

# Cohesive Sediment Characterization by Combined Sedimentation and Rheological Measurements

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**Abstract:** A laboratory characterization of cohesive sediment has been carried out in which data obtained from standard sedimentation and rheological measurements were combined in a determination of the critical solid concentration for the detection of elasticity in a weakly cohesive suspension. The corresponding storage modulus and shear stress are very critical in any in situ rheometry of sediments, especially in the study of mud-water surface erosion in a flume. Sedimentation results showed that particle size distribution rather than surface treatment controlled the rheological behavior of the suspension while the critical solid concentration for the appearance of three-dimensional space-filling network, showing some measurable elasticity in the suspension, occurred in the region of 0.015. This parallel between the consolidation behavior and shear rheology development for the flocculating system has been established. This technique could be an adjunct to the laboratory characterization of cohesive sediments for the estimation of critical shear stress for surface erosion, especially in a typical flume experiment under water wave pressure.

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## Introduction

Natural coastal sedimentation processes often result in consolidated cohesive beds. The various settling phenomena and associated rheological responses of soft moveable cohesive beds are vital to our understanding of their dynamic properties due to, for example, external pressure produced by water wave action in a flume. An important parameter of interest in such investigations is the stress level at which elasticity would be expected to become measurable in the suspension which would have implications for the commencement of mud-water surface erosion.

This technical note reports on a laboratory investigation of the characterization of sedimentation and viscoelastic behavior of kaolinite. This should contribute to a better understanding of particulate suspension consolidation under a different chemical environment in the determination of storage modulus values in the low-solid-concentration ranges hitherto immeasurable with conventional instruments. It involved measurement of sedimentation behavior at different solid concentrations which was correlated with rheological measurements in the determination of a critical solid concentration for the formation of a three-dimensional (3D) network structure that must not be exceeded in

order to prevent mud surface erosion in a flume.

There has been substantial interest in settling and sedimentation phenomena in which several experimental and theoretical works have been reported. The early fundamental works include the theoretical exposition of Kynch (1952), the group settling rate concept of Richardson and Zaki (1954), and the settling and settling rate analysis of Michaels and Bodger (1962) representing classic works on sedimentation behavior of cohesive sediment suspensions. The works of Toorman and Berlamont (1991) on the mathematical modeling of cohesive sediment settling and consolidation, Torfs et al. (1996) on the settling and consolidation of mud/sand mixture, and Smith and Cheung (2003) on the settling characteristics of calcareous sand have also made significant contributions to this subject. With increasing need for in situ investigation of the dynamic mechanical behavior of cohesive beds, especially in estuaries and other marine environments, the need for an accurate means of assessing the erosion parameters under laboratory conditions cannot be overemphasized.

Answers to numerous questions are required on the conditions under which a flocculated cohesive sediment mud could suitably be used in flume experiments involving high-frequency in situ viscoelastic measurements (Williams and Williams 1992; Babatope et al. 2006). The establishment of an accurate relationship between sedimentation behavior and shear rheology is as vital to in situ measurement of dynamic rheological response of sediment beds as other established methods. A theoretical proposition relating to this problem has been presented earlier (Babatope et al. 1999).

In a dilute suspension, the sediment particle group settling rate is given as (Richardson and Zaki 1954; Michael and Bodger 1962)

$$U_0 = \frac{g(\rho_F - \rho_w)d_A^2}{18\mu_w}(1 - C_{AS}C_F)^{1/\alpha} \quad (1)$$

where  $g$  = acceleration due to gravity;  $\alpha^{-1}$  = constant with the value of 4.65;  $\rho_F$  = aggregate floc density taken to be the solid

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density because we are dealing with dilute concentration of the suspension;  $\rho_w$ =density of water;  $C_{AS}$ =ratio of aggregate concentration to that of the solid;  $C_F$ =solid concentration by volume;  $d_A$ =average or equivalent aggregate diameter that remains constant once settling has started (Michael and Bodger 1962);  $\mu_w$ =viscosity of water; and  $U_0$ =settling rate.

Substituting appropriate values and making necessary approximations of the various parameters for kaolinite, single aggregate settling can be described by

$$U_{SA} = K \left( \frac{d_A^2}{C_{AF}} \right) \quad (2)$$

where  $U_{SA}$ =Stokes settling rate for a single aggregate;  $C_{AF}$ =ratio of the aggregate concentration to that of the floc; and  $K$ =constant obtainable using appropriate values of the parameters in Eq. (1). It follows directly from Eqs. (1) and (2) (Michael and Bodger 1962) that

$$U = U_0^\alpha = U_{SA}^\alpha (1 - C_{AS} C_F) \quad (3)$$

A plot of  $U$  against  $C_F$  in Eq. (3) would yield  $C_F$  for zero settling rates by extrapolation. This is the required critical volume fraction.

## Experimental Procedure and Methods

### Materials and Apparatus

The material used in this investigation is a China clay (supreme kaolinite) obtained from English China clay (ECC), Cornwall, United Kingdom. The material was further treated to remove unwanted ions and organics, and particles larger than 10  $\mu\text{m}$  were removed using the sedimentation-decantation method. The particle size distribution was measured with an MS 21 Mastersizer (Malvern Instruments Ltd., United Kingdom). A pulse shearometer (Rank Bros, Bottisham, United Kingdom) was used for the measurement of shear rigidity modulus of the suspensions.

### Sample Preparation and Procedure

Homoionic Na (sodium)-substituted kaolinite samples were prepared for a series of standard settling and consolidation experiments including aluminum ion concentration determination using a combination of procedures (Williams 1976; Schofield and Samson 1954; Benne 1988; and Dougan and Wilson 1974). The particle size distribution of the decanted batch used for all sedimentation and rheological experiments had 90% volume below 5  $\mu\text{m}$  and 98% volume below 10  $\mu\text{m}$ , and the average size was 1.65  $\mu\text{m}$ . All suspensions were prepared in a  $10^{-3}$  N NaCl electrolyte by adopting a uniform mixing cycle to ensure specimen uniformity (Williams and Williams 1989).

Conventional sedimentation experiments were carried out in 2.5 cm diameter glass tubes in different chemical environments (by varying the pH level) at different volume fractions. The volume fraction (volume per volume,  $C_F$ ) was obtained by calculating the solid volume using the measured density of the suspension as a fraction of the electrolyte used. Settling rates were obtained for different volume fractions enabling the determination of optimum conditions for maximum settling rate in the appropriate chemical environment.

The rigidity modulus  $G^*$  was obtained with the pulse shear wave propagation technique (James et al. 1987; Williams and Williams 1989), which uses two parallel disks mounted on a pi-

ezoelectric crystal and dipped in a cup containing the sample. The shear wave generating system and shear wave characteristics were monitored by means of storage oscilloscope. The shear wave was initiated by an electrical pulse to one of the crystals (sender), which induced very rapid waves of small amplitudes on the order of  $10^{-5}$  rad torsional displacement of one of the disks relative to the other (the receiver) at a frequency of approximately 220 Hz. The measurement was only valid under the conditions of low attenuation in which  $\lambda/x_0 \ll 1$ , where  $\lambda$  is the shear wavelength and  $x_0$  is the attenuation constant. Under such conditions, the complex rigidity modulus,  $G^* = \rho v^2$ , is equal to  $G_\infty$ , the high-frequency limit of the shear rigidity modulus at infinite frequency;  $\rho$  is the sample density; and  $v$  the shear wave velocity (Goodwin et al. 1986). Furthermore, this method was used as a means of monitoring changes taking place in the different test specimens as an accurate measure of changes occurring in the structure of the suspension.

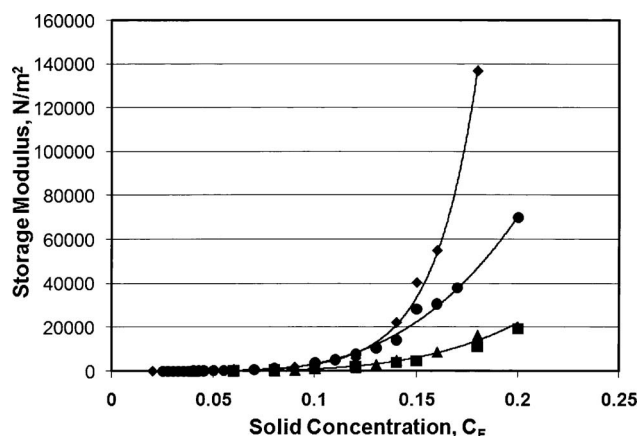
The shear wave propagation velocity was determined by measuring the time for the wave to travel in the clay between the disks that are separated at a given distance (Williams and Williams 1989; Whorlow 1992). The disk separation distance varied from a few millimeters to about 16 mm while the shear wave velocity  $v$  was calculated from the measured time of travel of the pulse. The shear wave velocity was determined for different volume fractions only when the measured amplitude variation was negligible. The wave attenuation constant  $x_0$  was determined with an oscilloscope by using a continuous wave from the shear wave generator while varying the path length in order to record the amplitude variation at a fixed frequency (Whorlow 1992).

## Results and Discussion

Standard settling experiments covering solid concentrations as measured by volume fractions in the dilute (0.003–0.005), intermediate (0.007–0.010), and high ( $>0.01$ ) range showed varying settling rates. Constant settling rates (the linear portion of settling height against time) were recorded in the dilute and intermediate ranges. The absence of constant settling at and above the volume fraction of 0.01 could signify the appearance of some elasticity in the suspension. However, available conventional rheological instruments would not be able to detect the onset of this particle interaction mainly due to the required high instrument resolution.

The shear wave propagation measurements carried out as a function of volume fraction shows the effect of particle size and surface treatment on the rheology of the suspension (Fig. 1). They suggest that surface treatment has only a negligible effect relative to size grading, because the highest shear rigidity modulus value was obtained in the case of samples that were untreated (but size graded) followed by the surface-treated samples. Furthermore, in the graded samples, the rheology appeared to be more sensitive to surface treatment as shown by comparison with the untreated specimens. It could therefore be concluded that the rheology of the suspension was sensitive to surface treatment for particle sizes substantially below 10  $\mu\text{m}$  which caused increased interparticle interaction that in turn led to higher rigidity modulus. In the comparative rheological and dynamic mechanical properties of a natural mud in a flume experiment, for example, sample surface treatment would have no significant effect.

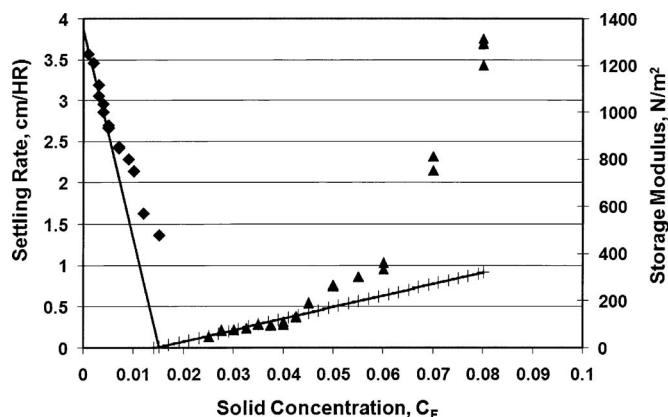
Fig. 2 shows the plots of both the settling rates (left curve) obtained from the average settling velocity in Eqs. (1) and (3) and wave rigidity modulus for dilute and intermediate solid concentration ranges against volume fraction  $C_F$ , for the surface-treated



**Fig. 1.** Shear rigidity modulus against solid concentration showing effect of particle size and surface treatment

sample. The settling experiment was carried out at pH levels between 4.5 and 6.5 in which the optimum pH was found to be 5 with respect to maximum settling rate. The results showed that the settling rate was not pH dependent, decreasing with increasing volume fraction. This is thought to be due to increased mutual hindrance of the particles during sedimentation as the concentration gradually increased.

Linear extrapolation of the two curves to zero settling rates, where the use of the power law as applied to the very dilute concentration of the suspension is applicable (and zero shear wave rigidity) suggests that within the limits of experimental error, there exists a volume fraction above which some elasticity is detectable in the suspension. This concentration, which is in the region of 0.015, can be regarded as a critical value for the onset of structure development due to the predominance of a 3D space-filling network and the appearance of measurable elasticity in the suspension. Here, a kind of “plug” settling became dominant during sedimentation which became more prominent at the transition between the intermediate and the high solid concentration regime. Here, sustained long-term floc formation and aggregation developed “card house-like” channels through which water gradually oozed upwards within the complex 3D network. As long as this



**Fig. 2.** Maximum settling rate (left axis) and wave rigidity modulus (right axis) against solid concentration for surface-treated and size-graded samples showing experimental and fitted curves and approximate critical volume fraction obtained by extrapolation to zero settling rate ( $\alpha$  is constant  $\approx 0.215$ ) (Michael and Bodger 1962)

dynamic could be sustained within the time available for rheological measurement, a finite value of rigidity modulus could be obtained depending on the sensitivity of the measuring instrument. Since this is not often realizable due to obvious limitations, the sedimentation data would then become an alternative means of estimating the value of rigidity modulus at the critical volume fraction, which is often very small. This value is useful in preparation for flume experiments for surface erosion study of suspensions.

It can be inferred from these measurements that the sedimentation and rheological data are internally consistent. It also shows that a critical volume fraction below which the structure would be completely fluid-like (i.e., no elasticity, zero storage modulus, and maximum loss modulus) could be determined using this method. It had been previously noted that there are parallels between the consolidation behavior and shear rheology development for flocculated systems (Buscall and White 1987). This work appears to present further evidence in support of this finding.

## Concluding Remarks

Rheological behavior of consolidating sediments could be captured using the reported technique and that it is just above the critical volume fraction that storage modulus,  $G'$ , becomes finite and measurable. A critical concentration of 0.05 for formation of the space-filling network had been reported for spherical particles (Buscall and White 1987); thus the lower value of about 0.015 obtained in this work could be attributed to the plate-like, flocculated, and anisotropic nature of kaolinite particles resulting in early development of structures due to a more efficient particle packing compared with spherical particles. The technique can be used as an adjunct in laboratory characterization of sediments for the determination of rheological parameters required in experiments for the estimation of erosion parameters for the prevention of surface erosion.

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