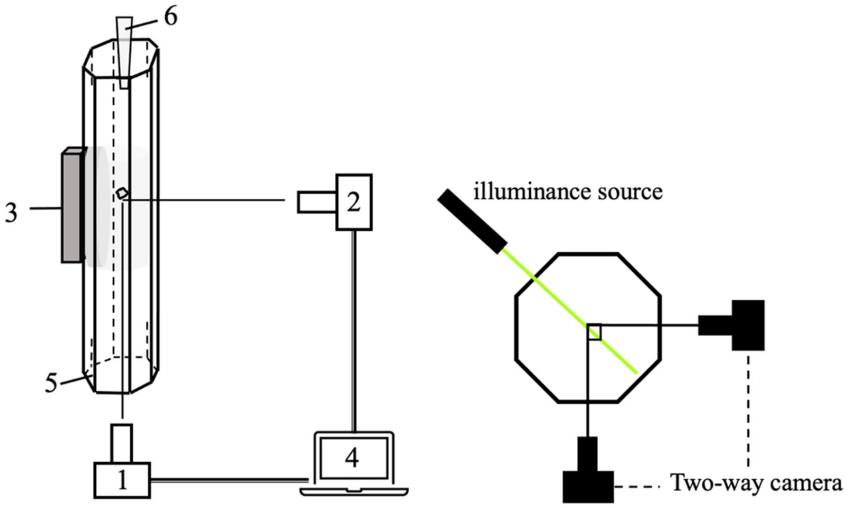


Fig. 2 Experimental setup for measuring the single floc sedimentation observed by the Two-way camera and its cross-section view. 1 and 2 are the cameras, 3 is the illumination source, 4 is the computer, 5 is the octagonal settling tube, and 6 is the floc sampler for releasing flocs.

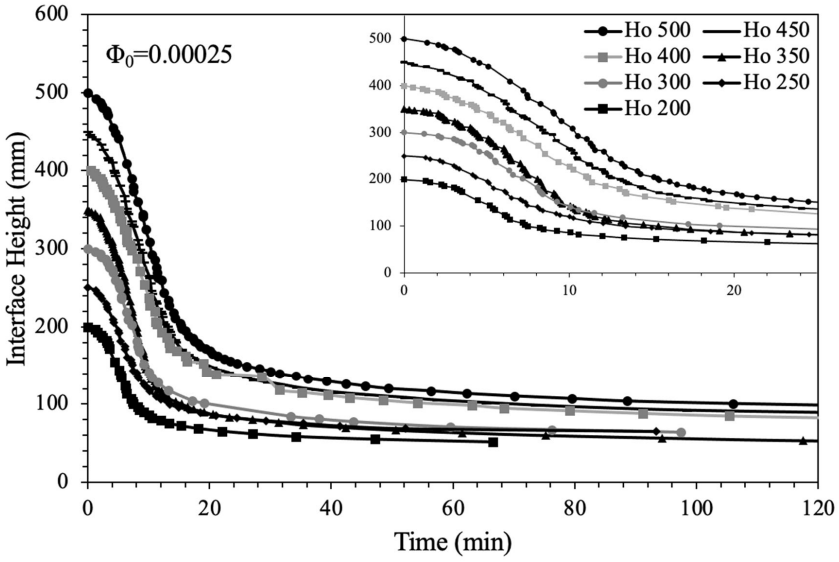


Fig. 3 Interface height as a function of elapsed time for different initial heights in the 5 cm cylinders. 1.0 M NaCl, Na-Montmorillonite 2.5×10^{-4}.

2.3 Measurement of the single floc sedimentation

In order to obtain an accurate relation between the floc diameter and the settling velocity, the single floc sedimentation experiment was conducted by applying the method of Two-way camera (Adachi and Daitokurin, 2020) installed in an octagonal settling tube (1200.00 mm Height; 80.00 mm inner diameter). The Two-way camera system consists of two CCD cameras (DU657M, Toshiba Teli Co., Japan) coupled with image processing software. Two cameras were orthogonally installed on the same horizontal plane as shown in Fig. 2. During the settling of a single floc, images from two directions were captured. The mean value obtained from the projections taken from two different directions was used for the analysis. The digital camera could acquire images with 4080-3072 pixels. DIPP macro II software (DITECT Co., Ltd., Japan), was used for the image processing. The settling rate of the single floc (Q_f) was calculated by the vertical displacement of the floc centroid moved between two points at a certain time. Floc diameter (d_f) was obtained as the diameter of a sphere with the equivalent area of the binarized projection.

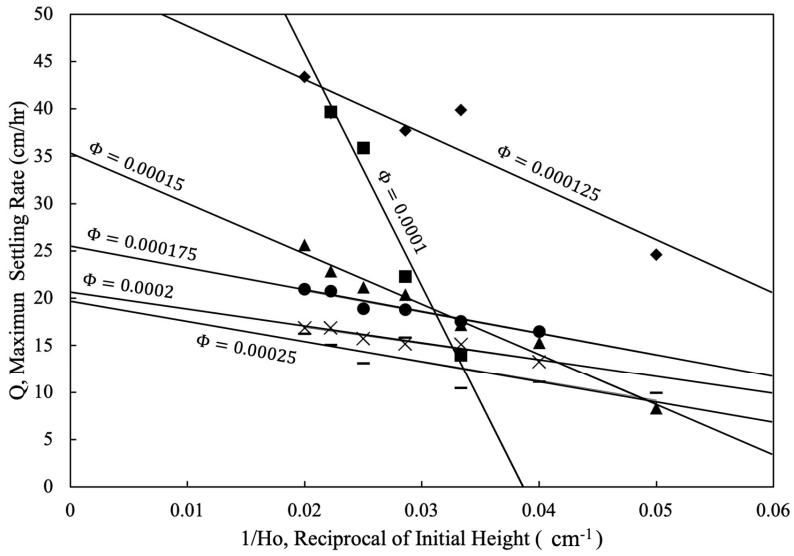


Fig. 4 Plot of maximum settling rate with the reciprocal of initial height.

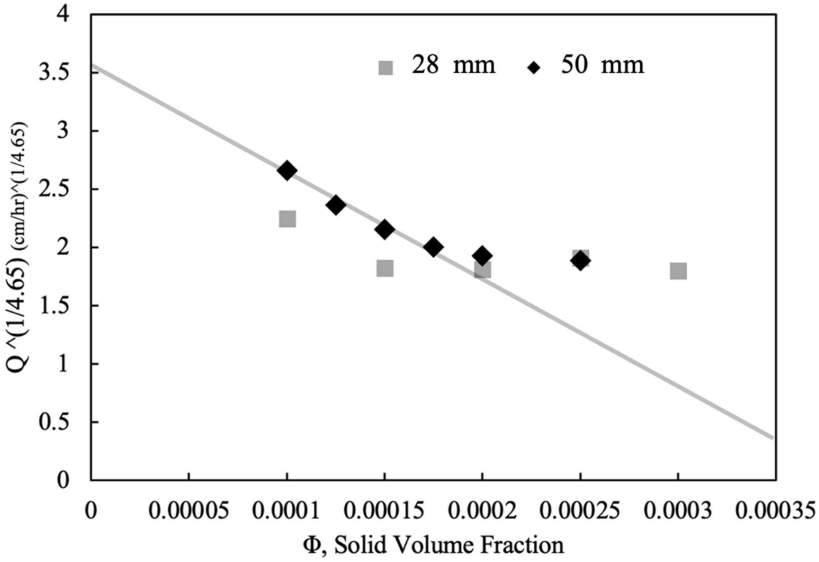


Fig. 5 Relations between the settling velocity of the interface ($Q^{1/4.65}$) and volume fraction of solid (Φ) at 1.0 mol L^{-1} NaCl concentration with inner diameter of cylinder 28 and 50 mm.

3. Results

3.1 Hindered sedimentation

Here, the result under a solid volume fraction of 2.50 $\times 10^{-4}$ was demonstrated in Fig. 3. Sedimentation curves under different initial settling heights were plotted, showing a similar trend as a sigmoidal curve, which is the descending speed of the interface changes from slow at the beginning to intense and then continues to slow as described in the introduction. The maximum interface settling rate (Q) was obtained from the slope of the relation curve between interface settling height and elapsed time during the rapid sedimentation stage. It was confirmed that the results match our earlier findings, in which the settling velocity increased linearly with an increase in the initial settling height (Ghazali et al., 2020).

The maximum interface settling rate (Q) with respect to the reciprocal of the initial settling height ($H_o{}^{-1}$) under different solid volume fractions is presented in Fig. 4. The maximum interface settling rate under infinite container height condition (Q') was obtained directly from the intercept of the tendency line. It is obvious that Q' increased sharply with the decrease of solid volume fractions from 1.75 $\times 10^{-4}$ to 1.00 $\times 10^{-4}$. In the more concentrated

cases, which are solid volume fractions under 2.00 $\times 10^{-4}$ and 2.50 $\times 10^{-4}$, Q' obtained from the data plots demonstrates a remarkable level of congruence.

3.2 A single floc sedimentation test using the method of Two-way camera

In this study, the direct measurement of a single floc settling experiment was also carried out to examine the validity of the obtained values through R-Z's modified equation. Typical images of the observed Na-Montmorillonite obtained by the method of Two-way camera are shown in Fig. 6. The floc diameters were calculated from the mean value of the two directions' projections. Our research proposes a novel approach to floc analysis by utilizing a two-way camera system to observe individual flocs from two different directions. By capturing images of flocs from multiple directions, we can obtain a more comprehensive and accurate understanding of their physical properties, including size, shape, and settling velocity. It is obvious that, even on the same floc the sizes we observed from two directions were different. This is particularly important given the complex and heterogeneous nature of flocs in water treatment processes, which can exhibit significant variation in their properties even within a single sample.

By assuming that the settling floc can be approximated as an impermeable sphere with a fractal structure, i, the number of primary montmorillonite particles composing a floc can be described as

$$i = \left(\frac{D_\mathrm{f}}{d_\mathrm{o}}\right)^{D} \tag{5}$$

Where d_0, D_f and D denotes the number of montmorillonite particles composing a floc (mm), the diameter of the montmorillonite particle (mm), and the fractal dimension, respectively. So, the swelling ratio of the floc (α) can be further expressed as

$$\alpha = \frac{\phi_\mathrm{f}}{\phi} = \left(\frac{D_\mathrm{f}}{d_\mathrm{o}}\right)^{3-D} \tag{6}$$

This equation means, that the volume fraction will increase with the increase of the floc diameter. So, equation (1) can be simply modified as

$$V_\mathrm{f} = \frac{g}{18\mu}(\rho_\mathrm{s} - \rho_\mathrm{w}){d_0}^{3-D}{D_\mathrm{f}}^{D-1} \tag{7}$$

Where ρ_s denotes the density of the montmorillonite particle (g mm^{-2}). The relationship between the floc diameter and settling velocity from the single floc sedimentation was plotted on a log-log paper in Fig. 7. The settling velocity increased with the increase of floc diameter, and the obtained relationship was confirmed to follow a power law. On the basis of equation (7), the fractal dimension was determined as 1.97 from the value of power of this relationship.

4. Discussion

4.1 Sedimentation behavior under different initial settling heights

The hindered sedimentation behavior can be characterized as a very slow flocculation stage in the beginning, followed by rapid sedimentation, and entirely comes back to

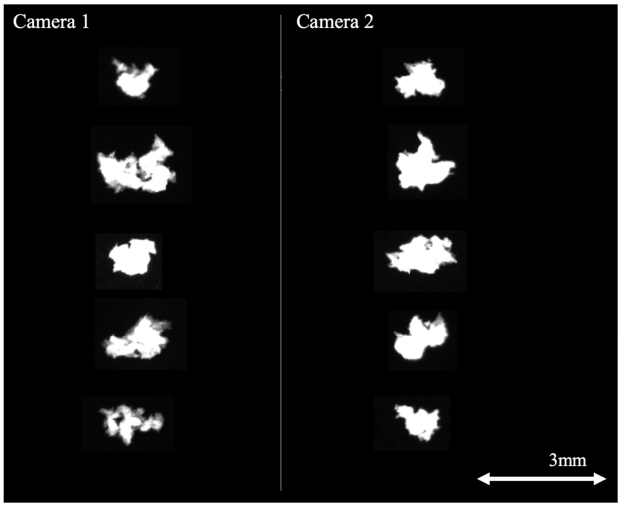


Fig. 6 Typical images of the floc projections taken from the Two-way Camera observation in the single Na- montmorillonite floc sedimentation in 1M NaCl. Here we show five sets of images, each set horizontally, of the same flocs viewed from different directions. The left column represents camera number one, and the right column represents camera number two.

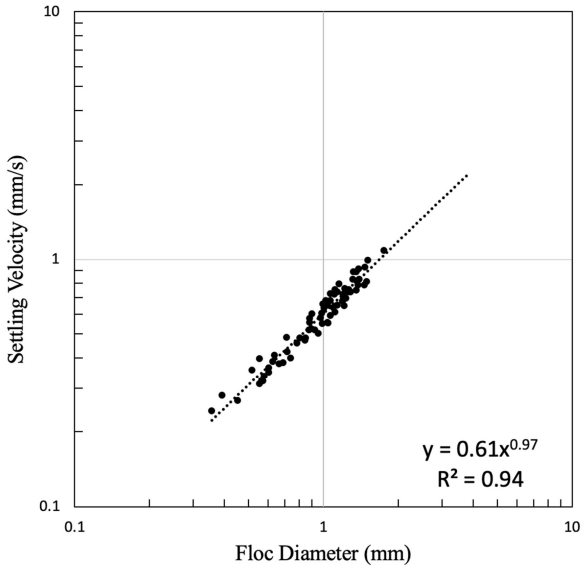


Fig. 7 Relationships between the settling velocity and diameter of a single Na-montmorillonite floc in 1M NaCl.

a very slow consolidation process. With the help of well-established visualization techniques in this study, detailed descriptions of this phenomenon could be given. The initial stage can be understood as the formation of a loosely connected network structure between flocs. However, the interface starts to move rapidly by the self-weight of the network. In the rapid settling stage, the flocs continuously transfer downward, and the liquid in the lower deposition is squeezed upward, resulting in strong convection. Then, in the third stage of the consolidation, the settling rate of the interface becomes relatively slow. This typical phenomenon of the three stages settling in the hindered sedimentation of the semi-dilute regime was clearly displayed using Na-montmorillonite suspension. Once the sedimentation starts, the whole system takes into action. As long as the settling continued the force was transferred upwards. The starting point of the rapid sedimentation period was clarified by our previous study (Wu and Adachi, 2016). To define the beginning of the consolidation stage, we can employ a straightforward procedure utilizing the standard sedimentation curve. This curve illustrates the relationship between the interface settling height and elapsed time. Firstly, we extract the fitting line from the rapid sedimentation stage, which provides the interface settling velocity based on its slope. Subsequently, we draw the fitting line representing the final consolidation stage. By determining the abscissa of the intersection point between these two lines, we can accurately identify the starting point of consolidation. While this method provides a quick way to determine the beginning of the consolidation stage, it is important to note that more precise research is required for accurate results. Determining the starting point of consolidation is crucial for predicting the formation of the final sediments' structure. Furthermore, the compression settlement stage is a common occurrence in nature, and analyzing it can offer a better understanding of physical phenomena in natural environments.

A same conditioned experiment was done in thinner cylinders (28.00 mm inner diameter) was conducted separately in Fig. 5, in which the Q' was smaller than the thicker cylinders' case. While in the less concentrated part 1.00×10^{-4} and 1.50×10^{-4} the slope shows nearly the same tendency as the thicker cylinders' condition. These results showing the disturbance by the formation of the large aggregates illustrate the evidence of a long-chain network structure that yields a long-distance stress chain in the flocculated sediment.

4.2 The validity of Michaels and Bolger's methods

Estimated results using R-Z's equation from hindered sedimentation were obtained from the fitting line between 1.00×10^{-4} and 1.75×10^{-4} in Fig. 5. A linear relationship between $Q^{1/4.65}$ and ϕ can be obtained through equation (4). Therefore, corresponding values of α, Q_f, and D_f can be derived from the slope of this relation, which was obtained as 2497.74, 0.93 mm s^{-1}, and 1.64 mm respectively. Eventually, the value obtained from hindered sedimentation matches the results of the single floc sedimentation Fig. 8. Miyahara's previous result was below the fitting line because the influence of the container height was not considered in the hindered sedimentation experiment at that time, 150.00 mm was adopted as a unified container height. So, in the present result, taking advantage of the infinite height measurement through M-B's method, the calculated value falls exactly on the fitting line of the relationship between floc diameter and settling velocity. We can see that the hindered sedimentation of Na-montmorillonite is highly correlated with the single floc sedimentation under 1.00 M NaCl condition.

5. Conclusion

To investigate the validity of R-Z's equation through M-B's method on Na-montmorillonite suspension, we systematically analyzed the sedimentation behavior of the flocculated suspension of Na-montmorillonite. The relationship between the interface sedimentation rate and the effective volume fraction of the suspension was determined. The estimated result using R-Z's equation obtained from hindered sedimentation was confirmed by the direct

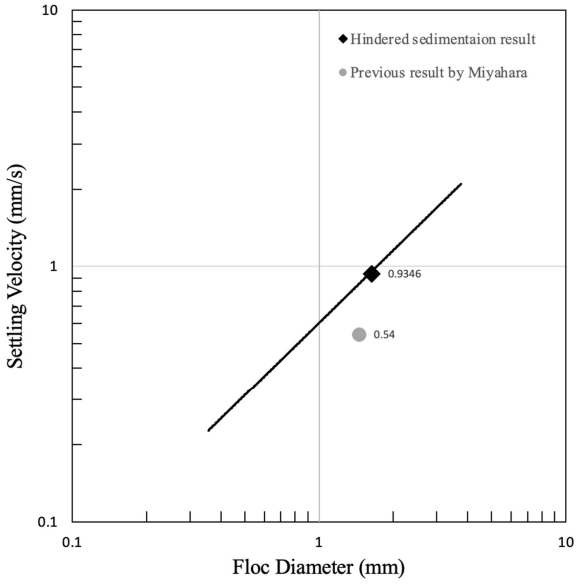


Fig. 8 Relations between the settling velocity and diameter of single flocs sedimentation. Estimated results using R-Z's equation obtained from the hindered sedimentation was plotted. Previous' result done by Miyahara was also shown (Miyahara et al., 2001).

measurement of single floc sedimentation. The estimated value is exactly in line with the measured results. Compared with our previous research done by Miyahara, the concept of infinite height was used for taking no influence of container height in this study. We could conclude that R-Z's equation is applicable to the clay suspension of Na-montmorillonite under 1.00M NaCl condition. The effect of container diameter on the settlement was confirmed, that is, thicker cylinders (50.00 mm diameter) have no effect while thinner cylinders (28.00 mm diameter) will greatly hinder the sedimentation due to the long-chain network structure of Na-montmorillonite. Furthermore, the unique approach of the Two-way camera observation to the single floc sedimentation provides an effective way for the accurate measurement of flocs. Analyzing the sedimentation behaviors of Na-montmorillonite under different conditions and the consolidation period in the hindered sedimentation will further elucidate the study on floc structure and illustrate the floc packing phenomenon.

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