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The influence of wall slip on yield stress and viscoelastic measurements of cement paste

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Abstract

The influence of wall slip on the shear yield stress and modulus of cement paste was investigated using a rotational rheometer with smooth-walled concentric cylinders and a vane. The results show that the concentric cylinders suffer from slip during yield stress measurements due to the formation of a water-rich layer at the walls of the cylinders. The use of a vane eliminates slip since shearing occurs within the material. Oscillatory tests were conducted to measure the viscoelastic properties of cement paste. The data for the vane and concentric cylinders are in excellent agreement at stresses below the yield point. It is difficult to determine the influence of slip for stress sweep measurements. Frequency sweep data for various materials show the general applicability of the vane method over several decades of modulus. © 2001 Elsevier Science Ltd. All rights reserved.

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1. Introduction

Controlling the flow behavior of cement paste is critical for successfully processing cement-based materials, including concrete, mortar, and extrudable products. The development of self-compacting concrete by Okamura [1] is one example of the tremendous benefits obtained by using rheology as a design parameter.

The rheology of fresh cement paste is controlled by the structure of the three-dimensional network of cement particles in water. A 'gel' structure forms immediately after water is introduced to cement powder. The structure arises from a combination of colloidal forces (e.g. van der Waals attraction and electrostatic repulsion), hydrodynamics, and chemical reactions producing calcium silicate hydrates [2]. The load-bearing capabilities of the network result in the viscoelastic properties of cement paste, common of ceramic suspensions [3].

At low stresses, cement paste is very viscous, resembling an elastic solid. Over a very narrow stress range, the viscosity drops several orders of magnitude and macroscopic flow occurs. The critical stress range is very small and often designated as a single point, called the 'apparent' yield stress [4].

Various empirical and theoretical models have been used to determine the yield stress of cement paste based on data obtained from flow curve measurements. Among the most widely used are the Bingham and Herschel–Bulkley models [2,5–8]. Although these models can provide a reasonable estimate of yield stress, the measurements are highly dependent on the model assumptions, accuracy of the experimental data, and rheometer specifications [8]. In addition, large errors in determining yield stress can result by selecting the wrong shear rate range to fit the models [4].

The best way to determine yield stress is to directly measure the stress required to initiate measurable flow. Several types of tests have been developed and Nguyen and Boger [9] present an excellent review of this subject. Creep recovery [10] and stress growth [11,12] are among the techniques that have been used to directly determine the yield stress of cement paste.

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At stresses below the yield stress, the viscoelasticity of the network structure can be evaluated using oscillatory testing by applying a sinusoidally varying stress[1] [Eq. (1)]:

$$\tau = \tau_o \sin(\omega \cdot t) \quad (1)$$

where τ_o is the maximum applied stress amplitude, ω is the angular velocity, and t is time. The measured strain is a sinusoidal function taking the form [Eq. (2)]:

$$\gamma = \gamma_o \sin[(\omega \cdot t) + \delta] \quad (2)$$

where γ_o is the maximum strain amplitude and δ is the phase shift representing the degree to which the strain is out of phase with the applied stress. The ratio of the applied stress to the maximum strain is called the complex modulus [Eq. (3)].

$$G^* = \frac{\tau_o}{\gamma_o} \quad (3)$$

The complex modulus can be divided into an elastic and viscous portion representing the magnitude of the strain in-phase and out-of-phase with the applied stress, respectively [Eq. (4)].

$$G^* = G' + iG'' \quad (4)$$

The elastic component is called the storage modulus [Eq. (5)] and the viscous component is called the loss modulus [Eq. (6)]. The phase shift is expressed as the ratio of the two moduli [Eq. (7)].

$$G' = G^* \cos\delta \quad (5)$$

$$G'' = G^* \sin\delta \quad (6)$$

$$\tan\delta = \frac{G'}{G''} \quad (7)$$

A perfectly elastic material exhibits no phase shift between the applied stress and resultant strain ($\delta = 0$). Conversely, a perfectly viscous material is 90° out of phase with the applied stress and $G^* = G''$. Below the yield stress, cement paste behaves like a viscoelastic solid with $G^* \sim G'$.

There have been relatively few studies examining the viscoelasticity of cement paste due primarily to equipment limitations [13]. In many of the reported studies, the experiments were not conducted within the linear viscoelastic region (i.e. below the yield stress). Consequently, the results do not accurately reflect the true viscoelastic properties of the paste [14–16]. Using a highly sensitive controlled-stress rheometer, Struble and Schultz [13] showed that the critical strain limit for cement paste is on the order of 10^{-4}. Assuming a yield stress less than 100 Pa, these results suggest that the complex modulus (G^*) of cement paste should be on the order of 10^5 Pa.

[1] Equivalently, a controlled strain can be applied and the resultant stress measured.

1.1. Wall slip

Concentric cylinders are normally used in rotating rheometers to measure the yield stress and viscoelastic properties of cement paste [Fig. 1(a)]. During testing, a solvent-rich layer develops due to the displacement of the cement particles away from the smooth walls of the cylinder. The development of this water-rich layer produces a lubricating effect, making flow easier and not representative of the bulk material. This phenomenon, called 'slip,' is most pronounced at stresses near the yield point [17].

Slip is the result of both static and dynamic effects. The static effects arise from the physical depletion of cement particles away from the area next to the cylinder walls. This depletion is the result of both particle-packing characteristics and the disruption of Brownian motion near the solid boundaries. Electrostatic and steric effects can also arise between the cylinder walls and the particles due to a variety of physical and chemical forces [17]. The dynamic effects stem from hydrodynamic forces moving particles away from the walls when torque is applied. As the torque is increased, a shear force gradient develops, contributing to the slip phenomenon. Gravitational forces can also enhance the slip effect in systems that are known to sediment, such as cement paste [17].

The width of the slip layer is normally on the order of 0.1–10 μm. As the solids concentration increases, the size of the slip layer decreases, but the influence of slip becomes more dominant [17]. The high solids concentrations, typical of most cement pastes, make slip a likely scenario in experiments using smooth-walled concentric cylinders.

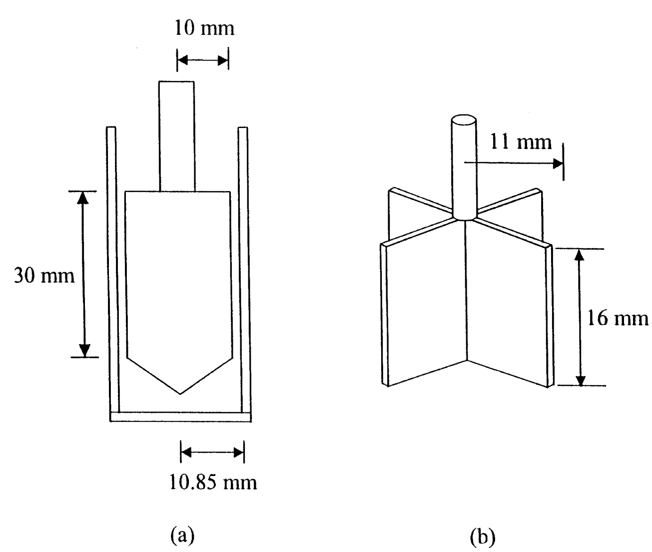

Fig. 1. (a) Concentric cylinders and (b) vane.

Slip was first shown to occur in rheological measurements of cement paste by Wesche et al. [18] who compared the results of flow curve experiments using smooth-walled and serrated cylinders. Slip was most pronounced at low strain rates and led to unusually low viscosity readings. As the strain rate was increased, the influence of slip decreased. Other studies have shown similar results for cement paste [19,20] and for a variety of suspensions [17,21,22].

1.2. Vane method

Slip can be reduced, if not completely eliminated, by profiling or roughening the walls of concentric cylinders [17]. An increasingly popular technique involves using a vane geometry, since slip is physically impossible [Fig. 1(b)].

A vane normally has four to six blades extending outward from the shaft at equal angles. The great advantage of the vane method is that slip is eliminated since shearing occurs completely within the material along the localized surface circumscribed by the vane [8]. Another advantage is that insertion of the vane results in much less disruption of the material in comparison to concentric cylinders. This is particularly advantageous due to the irreversible structural breakdown and thixotropy associated with shearing cement paste [2].

The torque on the vane surface is calculated by replacing the vane with a cylinder of equal dimensions. It is assumed that the force due to shearing is distributed uniformly over the entire cylindrical surface [23]. The torque is given by:

$$T = 2\pi r_v^2 H \tau_c + 4\pi \int_0^{r_v} \tau_e r^2 \mathrm{d}r \tag{8}$$

where T is the torque, r_v is the radius of the vane, H is the height of the vane, τ_c is the shear stress at the radius of the vane, τ_e is the shear stress at the top and bottom surfaces of the vane, and r is the radial distance from the center of the vane. Assuming that the stress is uniformly distributed along the top and bottom surfaces and τ_e equals τ_c when r equals r_v, Eq. (8) reduces to Eq. (9):

$$T = 2\pi r_v^3 \tau_c \left(\frac{H}{r_v} + \frac{2}{3}\right) \tag{9}$$

Keentok et al. [24] modeled the shearing surface around a vane in two dimensions by a finite element method. The results from the model agree with their experimental data, showing that shearing occurs along the cylinder circumscribed by the vane. The model also predicts stress concentrations at the blade tips. Finite element modeling by Christensen [25] also concluded that the yield surface existed along the circumference of the vane. However, the results suggest that the yielding zone is much smaller than what Keentok's model predicts. Yan and James [26] used a more detailed finite element mesh to model shearing in a vane for a variety of fluids. Their results confirm those of Keentok et al. and Christensen concerning the cylindrical conformity of the shearing surface. However, they go on to state that the concentration of shear stresses at the tips of the blades is not as pronounced as Keentok et al. suggests.

The vane method has become a popular technique for evaluating the yield stress of suspensions, emulsions, and pastes [27–31]. Haimoni and Hannant [11] measured the yield stress of cement paste using a vane and found it to be greater than twice the value measured using smooth-walled concentric cylinders. They also found the yield stress to be dependent on the rotational speed of the vane. Banfill and Kitching [32] also reported a large difference in yield stress between vane and parallel plate measurements, attributing the differences to slip.

There are very few studies reported where a vane has been used to measure viscoelastic properties. Yanez et al. [33] showed excellent agreement between vane and concentric cylinder measurements of G' for alumina slurries. The results did, however, show differences in the values of G'' over several decades of angular velocity and at low strains. Zhang et al. [34] used a vane to measure the modulus of foams and found no difference in G' or G'' between vane and roughened parallel-plate measurements. There are no studies reported where the vane has been used to measure the viscoelastic properties of cement paste.

The objective of this research program is to evaluate the influence of wall slip on yield stress and viscoelastic measurements of cement paste. A vane shear is used to eliminate slip, and results are compared with data obtained using smooth-walled concentric cylinders.

2. Experimental procedure

Type I Portland cement and deionized water were used for all experiments at water to cement ratios (w/c) of 0.30 and 0.40. Each batch was mixed in a mechanical mixer conforming to the specifications of ASTM C-305 [35]. The water was first placed in the mixing bowl and the cement was added over a 1-min period, while the mixer was at setting No. 1. The batch was then mixed at setting No. 2 for 2.5 min, after which the sides of the bowl were scraped with a rubber paddle. Finally, the sample was mixed for an additional 2.5 min for a total of 5 min of mixing at setting No. 2. This mixing procedure was strictly followed to avoid any experimental complications arising from incomplete mixing [36].

A Haake Rheostress 150 rheometer (Haake, Karlsruhe, Germany) was used for all of the testing. The dimensions of the concentric cylinders and vane used for both the yield stress and viscoelastic experiments are given in Fig. 1. The inner radius of the cup used with the vane was 22 mm, conforming to the suggested procedure of Dzuy and Boger [23]. A solvent trap was used with both

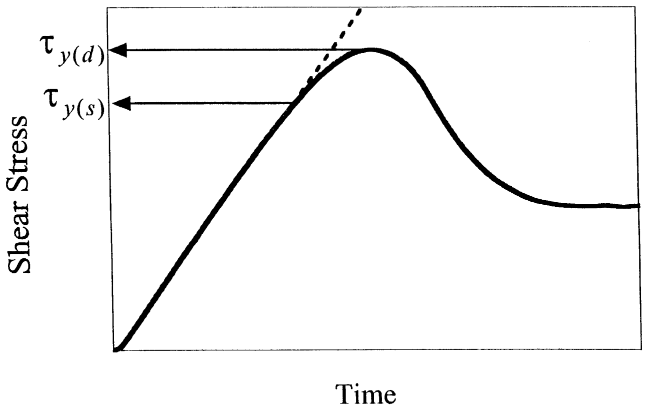

Fig. 2. Stress response for constant rotational speed yield stress measurements. The static yield stress ($\tau_{y(s)}$) represents the onset of viscoelasticity. The peak stress corresponds to the dynamic yield stress ($\tau_{y(d)}$).

the concentric cylinders and vane to prevent drying of the samples.

2.1. Yield stress measurements

Cement paste was placed in the rheometer immediately after mixing. Yield stress was measured via a stress growth experiment [9]. A constant rotational speed was inputted into the rheometer program ranging from 0.001 to 1 rad/s. Shear stresses developed in the sample as the rheometer tried to move the spindle to meet the designated rotational speed (Fig. 2). Initially, the sample deforms elastically due to stretching of the bonds in the network structure. At some point, the network begins to break under the shear stress as localized bonds reach their elastic limit. The development of viscoelastic effects is represented by the departure from linearity of the shear stress–time profile as shown in Fig. 2. At some point, the structure breaks down completely and a maximum stress is obtained. Finally, the stress decays to some equilibrium value as the vane begins to rotate [8].

Two yield stresses can be measured as shown in Fig. 2. The first yield stress ($\tau_{y(s)}$) represents the onset of viscoelasticity in the material. This is often referred to as the static yield stress since macroscopic flow has not occurred. The peak of the shear stress–time profile is called the dynamic yield stress ($\tau_{y(d)}$), denoting the onset of viscous flow. Traditionally, the dynamic yield stress is taken as the true yield stress of the material, since it represents the full breakdown of the structural network [28]. For this paper, the reported yield stress refers to the peak of the shear stress–time profile (i.e. dynamic yield stress).

2.2. Viscoelastic testing

The viscoelastic properties of cement paste were evaluated by oscillatory testing. As outlined in the Introduction, a sinusoidally varying stress was applied and the resultant strain was measured. For cement paste, the phase shift angle is very small, thus, the complex modulus is very close in value to the storage modulus in the linear viscoelastic region (i.e. below the yield stress).

Stress sweeps were performed using both concentric cylinders and a vane. For all tests, the frequency was 1.0 Hz. Frequency sweeps were also preformed at angular velocities ranging from 0.063 to 62.8 rad/s. The applied stress during the frequency sweeps was well below the yield stress of the particular sample.

As mentioned previously, there have been relatively few studies reported using oscillatory shear to probe the viscoelastic properties of fresh cement paste. This is a result of experimental difficulties in measuring modulus on the order of 10^5 Pa or higher [37]. Allowing the sample to sit in the rheometer for several minutes after mixing resulted in less data scatter and more reproducible measurements, since higher stresses could be applied during testing. For this reason, all oscillatory tests were started exactly 15 min after the cement powder was first added to the water.

3. Results and discussion

Fig. 3 shows results from a typical stress growth experiment. The data obtained using the vane are very similar to the expected behavior (Fig. 2). However, the curve obtained using the concentric cylinders clearly shows slip occurring. Initially, stress develops in the sample as expected with the concentric cylinders. At some point, there is an instantaneous decrease in stress due to the formation of a slip layer. The spindle rotates very slightly until its rotation is resisted by cement particles. The stress begins to develop again until slip occurs. This process continues, producing the characteristic 'sawtooth' type behavior. The large difference

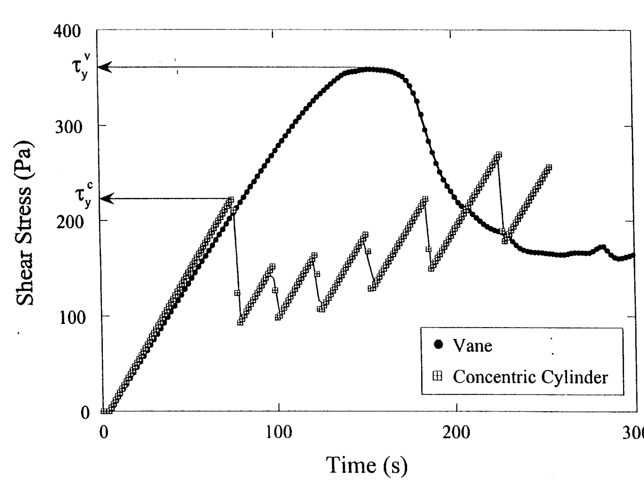

Fig. 3. Representative stress growth curves for concentric cylinders and vane. Rotational speed = 0.01 rad/s and w/c = 0.30.

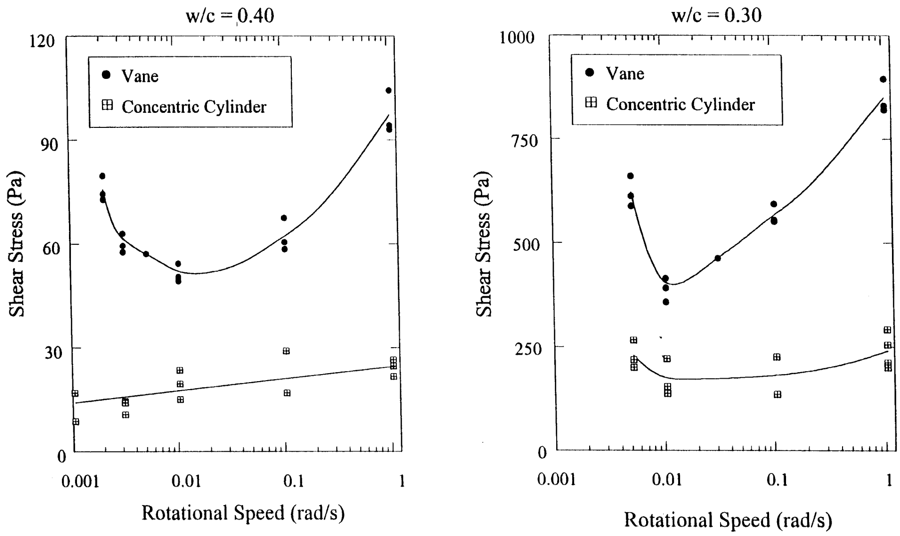

Fig. 4. Influence of rotational speed on yield stress.

between the peak stress measured using the vane and smooth-walled concentric cylinders can be attributed to the initial development of the slip layer.

The influence of rotational speed on yield stress measurements is shown in Fig. 4. The data for the vane show a minimum in yield stress at rotational speeds near 0.01 rad/s for both solids concentrations. At the minimum of the shear stress–rotation speed curves, the stress applied by the rheometer is just great enough to overcome the restoring forces due to reorientation of the particles into a more preferential, load-bearing structure, thixotropic recovery of broken bonds, and structural development due to hydration reactions. This minimum stress corresponds to the apparent yield stress of the paste.

The shape of the curves in Fig. 4 is very similar to the results of Liddell and Boger [28] who measured the yield stress of TiO_2 suspensions using a vane. The results also agree with the trends shown by Haimoni and Hannant [11] for cement paste.

The development of stress in the sample is extremely slow and the time scale of the measurement is much longer at low rotational speeds (Fig. 5). In this case, the sample is able to withstand higher stresses before yielding due to the reorientation, hydration, and recovery mechanisms. Conversely, at high rotational speeds, the time scale of the measurement is very short, approximately 1–3 s (Fig. 5). The stress applied by the rheometer rips apart the structural network bonds, leading to an increase in yield stress.

The yield stress measured using concentric cylinders appears to be largely independent of rotational speed (Fig. 4). This data agrees with the concept that a water-rich slip layer, with a solids concentration lower than the bulk material, forms during the experiment. At high rotational speeds, there is less structure present at the walls of the cylinders to plastically deform. Thus, the increase in yield stress at high rotational speeds is much lower in comparison to the vane measurements. For the same reason, the influence of thixotropic recovery or particle reorientation is not as influential at lower rotational speeds.

The yield stress measured at the minimum of the vane curves is almost twice as great as that measured using concentric cylinders. These results are in excellent agreement with the data of Haimoni and Hannant [11]. The results from Figs. 3 and 4 indicate that the measured yield stress of cement paste can be inaccurately low if slip is not eliminated.

Data from stress sweep experiments show excellent agreement between the vane and concentric cylinder mea-

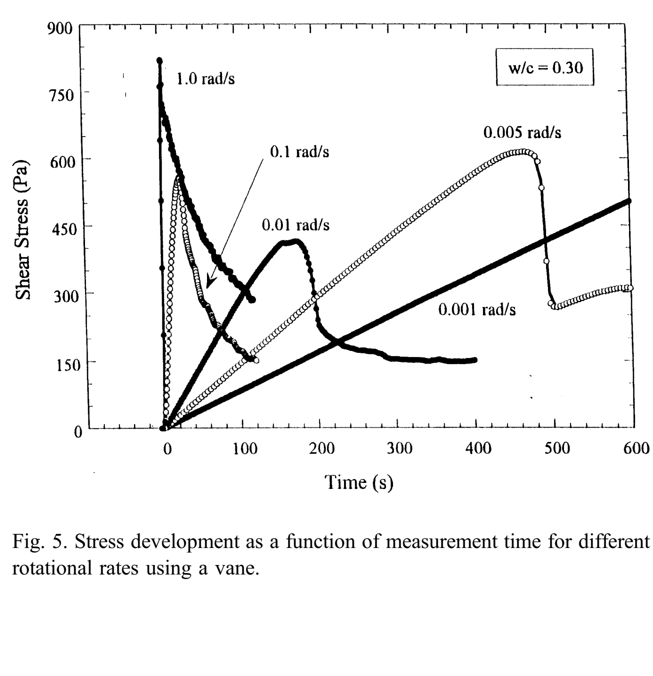

Fig. 5. Stress development as a function of measurement time for different rotational rates using a vane.

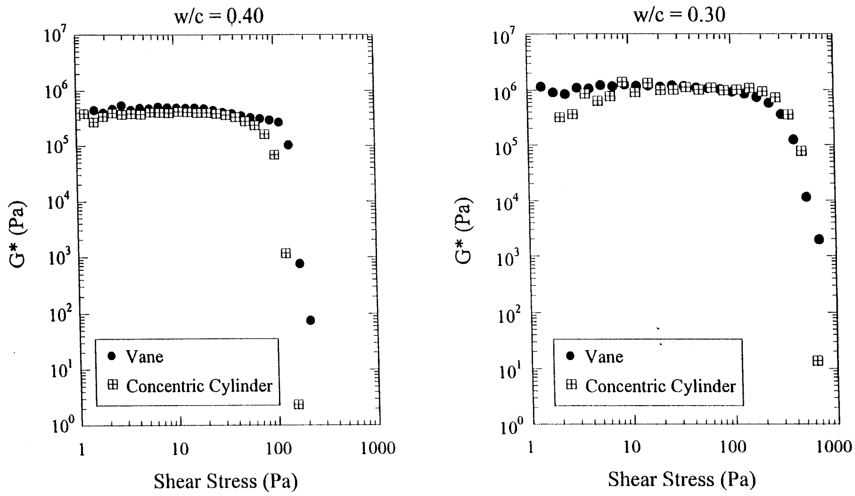

Fig. 6. Stress sweeps using concentric cylinders and a vane.

surements (Fig. 6). The modulus measurements at w/c of 0.40 are on the order of 3×10^5 Pa. Using the yield stress data from Fig. 4, the critical strain calculated using Eq. (3) is approximately 10^{-4}. These results are in excellent agreement with the data of Schultz and Struble [13].

It is very difficult to determine if slip is present as the samples begin to yield. As an example, at w/c = 0.40, the concentric cylinder measurement displays yielding before the vane. However, at w/c = 0.30, both the concentric cylinders and vane show yielding at approximately the same point. Several measurements were performed at each solids concentration and no discernable trends concerning the formation of a slip layer could be concluded.

Determining the onset of nonlinear behavior is one of the major problems while trying to identify slip in viscoelastic experiments. Most rheometer testing programs require a logarithmic distribution of measurement points to cover the wide stress range shown in Fig. 6. Such a distribution makes it difficult to determine the stress at which yielding occurs, in contrast to the more direct stress growth experiment discussed earlier.

Frequency sweeps were performed at stresses within the linear viscoelastic region (Fig. 7). The agreement between the vane and concentric cylinders data is encouraging, given the low strains required to perform these measurements. The limited scatter in the data between the vane and concentric cylinder measurements is well within the data scatter of an individual testing method. The value of the modulus remains relatively invariant over a wide range of angular velocities for both solids concentrations. This is in agreement with results for other suspensions [33,38].

To further test the general applicability of the vane method, frequency sweeps were performed on a standard low modulus emulsion using both the vane and concentric cylinders. The data are presented in Fig. 8 with the results from frequency sweeps of cement paste and an alumina slurry [33]. Once again, there is excellent agreement between the vane and concentric cylinder measurements.

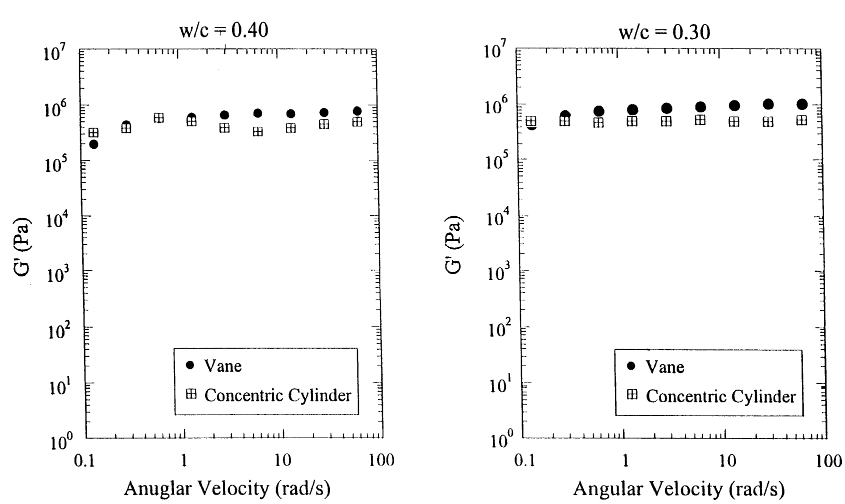

Fig. 7. Frequency sweeps using concentric cylinders and vane.

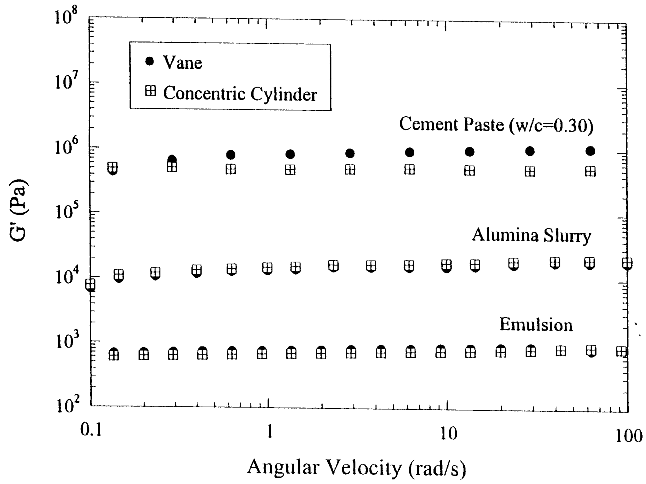

Fig. 8. Frequency sweeps using concentric cylinders and vane for a variety of materials (alumina data from Ref. [33]).

The results show that the vane can be used over several decades of modulus values.

4. Conclusions

In this study, the influence of slip on yield stress measurements of cement paste was determined using a rotational rheometer with smooth-walled concentric cylinders and a vane. The results indicate that a slip layer develops when the shear stress approaches the yield point. The yield stress measured using the vane is approximately twice the value measured using smooth-walled concentric cylinders. The rotational speed of the vane greatly influences the yield stress measurements, in agreement with published data for cement paste and other suspensions [11,28]. The yield stress measured using concentric cylinders was largely independent of rotational speed, further suggesting the development of a slip layer.

Viscoelastic measurements were performed to determine the extent of the slip phenomenon at stresses below the yield point. There was excellent agreement between the vane and concentric cylinder modulus measurements for oscillatory stress sweeps. The results suggest that slip is not prevalent in measurements below the yield stress of cement paste. Frequency sweeps were also performed on a low shear modulus emulsion to show the general applicability of the vane method. Modulus measurements made using a vane agree with measurements made using concentric cylinders over several orders of magnitude.

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References

[1] H. Okamura, Self-compacting high-performance concrete, Concr. Int. 6 (1997) 50–54 (July).
[2] G.H. Tattersall, P.F.G. Banfill, The Rheology of Fresh Concrete, Pitman Advanced Publishing, Boston, 1983.
[3] F.F. Lange, Powder processing science and technology for increased reliability, J. Am. Ceram. Soc. 72 (1) (1989) 3–15.
[4] C.W. Macosko, Rheology: Principles, Measurements, and Applications, VCH Publishers, New York, 1994.
[5] A. Papo, Rheological models for cement pastes, Mater. Constr. 21 (1988) 41–46.
[6] R.I.A. Malek, D.M. Roy, Modeling the rheological behavior of cement pastes: A review, in: S. Mindess (Ed.), Advances in Cementitious Materials, Ceram. Trans. American Ceramic Society, Westerville, Ohio, 16 (1991) 31–41.
[7] T.E.R. Jones, S. Taylor, A mathematical model relating the flow curve of cement paste to its water/cement ratio, Mag. Concr. Res. 29 (101) (1978) 207–212.
[8] N.Q. Dzuy, D.V. Boger, Yield stress measurement for concentrated suspensions, J. Rheol. 27 (4) (1983) 321–349.
[9] Q.D. Nguyen, D.V. Boger, Measuring the flow properties of yield stress fluids, Annu. Rev. Fluid Mech. 24 (1992) 47–88.
[10] L.J. Struble, M.A. Schultz, Using creep and recovery to study flow behavior of fresh cement paste, Cem. Concr. Res. 23 (1993) 1360–1379.
[11] A. Haimoni, D.J. Hannant, Development in the shear vane test to measure the gel strength of oil well cement slurry, Adv. Cem. Res. 1 (4) (1988) 221–229.
[12] J. Keating, D.J. Hannant, The effect of rotation rate on gel strength and dynamic yield strength of thixotropic oil well cements measured using a shear vane, J. Rheol. 33 (7) (1989) 1011–1020.
[13] M.A. Schultz, L.J. Struble, Use of oscillatory shear to study flow behavior of fresh cement paste, Cem. Concr. Res. 23 (1993) 273–282.
[14] T. Gregory, S.J. O'Keefe, Rheological measurements on fresh polymer-modified cement pastes, in: P.F.G. Banfill (Ed.), Rheology of Fresh Cement and Concrete, E and FN Spon, London, 1991, pp. 69–79.
[15] A. Saasen, C. Marken, N. Blomberg, Viscoelastic properties of oilfield cement slurries, in: P.F.G. Banfill (Ed.), Rheology of Fresh Cement and Concrete, E & FN Spon, London, 1991, pp. 170–177.
[16] B.H. Min, L. Erwin, H.M. Jennings, Hysteresis loops of cement paste measured by oscillatory shear experiments, Korean J. Rheol. 5 (2) (1993) 99–108.
[17] H.A. Barnes, A review of the slip (wall depletion) of polymer solutions, emulsions and particle suspensions in viscometers; its cause, character, and cure, J. Non-Newtonian Fluid Mech. 56 (1995) 221–251.
[18] K. Wesche, H. Flaatten, W. vom Berg, On the influence of spearations on the rheological investigations of cement paste and mortar in the rotation rheometer, in: A.M. Neville, D. Slater (Eds.), Fresh Concrete, Important Properties and their Measurement, vol. 2, RILEM, Leeds, England, 1973, pp. 1–11.
[19] B. Caufin, A. Papo, Rheological behavior of cement pastes, Zem.-Kalk-Gips 12 (1984) 656–661.
[20] R. Lapasin, A. Papo, S. Rajgelj, Flow behavior of fresh cement pastes; a comparison of different rheological instruments and techniques, Cem. Concr. Res. 13 (1983) 346–349.
[21] P. Coussot, J.-M. Piau, A large-scale field coaxial cylinder rheometer for the study of the rheology of natural coarse suspensions, J. Rheol. 39 (1) (1995) 105–124.
[22] R. Buscall, J.I. McGowan, A.J. Morton-Jones, The rheology of concentrated dispersion of weakly attracting colloidal particles with and without wall slip, J. Rheol. 37 (4) (1993) 621–641.

[23] N.Q. Dzuy, D.V. Boger, Direct yield stress measurements with the vane method, J. Rheol. 29 (3) (1985) 335–347.

[24] M. Keentok, J.F. Milthorpe, E. O'Donovan, On the shearing zone around rotating vanes in plastic liquids: Theory and experiment, J. Non-Newtonian Fluid Mech. 17 (1985) 23–35.

[25] G. Christensen, Modeling the Flow of Fresh Concrete: The Slump Test, PhD Thesis, Princeton University, 1991.

[26] J. Yan, A.E. James, The yield surface of viscoelastic and plastic fluids in a vane viscometer, J. Non-Newtonian Fluid Mech. 70 (1997) 237–253.

[27] M. Keentok, The measurement of the yield stress of liquids, Rheol. Acta 21 (1982) 325–332.

[28] P.V. Liddell, D.V. Boger, Yield stress measurements with the vane, J. Non-Newtonian Fluid Mech. 63 (1996) 235–261.

[29] A.S. Yoshimura, R.K. Prud'homme, H.M. Princen, A.D. Kiss, A comparison of techniques for measuring yield stress, J. Rheol. 31 (8) (1987) 699–710.

[30] N.J. Alderman, G.H. Meeten, J.D. Sherwood, Vane rheometry of bentonite gels, J. Non-Newtonian Fluid Mech. 39 (1991) 291–310.

[31] K.S. Avramidis, R.M. Turian, Yield stress of laterite suspensions, J. Coll. Int. Sci. 143 (1) (1991) 54–67.

[32] P.F.G. Banfill, D.R. Kitching, Use of a controlled stress rheometer to study the yield stress of oilwell cement slurries, in: P.F.G. Banfill (Ed.), Rheology of Fresh Cement and Concrete, E & FN Spon, London, 1991, pp. 125–136.

[33] J.A. Yanez, T. Shikata, F.F. Lange, D.S. Pearson, Shear modulus and yield stress measurements of attractive alumina particle networks in aqueous slurries, J. Am. Ceram. Soc. 79 (11) (1996) 2917–2924.

[34] X.D. Zhang, D.W. Giles, V.H. Barocas, K. Yasunaga, C.W. Macosko, Measurement of foam modulus via a vane rheometer, J. Rheol. 42 (4) (1998) 871–889.

[35] ASTM Designation C-305-94; mechanical mixing of hydraulic cement pastes and mortars of plastic consistency, in: Annual Book of ASTM Standards 04.01, Am. Soc. Test. Mater., Easton, MD, 1996, pp. 194–196.

[36] D.A. Williams, A.W. Saak, H.M. Jennings, The influence of mixing on the rheology of fresh cement paste, Cem. Concr. Res. 29 (2000) 1491–1496.

[37] A.W. Saak, Characterization and Modeling of the Rheology of Cement Paste: With Applications Toward Self Flowing Materials, PhD Thesis, Northwestern University, Evanston, IL, 2000.

[38] W.B. Russel, D.A. Saville, W.R. Schowalter, Colloidal Dispersions, Cambridge Univ. Press, Cambridge, 1989.