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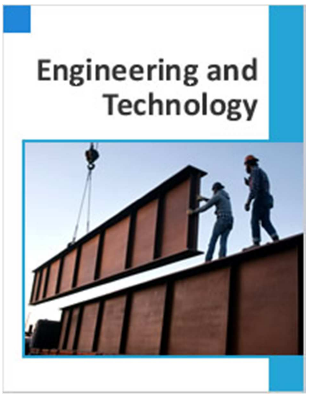

Effects of Slurry Conditions on the Measurement Accuracy of a Manometer-Type Hydrostatic Pressure Measurement System

Hiroshi Satone[1, *], Kenji Iimura[1], Koharu Satone[2], Junichiro Tsubaki[3], Takamasa Mori[4]

[1]Department of Chemical Engineering, Graduate School of Engineering, University of Hyogo, Himeji, Japan
[2]Department of Molecular Design and Engineering, Graduate School of Engineering, Nagoya University, Nagoya, Japan
[3]Department of Research, Nagoya Industrial Science Research Institute, Nagoya, Japan
[4]Department of Chemical Science and Technology, Faculty of Bioscience and Technology, Hosei University, Koganei, Japan

Email address
satone@eng.u-hyogo.ac.jp (H. Satone)
[*]Corresponding author

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Measurement Accuracy,
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Abstract
Evaluation of the slurry characteristics is very important for various industrial processes. However, there are merits and demerits to traditional evaluation methods. Therefore, a sensor-type hydrostatic pressure measurement system was developed. Although this system is very useful, the apparatus required for this system is expensive. Based on this, a manometer-type hydrostatic pressure measurement system, which is inexpensive and simple to construct, was developed. However, because of the transport of liquid on the slurry side and manometer side through the sediment on the filter surface, the measurement accuracy decreases, accurate evaluation is not possible. Therefore, it is necessary to correct the results obtained by the manometer. In this study, the effects of the sediment on the measurement accuracy of this system were investigated. The results showed that its measurement accuracy was decreased by an increase in the permeation resistance of the dispersed liquid by the existence of dense sediment on the filter. To resolve this problem, the transport of the liquid in the filter and the sediment was modeled. Using this model, the decrease in the measurement accuracy was successfully corrected. In addition, the effects of the slurry conditions on the measurement accuracy of this system were investigated by simulation. The results showed that the abovementioned adverse effect on accuracy occurs under all slurry conditions. Moreover, the magnitude of the effects of the slurry conditions on measurement accuracy was in the following order: packing fraction, initial concentration, particle diameter, and initial height of the slurry.

1. Introduction

In various industrial processes such as wet-forming, spray-drying granulation, and film formation, fine particles are usually applied in the state of slurry (fine particles dispersed in a dispersion medium) and become the final product after concentration, dehydration, and drying. Evaluation of the slurry characteristics is very important because these characteristics at the start of the process affect the characteristics of the final product. Conventionally, apparent viscosity measurement has been used to evaluate slurry characteristics; however, it was reported that the apparent viscosity of slurry does not always have a good relation with the packing fraction of the green bodies [1], [2], [3], and there are cases where the product characteristics cannot be accurately predicted by measuring apparent viscosity alone.

Against this background, previous studies have evaluated the settling behavior and packing characteristics of particles in slurries by using settling tests [4], [5], [6], [7] for a long time. However, when the particle diameter is small or the particle density is low, it can take several hours to perform the experiment, even when centrifugal force is applied. Moreover, because the settling behavior is observed from the change in settling interface, the settling behavior of only the smallest particles can be obtained. Therefore, when particles with a wide particle size distribution are used, the necessary information cannot always be obtained. Furthermore, when the liquid and the particles are of the same color, it is difficult to observe the settling interface and sediment in the first place.

In addition to these methods, various evaluation methods such as evaluation by particle size distribution measurement of slurry [7], [8] and an *in situ* solidification method [9], [10], [11] have been proposed. Regarding the former, especially, because the state of the primary particle diameter of the raw material powder used is measured in a state of perfect dispersion, the method is easy for users to understand. The latter is a method in which the slurry is solidified while maintaining a dispersed state of particles by gel casting, and is transformed into flakes and observed directly using the transmitted beam. Moreover, the method can evaluate the slurry several times. However, in both methods, the evaluable upper limit of the slurry concentration is not as high as that of the high-concentration slurry used in ceramic manufacturing processes, making it difficult to evaluate such slurry directly.

Therefore, a hydrostatic pressure measurement method was proposed [12], [13], [14], which predicts the settling behavior and packing characteristics of particles in a slurry in a short time by measuring the hydrostatic pressure of the slurry via a pressure sensor installed at the bottom of a settling tube. Because this method involves a mass of particles suspended in liquid being detected as hydrostatic pressure, the detailed settling behavior of particles in the slurry can be observed. Therefore, measurement of the particle size distribution of highly concentrated slurry without dilution has also been succeeded by analyzing the change in hydrostatic pressure over time [15]. Although the settling hydrostatic pressure method is very useful for evaluating slurry, because it requires a high-sensitivity sensor and an information processing terminal, the apparatus required for this approach is expensive.

Therefore, a manometer-type hydrostatic measurement system developed as a less expensive evaluation apparatus. The appearance and structure of the manometer- and sensor-type systems are shown in Figures 1 and 2, respectively. In the former system, a manometer is installed through a filter at the bottom of the settling tube from which the slurry is poured, which is simple and inexpensive compared to the sensor type.

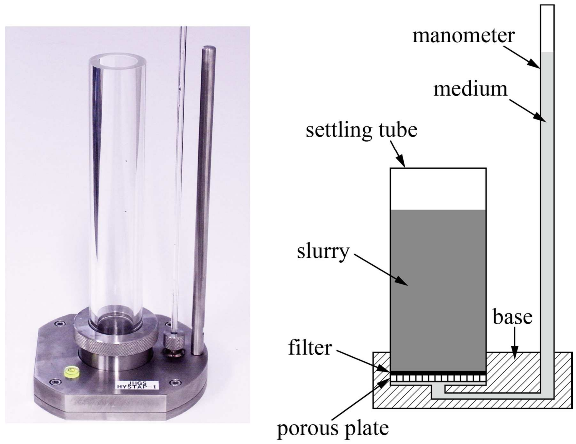


Figure 1. *Photograph and schematic of the manometer-type hydrostatic measurement system (HYSTAP-I, JHGS).*

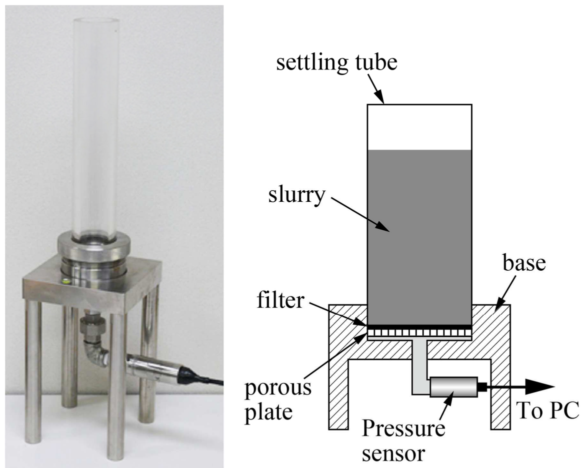


Figure 2. *Photograph and schematic of the sensor-type hydrostatic measurement system (HYSTAP-II, JHGS).*

However, when applying this approach, because the transport of liquid on the slurry side and manometer side occurs through the manometer-connecting part as the hydrostatic pressure changes, particles in the slurry settle, deposit, and form sediment on the filter surface, and thus, the permeation resistance of the dispersed liquid at the manometer-connecting part rises. As a result, because the change in the liquid surface on the manometer side does not catch up with that in the pressure and the accuracy decreases in comparison with that upon measurement by the sensor-type system, accurate evaluation is not possible. For this reason, it is necessary to correct the results obtained by the manometer.

Therefore, in this study, the hydrostatic pressure of slurries of the same lot was measured by using the sensor-type and manometer-type systems, and then clarified the preparation conditions of slurries associated with a decrease in measurement accuracy. Next, to clarify the relation between the settling phenomenon as well as the characteristics of the formed sediment in the slurry and the decrease in measurement accuracy, the formation of the sediment and the permeation phenomenon of the liquid at the manometer-connecting part were modeled and the validity of the model was examined by comparing the value calculated using the model with the test result. Furthermore, by simulating the change in hydrostatic pressure over time by using the proposed model, it was examined the effect of various slurry conditions such as sample powder, liquid, and initial height of the slurry on the measurement result.

2. Comparison of Sensor-Type with Manometer-Type Hydrostatic Pressure Measurement

2.1. Experimental

Slurries were prepared from abrasive alumina powder (JIS #4000: average particle size 3.0 μm; #2000: average particle size 6.7 μm, density 3960 $kg \cdot m^{-3}$; Fujimi) and ion-exchanged water. The solid concentration was 30 vol%. The values of the slurry pH were adjusted using HCl. Slurries with different dispersion states were prepared by changing the values of slurry pH. However, regarding the slurry in the aggregated state, because the settling velocity of particles was too high in #2000, it was difficult to measure the change in hydrostatic pressure over time. Therefore, it was assumed to be prepared only with #4000, and the value of this slurry pH was adjusted to 6.9, which is the isoelectric point. Both #4000 and #2000 were adjusted to pH 4.0 (zeta potential of 40 and 30 mV, respectively) as slurries with good dispersion. After mixing the powder sample, liquid, and a pH regulator to produce the specified concentration and pH, slurries were irradiated with ultrasonic waves for 10 min. It was confirmed that there were no changes in pH before and after ultrasonic irradiation, after which the slurry was considered suitable for testing. Next, the prepared slurries were poured into the settling tube, which is part of the manometer-type and sensor-type hydrostatic pressure measurement systems (HYSTAP-1 and HYSTAP-2; JHGS). The initial height of each slurry was 150 mm. After preparation, the change in hydrostatic pressure over time was measured. It was assumed that the test ends when hydrostatic pressure becomes constant. The filter (hydrophilic PTFE film, bore diameter 0.1 μm) was installed between the manometer part as well as the sensor part and the settling tube part. Accordingly, in the system, only liquid phase could move. Moreover, to prevent liquid evaporation during the measurement, liquid paraffin was poured on the surface of the slurry to a thickness of 1 mm. Furthermore, to preclude the effect of disturbance during the measurement, the measurement system was set on a vibration reduction mat.

2.2. Results and Discussion

Figure 3 shows the changes in hydrostatic pressure over time in the aggregated state of #4000 and dispersed states of #4000 and #2000. Comparing the slurry in the aggregated state with that in the dispersed state of #4000, because the settling velocity of the dispersed slurry was slow, the rate of decrease in the hydrostatic pressure of the dispersed slurry was slower than that of the aggregated state. Focusing on the difference between the sensor-type and manometer-type systems, in the dispersed slurry, the change of pressure was delayed for the manometer type compared with that for the sensor type and the measurement accuracy decreased. Regarding the slurry in an aggregated state, the time when the hydrostatic pressure of the manometer-type system became constant was later than that of the sensor-type, and decline in measurement accuracy occurred. However, although the amount of transport of liquid per unit time was greater than that of the slurry in the dispersed state, the degree of decline of measurement accuracy was low. The packing fractions of the sediment of the slurry in the dispersed state and that in the aggregated state were 0.581 and 0.344, respectively. Therefore, because the dense sediment was formed during settling and the sedimentation process, the permeation resistance of the dispersed liquid increased; for that reason, measurement accuracy decreased. Next, comparing the dispersed slurry of #4000 with that of #2000, with #4000 having a smaller diameter, there was a delay time of a maximum of about 5 h between the sensor type and the manometer type; on the other hand, there was a delay of about 1 h in #2000 having a larger diameter. In addition, the packing fraction of the sediment of #2000 was 0.575, which was approximately equal to that of #4000, although the rate of decrease in pressure, that is, the amount of liquid moving per unit time, was large. The reason for this result is that the permeation resistance of the dispersed liquid became lower than the case of #4000 because the gap in the sediment, that is, the width of the flow path of the liquid, was larger than that of #4000. From these findings, the cause of decline of the measurement accuracy is the formation of dense sediment with the settling of particles.

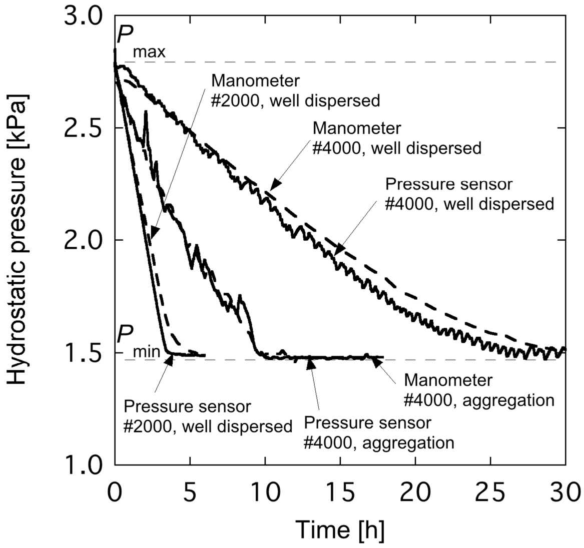


Figure 3. Time change in the hydrostatic pressure for various slurries.

3. Effects of the Slurry Conditions on the Measurement Accuracy

3.1. Mathematical Model

According to the results of the preceding section, when the sediment of a high packing fraction was formed on the filter on the manometer-type system, the permeation resistance of the dispersed liquid at the manometer-connecting part increased; therefore, the detectability decreased and an inability to measure the change over time arose. However, if the degree of detection delay can be clarified in advance using the characteristics of the slurry and sediment formed, it is possible to correct the measurement result to obtain an accurate value. Therefore, the detection delay was attempted to calculate by modeling the manometer-connecting part.

Schematic drawings for this model are shown in Figure 4. To simplify the model, the packing fraction distribution in the sediment was kept uniform. Because D>>d, where D is the inner diameter of the settling tube [m] and d is the inner diameter of the manometer [m], the change in the height of the liquid surface of the slurry side was ignored. Maximum hydrostatic pressure (the hydrostatic pressure at the bottom of the vessel at the time immediately after the slurry has been poured) P_{max} [kPa] and minimum hydrostatic pressure (hydrostatic pressure of only liquid phase reaching finally) P_{min} [kPa] are expressed by the following equations, using initial height of the slurry H_0 [m], liquid height of the manometer side h_0 [m], particle concentration ϕ [-], particle density ρ_{p} [kg·m^{-3}], liquid density ρ_l [kg·m^{-3}], and slurry density ρ_s [kg·m^{-3}]:

$$P_{\mathrm{max}} = \rho_{\mathrm{s}} g H_0 = \{\rho_l(1-\phi) + \rho_{\mathrm{p}} \cdot \phi\} g H_0 = \rho_l g (h_0 - h_{\mathrm{c}}) \quad (1)$$

$$P_{\mathrm{min}} = \rho_{\mathrm{l}} g H_0 = \rho_l g (h_0 - h_{\mathrm{c}}) \quad (2)$$

However, h_{c}[m] is the capillary height in the manometer, which is expressed using the following equation, using the surface tension of liquid γ [N·m^{-1}] and contact angle between the inner wall of the manometer and the liquid θ [rad]:

$$h_{\mathrm{c}} = \frac{4\gamma \cos\theta}{d \rho_l g} \quad (3)$$

In this study, a combination of a glass manometer with an inner diameter of 3 mm and water was used. In this case, when 72.8×10^{-3} N·m^{-1}, 10^3 m^3, and 0 rad were used as values of surface tension, density, and contact angle of liquid, respectively, the capillary elevation height became 9.9 mm.

Mass of suspended particles per unit area at t [s], namely M [kg·m^{-2}], is expressed by the following equation, using initial mass of suspended particles per unit area M_0 [kg·m^{-2}]:

$$M = M_0 - \int_0^t \mathrm{d}M \quad (4)$$

Slurry density at this time is expressed by the following equation, as the concentration distribution in the depth direction is uniform:

$$\rho_{\mathrm{s}} = \rho_l \left(1 - \frac{M/\rho_{\mathrm{p}}}{H_0}\right) + \rho_{\mathrm{p}} \cdot \frac{M/\rho_{\mathrm{p}}}{H_0} = \rho_l + \frac{\rho_{\mathrm{p}} - \rho_l}{\rho_{\mathrm{p}} H_0}\left(M - \int_0^t \mathrm{d}M\right) \quad (5)$$

Next, it is considered that the permeation resistance rising because of increased thickness via the settling and sedimentation of particles. Permeation resistance (permeation resistance at time t) R [m^{-1}] is expressed by the following equation, using permeation resistance of filter medium R_0 [m^{-1}]:

$$R = R_0 + \int_0^t \mathrm{d}R \quad (6)$$

Here, the rate of increase in R is expressed by the following equation based on the Kozeny–Carman equation, using packing fraction of the sediment Φ [-], specific surface area of the particle S_{v} [m^{-1}], thickness of the sediment L [m·s^{-1}], and particle diameter x [m]:

$$\frac{\mathrm{d}R}{\mathrm{d}t} = 5 S_{\mathrm{v}}^2 \frac{\Phi^2}{(1-\Phi)^3} \frac{\mathrm{d}L}{\mathrm{d}t} = \frac{180\Phi}{(1-\Phi)^3 x^2 \rho_{\mathrm{p}}} \frac{\mathrm{d}M}{\mathrm{d}t} \quad (7)$$

Next, it is considered that the transport of dispersed liquid. Considering the change in the cross-sectional area of the manometer-connecting part, the rate of transport of the liquid from the manometer side to the slurry side is expressed by the following equation, based on the Darcy equation, using the viscosity μ:

$$\frac{\mathrm{d}h}{\mathrm{d}t} = \frac{D^2}{d^2} \frac{g}{\mu R} \{\rho_l (h - h_c) - \rho_{\mathrm{s}} H_0\} \quad (8)$$

By sequentially calculating equations (5–8), the slurry density at the time can be calculated from the rate of change in height of the liquid surface of the manometer. From this, the hydrostatic pressure at the time can be corrected accurately.

Figure 5–7 show the results upon correcting the measurement results of Figure 3 by the above method. Under all conditions, the corrected values are in good agreement with the measurement results for the sensor type, which confirmed the validity of the proposed model.

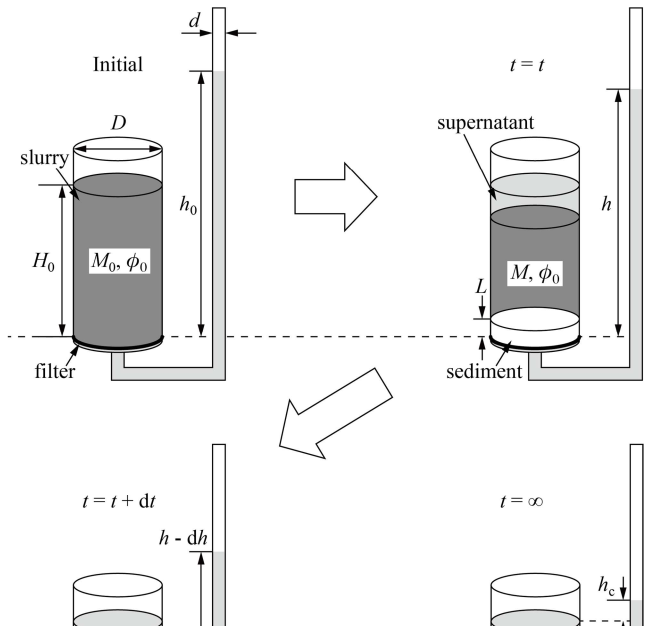


Figure 4. *Schematic illustration of settling and sedimentation process.*

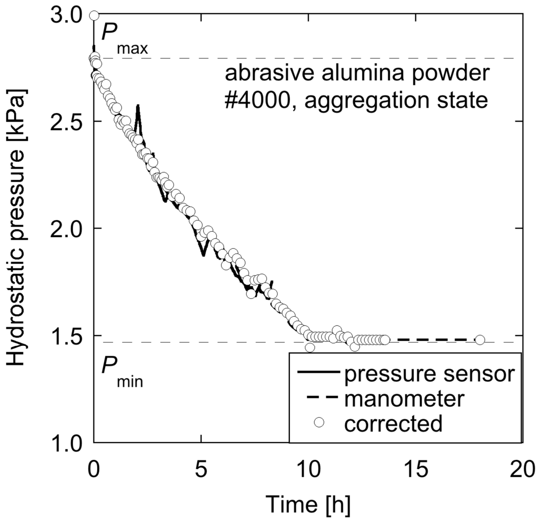


Figure 5. *Comparison between corrected and measurement values of the hydrostatic pressure for abrasive alumina powder #4000, aggregation state.*

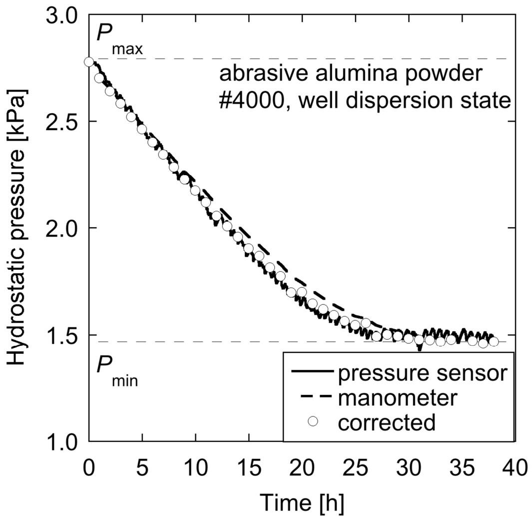


Figure 6. *Comparison between corrected and measurement values of the hydrostatic pressure for abrasive alumina powder #4000, in a well-dispersed state.*

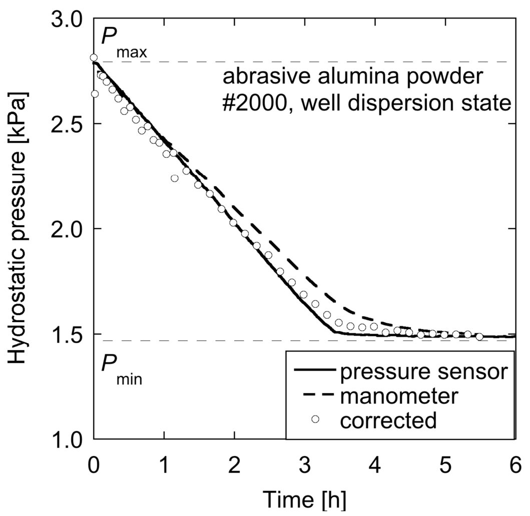


Figure 7. *Comparison between corrected and measurement values of the hydrostatic pressure for abrasive alumina powder #2000, in a well-dispersed state.*

3.2. Simulation Conditions

From equations (5), (7), and (8), it is predicted that various parameters such as particle diameter, packing fraction of the sediment, initial concentration, and initial height, as well as the existence of sediment, affect the measurement accuracy of the manometer type. Because it is exceedingly difficult to confirm these effects via testing, it was simulated by sequential computation using the model described in the preceding section, with which the effect of slurry conditions on measurement accuracy was examined.

For simulation, it is necessary to calculate the number of suspended particles per unit area in equation (4) from the settling velocity of the particles. However, in highly concentrated slurry as used in this study, settling is obstructed via the upward flow accompanying particle settling, for which correction is necessary. Therefore, change in the hydrostatic pressure of the slurry over time under the same conditions as those in Figure 3 was calculated by sequential computation using the correction formula [15], as shown in equation (9), which was proposed previously:

$$u_c = u_\infty (1-\phi)^{7.16} \tag{9}$$

To simplify the calculation, the particles in the slurry were assumed to be monodispersed. The calculation results are shown in Figure 8. As shown in this figure, for both the sensor type and manometer type, because the calculation results are in good agreement with the measurement results, there is no problem with the calculation method. Therefore, regarding the alumina slurry used in this study, the relation with the measurement accuracy was calculated using each of the initial height, particle diameter, particle concentration, and packing fraction of the sediment as parameters.

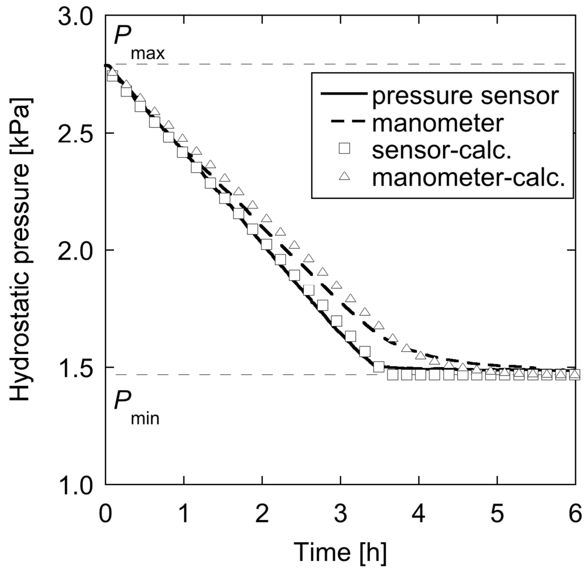


Figure 8. *Comparison between calculated and experimental values of the hydrostatic pressure for various measurement methods.*

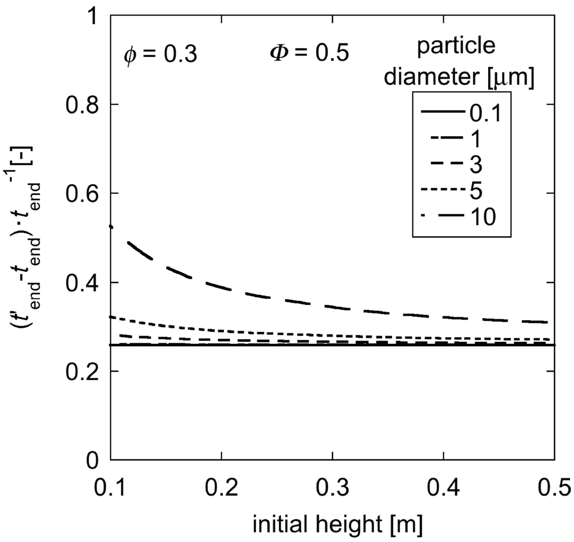
The range of each parameter is summarized in Table 1. To evaluate the measurement accuracy, the time until the hydrostatic pressure of the sensor type reached $P_{\min}$ is defined as t_{end} and the time until the hydrostatic pressure of the manometer type reached $P_{\min}$ is defined as t'_{end}, and the value of $(t'_{\mathrm{end}} - t_{\mathrm{end}})\cdot t_{\mathrm{end}}^{-1}$ was used. As this value increases, the decline of measurement accuracy is large.

Table 1. *Simulation conditions.*

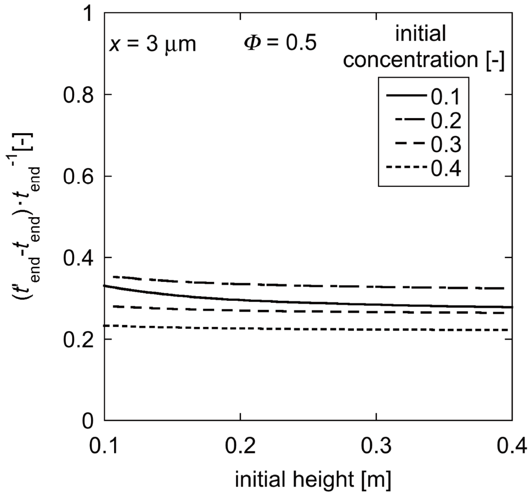
Parameter	Range
Initial height [m]	0.1–0.5
Particle diameter [μm]	0.1–10
Initial concentration [-]	0.1–0.4
Packing fraction [-]	0.1–0.7

3.3. Simulation Results and Discussion

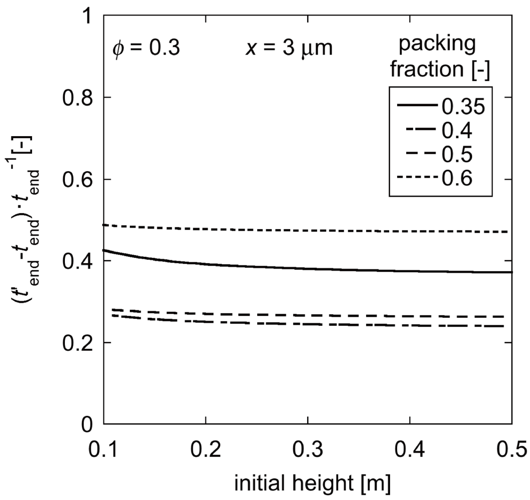
When the manometer-type hydrostatic pressure measurement system is used, it is easy to adjust the parameter of the initial height of the slurry. Therefore, first, to clarify the effect of the initial height on measurement accuracy, the relations among particle diameter, initial concentration, packing fraction of the sediment, and measurement accuracy when the initial height was changed was determined, as shown in Figure 9 (a–c). When the initial height was reduced, the measurement accuracy tended to decrease, and this tendency was remarkable under conditions in which the particle diameter was large. The reason for this result is as follows. When the initial height decreases, because the time to reach $P_{\min}$ becomes relatively short, the transport of liquid in the manometer does not catch up. The reason why this effect is so pronounced at a large particle diameter is that the amount of transport of the dispersed liquid per unit time increased because the time to reach $P_{\min}$ was further shortened; in addition, the rate of formation of sediment, that is, the rate of increase in permeation resistance of the dispersed liquid, increased owing to the settling velocity increasing with the particle diameter. However, under all conditions, the graph is nearly flat, except for the condition with a very low initial height. Therefore, initial height does not have a major impact on measurement accuracy.



(a) Particle diameter



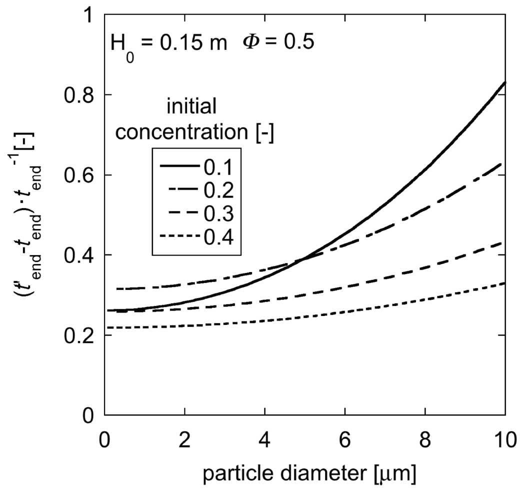
(b) Initial concentration



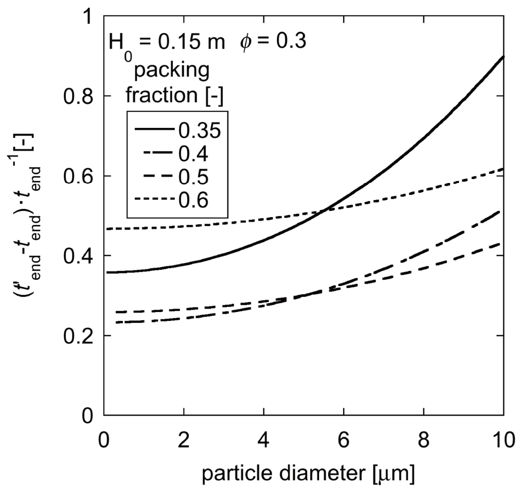
(c) Packing fraction

Figure 9. *Relation between the measurement accuracy and the initial height of the slurry for various parameters: (a) particle diameter, (b) initial concentration, (c) packing fraction.*

Next, to clarify the effect of particle diameter on measurement accuracy, the relations among initial concentration, packing fraction of sediment, and measurement accuracy when particle diameter changes was determined, as shown in Figure 10 (a, b). As a general tendency, as with initial height, the measurement accuracy decreases as the particle diameter increases under all conditions. As shown in Figure 10 (a), in the range where the particle diameter is relatively large, the measurement accuracy decreases as the initial concentration becomes lower. The reason for this result is that the amount of transport of the liquid and the rate of increase in the resistance increase because settling velocity becomes higher as initial concentration becomes lower owing to the effect of hindered settling. Meanwhile, under conditions with the initial concentration of 0.1, the measurement accuracy remarkably improves within the range of a small particle diameter. This is opposite to the condition of initial concentrations of 0.2 and 0.3. Although this condition is that the velocity lowering owing to hindered settling is much smaller than that of other concentrations, because the initial concentration is very low, the amount of transport of the liquid and the thickness of the sediment formed decrease. This result was obtained because of these multiple factors. Next, as shown in Figure 10 (b), although there is no clear correlation between the packing fraction of the sediment and measurement accuracy, as the packing fraction of the sediment increases, the width of decline of measurement accuracy with increasing particle diameter increases. The reason for this result is as follows. When the packing fraction is low, the effect of the particle diameter mentioned above appears. However, because the permeation resistance of the dispersed liquid is high since the initial stage of sediment formation, as the packing fraction increases, the effect of particle diameter is relatively low.



(a) Initial concentration



(b) Packing fraction

Figure 10. *Relation between measurement accuracy and particle diameter for various parameters: (a) initial concentration, (b) packing fraction.*

The above findings suggest that the main cause of the decline in measurement accuracy is the increase in permeation resistance of the dispersed liquid because of the existence of dense sediment. Therefore, to clarify the effect of the packing fraction of the sediment on measurement accuracy, the relation between the initial concentration and measurement accuracy when the packing fraction of the sediment is changed was determined, as shown in Figure 11. The results suggest that the measurement accuracy decreases when the packing fraction becomes extremely high. Moreover, when the packing fraction of the sediment becomes low and approaches the initial concentration, the measurement accuracy decreases sharply. The reason for this is as follows. The fact that the packing fraction of the sediment is close to the initial concentration means that the time required from the start of settling until its completion is very short and the amount of transport of the liquid per unit time becomes very large. Therefore, the transport of the liquid in the manometer cannot catch up. Because this condition is applicable to the slurry with a strong attractive force acting between the particles and the whole gel at the same time as the settling starts, it is necessary to pay attention when performing measurements on such slurry in a strongly aggregated state.

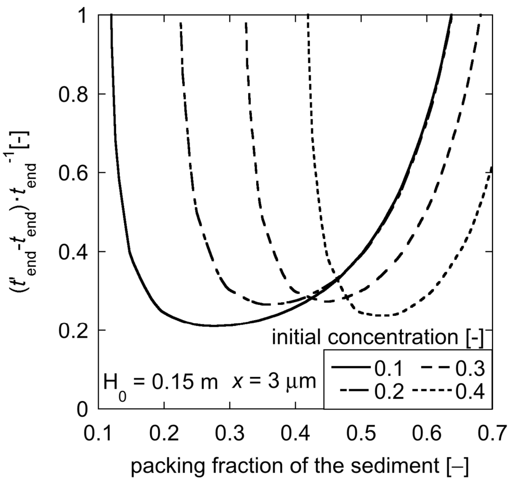


Figure 11. *Relation between measurement accuracy and packing fraction of sediment.*

From the above results, a certain degree of decline in measurement accuracy occurs with the manometer-type hydrostatic pressure measurement system under all slurry conditions. The packing fraction has the largest effect on the measurement accuracy. The effects of the initial concentration and particle diameter are the second largest. Regarding the initial height, measurement accuracy generally does not change irrespective of the level of this variable, although measurement when its value is extremely low should be avoided.

4. Conclusion

To clarify the effects of slurry conditions on the measurement accuracy of the manometer-type hydrostatic pressure measurement system, the hydrostatic pressure of slurries of the same lot was measured using the manometer-type and sensor-type systems. The results showed that the decline of measurement accuracy of the manometer-type system was due to the dense sediment formed as a result of the settling of particles. In addition, this phenomenon could be expressed using a relatively simple model of the sediment of particles and transport of the dispersed liquid, as proposed in this paper. From this model, it became possible to correct the measurement result, without which the measurement accuracy would be decreased.

Moreover, by simulating the change in the hydrostatic pressure over time using the proposed model, the effects of various slurry conditions on the measurement results were investigated. The results showed that a certain degree of decline in measurement accuracy occurs with the manometer-type hydrostatic pressure measurement system under all slurry conditions. Furthermore, the magnitude of the effects of slurry conditions on measurement accuracy was in the following order: packing fraction, initial concentration, particle diameter, and initial height of the slurry.

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Nomenclature

D	inner diameter of the test tube	[m]
d	inner diameter of the manometer	[m]
g	gravitational acceleration	[$m \cdot s^{-2}$]
P	hydrostatic pressure	[kPa]
P_{max}	maximum hydrostatic pressure	[kPa]
P_{min}	minimum hydrostatic pressure	[kPa]
H_0	initial height of the slurry	[m]
ϕ	particle concentration	[-]
ρ_p	particle density	[$kg \cdot m^{-3}$]
ρ_l	liquid density	[$kg \cdot m^{-3}$]
ρ_s	slurry density	[$kg \cdot m^{-3}$]
h_c	capillary height	[m]
γ	surface tension	[$N \cdot m^{-1}$]
θ	contact angle	[rad]
M_0	initial mass of suspended particles per unit area	[$kg \cdot m^{-2}$]
M	mass of suspended particles per unit area	[$kg \cdot m^{-2}$]
t	time	[s]
R	permeation resistance	[m^{-1}]
R_0	permeation resistance of filter medium	[m^{-1}]
Φ	packing fraction of the sediment	[-]
S_v	specific surface area of the particle	[m^{-1}]
L	thickness of the sediment	[m]
x	particle diameter	[m]
μ	viscosity of liquid	[$Pa \cdot s$]
u_c	hindered settling velocity of single particle	[$m \cdot s^{-1}$]
u_∞	settling velocity of single particle	[$m \cdot s^{-1}$]

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