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Potential errors in paste fill rheology measurements

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Abstract

Depending on the mining method being used, the required unconfined compressive strength (UCS) of paste fill can vary between 500 to 5000 kPa at 28 days. In order to achieve these upper limit UCS targets, high concentration cemented paste backfill must be placed underground, sometimes at significant distances from the backfill plant. The understanding of the flow behaviour (rheology) of these mixtures is therefore vital from a reticulation design and operational point of view.

There are many methods to determine the rheology of paste fill mixtures. Vane yield stress, along with bob and cup rotational viscometer measurements (yield stress and viscosity), are the most widely used methods due to the relatively simple test procedures and limited sample size required, but there are drawbacks to each method. Vane yield stress measurement will only provide the design engineer with yield stress values, not viscosity. Bob and cup rotational viscometer measurements will provide the design engineer with yield stress and viscosity, but one may experience slip at the wall of the rotational viscometer's bob at high concentrations, which will skew the test results.

To overcome the issue of slip at high concentration paste fill mixtures when using a bob and cup rotational viscometer, pipe loop tests can be conducted in at least two pipe diameters to accurately measure the rheology of these high concentration paste fill mixtures. This paper will discuss typical test results where the measurements of the bob and cup rotational viscometer underpredicted the yield stress and overpredicted the viscosity when the results were compared to a pipe loop test campaign completed on the same material.

Keywords: *unconfined compressive strength, paste fill, rheology, yield stress, slip*

1 Introduction

Testwork programs are conducted to obtain the necessary design input information to successfully design and implement a paste fill underground reticulation system. Various tests are conducted as part of such a program and the correct test methods must be selected to obtain the correct design information. For this particular study, the mine design called for unconfined compressive strengths (UCS) of 1,000 kPa (majority) and 2,500 kPa (selected stopes) at 28 days. The testwork program was prepared to ensure that sufficient test data based on these strength threshold targets was obtained. Material property tests were conducted and used as inputs to the mix designs for the strength and rheology tests.

The UCS tests were conducted at 6, 9 and 12% cement addition at mixture mass concentrations of 76, 79 and 82%m. Curing days of 7, 14, 28 and 56 days were tested to track the strength gain over time. For each mixture, rotational viscometer rheology tests were conducted to measure the yield stress and plastic viscosity. The rotational viscometer rheology test results indicated that above 79%m, the plastic viscosity of the mixes appeared to increase significantly which was a concern from a cemented paste fill transport point of view. In addition to this, the cost of the cement must also be taken into consideration as a majority of the stopes only require 1,000 kPa at 28 days. Based on this, the decision was taken to conduct pipe loop tests at a cement addition of 5% and confirm the rotational viscometer rheology measurements for similar water/cement (W/C) ratios.

This paper presents the potential errors that can occur if the rotational viscometer software outputs are not corrected when testing is based on the bob in an infinite cup method. In addition to this, the paper also presents the difference in rotational viscometer and pipe loop data where one can easily underpredict the yield stress and overpredict the viscosity of mixtures when only rotational viscometer test data is available.

2 Material property tests

2.1 Solids density

The material used as tailings for the paste fill mixture was zinc flotation tailings. The solids density was determined using a helium pycnometer (ASTM D5550-06), which measures the skeletal solids density. The tailings and cement had solids densities of 2,941 and 3,135 kg/m^3, respectively. Figure 1 shows the relationship between slurry density (ρ_m), mass solids concentration (C) and volumetric solids concentration (C_v) at a solids density of 2,941 kg/m^3 for the uncemented zinc flotation tailings only.

2.2 Particle size distribution

The particle size distribution (PSD) of the tailings was determined by wet sieving to 25 µm as well as laser diffraction. Figure 2 presents the PSD indicating a top size and d_{50} of ~200 and ~25 µm, respectively. The results show good agreement between the two PSD measurement methods.

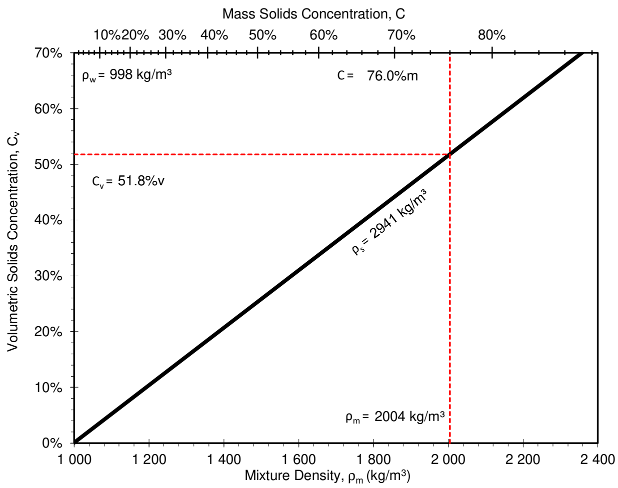

Figure 1 Relationship between ρ_m, C and C_v

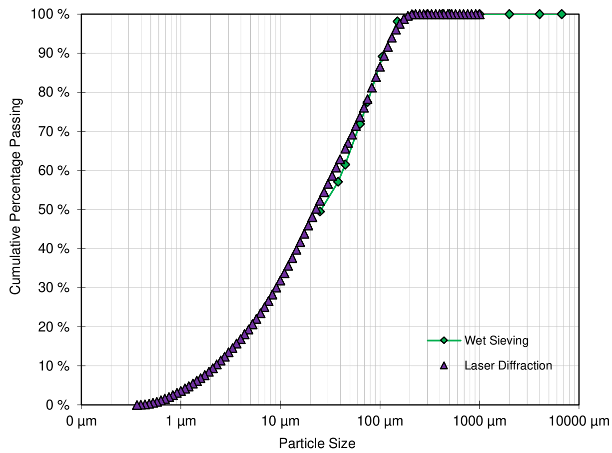

Figure 2 Tailings PSD

2.3 Slurry pH and temperature

The pH and temperature of the slurry were measured by using a Hanna handheld meter. The average slurry pH before and after cement addition was 8.76 and 12.05, respectively. The temperature was, on average, 24.5°C before and after cement addition.

2.4 Mineralogy

Mineralogy by x-ray diffraction analysis (XRD) tests were conducted on the tailings to better understand the behaviour of the material. The mineralogy indicated that the tailings mostly consist of quartz (41%), dolomite (22%) and pyrite (11%). When oxidised, pyrite releases sulfuric acid that can affect the long-term strength of the backfill due to sulfate attack.

3 Strength tests

3.1 Cement quality tests

A CEM II/A-LL 42,5N cement was supplied for the testwork. Before commencing the testwork, cement mortar tests, according to EN 196-1:2005 Edition 2, were carried out to determine the quality of the cement.

The 2- and 28-day results showed a compressive strength of 28.4 and 46.9 MPa; the latter exceeding the minimum required compressive strength of 42.5 MPa, indicating that the cement complied with the standard requirements.

3.2 Cement addition, W/C ratio and mass concentration

The percentage cement addition, W/C ratio and mass concentration are calculated using the following formulas:

$$Percentage\ cement = \frac{mass\ of\ cement}{mass\ of\ tailings + mass\ of\ cement} \tag{1}$$

$$Water/cement\ ratio = \frac{mass\ of\ water}{mass\ of\ cement} \tag{2}$$

$$Mass\ conc. = \frac{mass\ of\ tailings + mass\ of\ cement}{mass\ of\ tailings + mass\ of\ cement + mass\ of\ water} \tag{3}$$

3.3 Unconfined compressive strength

UCS tests were conducted according to ASTM D 4832-02 Standard test method for preparing and testing of controlled low strength material (CLSM) test cylinders.

Initially cylinders were cast for 7, 14, 28 and 56 days to determine the change in strength over time at 6, 9 and 12% cement addition. The mass concentrations of the UCS sample matrix were 76, 79 and 82%m, respectively, with rotational viscometer rheology being measured for each mixture. However, the rotational viscometer rheology test results indicated that above 79%m, the plastic viscosity of the mixes appeared to increase significantly, which was a concern from a cemented paste fill transport point of view. A decision was then taken to conduct pipe loop tests at a cement addition of 5% to confirm the rotational viscometer rheology for similar W/C ratios.

Table 1 presents the UCS data from 7 to 56 days at mass concentrations of 76, 79 and 82%m and at cement additions of 6, 9 and 12% for the initial casting. Also shown are the additional cylinders that were cast from the pipe loop tests which varied from 76 to 80%m at 5% cement addition. The UCS test data shows that there is a continuous increase in strength from 7 to 28 days, but that there is no significant increase in strength from 28 to 56 days, which can most likely be attributed to the pyrite present in the tailings.

Figure 3 presents the UCS at 7, 14, 28 and 56 days for 5, 6, 9 and 12% cement addition. Figure 4 presents the UCS versus W/C ratio at 7, 14, 28 and 56 days.

Table 1 UCS test results

Mix number	Cement addition (%)	Mass conc. (%m)	W/C ratio (m/m)	7 days UCS (kPa)	14 days UCS (kPa)	28 days UCS (kPa)	56 days UCS (kPa)
1.1	12	82.0	1.83/1	4,684	5,100	5,565	5,806
1.2	12	79.0	2.22/1	3,026	3,332	4,002	4,290
1.3	12	76.0	2.63/1	1,957	2,052	2,595	2,737
2.1	9	82.0	2.44/1	2,539	2,854	3,353	3,431
2.2	9	79.0	2.96/1	1,645	1,851	2,237	2,326
2.3	9	76.0	3.51/1	1,191	1,370	1,613	1,694
3.1	6	82.0	3.66/1	1,284	1,331	1,764	1,911
3.2	6	79.0	4.43/1	844	1,101	1,341	1,404
3.3	6	76.0	5.27/1	560	685	878	934
4.1 (pipe loop)	5	80.0	5.00/1	853	960	1,070	1,211
4.2 (pipe loop)	5	79.4	5.19/1	794	864	1,028	1,099
4.3 (pipe loop)	5	78.2	5.58/1	731	827	911	939
4.4 (pipe loop)	5	77.3	5.88/1	624	699	786	796
4.5 (pipe loop)	5	76.0	6.32/1	475	534	688	713

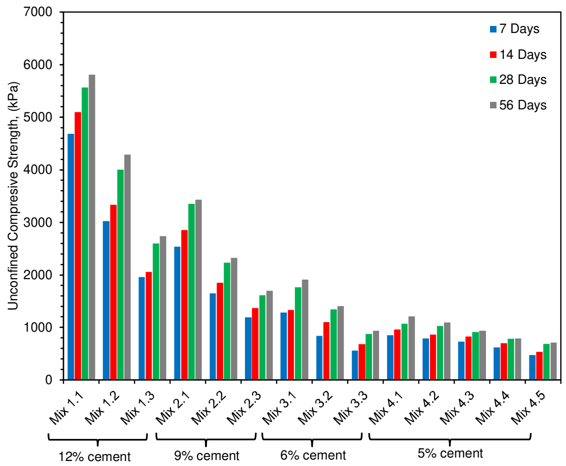

Figure 3 UCS at 7, 14, 28 and 56 days

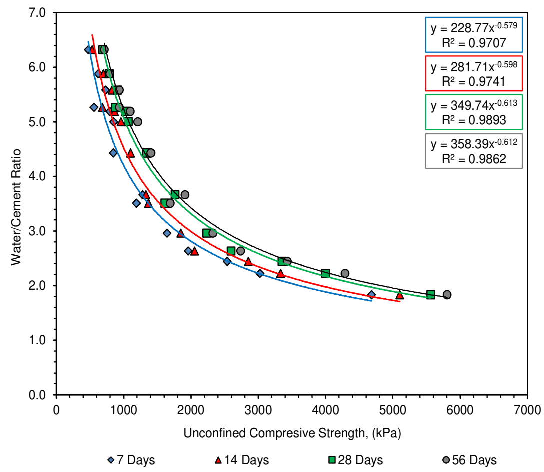

Figure 4 UCS versus W/C ratio

4 Rotational viscometer rheology tests

4.1 Geometry

The rotational viscometer rheology tests were conducted using an Anton-Paar RheolabQC rotational viscometer with a maximum torque of 75 mNm. Depending on the yield stress and plastic viscosity of the material, the testwork is conducted using either a CC39 or CC27 bob with dimensions as shown in Table 2.

Table 2 Rotational viscometer bob dimensions

Type of bob	Diameter	Height
CC39 bob	17.5 mm	52.5 mm
CC27 bob	11.0 mm	33.0 mm

4.2 Rheogram

Rotational viscometer tests can be conducted using either the conventional bob and cup or the bob in an infinite cup method Chhabra & Richardson (1999). Typically, for slurries with yield stresses higher than 100 Pa (Zengeni et al. 2012), the bob in an infinite cup method is a more accurate method to measure the slurry's rheology.

The bob was immersed in a beaker at least twice the diameter of the bob and twice the height of the bob. The torque was then recorded over speeds that ranged from 0–500 RPM. The measured speed was converted to angular velocity (Ω) and the following basic equation for coaxial rotational viscometers considered:

$$\frac{d\Omega}{d\sigma_b} = \frac{f(\sigma_b)}{2\sigma_b} \tag{4}$$

where:

Ω = angular velocity (rad/s).

σ_b = shear stress at the bob (Pa).

Solving Equation 4 for the shear rate at the bob and multiplying the numerator and denominator by Ω, and simplifying Equation 4, the following equation is obtained and can be used to determine the shear rate at the bob in the infinite cup (annulus is determined where the yield stress is equal to the shear stress) method:

$$f(\sigma_b) = \gamma_b = (2\sigma_b)\frac{d\Omega}{d\sigma_b} = \left(\frac{2\Omega\sigma_b}{\Omega}\right)\frac{d(\Omega)}{d\sigma_b} = (2\Omega)\frac{d(ln\Omega)}{d(ln\sigma_b)} \tag{5}$$

It is important to note that the rotational viscometer's software will not do these calculations and the data must be corrected using the raw speed and torque values recorded for each test. Figure 5 shows the typical error that can made if one uses the software output from the rotational viscometer without correcting the data. By not correcting the test data, the viscosity of the material will be underpredicted.

Figure 6 presents the corrected rotational viscometer test data for the cemented mixture. At 76%m it can be seen that there was a slight difference in the measured flow curves and, as the mass solids concentration increased, the effect of increasing cement addition increased slightly. In addition, the data also indicated that there was a significant increase in the plastic viscosity of the material above mass concentrations of 79%m.

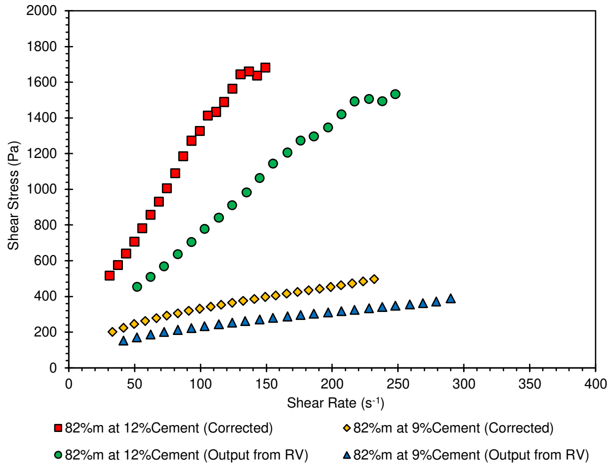

Figure 5 RV software output versus corrected data

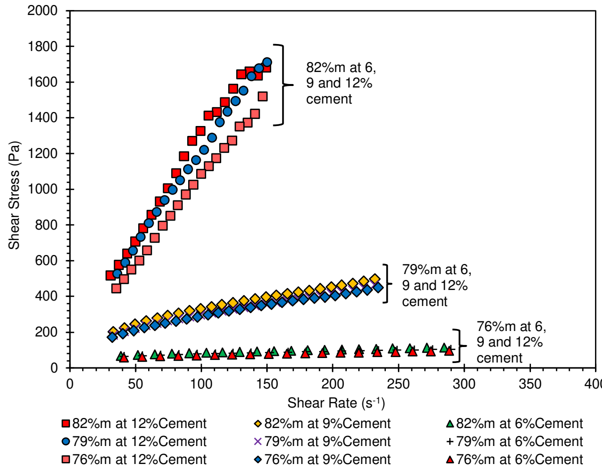

Figure 6 Corrected rotational viscometer data

4.3 Rheological characterisation

The rheological properties of the cemented mixes were determined from the rheogram. The data was analysed by applying the Bingham plastic model, which is a two-parameter model describing the slurry rheology. The two parameters are the Bingham plastic viscosity (K_{BP}) and Bingham yield stress (τ_y). The Bingham plastic equation is as follows:

$$\tau = \tau_{yBP} + K_{BP}\,\gamma \tag{6}$$

where:

τ = shear stress (Pa).

τ_{yBP} = Bingham plastic yield stress (Pa).

K_{BP} = Bingham plastic viscosity (Pa.s).

γ = shear rate (s^{-1}).

Table 3 presents the rotational viscometer rheology test results. The data shows that as the concentration increase, the yield stress and plastic viscosity increase. In addition to this, the data also indicate that there is an increase in yield stress and plastic viscosity with the increase in cement addition at mass concentration higher than 79%m.

Table 3 Rotational viscometer rheology results

Mix number	Cement addition (%)	Mass conc. (%m)	Water/cement ratio (m/m)	Yield stress (Pa)	Plastic viscosity (Pa.s)
1.1	12	82.0	1.83/1	209	10.7
1.2	12	79.0	2.22/1	191	1.3
1.3	12	76.0	2.63/1	60	0.2
2.1	9	82.0	2.44/1	180	10.4
2.2	9	79.0	2.96/1	170	1.3
2.3	9	76.0	3.51/1	58	0.2
3.1	6	82.0	3.66/1	161	9.1
3.2	6	79.0	4.43/1	154	1.3
3.3	6	76.0	5.27/1	56	0.1

5 Pipe loop tests

5.1 Equipment

The objective of pipe loop tests is to collect pressure gradient data over a range of flow velocities and mass concentrations (W/C ratios). A progressive cavity pump with a 90 kW motor (variable frequency drive driven) was used to circulate the material in the pipe loop. The pipe loop has the following components:

- Pressure tappings: Differential pressure measurements in the horizontal sections of pipe are made via static pressure tappings through the pipe wall. The tappings are burr-free inside the pipe. Each tapping is connected to a solids trap to ensure that the differential pressure transducers are isolated from the slurry in the pipe.
- Magnetic flowmeter: The slurry flow rate is measured using a magnetic flowmeter. The magnetic flowmeter, which consists of a detection head and signal converter, provides a current output that is proportional to the mean mixture velocity independent of the type of fluid (provided the fluid is conductive and not magnetic).
- Pressure gradient measurement pipes: 40.7 and 62.4 mm ID carbon steel pipes were used as the test pipes for this work.
- Slurry density measurement: The slurry density and calculated mass concentration measurements were taken at the return pipe when discharging back into the sump.
- Data acquisition system: All instrumentation is connected to a Paterson & Cooke data logging unit. The unit accepts analogue 4 to 20 mA inputs from the field instruments. The 4 to 20 mA signals are converted to 1 to 5 V DC signals linked directly to a data acquisition unit.

5.2 Clear water tests

The correct operation of the flowmeter, differential pressure transducer and pressure tappings is confirmed by conducting clear water tests prior to the slurry tests. The average pipe roughness is calculated from the measured water test data points using the Colebrook-White friction factor formulation and the Darcy equation.

The measured hydraulic pipe roughness for the 40.7 and 62.4 mm ID pipes are 8 and 40 µm, respectively, as shown in Figures 7 and 8. These hydraulic roughnesses are similar to previous test campaigns which indicated that the flowmeter, differential pressure transducer and pressure tappings were all working correctly.

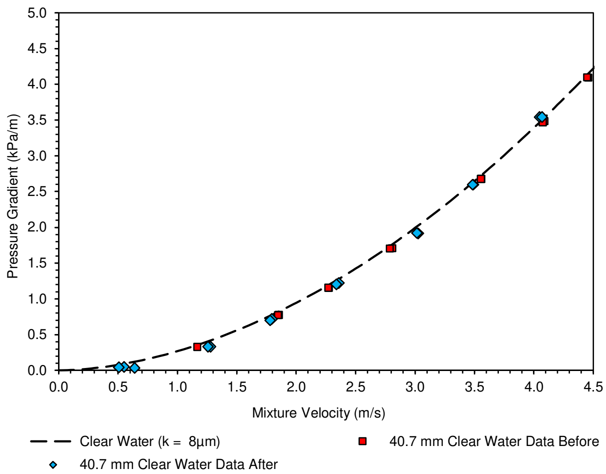

Figure 7 Clear water test – 40.7 mm ID

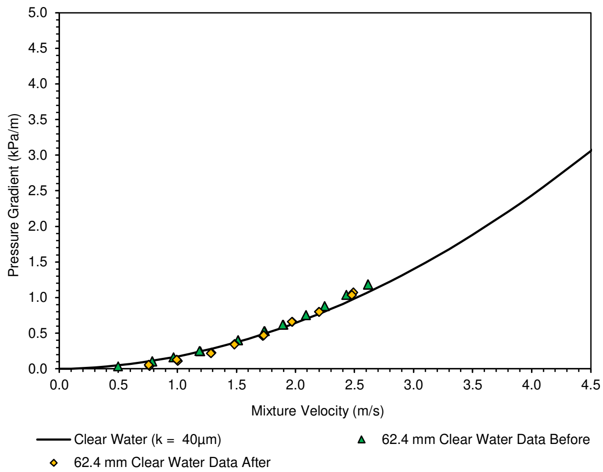

Figure 8 Clear water test – 62.4 mm ID

5.3 Slurry pressure gradients

The required target strengths are 1,000 kPa (majority) and 2,500 kPa (selected stopes) at 28 days. Based on the UCS test results, 1,000 and 2,500 kPa at 28 days can be reached at W/C ratios of 5.07/1 and 2.89/1, respectively.

The target strength of 1,000 kPa (W/C ratio of 5.07/1) could be reached at 79.0%m at 6% cement or 80.0%m at 5% cement, with the target strength of 2,500 kPa (W/C ratio of 2.89/1) being reached at 76.0%m with 12% cement or 80.0%m with 9% cement.

The rotational viscometer rheology test results indicated that above 79%m, the plastic viscosity of the mixes appeared to increase significantly, which was a concern from a cemented paste fill transport point of view. In addition to this, the cost of the cement must also be taken into consideration. Since the majority of the stopes only required 1,000 kPa at 28 days, the decision was taken to conduct pipe loop tests at a cement addition of 5% to confirm the rotational viscometer rheology for similar W/C ratios.

The pipe loop tests were conducted in two pipe diameters starting at a concentration of 82%m to match the W/C ratios tested during the rotational viscometer rheology tests. The pipe loop tests at 82%m showed that the material could not be pumped as the yield stress and plastic viscosity were too excessive and the material was therefore too dry. The starting mass concentration was therefore reduced to 80.0%m to achieve a pumpable mixture.

Figures 9 and 10 present the pressure gradient test results from the 40.7 and 62.4 mm ID pipes at a cement addition of 5%. The testwork was conducted at mass concentrations (W/C ratio) ranging from 76.0%m (6.32/1) to 80.0%m (5.00/1).

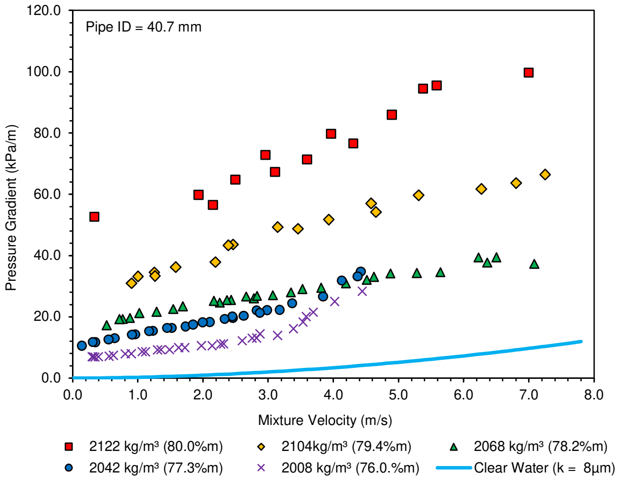

Figure 9 Pressure gradient versus mixture velocity 40.7 mm ID pipe

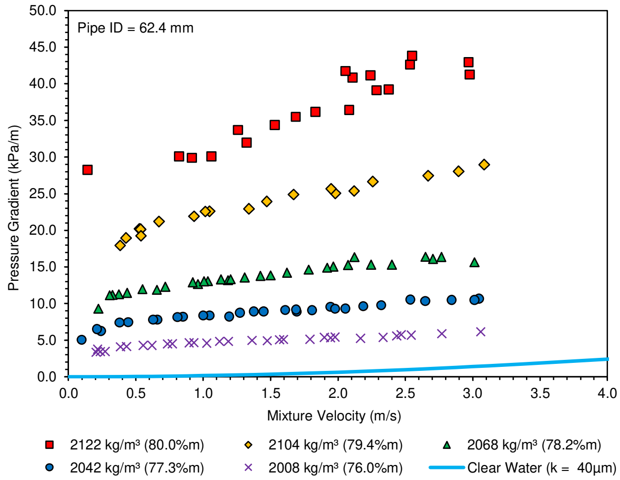

Figure 10 Pressure gradient versus mixture velocity 62.4 mm ID pipe

5.4 Wall shear stress and pseudo shear rate

The measured pressure gradient and mixture velocity were converted to wall shear stress and pseudo shear rate (pseudo shear diagram) by using the following formulas:

$$\tau_0 = \frac{\Delta PD}{4L} \qquad (7)$$

$$\Gamma = \frac{8V}{D} \qquad (8)$$

where:

τ_0 = wall shear stress (Pa).

ΔP = differential pressure (kPa).

D = internal pipe diameter (m).

L = tapping distance (m).

Γ = pseudo shear rate (s^{-1}).

V_m = mixture velocity (m/s).

5.5 Rheological characterisation

The results from the pipe loop tests were used to determine the Bingham plastic model parameters for comparison with the results from the rotational viscometer test results. Only laminar flow (Govier & Aziz 1972) data are presented on a pseudo shear diagram which is a plot of wall shear stress versus pseudo shear rate. The form of the Buckingham equation is as follows:

$$\frac{8V_m}{D} = \frac{\tau_0}{K_{BP}}\left[\left(1 - \frac{4}{3}\left(\frac{\tau_{yB}}{\tau_0}\right) + \frac{1}{3}\left(\frac{\tau_{yB}}{\tau_0}\right)^4\right)\right] \qquad (9)$$

where:

D = internal pipe diameter (m).

V_m = mixture velocity (m/s).

τ_0 = wall shear stress (Pa).

τ_{yB} = Bingham yield stress (Pa).

K_{BP} = Bingham plastic viscosity (Pa.s).

Figure 11 shows a comparison between the pseudo shear diagrams for the 40.7 (no fill) and 62.4 mm ID (solid fill) pipes. Figure 11 shows that the measured data from both pipes coincide with each other in the laminar flow region below 78.2% and then appear to diverge slightly at higher concentrations. If the data are not coincident it is likely due to some slip[1] at the pipe wall that may occur when pumping highly viscous materials.

Figure 12 presents the yield stress and plastic viscosity as a function of mass solids concentration, respectively. The data shows that a small increase in mass solids concentration results in a significant increase in yield stress and plastic viscosity over the concentration range tested.

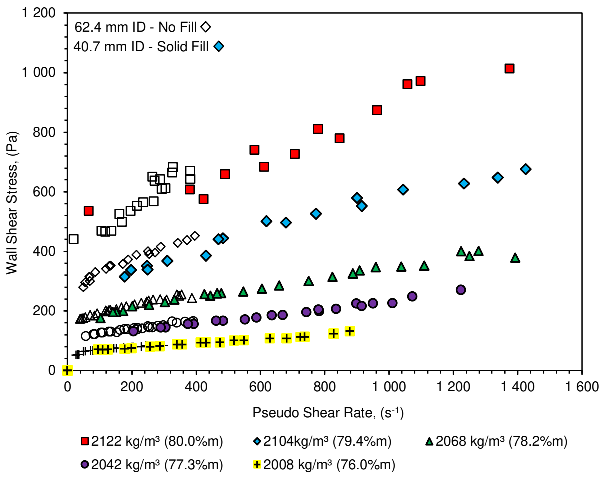

Figure 11 Pseudo shear diagram – 40.7 and 62.4 mm ID pipe data comparison

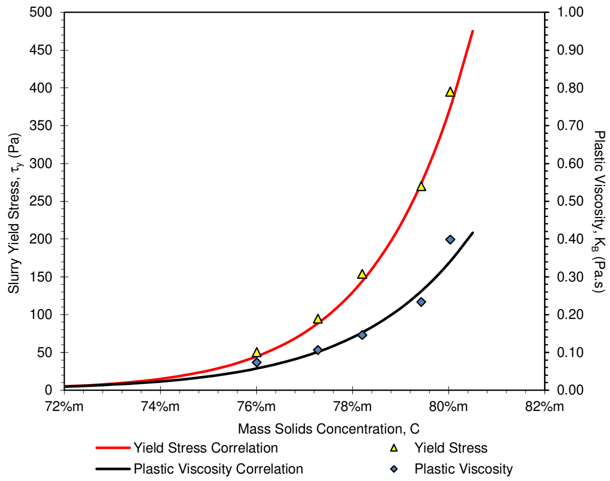

Figure 12 Yield stress and plastic viscosity versus mass concentration

6 Pipe loop versus rotational viscometer rheology

6.1 Shear rate and pseudo shear rate

Care must be taken when comparing pipe loop and rotational viscometer test data. A pipe loop measures volumetric flow rate and friction loss that is converted to wall shear stress and pseudo shear rate where a rotational viscometer measures torque and rotational speed. The torque is translated to shear stress at the cup (bob in an infinite cup method) and the shear rate is estimated from a velocity profile in the annulus (annulus is determined where the yield stress is equal to the shear stress).

The Rabinowitsch–Mooney transformation is used to obtain the shear rate at the wall of the pipe loop using the following equation:

$$-\left(\frac{du}{dr}\right)_0 = \left(\frac{3n'+1}{4n'}\right)\frac{8V}{D} \tag{10}$$

and

$$n' = \frac{d(\ln \tau_0)}{d\left(\ln \frac{8V}{D}\right)} \tag{11}$$

[1] Wall slip occurs in viscous fluids when the shear stress at a given shear rate is lower than expected due to the formation of a thin layer of lubricating fluid at the wall.

Note that n' is the slope of a logarithmic plot of the wall shear stress, τ_o, versus the pseudo shear rate, 8V/D. The slope n' is not necessarily constant and must be determined for each data point value of 8V/D. In practice n' is often constant over a wide range of shear rates for non-Newtonian liquids. If this is the case it is possible to use the average slope and make a correction across a series of shear rates. If the slope is not constant then it must be determined at each pseudo shear rate.

Once n' has been calculated, the true shear rate is easily determined using Equation 10, and can be plotted on a rheogram and compared to data from a rotational viscometer.

6.2 Data comparison

Figures 13 and 14 present comparisons between the pipe loop and rotational viscometer at mass concentrations of 76 and 79%m. The data shows that at 76%m, the pipe loop data at 5% cement addition compares fairly well with the rotational viscometer data at 6% cement addition. However, when the mass concentration is increased to 79%m, the test data shows that there is a significant difference between the pipe loop and rotational viscometer data. The data clearly shows that the rotational viscometer underpredicts the yield stress and overpredicts the plastic viscosity. This can be attributed to slip occurring at the wall of the bob.

Figures 15 and 16 present the yield stress and plastic viscosity data for the pipe loop at 76, 79 and 80%m versus the rotational viscometer test data at 76, 79 and 82%m. As seen from Figures 13 and 14 there is a clear deviation between the two methods at 79%m and, based on measured data and pumping limitations, it was recommended that the maximum solids concentration be limited to 79%m.

The required strength can then be achieved by varying the cement addition as the test data indicated, and there is only a slight difference between the yield stress and plastic viscosity as the cement addition increases.

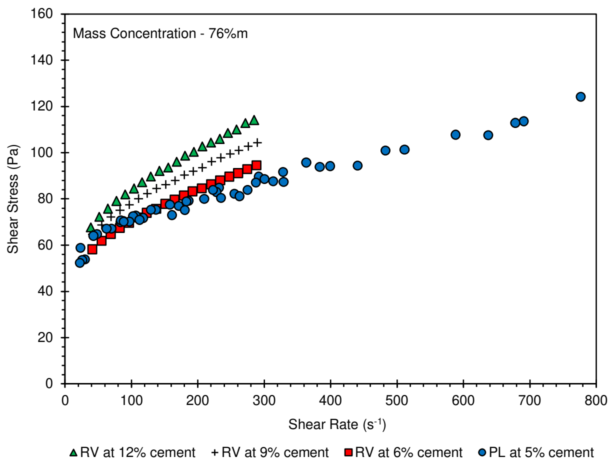

Figure 13 **Rotational viscometer versus pipe loop at 76%m**

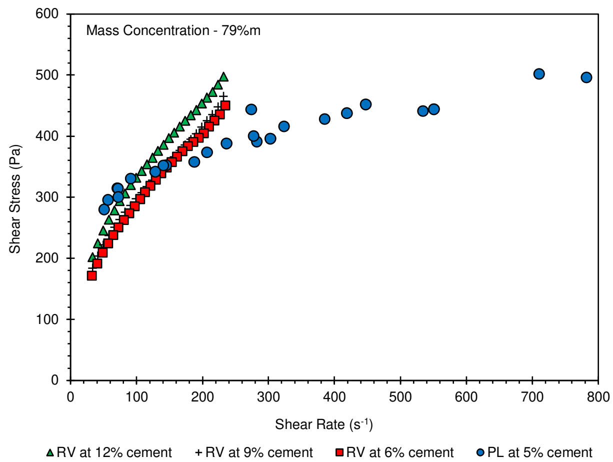

Figure 14 **Rotational viscometer versus pipe loop at 79%m**

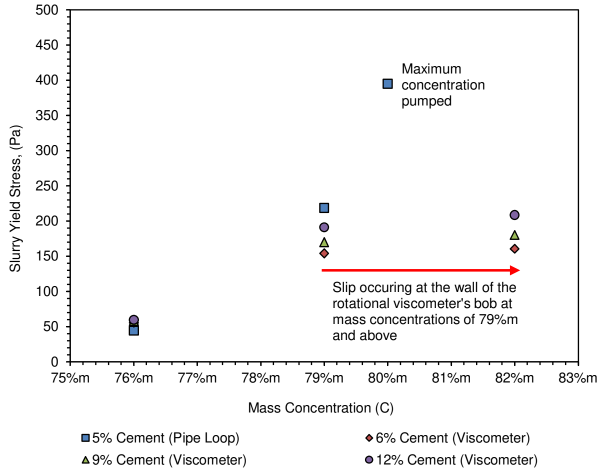

Figure 15 Yield stress versus mass concentration

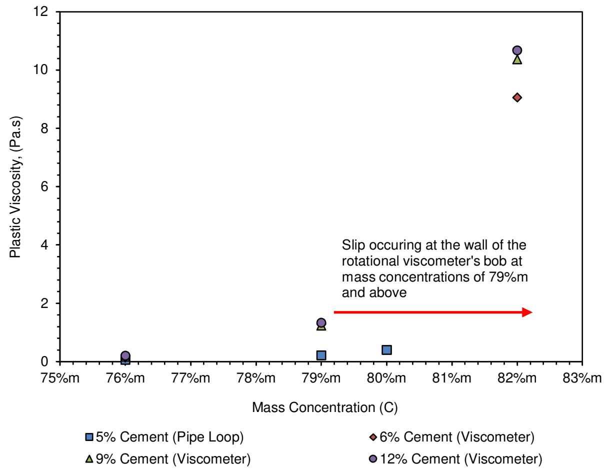

Figure 16 Plastic viscosity versus mass concentration

7 Conclusion

This paper presents the results of a series of tests conducted to ultimately determine the rheology for the cemented paste fill mixture. The main conclusions are as follows:

1. When conducting rotational viscometer measurements it is important to select the correct test method. For very viscous materials (>100 Pa), the typical bob and cup method will not provide accurate data. Better data will be obtained using the bob in an infinite cup method. It is important to note that the rotational viscometer software will not do these calculations and the data must be corrected using the raw speed and torque values recorded for each test. At high concentrations, the bob in an infinite cup might still provide inaccurate results due to slip on the wall of the bob as seen from these test results.
2. The pipe loop tests indicated that above ~78%m, the measured data from the two pipes appears to diverge slightly from each other, which could be attributed to slip occurring at the pipe wall. However, when the pipe loop rheology is compared to the rotational viscometer rheological test data, it is clear that at 79%m and above the rotational viscometer test data overpredicts the plastic viscosity and underpredicts the yield stress. If the rotational viscometer data is applied without realising that there are errors associated with slip, the yield stress can be significantly underpredicted and the plastic viscosity significantly overpredicted.
3. In addition to this, the pipe loop tests also indicated that with 82%m at 5% cement addition, the paste fill cannot be pumped due to excessive yield stress viscosity values. The maximum pumpable concentration was found to be 80%m. The data finally indicated that the maximum solids mass concentration should be limited to 79%m as this allows for a range of cement additions to achieve the required strength.

Acknowledgement

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