A fifty cent rheometer for yield stress measurement

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Synopsis

The slump test, initially developed to determine the flow properties of fresh concrete, has been adopted as a means of accurately measuring the yield stress of strongly flocculated suspensions. The slump test offers a quick and easy way of measuring yield stress without the need for sophisticated electronic equipment, thereby giving plant operators an effective tool for determining yield stress. The model used to predict the yield stress from the conical slump test was devised by Murata (1984) and corrected by Christensen (1991). In the present case the theory has been adapted for a cylindrical geometry. Yield stress measurements obtained with the vane for numerous mineral suspensions under known surface chemistry conditions are compared to the slump measurements and theoretical prediction. Good agreement is obtained. © *1996 Society of Rheology.*

I. INTRODUCTION

Several semiempirical models have been devised to characterize the rheological behavior of time-independent viscoplastic fluids [Nguyen and Boger (1992); Walters *et al.* (1989); Bird *et al.* (1983)]. Fundamental to all such equations is the existence of a yield stress. The yield stress is defined as the minimum stress required to cause the fluid to flow like a viscous material with finite viscosity. When the stress applied to the fluid is less than the critical stress (τ_y) the fluid does not flow but deforms plastically like a solid, with definite strain recovery upon the removal of the stress. The measurement of the yield stress is essential in understanding the flow properties of such materials.

Knowledge of the yield stress is essential in engineering design and operation where the handling and transport of industrial suspensions are involved. In particular, recent techniques developed for the disposal of mineral tailings suspensions depend critically on the yield stress [Williams (1992); Robinski (1975, 1979)]. Thickened discharge techniques [Cooling and Glenister (1991); Wood and Mcdonald (1986)] currently implemented as a means of tailings disposal in numerous mineral processing plants require the dilute mineral tailings leaving the plant to be concentrated to a high volume fraction before disposal. The yield stress of the resulting material is of critical importance in the implementation and control of the entire disposal strategy.

The current paper develops the slump test for yield stress measurement. The slump measurement consists of filling a cylindrical frustum with the material to be tested in the specified way, lifting the frustum off and allowing the material to collapse under its

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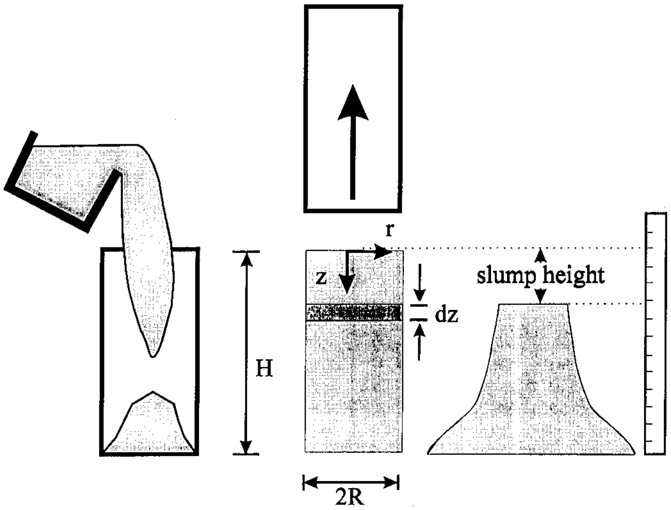

FIG. 1. Schematic diagram of the slump test with reference axis.

weight. The height of the final deformed, or slumped, material is measured. The difference between the initial and final heights is termed the slump height. Figure 1 illustrates schematically the experimental procedure.

The slump test is used extensively by civil engineers to estimate the "workability" of fresh concrete. Workability is a qualitative term used to describe a combination of effects due to varying yield stress and viscosity. The term arises from the need to know that fresh concrete will flow to its final position: if too stiff, the mixture will not flow into tight corners of moulding; conversely, if the concrete is too runny (water content is in excess), the ability of the concrete to flow is increased but the strength of the final hardened concrete is reduced. Therefore, it is necessary to strike a balance to optimize the flow properties. Similarly, the flow properties of a tailings in a waste disposal scheme need to be tailored for slope deposition [Ritcey, (1989)]. Too thin a material will result in little if any slope, while too thick a tailing will result in the material being deposited around the discharge point and not flowing over the disposal area.

The adaptation of the slump test to a circular cylindrical geometry was first made by Chandler (1986) in the alumina industry and is adopted here as the preferred geometry because of simplicity. Chandler failed to analytically relate the slump height to the yield stress but realized that the slump height was representative of the flow behavior of material he was examining, bauxite residue: "...it does measure a property which correlates well with the behaviour of mud on the drying site."

The theoretical analysis relating the slump height to the yield stress was first undertaken by Murata (1984) for a conical geometry. The materials used in the experiments were limited to concrete, and the results remain largely neglected by industry. Christensen (1991) corrected a simple integration error in the original Murata theory. Experimental results presented by Christensen exhibited a large degree of scatter because of uncertainties associated with the yield stress data provided by W. R. Grace and Company and from the original Murata paper (1984). In both cases, rotational devices were used for independently measuring the yield stress.

The slump experiments have been conducted using a cylindrical geometry for three well-characterized suspensions. Yield stress for each of the suspensions was indepen-

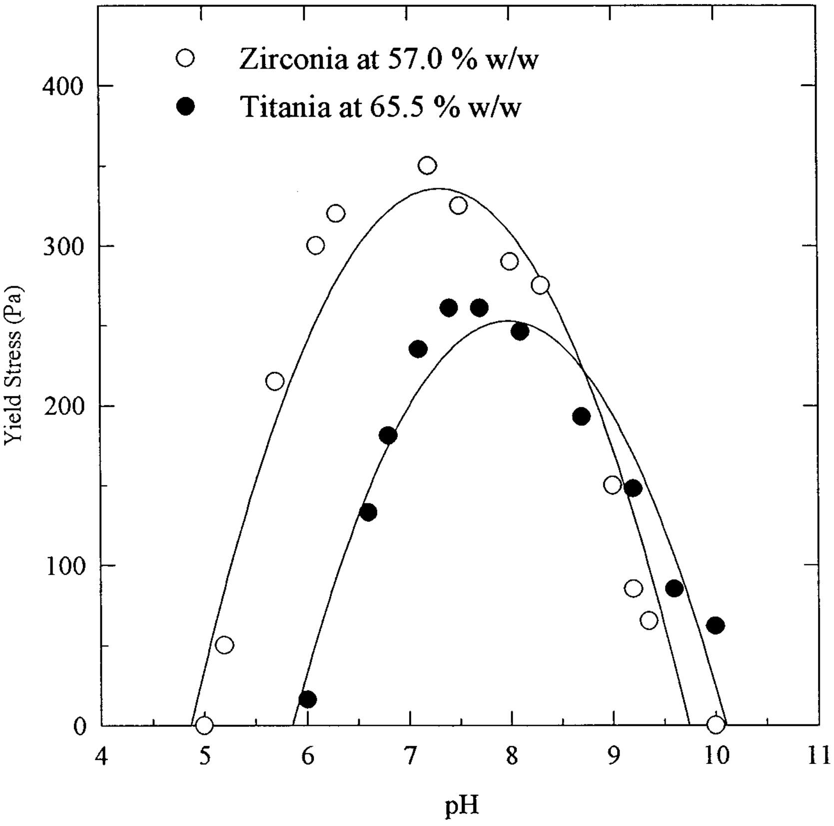

FIG. 2. Yield stress vs *p*H for titania and zirconia, from Liddell and Boger (1994) and Leong *et al.* (1993a, 1993b).

dently measured using the vane technique, a technique now well established for accurate yield stress measurement. The theory developed is then used to predict the yield stress from the measured slump, and finally, a comparison is made between the yield stress predicted from the slump measurement and the independently measured value with the vane technique. Good agreement is obtained.

II. MATERIALS, MEASUREMENTS, AND EXPERIMENTAL PROCEDURE

A. Materials

The data presented refer to experiments on well-characterized mineral suspensions. Experiments were conducted using three different mineral suspensions: titania, zirconia and bauxite residue from the Bayer process (red mud). The extensive synthetic flocculation of red mud with polyacrylamide during processing ensures the final material is strongly flocculated. The flocculation of the titania and zirconia is via *p*H adjustment. Figure 2 illustrates the yield stress, *p*H behavior of the two metal oxide samples. The results highlight the maximum in the yield stress behavior for the two samples. The *p*H at which the maximum occurs has been shown, via electroacoustic measurements, to coincide with the isoelectric point [Hunter (1993)]. At the isoelectric point the net surface charge on the particle is zero, and the interparticle forces are dominated by the van der

Waals forces of attraction [Hunter (1991); Shaw (1993)] resulting in the maximum in the yield stress. At the isoelectric point, a state of strong flocculation is known to exist, hence, all experiments presented are for strongly flocculated suspensions.

Titanium dioxide (TiO_2) supplied by Tioxide Chemicals Pty Ltd, was used. The material has been characterized by Liddell and Boger (1994), and has an isoelectric point of 7.6, and density ca. 4000 kg/m^3. Zirconia dioxide (ZrO_2) supplied by Z-Tech (an ICI Advanced Ceramics Australia subsidiary) was also used, and this material has been well-characterized by Leong *et al.* (1993a, 1993b) and found to have an isoelectric point of 7.2 and density ca. 5800 kg/m^3.

The samples were prepared by placing a known mass of dry solids into a large beaker and adding the required amount of Millipore "Milli-Q" water. Sonication was performed on the suspension for approximately 15 min with a high intensity sonic probe (Sonifier B30, 20 kHz, 350 W), creating a smooth, homogeneous suspension. Suspension pH was altered by addition of concentrated HNO_3 or NaOH ($\sim 10 \text{ mol dm}^{-3}$). All slump test experiments with titania or zirconia were conducted at the isoelectric point. The yield stress of these suspensions was varied by dilution with water along with the correct amount of acid\base to maintain the isoelectric point pH. The suspension was then sonicated for approximately 5 min, and allowed to stand for at least 2 h before conducting an experiment.

An industrial suspension of Bayer process residue was also used in a series of experiments. The material was supplied by Alcoa Australia's Kwinana operations, and is representative of a typical final stage thickener underflow. The material consists primarily of iron and silica oxides and has a solids density of ca. 3200 kg/m^3. The pH of the material was approximately 13.5. The material is an irreversible thixotropic material with a yield stress that is sensitive to shear history [Pashias and Boger (1994); Nguyen (1983)]. The yield stress of the bauxite residue was varied in two ways. In one instance dilution via the addition of excess plant liquor was undertaken. In another case the suspension was mixed for prolonged periods of time, causing structural decay and consequently a reduction in yield stress; this is referred to as the "reduced state." In flocculated bauxite residue suspensions the rate of structural breakdown in a shear field is rapid compared to structural recovery (irreversible thixotropy).

B. Measurement

Independent yield stress measurements were made using the vane technique [Nguyen and Boger (1983, 1985)]. An Orion pH meter was used for pH measurement. Four slump cylinders were used, with aspect ratios of 0.78, 0.97, 1.17, and 1.28. Three of the cylinders were constructed from PVC pipe, while the fourth was made from stainless steel.

C. Experimental procedure

The experimental procedure for the slump test is outlined in British Standard 1881 (1970). In the present case a cylindrical frustum was filled with the material to be tested. After filling the cylinder, a thin spatula was used to remove any air bubbles trapped in the material, the top of the cylinder was then smoothed over, and the frustum lifted off manually. Care was taken in order to lift the cylinder evenly; the resulting height was then measured with a ruler. Due to the uneven nature of the resulting top surface, the middle point of the slumped material was taken as the representative height. Heights were measured to the nearest 0.5 mm. Density and concentration were also measured at the time of testing.

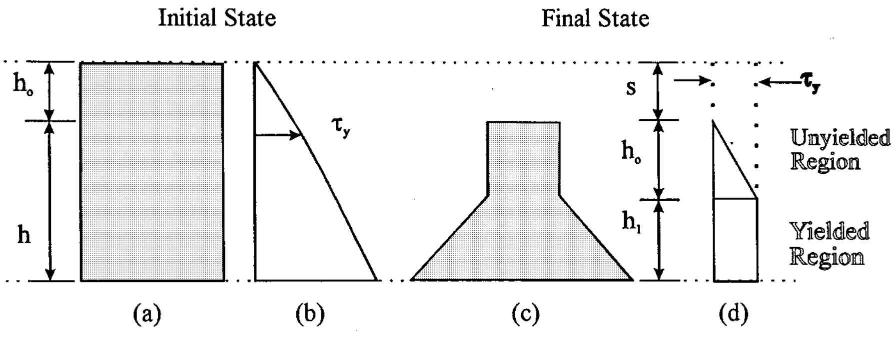

FIG. 3. Schematic diagram of the initial and final state stress distribution.

III. THEORY

The analytical model devized by Murata (1984) relating yield stress to the slump height has been adopted for a cylindrical geometry [as pioneered by Chandler, (1986)]. Figure 1 schematically represents the undeformed material. At any given height, z, the pressure, P, can be approximated as

$$P|_z = z\rho g. \tag{1}$$

For an ideal elastic solid the maximum shear stress that can act on a body when a pressure (P) is applied to it in a normal direction is equal to half the pressure [Hibbeler (1991)]. Hence, the maximum shear stress (τ) at a height z can be expressed as

$$\tau|_z = \tfrac{1}{2} z\rho g. \tag{2}$$

Scaling the stress with $\rho g H$, Eq. (2) in dimensionless form becomes

$$\tau|_z' = \tfrac{1}{2} z', \tag{3}$$

where $\tau|_z'$ and z' are the scaled variables.

Equation (3) illustrates that there is a linearly increasing stress distribution along the height of the cylinder, ranging from zero at the top to a maximum at the base. Figure 3 illustrates schematically the slumping behavior and the corresponding stress profile throughout the material. At some point along the height of the undeformed cylinder [Fig. 3(a)], the material will experience a stress that is larger than the yield stress [Fig. 3(b)], while above this point the stress experienced by the material is less than the yield stress. Once the slumping is complete [Fig. 3(c)], the stress distribution is as shown in Fig. 3(d). In the upper, unyielded section the stress distribution is identical to that of the undeformed material, while flow occurs in the remaining material until the stress is reduced to the yield stress. The height of the unyielded material, denoted by the symbol h_0, or h_0' in dimensionless form, can be calculated by substituting τ_y' in place of τ_z' into Eq. (3).

The interface layer between the yielded and unyielded material is assumed to be a flat surface that moves down as the material beneath it flows. The initial height is reduced to a total final height (h) that is made up of two components, the height of the yielded region (h_1) and the remaining nonyielded region (h_0). In the portion of the material that undergoes yielding, the height can be divided into elements of thickness dz, which reduce to a thickness dz_1 once yielding is complete. During the deformation stage it is assumed that all horizontal sections remain horizontal, and slumping is only due to radial flow.

By assuming incompressibility, the thickness dz_1 can be related to dz by

$$dz_1 = \left(\frac{r|_z}{r|_{z1}}\right)^2 dz. \tag{4}$$

The height h_1 can then be evaluated by integrating dz_1,

$$h_1 = \int_{h_0}^{H} dz_1. \tag{5}$$

Since it is assumed that there is no flow between horizontal planes, the amount of material above any given plane will be the same before and after the slump. Flow occurs until the cross-sectional area increases so that the stress required to support the weight is reduced to the yield stress. Thus, another relationship between the original and final elements is that the product of stress times the cross-sectional area is proportional to the weight of material above the plane resulting in the following expression:

$$(r|_z)^2 \tau|_z = (r|_{z1})^2 \tau_y. \tag{6}$$

By substituting Eqs. (3), (4), and (6) into Eq. (5), the following equation in dimensionless form is obtained:

$$h_1' = \int_{h_0'}^{1} \frac{\tau_y'}{\frac{1}{2}z'} dz'. \tag{7}$$

Integrating Eq. (7) yields

$$h_1' = -2\tau_y' \ln(h_0'). \tag{8}$$

The value of h_0' to be used in Eq. (8) can be evaluated from Eq. (3) as

$$\tau_y' = \tfrac{1}{2}h_0'. \tag{9}$$

The dimensionless slump value can then be calculated via

$$s' = 1 - h_0' - h_1'. \tag{10}$$

Solving Eqs. (8)–(10) yields

$$s' = 1 - 2\tau_y'[1 - \ln(2\tau_y')]. \tag{11}$$

IV. RESULTS AND DISCUSSION

A series of tests were designed to determine if the slump test is suitable for yield stress measurements in the field. The sensitivity of the slump height to sample structure, material, aspect ratio, lift rate, and measurement time were investigated. The experimental results obtained are compared to the theoretical predictions from Eq. 11 and to the independent yield stress measurements. Results from slump test experiments undertaken to determine the validity of the theoretical derivation are illustrated in Fig. 4. A typical bauxite residue suspension was initially used as the test fluid.

Figure 4 compares the dimensionless slump height with yield stress (as measured using the vane technique) for red mud in two structural states. The slump height is found to decrease with increasing yield stress; that is, a material with a large yield stress undergoes only a small amount of slumping compared to a material with a lower yield stress. The extensive dependence of the red mud flow properties on the structured state is ideal for measuring yield stress as a function of structural state to determine if the slump height is a unique function of the yield stress. Despite the inability to determine the exact

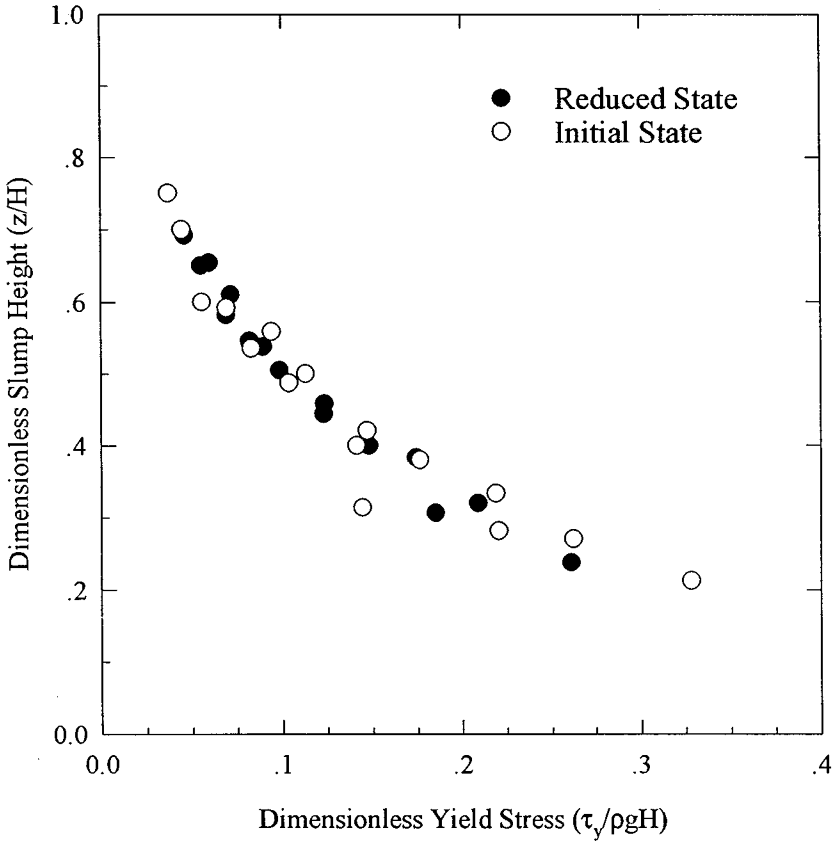

FIG. 4. Dimensionless slump height as a function of yield stress for a typical bauxite residue (red mud) sample.

structural state of a given red mud sample, slump measurements with this material provide an excellent test to establish that the slump height is a unique function of yield stress.

To further confirm that the slump height is dependent on yield stress, a series of experiments using red mud was undertaken to measure the yield stress and slump height as a function of agitation time of the red mud. In order for structural breakdown to occur, the sample was mixed with the aid of an anchor agitator for approximately 140 h. Yield stress measurements were performed at various times throughout the mixing process using the vane technique and slump test. Figure 5 shows the yield stress and slump height as a function of agitation time.

Figure 5 highlights the similar behavior of the dimensionless yield stress and dimensionless slump height with agitation time. Initially, the material exhibits a high yield stress resulting in a small slump height. As the material is sheared, the yield stress decreases and consequently the slump height is increased. Figure 5 suggests that the slump test is capable of measuring yield stress regardless of the time dependent nature of the material.

Figure 6 illustrates the slump height as a function of yield stress for numerous suspensions (titania, zirconia, and red mud) along with the theoretical model derived earlier. All data are presented in dimensionless form. The various suspensions tested fall on a

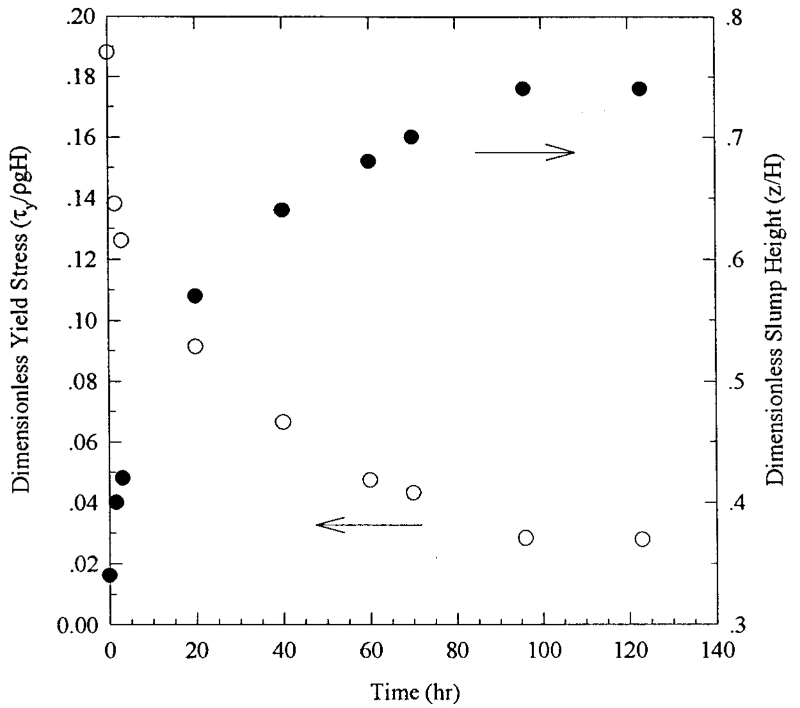

FIG. 5. Yield stress and slump height as a function of agitation time for a bauxite residue.

common curve, confirming that the slump height is independent of the material being tested and is a unique function of the yield stress. As a consequence, the slump test offers a direct measurement of the yield stress as no prior knowledge of the system is required.

The range of yield stress values capable of being measured in this work is from approximately 30–800 Pa, depending on which material and slump cylinder are used. The range is primarily limited by physical constraints. At very low yield stress values the material will spread over a large area leaving only a thin layer of unyielded material, thereby making it difficult to measure the resultant height. For yield stress values greater than 800 Pa, the material will slump only slightly, making the difference between the initial and final heights minimal.

Also shown in Fig. 6 is the slump prediction as a function of yield stress [Eq. (11)]. Agreement of the experimental results with the theoretical prediction is excellent at dimensionless yield stress values less than 0.15, with the error being approximately 10%, which represents a true yield stress value of approximately 200 Pa. Beyond this point the experimental results deviate from the exact solution. At higher yield stress values of 0.3, the experimental results again approach the exact solution.

For those wanting a simple expression for the yield stress as a function of slump, Eq. (11) can be approximated by expansion of $\ln 2\tau'$ into the infinite series

$$\ln 2\tau_y' = (2\tau_y' - 1) - \tfrac{1}{2}(2\tau_y' - 1)^2 + \tfrac{1}{3}(2\tau_y' - 1)^3 + \cdots$$

for $1/2 \geqslant \tau_y > 0$.

Using only the first term of the expression reduces Eq. (11) to

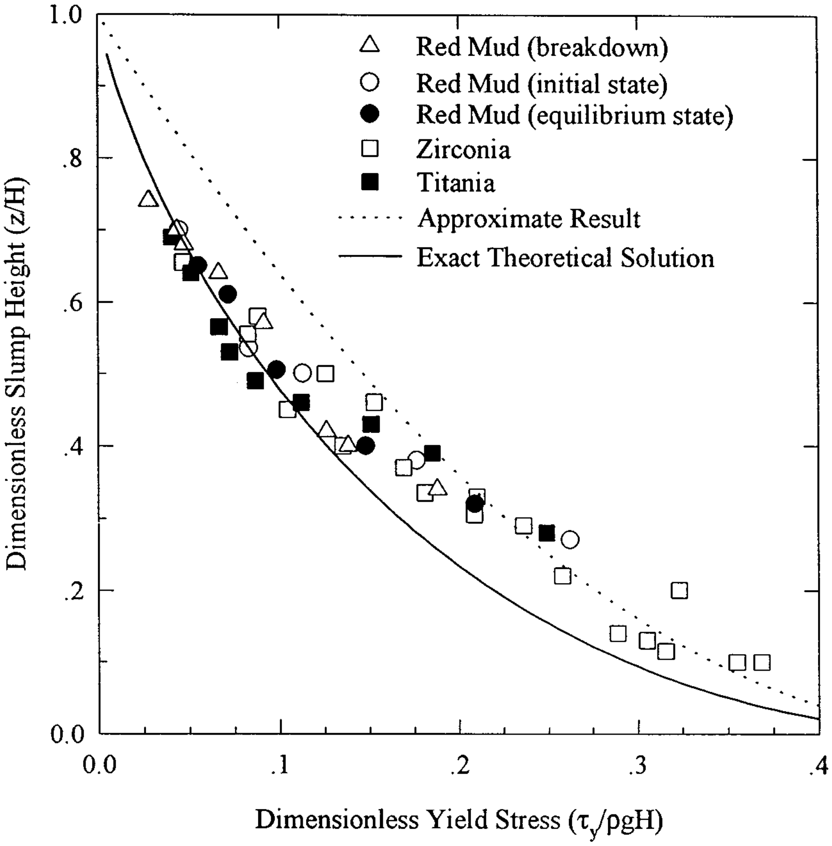

FIG. 6. Dimensionless height as a function of dimensionless yield stress for various suspensions.

$$\tau'_y = \tfrac{1}{2} - \tfrac{1}{2}\sqrt{s'}. \qquad (12)$$

The dotted line in Fig. 6 shows the approximate prediction.

The explanation why the theoretical model fails to predict the experimental behavior may be due to the simplified approximations made in the theoretical derivation. First, the relationship between the pressure distribution in the cylinder to the stress distribution may not be entirely valid, and is likely to be an over simplification for the system being modeled. The theoretical assumption made is valid for an ideal elastic solid. Clearly, the particulate system at hand does not fall within this category as the so called solid begins to flow at shear stress values greater than the yield stress. Moreover, the assumption that all horizontal planes remain horizontal may also lead to erroneous conclusions. Clearly, during the lifting of the frustum the friction between the walls and the suspension causes the outer layer of the material to be dragged up with the frustum. Furthermore, the theory fails to take into consideration any effect of friction with respect to the base and wall surfaces.

Experiments were undertaken to quantify the effect of the speed of removing the frustum on the final slump height. The frustum was lifted at various speeds with the aid of a variable speed motor and pulley system. Velocities from 0.1 to 30 m/s were tested. The velocity was found not to have any effect on the final slump height provided it remained less than ca. 10 m/s. At velocities greater than this, the slump height (s')

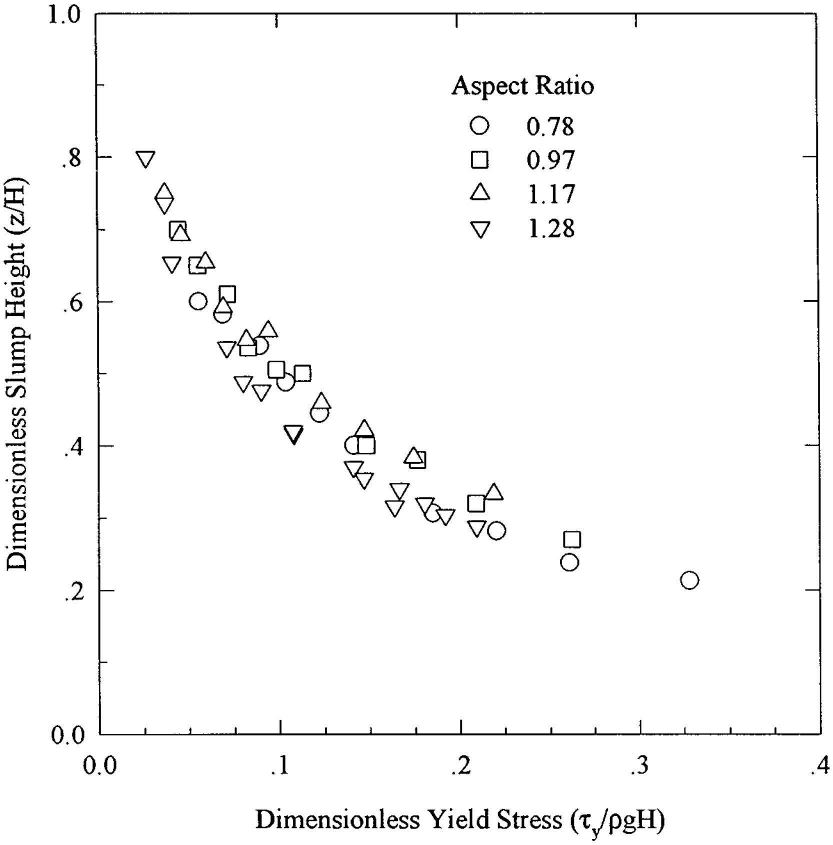

FIG. 7. Dimensionless slump height as a function of dimensionless yield stress for various aspect ratio slump cylinders.

increases linearly with velocity. The average manual (by hand) lift of a cylinder would be less than 1 m/s, well below that required to see any effect. Hence, for all practical purposes, the final slump height is independent of the velocity with which the frustum is lifted.

The time taken to measure the slump height after flow was also varied to determine whether the final height deviated with time. The height of the slump was measured at various intervals over a 1 h period. No measurable difference was detected in slump height. The surface on which the tests were conducted was also varied to determine if this has any effect on the final height. Various surfaces were tested ranging from rubber, smooth wood, rough wood, and stainless steel, and again no measurable difference was found. Consequently, it may be concluded that the frictional resistance to flow at the base is not an important variable and, hence, need not be built into the model.

Experiments were conducted on red mud to determine the effect of aspect ratio ($H/2R$) on the slump height–yield stress behavior. Figure 7 shows the results obtained using four cylinders. The aspect ratios considered were restricted to a value of approximately 1, due primarily to practical constraints: too large an aspect ratio would result in the collapse of the cylinder rather than the material flowing. Conversely, too small an aspect ratio would result in only a small degree of slumping and, consequently, only a small range of yield stress values being measured.

Figure 7 suggests that there is little (if any) effect of aspect ratio on the dimensionless quantities measured. It is difficult to conclude whether the scatter in the data is due to an aspect ratio dependence or the experimental errors associated with the experiment.

V. CONCLUSION

The slump test has been shown to provide a simple inexpensive and effective method for measuring yield stress for highly flocculated suspensions. The method is particularly useful where the provision of sophisticated electronic equipment is not possible. The measurement is insensitive to the type of flocculated material being tested. Furthermore, the velocity with which the frustum is lifted and the surface on which the experiment is undertaken have no effect on the final slump height. The slump test offers a robust and inexpensive method of directly measuring the yield stress, which plant operators can immediately and confidently use.

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