Batch Gravitational Sedimentation of Slurries

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We investigated the batch settling behavior of the kaolin slurry and the UK ball clay slurry at various initial solids fractions (φ₀) using a computerized axial tomography scanner (CATSCAN). The spatio-temporal evolutions of solids fractions in the consolidating sediments were continuously monitored. Since the interface between the sediment and the supernatant of the investigated slurries was blurred, an averaging procedure was employed to estimate their null-stress solids fractions (φ₀ g). Besides the rather slow settling for the high-φ₀ g slurries, the basic settling characteristics resemble each other regardless of whether φ₀ > φ₀ g or vice versa. The above-mentioned experimental data reveal that the investigated slurries are neither purely elastic nor purely plastic in rheological behavior. On contrary to most model works a blurred supernatant-sediment interface makes difficulty in the gel point determination. During initial settling the high-φ₀ g slurries clearly exhibit a finite yield stress to resist deformation. That is, the slurries are plastic fluids. However, the network structure in the slurries deteriorates gradually in the subsequent settling stage while the final, equilibrated sediment reveals a continuous distribution in solids fraction. Restated, the final sediment possesses as a purely elastic characteristic. The model parameters of theory by Buscall and White were regressed by the dynamic consolidating sediment data, while those by Tiller and Leu were obtained using the final equilibrated sediment data. Calculations from both models reveal that ball clay slurry is more compressible than is the kaolin slurry. The high-φ₀ g slurry would yield the less compressible sediment.

Key Words: CATSCAN; sedimentation; solids fractions; yield stress.

INTRODUCTION

Sedimentation characteristics of slurries are important in settler/thickener design and operation. Theorists like Coe and Cleverenger (1), Kynch (2), and Talmadge and Fitch (3) ignored the role of the rising sediment. Tiller (4) refined the Kynch theory to consider the effect of increasing sediment during sedimentation. Fitch (5) further investigated this theory and modified the procedures to determine the characteristic lines. Font (6, 7) reviewed these works and discussed the compression zone effects in batch sedimentation.

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The way the solids fraction (volume fraction of solid phase, φ) changes with solid pressure (Pₛ) governs the rheological characteristics of the consolidating sediment. As Koenders and Wake- man (9) revealed, most related works assumed an elastic filter cake having the following power-law type constitutive equation:

$$\phi = \phi_g \left(1 + \frac{P_S}{P_0}\right)^n$$  \[1\]

where a can be 0 or 1. φ₀ is the null-stress solids fraction or the gel point if a = 1 (9). P₀ represents the characteristic solid pressure (10). Equation [1] assumes that φ depends solely on the local solid pressure Pₛ. Lee and Wang (11) summarized the literature works adopted constitutive equations following Eq. [1]. Kos (12) further assumed that the solids fraction should depend not only on the local solid pressure, but also on its gradient. Shirato et al. (13) refined Eq. [1] for describing the time-dependent response of filter cake using a linear visco-elastic model. Tien et al. (14) modified Eq. [1] to incorporate the fine migration effects.

Buscall and White (15) and Ayzerais et al. (16) proposed a consolidation model considering the flocculated slurry as a purely plastic body that possesses a yield stress (Pᵧ(φ)). If the local pressure of sediment exceeds Pᵧ, the network structure would yield. Buscall and White (15) proposed the following constitutive equation:

$$\frac{D\phi}{Dt} = k(\phi, P)[P - Pᵧ(\phi)] , \quad P > Pᵧ(\phi)$$  \[2a\]

$$\frac{D\phi}{Dt} = 0 , \quad P \leq Pᵧ(\phi) ,$$  \[2b\]

where D( )/Dt denotes the material derivative and k is the “dynamic compressibility” of the suspension (9). This model had been extensively applied to sedimentation processes of flocculated suspensions (see, for instance, (17–21)).

The governing equations controlling the liquid flows in the suspension or in the sediment are different (4). The null-stress solids fraction (φ₀) represents the threshold value above which the particles in a suspension physically contact each other and form a continuous phase through which the solid pressure could be directly transmitted. Tiller and Khatib (22) proposed two extrapolating procedures for estimating φ₀ values. Lu et al. (23)
adopted low-head filtration test to determine the surface cake porosity and examine the effects of slurry concentration and operating conditions. Determination of $\phi_0$ for certain slurries is extremely difficult since the interface between the sediment and the suspension could be blurred. The sudden jump in solids fraction when moving from settling slurry to sediment as suggested by the idealized theory may not be easily observed (as discussed later).

Researchers adopted nondestructive techniques to obtain the relevant variable in the solid/liquid separation processes as a function of space and time, such as the use of $\gamma$-ray (18, 24), NMR (25–27), and X-ray (17, 28–33). Buscall (34) reviewed the pertinent literature before 1990. Shen et al. (19) adopted the yield-stress model by Buscall and White (15) for interpreting their settling data, and noted that the model was valid during the initial phase of settling/filtration of the “pliant” systems (with initial solid weight exceeds the corresponding yield stress). For the “stiff” systems (with initial solid weight being lower than the yield stress), on the other hand, no consolidation occurred in the initial stage of experiments.

We consider the cases at $\phi_0 > \phi_c$ with all particles in the initial suspension physically contact with each other and form a network matrix. Figure 1a represents the one-dimensional sedimentation process with the purely elastic sediment, in which $z = 0$ denotes the bottom and $z = h_0$ represents the slurry surface. Since the transmission of solid pressure through the network the sediment would continuously yield at the beginning of the test. Restated, no portion of the sediment could be kept at $\phi_0$ while no constant-$\phi_0$ regime could be noticeable in the sediment. Moreover, the solids fraction in the final sediment should be of a continuously varying manner with no apparent constant-$\phi_0$ regime (like Fig. 1a). For a purely plastic sediment with a finite yield stress, on the other hand, even at $\phi_0 > \phi_c$ the sediment would not deform if the local solid pressure has not exceeded $P_y(\phi_0)$. Therefore, a constant-$\phi_0$ regime could persist during settling (Fig. 1b). The final, equilibrated sediment would also possess the constant-$\phi_0$ regime (like Fig. 1b). The batch sedimentation tests of high-$\phi_0$ slurries could therefore provide the information on the validity of the above-mentioned model frameworks.

This work employed the computerized axial tomography scanner (CATSCAN) for investigating the batch-settling behavior of kaolin slurry and UK ball clay slurry, particularly on the effects of $\phi_0$. We first demonstrated that, since the two investigated slurries exhibit a blurred supernatant–sediment interface due to marked back-diffusion effect, the determination of their $\phi_0$ values were made through an averaging procedure. Then, we revealed the feasibility of using the purely elastic or the purely plastic constitutive models to interpret the obtained sedimentation data.

**EXPERIMENTAL**

**Samples**

Kaolin slurries and UK ball clay slurries were prepared by mixing fixed amount of powders with 0.1 M NaClO$_4$ solution in distilled water to provide a high ionic strength to prevent the interference of other ions that might be released from the particle surfaces. The pH values were fixed at 7.0. The particle size distribution of slurry was determined by a Sedigraph.

**FIG. 1.** Schematics of slurry settling. (a) Purely elastic sediment; (b) purely plastic sediment.

**FIG. 2.** The relationship between CT number and solids fraction in kaolin and clay slurries.
5100 C (Micromeritics), giving a mean diameter of 6.3 \(\mu m\) for kaolin slurry and 2.5 \(\mu m\) for clay slurry. An Accupyc Pycnometer 1330 (Micromeritics) measured the true solid density. The results are 2730 kg/m\(^3\) for kaolin powders and 2580 kg/m\(^3\) for ball clay powders. The Zetasizer 2000 (MARLVERN) measured the \(\zeta\)-potentials of suspension. The results are \(-17.7\) mV for kaolin slurry and \(-22.1\) mV for clay slurry.

**CATSCAN**

Batch settling experiments were conducted in a settling cylinder that had a height of 20 cm and a diameter of 9.5 cm, and were continuously monitored with the CATSCAN (DeltaScan 2060; 100 kV, 75 mA). The X-ray attenuations, which are normally presented as dimensionless scale, the CT number (35), could be obtained from a horizontal section. Bergstrom (18) adopted similar procedures using their \(\gamma\)-ray apparatus. Preliminary tests revealed that the CT number for the 0.1 M NaClO\(_4\) solution is essentially zero since the dissolved salts do not absorb a significant amount of X-ray. Furthermore, the correlations between CT number and the solids fraction are noted for the kaolin and clay slurries, as demonstrated in Fig. 2. The uncertainties in axial position measurement and the local solid volume fraction...
TIME EVOLUTIONS

Figures 3 and 4 depict the spatio-temporal evolutions of cross-sectional average solids fraction measured for kaolin and clay slurries during the first 6000 s and those at equilibrium. The mean solids fraction is interpreted as follows:

$$\phi_{\text{mea}} = \frac{\int_0^{h_0} \phi \, dz}{h_0}, \tag{3}$$

where $h_0$ is 20 cm, the height of the slurry. The calculated $\phi_{\text{mea}}$ data all range from 0.98 to 1.03$\phi_0$, which partially support the accuracy of the solids fraction distributions presented in Figs. 3 and 4.

For both slurries, the settling rates decrease substantially with increasing $\phi_0$, while the effects are more significant for the clay slurries than for the kaolin slurries. For all slurries initially at $\phi_0$, a sediment layer appears at the tube bottom during settling, while a constant-concentration regime at $\phi_0$ retains above this layer regardless of the range of $\phi_0$. The basic characteristics of settling are similar for slurries at $\phi_0 > \phi_g$ to those at $\phi_0 < \phi_g$.

Figures 5a and 5b depict the solids fraction distributions in the final, equilibrated sediment. The clay slurry at $\phi_0 = 0.15$ exhibits a very blurred region above the observed sediment and is excluded in Fig. 5b. For most slurries examined herein, the interface between the sediment and the supernatant (if any) is blurred. No apparent jump in solids fraction over the so-called “supernatant-sediment” interface is noted on all curves. Previous studies could neither illustrate such a jump (9, 17, 32, 33, 36). Since the X-ray beam is of a finite thickness, the length of the transition zone should be less than the resolution provided by the present CT scanner if a distinct interface does exist. Also the Brownian motion of colloidal particles may provide a “diffusion” effect on the settling sediment (37). The CATSCAN profiles could not directly measure the $\phi_g$ values for the present slurries. Before the position of the interface can be determined, no reliable theoretical analyses could be made.

As Fig. 5 reveals, an “interfacial zone” of width approximately 1 cm exists in all equilibrated solids’ fraction curves, which bridges the supernatant regime and the sediment regime. Although one could not distinctly identify the $\phi_g$ values from Fig. 5, it is clear from these curves that the portion of slurry exhibiting a solids fraction exceeding 0.10 should be definitely regarded as the sediment. A procedure is adopted herein to estimate the $\phi_g$ values. Since the present ultimate error for solids fraction estimation is 0.1%, the position where $\phi = 0.1\%$ is taken as the upper surface of the equilibrium sediment. The “interfacial zone” between the suspension and the sediment is taken from the surface ($\phi = 0.1\%$) to 1 cm below. The average solids fraction over this 1-cm zone is defined as $\phi_{\text{av}}$. The corresponding vertical distance from bottom to the point at which $\phi = \phi_g$ is termed as $l_0$ (sediment height). The average solids fraction in the sediment $\phi_{\text{av}}$ is defined as

$$\phi_{\text{av}} = \frac{\int_0^{l_0} \phi \, dz}{l_0}, \tag{4}$$

The estimated null-stress solid fractions are presented as circles in Figs. 5a and 5b and are illustrated together with the $\phi_{\text{av}}$s in Figs. 6a and 6b. Tiller and Khatib (22) proposed that as $\phi_0$ increases the $\phi_g$ would first decrease, after passing a minimum point (approximately at $\phi = 0.1\%$), and then increase with increasing $\phi_0$ up to 0.5. Apparently the values depicted in Figs. 6a and 6b do not change much with $\phi_0$. Hypothesis tests reveal that there exists no apparent correlation between the $\phi_g$ values and the $\phi_0$ data at a confidence interval of 95%.

The dashed lines in Figs. 6a and 6b represent the 45° lines at $\phi_0 = \phi_g$, which intercept the estimated $\phi_g$ at 0.074 and 0.082 for kaolin and clay slurries, respectively. Restated, although the procedures to determine $\phi_0$ deem arbitrary, a threshold solids fraction of $\phi_g \approx 0.08$ could be used to distinguish slurry from its sediment. This value correlates with the results of Buscall and White (15) that the $\phi_0$ ranges 0.05 to 0.3 for particulate suspensions. At $\phi_0 > 0.08$, the particles in the present slurries would physically contact each other and the whole slurry should be regarded as the sediment body at the start of the test.

The experimental range (0.01–0.15) has covered both the $\phi_0 < \phi_g$ and the $\phi_0 > \phi_g$ cases. Hence, the existence of the constant-$\phi_0$ regime in Figs. 3 and 4 represents the yield stress...
of the settling sediment, thereby supporting the purely plastic model by Buscall and White (15). As settling proceeds, the constant-\(\phi_0\) regime in Figs. 3 and 4 gradually shrinks, accompanied by the expansion of the supernatant and sediment zones. Equilibrium could be reached at settling time ranging from 48 h to 1 week, depending on the slurry species and the \(\phi_0\). It is noticeable that the final sediment exhibits no constant-\(\phi_0\) regime. This observation, on the other hand, favors the purely elastic model by Tiller and Leu (10). Shen et al. (19) also questioned the existence of the yield stress. Apparently neither the purely elastic nor the purely plastic constitutive equations could satisfactorily interpret the whole range of sedimentation process. At the start of the high-\(\phi_0\) tests a network of finite yield stress apparently exists. However, this structure seems degraded continuously at the bottom of the constant-\(\phi_0\) regime, evidenced by the increasing \(L_c\) values in Figs. 3 and 4.

**FINAL SEDIMENT**

As Fig. 3 demonstrates, the equilibrated solids fractions for kaolin slurries at bottom are all around 0.2 regardless of their \(\phi_0\) values, indicating that the kaolin slurry is not very compressible
to the applied compressive pressure. On the other hand, clay slurries could be compacted more readily to form the sediment of higher solids fraction when $\phi_0$ increases (for instance, as depicted in Figs. 4a–4e, at 6000 s $\phi = 0.28$ and 0.34 for $\phi_0 = 0.02$ and 0.15, respectively). Restated, the clay sediment is more compressible than is the kaolin sediment.

Quantitative evaluation of sediment compressibility requires the local solid pressure data for the slurries. At equilibrium, the solid pressure gradient could be stated as follows:

$$\frac{\partial P_S}{\partial t} = -\Delta \rho g \phi,$$

[5]

where $\Delta \rho$ is the density difference between the solid and liquid. Hence, the local solid pressure at height $z$ is expressed as follows:

$$P_S = \Delta \rho g \int_0^z \phi \, dz.$$

[6]

The solid pressure at bottom ($P_S |_{z=0}$) could be estimated based on the solids fraction data in Fig. 5 and Eq. [6] with $z = 0$. Figures 6a and 6b also represent the estimated $P_S |_{z=0}$ data as functions of $\phi_0$. All $P_S |_{z=0}$ data at equilibrium are proportional to $\phi_0$, or are equal to the buoyant weight of the solid particles. This also confirms the accuracy of the present experimental measurement.

Local solid pressure data could be evaluated based on Eq. [6]. Figures 7a and 7b illustrate the $\phi$ versus $P_S$ curves in the final sediment. Despite certain data scattering, the local solids fraction decreases continuously with increasing $\phi_0$ at some fixed effective pressure. Comparisons between clay and kaolin slurries reveal that the former exhibits a more compressible structure than is the latter.

The local structure in the sediment would be degraded owing to gravity consolidation to a level counterbalancing the corresponding yield stress. Therefore, the $\phi - P_S$ curves in Fig. 7 represent the $\phi - P_y(\phi)$ correlations. As Fig. 7 reveals, the sediment yields more readily at the low solids fraction regime than the high regime. The sediment was consolidated from the gel point to around 12–15% at a pressure less than 1–2 Pa. At a higher pressure, the sediment yields more slowly with the applied pressure. For instance, for kaolin slurry at $\phi_0 = 15\%$ the solids fraction increases from 15 to 20% over a pressure up to
400 Pa. In fact, the structure of the final sediment at high \( \phi_0 \) would depend only weakly on the applied pressure.

Empirical equations could correlate the \( \phi - P_S \) curves in Fig. 7. Here we employ Eq. [1] in sample calculations since the fitting could be achieved using graphical method proposed by (10). The obtained parameters according to the graphical method were adopted as the initial guess to nonlinear regression of Eq. [1] with the experimental data. Table 1 lists the obtained results. The parameter \( P_0 \) is rather low, generally around or less than 1 Pa, indicating that these slurries would largely deform at the low-pressure regime. Also, the \( \beta \) values are lesser for clay slurries than those for the kaolin slurries, stating that the former is more compressible than the latter. Finally, the higher \( \phi_0 \) would yield the lower \( P_0 \) and \( \beta \). Restated, the slurry with a high initial solid concentration would deform quickly at the low solid pressure, but would resist more readily the external applied pressure at a greater solid pressure. Therefore, in the practical regime the high-\( \phi_0 \) systems would pack more readily when initially form, but become rather “stiff” owing to the associated large yield stress.

**CONSOLIDATING SEDIMENT**

For demonstrating the sediment compaction effects, only the portion of sediment with \( \phi > \phi_g \) would be herein considered.

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**FIG. 6.** The null-stress solids fraction \( \phi_g \), average solids fraction \( \bar{\phi} \) in sediment, and solid pressure at bottom \( P_S \) at different \( \phi_0 \): (a) kaolin slurry and (b) clay slurry.

**FIG. 7.** The solids fraction distributions in sediment versus solid pressure at different \( \phi_0 \): (a) kaolin slurry, and (b) clay slurry.
## Table 1

Model Parameters Obtained in This Work

<table>
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<tr>
<th>φ₀</th>
<th>φ₀</th>
<th>U₀K(φ₀) (m/s)</th>
<th>P₀ (Pa)</th>
<th>P₀ (Pa)</th>
<th>β</th>
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<td>UK ball clay slurrys</td>
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The solid velocity of sediment at φ₀ by Landman and Russell (38) is stated as follows:

\[
\frac{dy}{dt} = -U₀K(φ₀) \left(1 - \frac{∂P_y}{∂z} \frac{∂z}{Δρgφ₀} \right)
\]

where \(U₀\) is the terminal velocity of individual particle; \(K\) is the hindered factor; \(Δρ\), the density difference between the solids and the liquid; \(g\), the gravitational acceleration; \(y\), the upper interfaceal height where \(φ = φ₀\); and \(L_c\), the height where \(φ\) deviates from \(φ₀\) (19). Equation [5] is valid for the consolidating sediment with \(φ > φ₀\). A regime of constant \(φ₀\) is assumed to exist in the consolidating sediment over the range of \(z = y\), where \(P_s = 0\) to \(z = L_c\), where \(P_s = P_y(φ₀)\). Hence, the plot with \(dy/dt\) versus \(1/(y - L_c)\) estimates the \(U₀K\) and \(P_y\) at \(φ = φ₀\) with linear regression analysis.

The investigated slurries possess a blurred suspension, while the solid pressure accumulated at \(y\) changes, where \(φ = φ_y\). Therefore, Eq. [5] needs correction as follows:

\[
\frac{dy}{dt} = U₀K(φ₀) \left[1 - \frac{P_y(φ₀)}{Δρgφ₀(y - L_c)} \right],
\]

where \(P_y\) is the accumulated solid pressure at \(y\) and could be approximated as

\[
P_y = P|_{z=y} \approx Δρg \int_{y} φ dz.
\]

If Eq. [8] were valid for describing the dynamics of the consolidating sediment, the curves for \(dy/dt\) versus \(1/(y - L_c)\) data should reveal a linear character. Figure 8 shows some \(dy/dt\) versus \(1/(y - L_c)\) data sets of \(φ₀ > φ_y\) tests. Apparently the curves in Fig. 8 reveal a linear relation during early stage of settling. For instance, for kaolin slurry at \(φ₀ = 8%\), Eq. [1] could describe the consolidation at testing time less than 1000 s. After the initial stage the settling becomes very slow (very small \(dy/dt\)) while the linear relationship breaks down. This observation corresponds to the observation by Shen et al. (1994). The yield stress of the investigated slurries could not sustain the integrity of the constant-\(φ₀\) regime which deteriorates continuously with time.

Table 1 lists the best-fit \(P_y(φ₀)\) and \(U₀K(φ₀)\) for the investigated slurries in their regimes. For both slurries \(P_y\) increased and \(U₀K\) decreased with increasing \(φ₁\). At the same \(φ₀\) kaolin slurries have the higher \(P_y\) but the lower \(U₀K\) than the ball clay slurries. Therefore the kaolin slurries are “stiffer” than the clay slurries. This observation corresponds to the greater \(β\) values for the clay slurries in Table 1 when compared with the kaolin slurries. This plastic characteristic apparently degrades in the subsequent settling stage. The \(dy/dt\) versus \(1/(y - L_c)\) data deviate from the linear regime at \(t > 1000\) s.

### Conclusions

This study utilized a CATSCAN to measure the spatio-temporal distributions of solids fraction for kaolin and clay slurries at various initial solid concentrations, \(φ₀\). The CATSCAN could not directly measure the null-stress solids fraction (\(φ₈\)) for the investigated slurries, whose values are estimated using an averaging technique to all around 0.07–0.08, regardless of the \(φ₀\).

The experimental range of \(φ₀\) (0.01–0.15) has covered both the \(φ₀ < φ₈\) and \(φ₀ > φ₈\) cases. The basic settling characteristics are similar for slurries with \(φ₀ > φ₈\) and vice versa. A constant-\(φ₀\) regime is noted for both cases, beneath which a sediment
layer forms. There existed a finite yield stress of the settling sediment indicating that the investigated sediments are plastic fluids. However, the final sediment equilibrated with gravity exhibited no constant-$\phi_0$ regime, which indicated purely elastic sediment. The network structure in the slurries degrades gradually in the settling process.

The model parameters of the theory by Buscall and White were regressed from the consolidating sediment data, while those by Tiller and Leu were obtained using the final sediment data. Both results indicate that the clay slurries are more compressible than the kaolin slurries. The higher initial solids fraction would also yield a “stiffer” sediment.

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