The Bucket Rheometer for Thickened Tailings and Paste Flow Curve Determination

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ABSTRACT

Obtaining valid shear stress versus shear rate data for thickened tailings and pastes can be difficult using conventional rotational rheometer geometries due to issues such as particle migration, particle size, settling and slip. Furthermore, for highly structured suspensions, the very act of performing a measurement can lead to destruction of the material structure and alteration of the rheological properties.

To eliminate or at least minimise the effects commonly observed in rheological measurements with rotational rheometers, a vane in an infinite medium (for example a bucket) can be employed. The vane is a convenient, portable and simple device for the determination of the shear stress versus shear rate characteristics of a sample and construction of flow curves for industrial materials when used in an infinite medium. Data for model and industrial suspensions is presented to confirm the conclusion that a vane rotated in a bucket of fluid is a practical and accurate industrial rheometer.

1 INTRODUCTION

The importance of rheology in the design and operation of thickened tailings and paste systems is well established, however much confusion still exists when it comes to selecting an appropriate and convenient geometry for taking measurements and the appropriate and correct data analysis and interpretation.

Many workers have found capillary rheometry an effective method for determining the flow properties of mineral suspensions and the similarity with pipeline flow makes it particularly attractive for this application. The major drawbacks of the capillary rheometer are the large sample volumes required and the fact that it is typically a laboratory device. Consequently, a quick, simple, transportable and accurate measurement technique is required for small sample volumes and on-site flow measurements.

Rotational rheometers are extensively used for rheological measurements in both laboratories and industrial settings due to the relative portability and compactness of the devices, ease of completing measurements and the relatively small sample volumes required. Rotational techniques often employ concentric cylinder geometry (bob and cup or Couette) with small gap widths where the shear stress is relatively constant across the gap. Industrial suspensions commonly contain a proportion of large particles so there is a need to utilise larger gap sizes to eliminate particle size effects. Results from conventional rotational rheometers, however, are often questionable as thixotropic breakdown, particle size effects, particle migration and settling and wall slip often go unnoticed in the reported data. The presence of a yield stress further complicates matters, as at low rotational rates the material within the gap may not be completely sheared. Conventional methods for shear rate calculations do not consider the presence of a variable effective gap width and so can lead to substantial error.

In an effort to overcome some of the effects commonly observed in rheological measurements with rotational rheometers, Barnes and Carnali (1990) analysed the vane in cup as an alternative to bob and cup rheometer geometry. The vane in cup is a modified concentric cylinder geometry where a four-bladed vane replaces the traditional solid bob. There were three important motivating factors for proposing the vane in cup:

- Insertion of a vane causes less sample disturbance than a cylindrical bob, minimising thixotropic breakdown.

- The vane in cup is less susceptible to artefacts arising from large particle sizes.
- Yielding occurs between layers of fluid, minimising the effects of wall slip.

Barnes and Carnali (1990) conducted a numerical simulation of the vane in cup geometry for shear thinning indices (n) ranging from 0.1 to 1. For the Newtonian case, the streamlines are not circular, leading to transfer of fluid from within the vane to outside the vane periphery. However, for a sufficiently shear-thinning fluid ($n \leq 0.5$), the streamlines are circular and the sample within the vane periphery rotates with the vane as a solid body.

Other workers have performed both experimental work (Keentok et al., 1985) and theoretical modelling (Yan and James, 1997) of a rotating vane in various yield stress materials and found that the assumption of a uniform stress distribution over a cylindrical yield surface is valid for both plastic and viscoelastic fluids with a yield stress.

This paper extends on the previous study by Barnes and Carnali (1990), with the major focus on the vane in an infinite medium geometry, rather than the vane in cup geometry. The vane in an infinite medium geometry is perceived as the more suitable geometry for measurements with complex fluids as experiments are easier, particle size effects are minimised and the need to deal with the yield stress during shear rate determination is eliminated. An additional advantage is that the shear rate can be explicitly defined using the bucket rheometer, whereas in the standard Couette narrow gap instrument the shear rate is implicit.

The vane is a convenient, portable, quick and simple device for the determination of the shear stress versus shear rate characteristics of a sample and construction of flow curves for industrial materials when used in an infinite medium. Data for model and industrial suspensions is presented to confirm the conclusion that a vane rotated in a bucket of fluid is a practical and accurate industrial rheometer. Analyses were performed with capillary rheometry tests as a comparator.

2 THEORY

Numerical simulations by Yan and James (1997) and Van Wazer et al. (1963) have shown that for sufficiently shear thinning fluids and for shear thinning fluids with a yield stress, the vane and encompassed fluid essentially rotate as a solid body. Consequently, equations developed for the concentric cylinder geometry can be directly adapted to the vane in either a cup or an infinite medium (see for example Kreiger and Maron, 1952).

Figure 1 displays a schematic of a concentric cylinder system, where an inner cylinder of radius R is rotated at a constant angular velocity (Ω) in a cup of radius εR.

The presence of a yield stress in the test material means that either complete shearing or partial shearing of the gap is possible. The prevalent mode of shearing is dependent on the magnitude of the shear stress and the yield stress. Complete shearing of the gap occurs for the conditions $\tau_1 > \tau_2 > \tau_y$, where the shear stress at all points in the gap exceeds the yield stress. Partial shearing across the gap will occur for the conditions $\tau_1 > \tau_y > \tau_2$, where the shear stress at the cup surface is below the yield stress. This partial shearing case is illustrated in Figure 1 where the shear stress reduces from τ_1 at the inner cylinder wall to τ_y at R_y, the interface between the sheared and unsheared regions. If the container holding the sample is sufficiently large, the stress induced on the sample by the rotating vane will decay either to that below the yield stress, or effectively to zero before the container walls. The sample volume is therefore referred to as infinite, as the measurement is not influenced by the container walls.

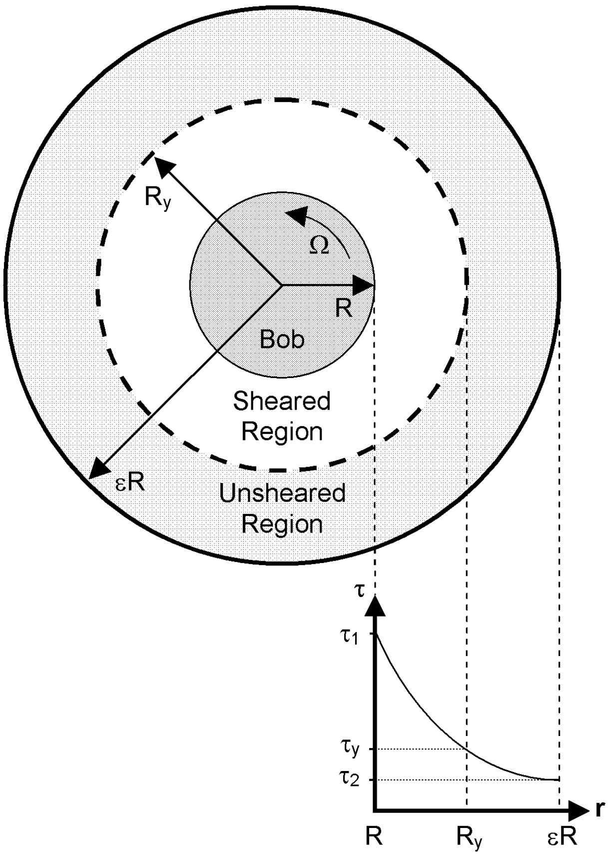

Figure 1 **A schematic of a concentric cylinder system for the flow of a yield stress fluid in a large gap system. The inner cylinder of radius R is rotated at a constant angular velocity (Ω) in a cup of radius εR. The radius of shearing (R_y) denotes the radius beyond which the fluid is static. The stress profile across this gap is also presented**

For the infinite medium scenario, the system is evaluated across the fully sheared region and a direct relationship between angular velocity and shear rate can be derived as shown in Equation 1.

$$\Omega = \int_{R}^{R_y} \frac{\dot{\gamma}}{r} dr = -\frac{1}{2} \int_{\tau_1}^{\tau_y} \frac{f(\tau)}{\tau} d\tau \qquad (1)$$

Both sides of Equation 1 can be differentiated with respect to τ_1 to yield the direct relationship between angular velocity and shear rate such that (Krieger and Maron, 1952):

$$\dot{\gamma}_1 = 2\tau_1 \frac{d\Omega}{d\tau_1} = \frac{2\Omega}{n} \qquad (2)$$

where n is the local gradient of a log-log plot of torque versus angular velocity.

Equation 2 is identical to the expression derived by Nguyen and Boger (1983) for a cylindrical bob rotating in an infinite medium. The same solution is derived because the condition for differentiability of the generalised relationship between the angular velocity and the shear rate is that one limit of the integral be constant. The constant (upper limit) for the infinite medium is 0 while the constant (upper limit) for the partially sheared case is τ_y. The relationship, expressed in Equation 5, is therefore valid for any fluid, with or without a yield stress. The only requirement for utilising Equation 5 is that the infinite medium conditions are satisfied.

3 EXPERIMENTAL TESTWORK

3.1 Materials

Metal oxide suspensions such as alumina are commonly considered to be ideal suspensions for particulate rheology due to the presence of a very narrow particle size distribution, the ease of controlling the surface chemistry and the fact that they can be reproducibly prepared. Alumina (Al_2O_3) AKP-50, produced by the Sumitomo Chemical Company was used to prepare model suspensions. Alumina suspensions have been well characterised by Zhou et al. (2001). The density of the alumina was 3900 kg/m^3 and the mean particle size was 0.18 μm with 100% passing 0.7 μm. The concentration was chosen to give a final yield stress of approximately 90 Pa. The pH of the sample at the time of testing was 7.60. For a second set of alumina tests, to maintain a relatively constant solids concentration and obtain a different yield stress, 4 L of the sample was placed in a baffled glass vessel and mixed with an anchor impeller. 5 M HNO_3 was added to the sample to move the sample pH further away from the isoelectric point at pH 9.2, to give a yield stress of approximately 30 Pa at pH 7.40.

The three nickel laterite samples used in this work were obtained from a single deposit. The red limonite, transitional limonite and rocky saprolite represent the top, middle and bottom layers of the nickel laterite deposit. While the red limonite and rocky saprolite represent the top and bottom layers of the deposit, they also exhibit the highest and lowest yield stress characteristics at a given solids concentration respectively. The samples had been slurried with high salinity water, ground and screened, but had settled during transportation and were resuspended using a laboratory mixer. Before testing, the samples were mixed in a bucket using an anchor impeller at a low rate for at least 24 hours to ensure homogeneity, and the sample yield stress was monitored to ensure that the sample was stable.

3.2 Rheometers Used for Comparison

A common use of rotational viscometer results for mineral slurries is in pipeline design and prediction of pipe flow. However, as the geometries are different there is often some doubt as to the applicability of using rotational results for the prediction of pipe flow. The simplest method to determine if the results produced with the rotational geometry are representative of pipe flow is to compare the rotational results with capillary viscometer results. The capillary is effectively a small pipeline so the results should be representative of pipe flow.

A Haake VT550 controlled rate rheometer was used for rotational measurements (yield stress and shear stress versus shear rate) with both a vane and bob and cup. In bob and cup geometry, end effects are minimised by using a hollow ended bob which traps air and reduces the base stress and therefore the end effect.

Vane end effects are normally checked via the use of different length vanes of the same diameter. However, the vane in an infinite medium is most likely to be adopted for on-site and laboratory measurement of flow curves for suspensions exhibiting a yield stress. For such applications it is desirable to complete testing with only one vane geometry. When using a vane for flow measurements, Clayton (2002) has shown that maintaining a vane L/D ratio of 6 limits the end effect error to approximately 5%, which is considered acceptable for mineral suspension systems. For the vane in infinite medium testing, the vane used had a height and diameter of 60 mm and 10 mm respectively.

Yield stress measurements were made using the vane technique (Nguyen and Boger, 1981; Nguyen and Boger, 1985) and a vane with a height and diameter of 40 mm and 15 mm respectively.

The capillary rheometer used in this work is the same as that reported in earlier works by Sofrà and Boger (2002) and de Kretser and Boger (1992). All capillary rheometer tests were performed with three capillaries, these being capillary G5 (H x D = 835 x 4.6 mm), capillary F5 (835 x 4.1 mm) and capillary F3 (550 x 4.1 mm). Each sample was tested using capillary G5, F5, F3 and then G5 again to confirm that the sample had not changed significantly during the course of testing.

3.3 Results

Two alumina suspensions were prepared denoted AKP50c and AKP50d. AKP50c was prepared at 50.2 wt% at pH 7.6 and the yield stress was measured to be 91 Pa. AKP50c was pH adjusted to pH 7.4 and the resulting AKP50d sample had a solids concentration of 50.6 wt% and a yield stress of 32 Pa. Figures 2 and 3 show the flow curves generated via vane in a infinite medium and capillary rheometry on linear and log coordinates respectively. Figure 2 shows excellent agreement between the two methods where the data overlap at shear rates below 1000 s^{-1}. Figure 4 is presented to show the consistency of the results at low shear rates and agreement with the vane yield stress.

The Herschel-Bulkley model was fitted to all of the data for each sample, including the vane in infinite medium, the capillary rheometer and the yield stress results for AKP50c (Equation 3) and AKP50d (Equation 4):

$$\tau = 9.48\dot{\gamma}^{0.372} + 93.1 \qquad (3)$$

$$\tau = 5.03\dot{\gamma}^{0.427} + 28.5 \qquad (4)$$

This model shows good agreement with the capillary rheometer and yield stress results down to low shear rates, at which cup and bob would usually show signs of slip or partial shearing within the gap.

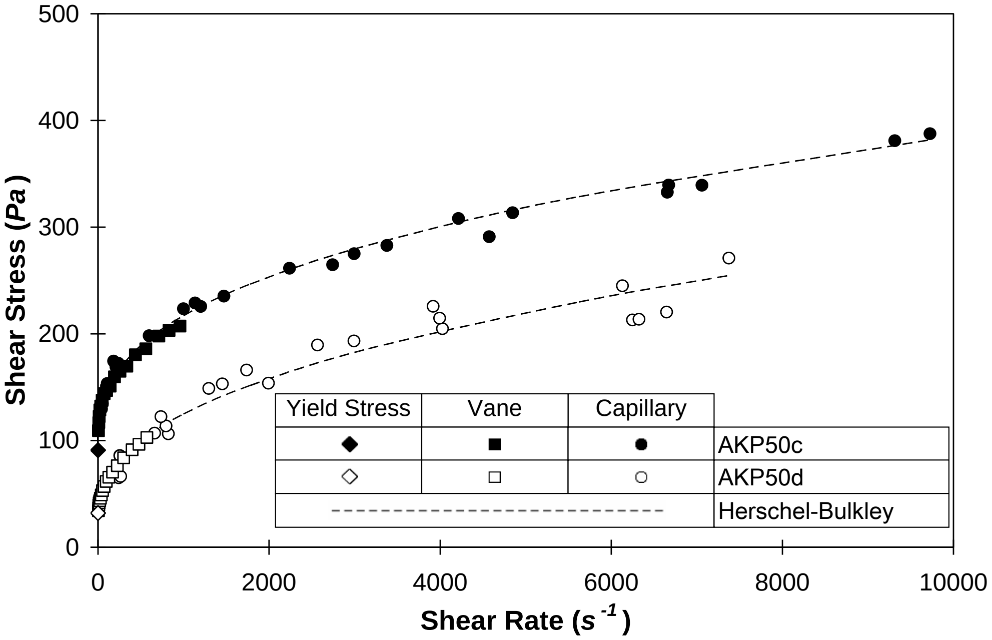

Figure 2 **Alumina AKP50 shear stress versus shear rate profiles with Herschel-Bulkley models fitted on linear coordinates (Fisher et al., 2006)**

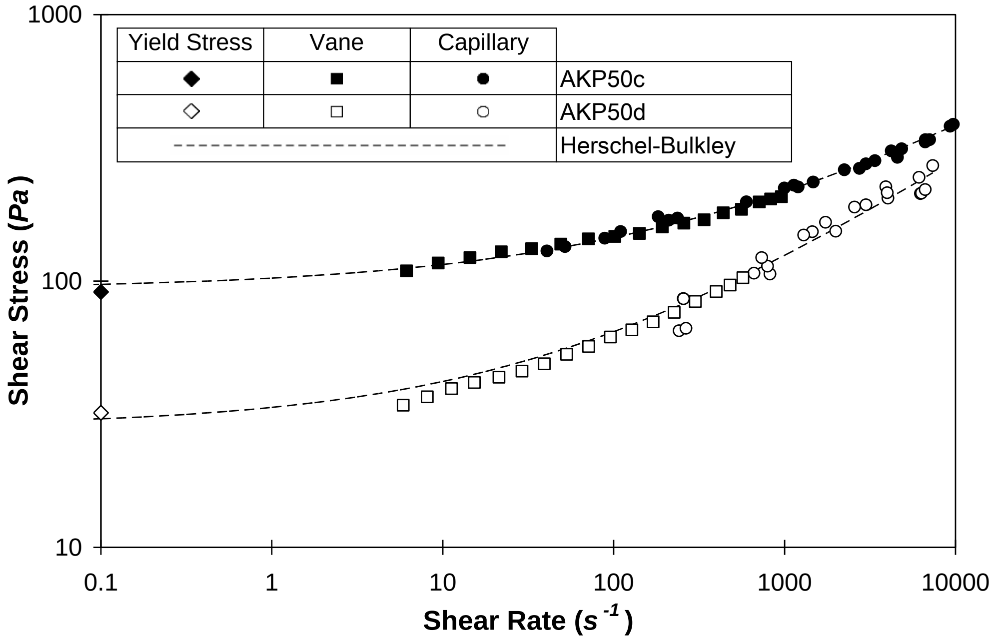

Figure 3 **Alumina AKP50 shear stress versus shear rate profiles with Herschel-Bulkley models fitted on log coordinates (Fisher et al., 2006)**

The results for three industrial nickel laterite samples, S1, L2 and L2 are shown in Figures 4 and 5 on linear and log coordinates respectively. Again, the vane in infinite medium data correlates with the medium range shear rate data of the capillary rheometer data and the vane yield stress data at low shear rates. In all three cases the correlation between all three data sources was excellent, which confirms the vane in infinite medium as a suitable method of testing for both model suspensions as well as industrial samples.

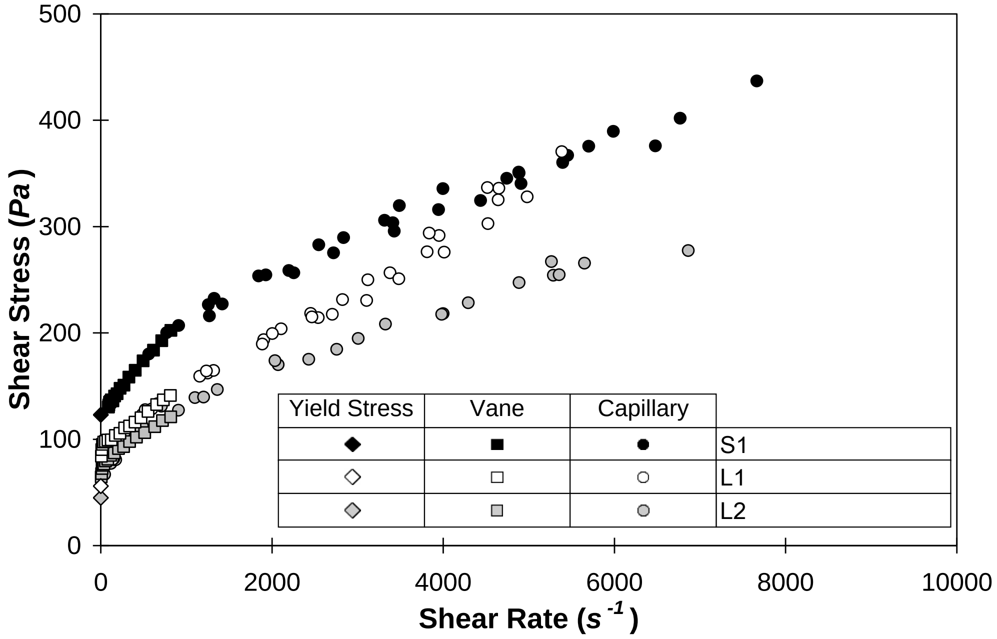

Figure 4 **Flow curves, linear coordinates determined via the vane in an infinite medium method and capillary rheometer for three nickel laterite suspensions. L2 at 41/3% $^{w}/_{w}$ and pH 6.35, L1 at 45.1% $^{w}/_{w}$ and pH 6.34 and S1 at 55.6% $^{w}/_{w}$ and pH 7.56 (Fisher et al., 2006)**

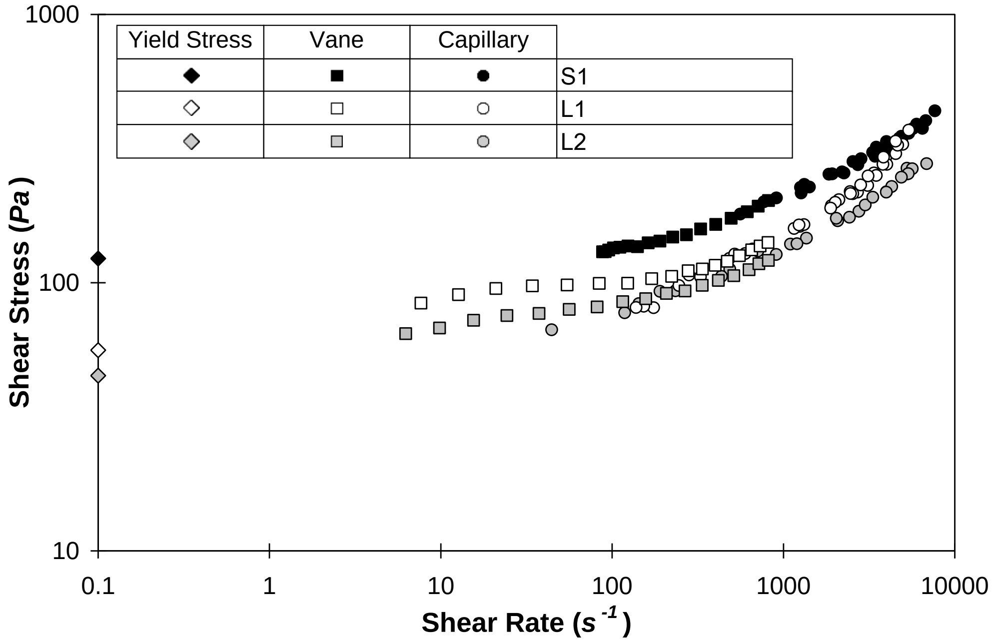

Figure 5 **Flow curves, log coordinates, determined via the vane in an infinite medium method and capillary rheometer for three nickel laterite suspensions. L2 at 41/3% $^{w}/_{w}$ and pH 6.35, L1 at 45.1% $^{w}/_{w}$ and pH 6.34 and S1 at 55.6% $^{w}/_{w}$ and pH 7.56 (Fisher et al., 2006)**

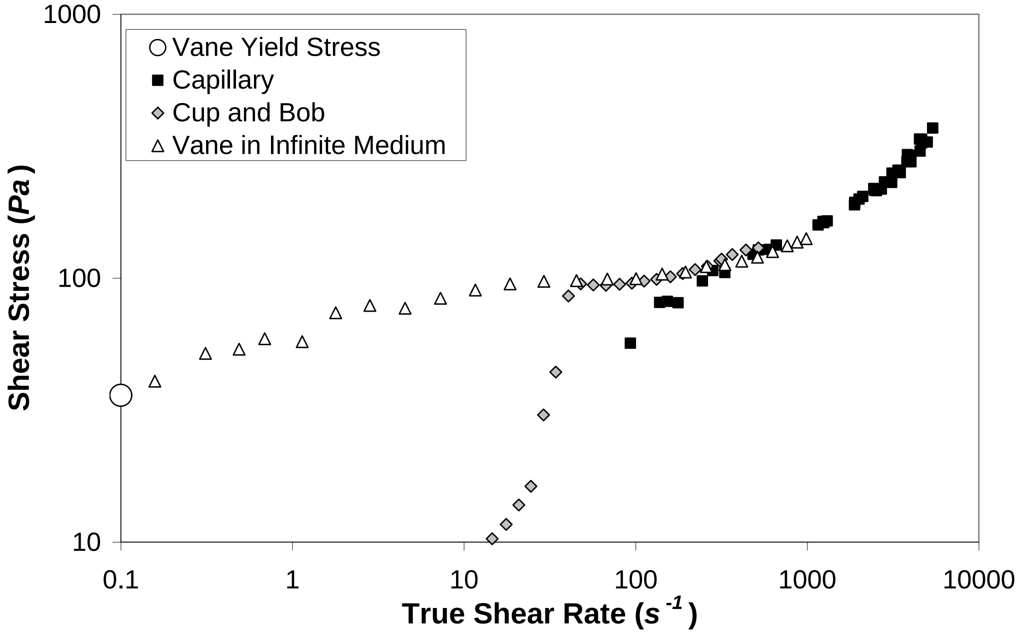

Figure 6 **Flow curves showing a comparison of vane yield stress, capillary, cup and bob and vane in an infinite medium geometry**

Data obtained using four measurement methods over a wide shear rate range are shown in Figure 6 for a beneficiated nickel ore sample. All of the data overlap at high shear rates. However, at low shear rates (less than approximately 200 s^{-1}), slip and/or incomplete shearing are clearly evident in the capillary and cup and bob data, whereas the vane in an infinite medium data show excellent agreement with the measured yield stress.

As yet, no commercially available rheometer software has the capability to correctly calculate the shear rate for a vane in an infinite medium. Figure 7 shows a comparison between vane in an infinite medium data calculated using Equation 2 and the output of a rheometer software package. Although the shear stress values of each measurement are the same, the rheometer software does not give the correct shear rate, as stipulating a vane fixture in the software results in the shear rate being calculated as a scalar multiple of the rotational rate.

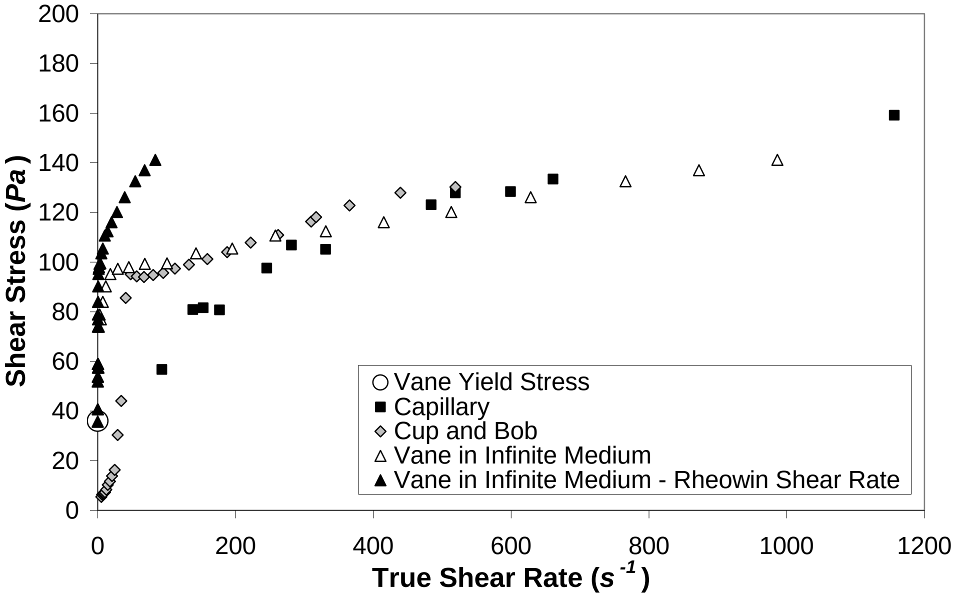

Figure 7 Comparison of rheometer geometries and the Rheowin software vane in an infinite medium shear rate output

4 DISCUSSION – USING THE ‘VANE IN A BUCKET’

By definition, using the vane in an infinite medium or bucket requires that that the diameter of the bucket is sufficiently large to ensure that the yield stress is exceeded within the annulus between the vane and the bucket. The radius of shearing (R_y) can be found using Equation 5.

$$R_y = \sqrt{\frac{\tau_1}{\tau_y}}.R \qquad (5)$$

If the shearing container is smaller than R_y, the sample within the annulus will be fully sheared, if the container is larger than R_y, the sample beyond R_y will experience a stress less than the sample yield stress, and hence will be static. Therefore for a given system, if the sample container has a radius larger than R_y, the sample container can be considered infinite.

One of the major benefits of the vane in a bucket is that a large container can be used that allows point measurements to be obtained at one shear rate prior to inserting the vane in undisturbed areas of the sample for successive high shear rate measurements. This procedure is particularly useful for highly shear history sensitive samples where continuous testing will lead to a break down in the material structure. For highly shear history sensitive suspensions, the very act of taking continuous measurements can change the rheological properties. Depending on the rate and duration of successive shear rates the rheology of the sample being tested can change to such an extent that the material may appear to have a negative viscosity which is nonsensical.

It is of utmost importance to note that no commercially available rheometer software exists that can perform the vane in an infinite medium calculation. Rheometer software packages are capable of calculating the correct shear stress from the torque measured and the vane dimensions, but the shear rate calculated will be incorrect as it will be merely a scalar multiple of the rotational rate. At the present time shear rate calculations must be performed manually from the instrument torque and rotational rate.

5 CONCLUSION

The vane in an infinite medium geometry has been shown to eliminate wall slip effects and provide a means for the accurate flow curve determination of complex materials. Using the vane, flow curves can also be generated for shear history sensitive materials due to minimal sample disturbance as the vane is inserted and the ability to test undisturbed sections of a large sample at various shear rates. Importantly, the vane in an infinite medium geometry also provides an accurate measure of the material yield stress and does not require the assumption of a fluid model for results calculation. However, care must be taken to ensure that correct data analysis methods are used and that the infinite medium condition exists. Performed correctly, the vane in an infinite medium geometry offers a simple, accurate and portable method for flow measurements, both in the laboratory and in the field.

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