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Continuous on-line rheological measurements for rapid settling slurries ☆

T.J. Akroyd, Q.D. Nguyen *

Department of Chemical Engineering, School of Chemical Engineering, University of Adelaide, Adelaide, SA 5005, Australia

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Abstract

A continuous flow rheometer based on the principles of helical flow has been developed for measuring the rheological properties of settling slurries. A special feature of the rheometer is its method for data analysis that allows the fundamental rheological parameters to be determined accurately and directly from experimental data without relying on any calibrations. Extensive testing with various fluids and mineral slurries has demonstrated the capability of the instrument for flow property characterisation of complex fluids. A comparison of the results obtained from the helical flow rheometer with those measured independently using a modified tube viscometer and a cone-plate rheometer shows excellent agreement. The numerical procedure developed for data analysis applies to fluids exhibiting any rheological behaviour, including viscoplastic fluids with a yield stress. Particle migration, which is known to affect rheological measurements of particulate systems, is found to have a minimal effect in the rheometer developed.

Keywords: Mineral slurries; On-line analysis; Process instrumentation; Shear forces; Tailings

1. Introduction

Mineral solids are usually processed, produced or handled in the form of two-phase slurries. The ability to handle, process and optimise equipment performance critically depends on the viscous and flow behaviour of the slurries. It is therefore essential that the rheological properties of these slurries be determined as accurately as possible under conditions that resemble closely those found in actual processes. This, however, is not an easy task when dealing with heterogeneous suspensions where there are large and high-density solid particles. Accurate and reliable measurements of the rheological properties of these slurries using conventional viscometric instruments can be difficult, if not impossible, since the solid particles settle rapidly under the influence of gravity. Thus an important feature of any rheometer that is to be used to measure the flow properties of a slurry is that it must keep the solid particles in a homogeneous suspension.

A variety of viscometers have been developed in recent years for measurement of the flow properties of settling slurries. The simplest and most popular designs employ mixing impellers to keep the particles suspended and maintain slurry homogeneity. The torque required to rotate the impeller at a certain speed is measured from which a measure of the slurry viscosity can be determined. Calibrations are always required with these mixer-viscometers either by using standard Newtonian liquids of known viscosities or by employing the average mixing shear rate method previously developed for agitation of non-Newtonian liquids (Metzner and Otto, 1957; Metzner and Taylor, 1960). Thus, while numerous data and correlations have been reported and used for engineering design, they are highly empirical and system-specific (Calderbank and Moo-Young, 1961; Kemblowski et al., 1988). In some instances, results have been obtained and analysed based on data generated with flow fields that are obviously in a highly turbulent condition, which is necessary in order to keep the particles in suspension (Shi and Napier-Munn, 1996). In general, however, given that the only measurable quantity is the average slurry viscosity, the results while

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* Corresponding author. Tel.: +61-8-8303-5456; fax: +61-8-8303-4373.

E-mail address: dnguyen@chemeng.adelaide.edu.au (Q.D. Nguyen).

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Nomenclature

A	consistency parameter of the power-law model (Pa s^n)
b	constant of integration
g	gravitational acceleration (m/s^2)
M	torque per unit length (Nm/m)
n	flow behaviour index of the power-law model
p	pressure (Pa)
Q	flow rate (m^3/s)
R	radius of inner cylinder (m)
r	radial distance (m)
u	fluid velocity (m/s)
$\mathbf{v}$	velocity vector (m/s)
z	axial distance (m)
ε	radius ratio between outer cylinder and inner
$\dot{\gamma}$	shear rate (1/s)
Ω	angular velocity of inner cylinder (rad/s)
ω	angular fluid velocity (rad/s)
τ	shear stress (Pa)
τ_y	yield stress (Pa)

Subscripts

r	cylindrical co-ordinates radial direction
z	cylindrical co-ordinates axial direction
θ	cylindrical co-ordinates angular direction
w	wall

adequate for some simple fluids may be quite misleading when the slurry exhibits more complex flow behaviour.

Several rheometers based on the concentric cylinder (Couette) geometry have been developed in attempts to measure more reliably the rheological properties of settling slurries. If the settling rate of the solids is known and is relatively slow then a rotating bob may be placed in the hindered settling zone where the solids concentration is known and stable (Klein et al., 1995). Alternatively the entire concentric cylinder arrangement may be placed within a concrete mixing bowl, the action of which keeps the particles suspended (Renehan et al., 1987). However, the flow patterns within the rotating mixing bowl especially at higher bowl rotational speeds, often required to keep the particles suspended, are very complex and can have significant influence on the accuracy of results obtained.

If an axial annular flow is superimposed on Couette flow then helical flow will result. This additional axial flow component should keep particles suspended within the system provided that the flow rate is high enough. A number of slurry rheometers, based on the helical flow concept have been developed to measure the properties of settling suspensions (e.g. Ferrini et al., 1979; Reeves, 1985; Blaszczyk and Petela, 1986; Shi and Napier-Munn, 1996). In these developments, while the axial flow component is generally recognised as important in affecting the overall shear field, calculations of the rheological parameters usually ignore the contribution due to axial flow and rely on calibrations using fluids with known properties. A different approach has been employed by Nguyen et al. (1999) in a recent development of a continuous flow rheometer for settling slurries based on the principles of helical flow. They presented a numerical method for data analysis that takes into account the contributions of both the tangential and axial flow component to extract the fundamental rheological properties from experimental data without the need for any calibrations. Akroyd and Nguyen (2003) have made further modifications to both the design of the rheometer and the data reduction procedure to account for wide-gap systems and non-Newtonian fluid mechanics effects. These changes led to improvements in the operating capability of the instrument and in the overall accuracy of the data analysis. However, when applied to slurries that have a yield stress, the calculations showed a reduction in accuracy especially in the low shear rate region.

This paper reports on modifications made to both the rheometer and to the data reduction procedure presented in Akroyd and Nguyen (2003), to improve the accuracy of the instrument when measuring the rheological properties of concentrated slurries mineral, which usually exhibit viscoplastic flow behaviour. A variety of fluids including yield stress liquids and slurries are examined using the flow rheometer, the results of which are compared with those obtained independently using other instruments.

2. Theory

Details on theoretical development of the helical flow rheometer and the numerical method for calculating the rheological properties from experimental data have been previously described by Nguyen et al. (1999) and Akroyd and Nguyen (2003). Here we summarise only the essential features which are pertinent to the present work. As shown diagrammatically in Fig. 1, the helical flow is formed when the fluid is flowing axially through the annulus between two coaxial cylinders, the inner of which is rotating at a constant angular velocity. From the theory of helical flow by Coleman and Noll (1959), the total shear stress experienced by the fluid consists of the Pythagorean addition of the axial component τ_{rz} and the tangential component $\tau_{r\theta}$ such that

$$\tau = \sqrt{\tau_{rz}^2 + \tau_{r\theta}^2} \quad (1)$$

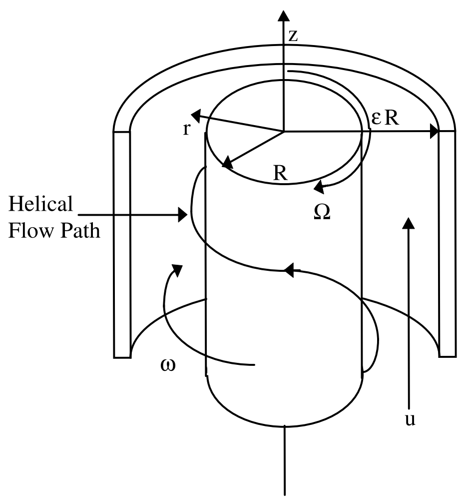

Fig. 1. Helical flow geometry.

where

$$\tau_{rz} = \frac{b}{r} - \left(\frac{\partial p}{\partial z} + \rho g\right)\frac{r}{2} \tag{2a}$$

$$\tau_{r\theta} = \frac{M}{2\pi r^2} \tag{2b}$$

and b is a constant of integration that must satisfy the following condition:

$$\left(\frac{\partial p}{\partial z} + \rho g\right)\frac{R^2}{2} > b > \left(\frac{\partial p}{\partial z} + \rho g\right)\frac{(\varepsilon R)^2}{2} \tag{3}$$

Similarly the total shear rate is defined as

$$\dot{\gamma} = \sqrt{\dot{\gamma}_{rz}^2 + \dot{\gamma}_{r\theta}^2} \tag{4}$$

where

$$\dot{\gamma}_{rz} = -\frac{\mathrm{d}u}{\mathrm{d}r} = -u'(r) \tag{5a}$$

$$\dot{\gamma}_{r\theta} = -r\frac{\mathrm{d}\omega}{\mathrm{d}r} = -r\omega'(r) \tag{5b}$$

For a typical experiment with the flow rheometer, the independent variables are the rotational speed of the inner cylinder, Ω, and the pressure gradient, $(\partial p/\partial z)$. The measured variables are the torque per unit length of the rotating inner cylinder, M and the axial flow rate, Q. The objective is to determine a rheological relationship for the fluid, $\tau = f(\dot{\gamma})$, from the experimental data $\{\Omega, M, (\partial p/\partial z), Q\}$. To simplify the analysis, it is assumed that the rheological properties of the fluid generally can be described by the power-law model

$$\tau = A(\dot{\gamma})^n \tag{6}$$

It is expected that this model will not be valid over the entire range of shear rate but over the usually narrow range of shear rates for each applied pressure gradient, Eq. (6) should be sufficient to describe the data.

A computer program written in Pascal (Press et al., 1990), utilising the Levenberg–Marquardt technique for non-linear regression (Marquardt, 1963) and an iterative procedure based on a proposal by Huilgol (1990) is used to determine the values of A and n that best characterise the rheological properties of the fluid by optimising the following relationship between the cylinder rotational speed and the measured torque and pressure gradient:

$$\Omega = \frac{M}{2\pi}\left(\frac{1}{A}\right)^{1/n}\int_{\kappa R}^{R}\left(\left[\frac{M}{2\pi r^2}\right]^2 + \left[\frac{b}{r} + \left(\frac{\partial p}{\partial z} + \rho g\right)\frac{r}{2}\right]^2\right)^{(1-n)/2n}\frac{1}{r^3}\,\mathrm{d}r \tag{7}$$

The optimum sets of A, n and b obtained for different pressure gradients or flow rates are next used to calculate the final shear stress and shear rate values.

3. Experimental

3.1. Equipment

The experimental set-up of the flow rheometer is shown in Fig. 2. The design has been previously presented in Akroyd and Nguyen (2003), though several modifications to the apparatus to improve the accuracy have been made. The helical flow rheometer was connected to an existing Haake VT550 viscometer, which provided the driving mechanism and means for torque measurement. The measuring system of the rheometer consisted of a stationary outer cylinder with diameter of 38 mm and a rotating inner cylinder (bob) 130 mm long. Two interchangeable bobs were available, with diameters of 36 and 32 mm, to increase the range of viscosities that can be measured and also to accommodate various

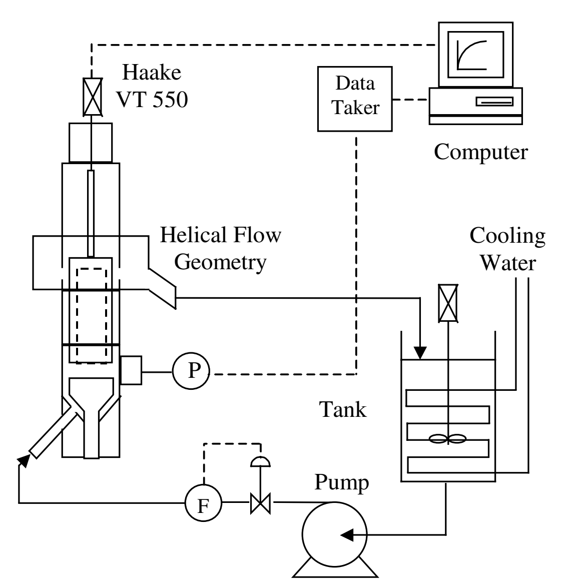

Fig. 2. Schematic diagram of the flow rheometer and experimental system.

particle sizes. Pressure measurements were obtained from a pressure transducer (manufactured by IMT, Germany). A data logger (DT50, by DataTaker Pty Ltd, Australia) recorded and transferred the pressure readings to a desktop computer. A helical rotor (positive displacement) pump (by Mono Pumps, Australia, rated at 0–1 l/min) was employed to circulate the fluid through the rheometer. A cooling coil was incorporated into the storage tank to reduce the effect of viscous heating.

In order to test the accuracy of the flow rheometer and verify its applicability as a scientific instrument, the results obtained with the various fluids tested, where possible, were compared with data obtained using other standard instruments. A Bohlin controlled stress rheometer (Model CVO-50) fitted with various measuring geometries was one of the instruments used for this purpose. For liquids a cone and plate geometry was used because it provides a constant shear rate across the measurement gap.

For slurries with a slow particle-settling rate a modified tube rheometer was used. The experimental arrangement of the tube rheometer is shown in Fig. 3. The interior of the tube rheometer vessel was equipped with a Rushton turbine impeller and an anchor impeller, driven by a 1/4 hp wash down DC motor (Baldor Electric Corporation, USA), to agitate the slurry and keep the particles suspended during flow property measurements. The tube rheometer vessel had approximately a 7 l capacity with a diameter of 160 mm and a depth of 360 mm. The interchangeable tubes ranged from about 4 to 8 mm in inner diameter and with lengths of 1000 mm. High-pressure air was controlled and supplied to the vessel through a high flow rate, high precision pressure regulator (manufactured by Norgren, UK) in conjunction with a Norgren 1 in. pressure gauge. A mass balance (manufactured by Ohaus Corporation, USA) was connected to a computer and used to measure the flowrate.

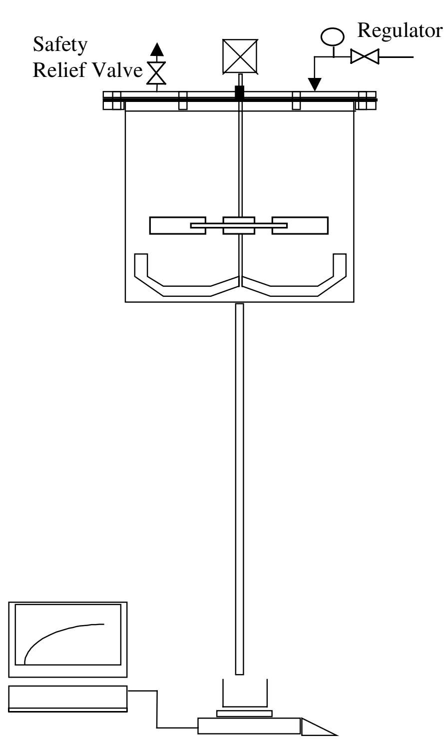

Fig. 3. Schematic diagram of the tube rheometer for settling slurries.

The yields stress of the slow settling slurries and yield pseudoplastic liquid was determined using the vane method developed by Nguyen and Boger (1985). The vane was 20 mm in diameter and 40 mm in length and was connected to an existing Haake VT550 viscometer, which controlled the speed of rotation at 0.5 rpm and measured the torque acting on the vane. For yield stress measurements it is important that the cup is at least twice the diameter of the vane (Nguyen and Boger, 1985) and for these tests a 42 mm diameter cup was used.

3.2. Materials

The experimental fluids used in this study included a Newtonian liquid (a 100% Glycerol solution), a yield pseudoplastic material (a 1.5 wt% Carbopol solution, pH 2.7) and three mineral slurries (a 71 wt% clay-water slurry, a 68 wt% fly ash–water slurry and a 49 wt% diamond mine tailing slurry). The size distribution data of the slurries are shown in Fig. 4. The properties of all but the diamond mine tailing slurry were measured using either the Bohlin rheometer or the tube rheometer and are summarised in Table 1 as are the solid particles settling velocities where applicable. The diamond mine tailings sample contained both fine and coarse particles but the settling rate of the coarse fraction, as shown in Table 1, was too high for either of these instruments so it was only tested using the flow rheometer.

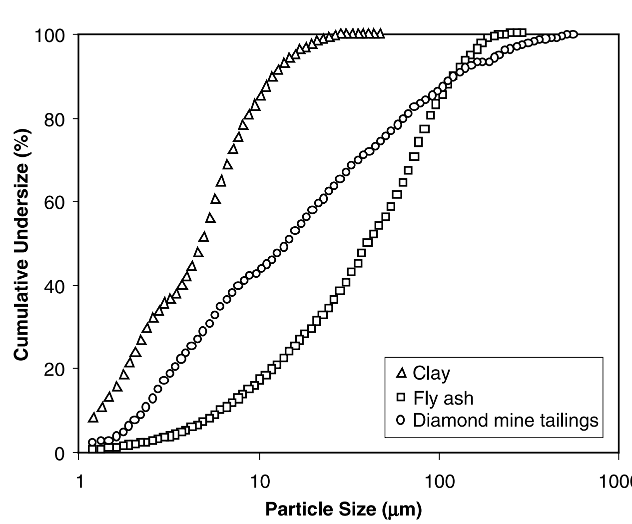

Fig. 4. Particle size distribution data for the solid particles in the slurries tested.

Table 1
List of fluids tested

Test fluid	τ_y (Pa)	A	n	Rheometer	Settling rate (mm/s)
100% Glycerol	–	0.773	1.0	CVO	N/A
1.5 wt% Carbopol, pH 2.7	5.5	0.865	0.6	CVO	N/A
71 wt% Clay–water	6.8	3.02	0.49	Tube	Negligible
68 wt% Fly ash–water	3.1	0.39	1.0	Tube	0.17
49 wt% Diamond mine tailings	6.3	0.44	0.48	Flow	13.3

3.3. Experimental technique

For a typical experiment with the flow rheometer, the test fluid was first circulated through the rheometer with the bob (inner cylinder) stationary. The pump speed was adjusted until the required pressure difference measured across the bob was reached. The bob was then set to rotate at a constant speed and the torque acting on the bob, measured as a function of time until a steady value was observed. If the additional shear due to the rotating cylinder caused the differential pressure across the instrument to change, the flow rate was adjusted accordingly to maintain a constant value of differential pressure. Between eight and ten different rotational speeds were selected at a particular value of differential pressure. For yield stress materials these tests were broken down into smaller groups over a smaller range of rotational speeds (or shear rate), for example three groups of five tests at a particular value of differential pressure were performed instead of the standard eight to ten tests. A new value of differential pressure was then chosen and the procedure repeated. The raw data were analysed in an Excel spreadsheet, which performed the data reduction procedure. For the yield stress materials each smaller test was individually analysed and the results were later combined. In this way the two-parameter power-law model can provide a better fit for the rheological properties of yield pseudoplastic materials, which would normally require a three-parameter model (e.g. Herschel–Bulkley model).

For an experiment with the tube rheometer, the test fluid was added to the storage vessel and the depth of the fluid recorded. If a slurry material was being examined then the motor and impeller were also set rotating, such that homogenous conditions were maintained within the rheometer vessel. The desired pressure was set using the regulator and the liquid allowed to flow out of the vessel. If the impeller was being rotated this operation was terminated for the duration of the test as the impeller rotation was found to affect the results. The mass flowrate was recorded until a steady state value was reached. Once the test was complete the impeller was rotated until the next pressure selected and the fluid allowed to again flow out through the tube. This procedure was repeated for at least eight different pressures settings, ranging from 0.1 to 1.6 bar depending on the actual observed flowrates. The raw results were imported into an Excel spreadsheet, which calculated the appropriate shear stress and shear rate values.

For an experiment with the vane, the test fluid was loaded into the cup (outer cylinder). A spatula was used to stir the fluid to ensure that any structure within the fluid was broken down. The vane was then quickly positioned within the fluid while taking care to minimise any stresses due to loading. The vane was then set rotating at 0.5 rpm and the torque acting on the vane recorded. The peak in the curve of torque versus time was used to determine the yield stress of the fluid following the procedure developed by Nguyen and Boger (1985).

4. Results and discussion

Figs. 5–9 shows results obtained for all the fluids tested using the helical flow rheometer, presented in terms of shear stress versus shear rate plots. The data collected from the flow rheometer at different axial flow rates are distinguished by different operating values of differential pressure. The data obtained using the 36 mm-diameter bob are denoted by closed symbols and those with the 32 mm-diameter bob by the open symbols. In Fig. 5, results obtained for the Glycerol solution, a Newtonian liquid, are compared with those obtained independently using the Bohlin rheometer. There exists an average error in viscosity values of 0.7% between the two instruments with a standard deviation of 2.2% in the flow rheometer results. Results for the Carbopol solution, which exhibits viscoplastic flow behaviour with a yield stress, are presented in Fig. 6. An excellent agreement between the flow rheometer and the Bohlin rheometer can be observed with an average error of 0.8% and a standard deviation of 2.5% in viscosity results.

Results from tests with the fly ash–water slurry are presented in Fig. 7, which compares data obtained from the flow rheometer using two bob sizes and from the tube rheometer with two tube diameters. First, the data indicate that dimensions of the systems had no observable effects on the results from both instruments. An average error of 1.2% exists between results from the tube rheometer and the flow rheometer with the flow

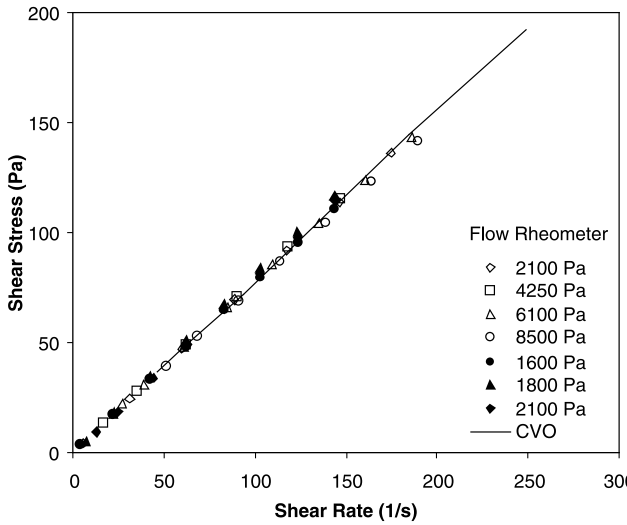

Fig. 5. Comparison between the helical flow rheometer results (data points, at different pressure differentials and gap sizes) and the Bohlin-CVO rheometer data (continuous line) for the Glycerol solution.

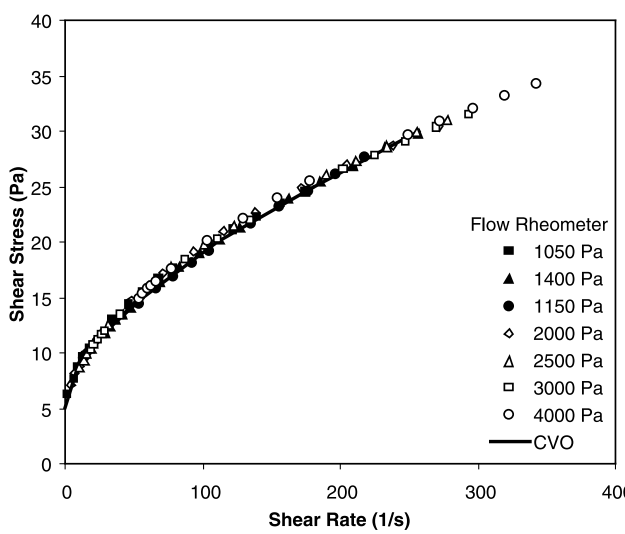

Fig. 6. Comparison between the helical flow rheometer results (data points) and the Bohlin-CVO rheometer data (continuous line) for the 1.5 wt% Carbopol Solution.

rheometer results subject to a standard deviation of 3.5%. Similarly excellent agreement was obtained with results for the clay–water slurry as shown in Fig. 8. An average error between the tube rheometer and the flow rheometer of 2.2% is present with a standard deviation of 4.3% in the flow rheometer results. The flow curve indicates that the clay slurry is viscoplastic with a yield stress of 7.0 Pa, extrapolated from the shear stress–shear rate data. This value agrees well with the yield stress of 6.8 Pa, obtained by direct measurement using the vane method (see Table 1).

The 49 wt% diamond mine tailings slurry contained heavy solid particles that settled so rapidly that meaningful measurements could not be made using either the Bohlin instrument or the tube rheometer. The results obtained only from the flow rheometer are presented in Fig. 9. Data obtained at different pressure differences collapse into a single curve, with a standard deviation of 1.5%, indicating the applicability of the rheometer for settling slurries and validating the method used for data analysis. The flow rheometer data clearly suggest that the tailings slurry behaves as a viscoplastic fluid with a yield stress of 6.5 Pa, which compares favourably with a value of 6.3 Pa measured by the vane method.

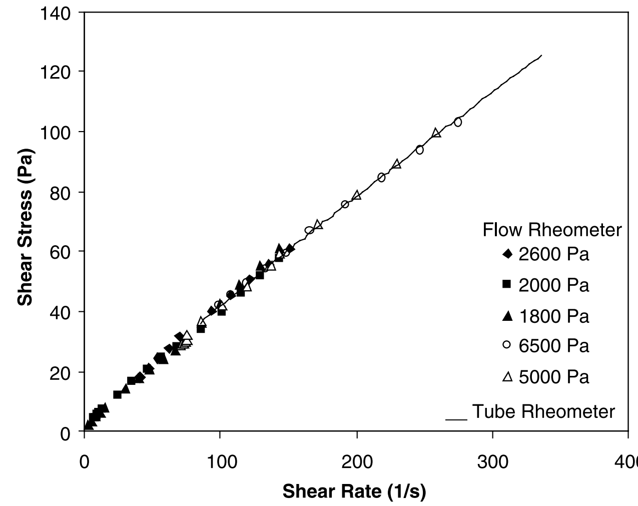

Fig. 7. Comparison between the helical flow rheometer results (data points) and the tube rheometer data (continuous line) for the 68 wt% fly ash—water slurry.

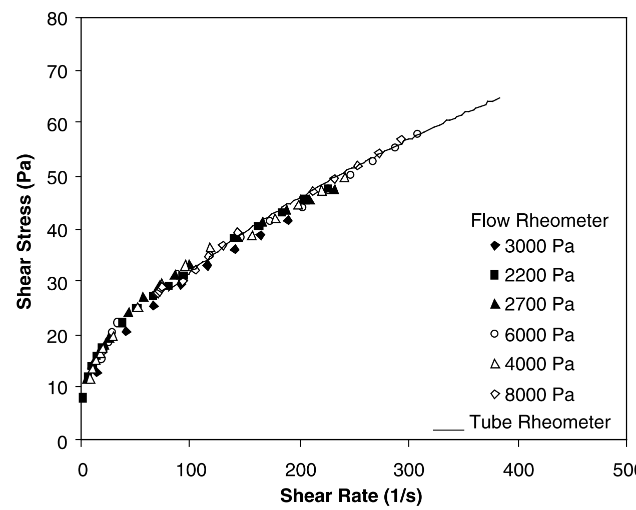

Fig. 8. Comparison between the helical flow rheometer results (data points) and the tube rheometer data (continuous line) for the 71 wt% clay—water slurry.

A significant improvement over the results presented in Akroyd and Nguyen (2003) can be noted for the yield stress fluids. The results presented previously showed errors nearly as high as 6% for a previously examined

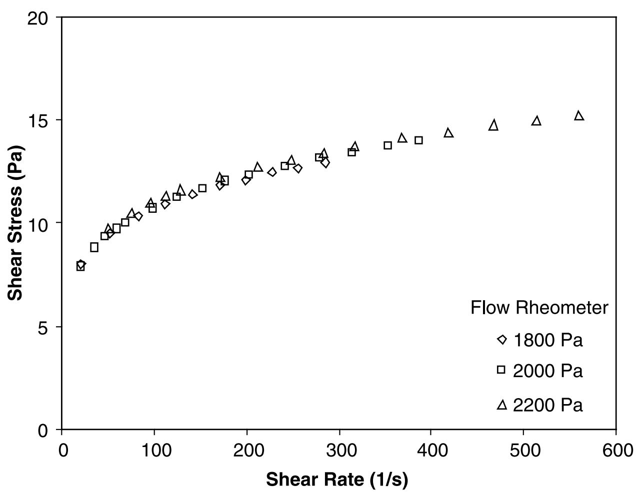

Fig. 9. Helical flow rheometer results for the 49 wt% diamond mine tailings slurry.

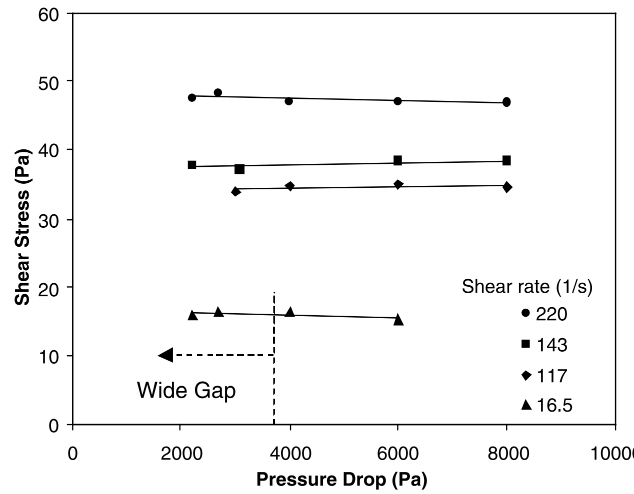

Fig. 10. Demonstration of insignificant particle migration effect. Data taken from the results for the clay–water slurry.

yield stress slurry. The improved accuracy of the results presented here can be attributed to the modified testing procedure presented for yield stress fluids. By breaking a single large test into smaller fractions the two-parameter pseudoplastic fluid model, Eq. (6), can be used to fit rheological data that would normally require a three-parameter model. Thus the accuracy of the flow rheometer has been improved without significant modifications to the already complex data analysis procedure.

Particle migration has been known to affect rheological measurements in Couette flow (Gadala-Maria and Acrivos, 1980; Leighton and Acrivos, 1987; Tetlow et al., 1998; Abbott et al., 1991) and tube flow (Sinton and Chow, 1991; Han et al., 1999; Allende and Kalyon, 2000), although little is known about this effect on helical flow. In the flow rheometer employed, the fluid including the particles is being circulated through the rheometer; the residence time of the fluid is therefore not long enough for significant particle migration to occur. To substantiate this theory some of the results for the clay–water slurry have been replotted in Fig. 10 in terms of shear stress values at different axial flow rates (pressure drops), for approximately constant values of shear rate. The shear rate values from the different runs are not exactly the same and this probably accounts from some of the deviation observed. In general, it is observed that the shear stress is effectively constant for the various values of the total shear rate, which is a Pythagorean addition of the axial and the Couette components. If particle migration was occurring it would be expected that as the ratio of the axial and Couette components of the shear rate changed the measured total shear stress would also change due to variations in both the rate and the degree of migration. The data in Fig. 10 indicate that this is not the case and further suggest that particle migration, if any, produced a minimal effect on the helical flow within the rheometer.

5. Conclusions

The helical flow rheometer and data reduction procedure have been modified to more accurately determine the properties of settling slurries, which also exhibit yield stress behaviour. Extensive testing with a variety of liquids and slurries has shown good comparison of results between the flow rheometer and other laboratory instruments. Particle migration, which is known to affect rheological measurements of particulate systems, has been shown to have a minimal effect. Work is continuing to determine the limits of the stability of the helical flow within the geometry, so that reliable results for as wide a range of shear rates as possible can be obtained.

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