Rheological properties of thickened tailings and cemented paste tailings and the effects of mixture characteristics on shear behavior

Drissa Ouattara, Mamert Mbonimpa and Tikou Belem
Department of Applied Sciences - Université du Québec en Abitibi-Témiscamingue (UQAT), Rouyn-Noranda, Québec, Canada

ABSTRACT
This experimental study assesses the rheological behaviour of thickened tailings (TT) and cemented paste backfills (CPB) in order to better understand the complex interactions between mixtures and shearing procedures. A torsional rheometer equipped with parallel plates was used. Two types of binder (general use Portland cement CP and a blend of CP and Slag in the ratio 1:4) and three binder contents (2, 4.5 and 6 wt%) were tested at 0, 1h, 2h and 4 hours curing times. The results showed that the studied CPBs present some thixotropic behaviour and that increased binder content increases the rheological parameters. In addition, the yield stress and viscosity strongly depend on binder type (CP or CP-Slag).

RÉSUMÉ
Cette étude expérimentale évalue le comportement rhéologique des résidus épaissis (RE) et des remblais cimentés en pâte (RPC) afin de comprendre l'interaction complexe entre les mélanges et les caractéristiques de cisaillement. Un rhéomètre à torsion équipée avec des plaques parallèles a été utilisé. Deux types de liant (ciment Portland ordinaire CP et un mélange CP-Slag) et trois teneurs en liant (2, 4,5 et 6 wt%) ont été testés à 0, 1, 2 et 4 h de temps de cure. Les résultats montrent que les RPC étudiés présentent un comportement thixotrope et que l'augmentation de la teneur en liant semble amplifier les paramètres rhéologiques. Aussi, le seuil de cisaillement et la viscosité dépendent fortement du type de liant.

1 INTRODUCTION

In order to ensure environmental protection, workers security and improve productivity, mine tailings can be densified to thickened tailings (TT) for surface deposit or cemented paste backfill (CPB) for underground disposal (Landriault et al. 1997, Robinsky 1999, Aubertin et al. 2003, Belem and Benzaazoua 2003, Benzaazoua et al. 2004, Kesimal et al. 2004). Densified tailings technology aims to enhance and stabilize the hydrogeotechnical properties of tailings by reducing the water content to obtain thickened tailings, paste, or filter cake. The paste can be mixed with different binder types (cemented paste backfill) and used to fill mine stopes. The TT and CPB are usually delivered by pumping or gravity, which requires a pipeline reticulation system. Because densified tailings behave as non-Newtonian fluids, the design parameters for pipeline transportation depend on the control of the tailings rheological properties (i.e., yield stress and viscosity) and the tailings deposition slope (Sofra and Boger 2002, Paterson et al. 2004). Numerous factors such as chemical functional groups, pore volume, pore fluid pH and solid particles surface potential can affect the rheological properties of the material (Goudoulas et al. 2003, Huynh et al. 2006). Some authors have investigated the effect of different factors on tailings rheological parameters. Kwak et al. (2005) used vane geometry to determine the effect of water content on the rheological behaviour of mine tailings compared to that of kaolin clay. These authors measured yield stress to predict the surface deposition slope of tailings. Huynh et al. (2006) studied the effect of polymer addition on dewatering thickened tailings to reduce the rheological parameters. Cooke et al. (1992) examined the effect of cement addition on the rheological behaviour of classified backfill. They concluded that adding cement to the mixture increases both yield stress and apparent viscosity. Many other authors have explored the rheological behaviour of cement pastes that show thixotropic behaviour (Legrand 1972, Shaughnessy and Clark 1988 and Chougnet et al. 2007). When sheared, cement pastes exhibit an apparent yield stress and shear thinning behaviour. This shear thinning can be explained by disaggregation due to increasing shearing forces that progressively break down the material structure and induce lower viscosity. Some authors have reported shear thickening in cement and tailings pastes at high shear rates and high solid concentrations (Hébraud and Lootens 2005, Hallbom 2008). This behaviour was due to the turbulent regime for high shear rates. However, for high solid concentrations, shear thickening can be explained by increased aggregate size under shear or by reduced free space between particles, which are forced to jump over each other, hence increasing the apparent volumetric space (Shaughnessy and Clark 1988, Chougnet et al. 2007). This is related to the concept of maximum packing concentration of the solid volume fraction. It is therefore important to investigate the influence of cement addition on the variation in rheological parameters of densified total tailings.

The objective of this study was to investigate the effect of binder type and amount, curing time and shearing procedure on the rheological properties of TT and CPB, using a rotational rheometer equipped with parallel plate geometry. The study also emphasises on the relevance and reliability of this geometry to ensure appropriate test data.

2 MATERIALS AND METHODS

2.1 Tailings sample

The main relevant properties of the mine tailings used are presented in Table 1. The initial water content was 22.7% (solid concentration of 81.51%) and the specific gravity G_s (or Dr) was 2.81. The grain-size volume distribution (GSD) was determined using a Malvern Mastersizer laser granulometer. The obtained cumulative GSD curve was unimodal and gave the average particle diameter D_{50} = 23.44 µm, D_{60} = 33.28 µm and D_{10} = 3.13 µm, corresponding to 50%, 60% and 10% of particles passing on the cumulative curve, respectively. The coefficient of uniformity C_U (= D_{60}/D_{10}) was 10.64. As explained below, another important GSD parameter in rheological characterization is the D_{90} (= 119.13 µm), corresponding to 90% particles passing. The tailings appear to consist of typical dispersed silty material. The main minerals present in the tailings are quartz (51.72%), albite (23.20%) and muscovite (17.69%). The only sulphurous mineral detected was pyrite at 2.77%.

Table 1. Physical properties of tailings sample

Parameter	Value
Initial water content (%)	22.7
Initial solid content (%)	81.51
D_r (-)	2.81
D_{10} (µm)	3.13
D_{50} (µm)	23.44
D_{60} (µm)	33.28
C_U (-)	10.64
D_{90} (µm)	119.13
Quartz (%)	51.72
Albite (%)	23.20
Muscovite (%)	17.69
Pyrite (%)	2.77

2.2 Torsional rheometer

The rheological measurements were performed using a torsional rheometer (AR2000, TA Instruments, USA). Only parallel plate geometry was used with the serrated stator. A 40 mm diameter serrated stainless steel upper plate was attached to the rotational axis of the rheometer. The automatic zero gap mode was used after bearing and inertia calibration. The plate geometry gap must be at least 10 times higher than the largest grain size of the mixture, which was assumed to correspond to D_{90}. Once the gap is set, a rotational geometry mapping is implemented and the apparatus is ready for operation.

Before testing, the temperature, geometry dimension and gap must be set. All tests were performed at a constant temperature of 20°C (±0.1°C accuracy). Preliminary testing showed that the gap must be kept at 2000 µm to minimize the plate geometry effect. Ghezzehei and Or (2001) and Goudoulas et al. (2003) used the same gap for a similar material. This gap is about 16 times higher than the D_{90} given in Table 1.

2.3 Experimental procedure

2.3.1 Calibration tests

Prior to testing the TT and CPB materials, the rheometer was first calibrated using certified Newtonian fluids from Cannon Instrument Company (USA) and the non-Newtonian fluid NNTF1 manufactured by Physikalisch-Technische Bundesanstalt (PTB, Germany). The objective was to assess, calibrate and validate the rheometer setting and any potential disruptions, including slip effect. Parallel plate geometries with smooth and serrated stators were used to quantify the potential slip effect.

2.3.2 TT and CPB recipes preparation

The uncemented thickened tailings (TT) and cemented paste backfill (CPB) mixtures were prepared based on ingredient mass proportion. The solid mass concentration for all recipes was 70 wt%. For each mixture, tailings mass was calculated for the preparation of 200 ml specimens of TT or CPB for rheological measurements. Two binder types were used: a general use Portland cement CP and a blend of CP and ground granulated blast furnace slag at a ratio of 1:4. For each binder type, three binder contents by dry mass of tailings were tested: 2, 4.5 and 6 wt%. Binders were selected for their widespread use at several mine backfill plants. Slag is increasingly used in the mining industry for its pouzzolanic reaction and its beneficial effect on backfill hydration, especially for sulphide-rich tailings. The binders were added to the tailings and mixed with deionized water using a high velocity mixer at 1000 rpm until samples were completely deflocculated. Tests were performed at different curing times: 0, 1, 2 and 4 hours after mixing.

2.3.3 Shearing procedures and rheological data analysis method

The experimental program involved various measurement conditions, including shearing procedures such as continuous ramp flow, stepped flow step and steady state step flow (see Figure 1). For the continuous ramp shear path, shear stress was increased continuously and data points were sampled at a fixed time interval. For the stepped step flow, a logarithmic stress increase was applied to the specimen and data points were sampled at the middle of a fixed time interval, and for the steady state step, data points were collected at the end of a fixed time interval.

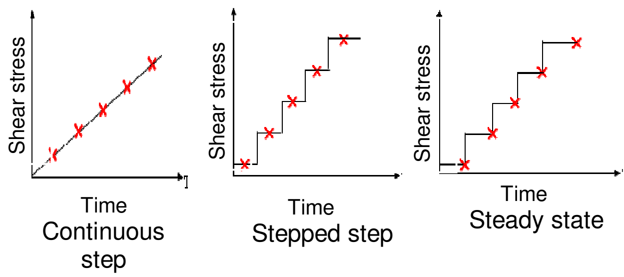

Figure 1. Shearing procedures using the AR 2000 torsional rheometer (crosses indicate rheological parameter sample points).

Based on the calibration tests, these three shear loadings recorded good results, and the shearing paths were implemented during the preliminary CPB shear behaviour investigation. The continuous ramp procedure was the most appropriate shear loading path for recording entire experimental rheological parameters because of the evolving nature of the material, due to either sedimentation or cementation over time. According to Ghezzehei and Or (2001), two criteria can be used to select the appropriate stress range for mixtures during pilot testing. First, adequate points were required near the yield point to better determine the yield stress. Thus, stress ramp appeared more appropriate than shear rate variation. The second criterion is related to the maximum stress obtained in intact material between the plates (at higher stress rates, failure results from high centrifugal forces).

Samples were loaded onto the roughened stator and the upper geometry was lowered to the prescribed gap of 2000 µm. The excess material was carefully removed from the stator before measuring. For all measurements, 30 points were recorded in 3 minutes. For each specimen, up flow and down flow tests were performed to measure both static and dynamic yield stresses and to assess the thixotropic behaviour of the material. Down flow curves are generally recommended for comparison purposes because of their stability and reproducibility (Ragouilliaux et al. 2006), while the up flow curves are influenced by sample placement artefacts. For the thickened tailings samples, a typical shear stress range of 0–150 Pa was found to be appropriate. Beyond the maximum stress of 150 Pa, the sample was expelled from the plate geometry. However, for CPB samples, typical stress ranges were: 0–250 Pa (up step) and 250–0 Pa (down step) for 0 hours of curing. Other shear stress ranges of 0–300 Pa or higher were sometime used with up and down shearing for tests conducted after 2 hours curing time on some CPB samples.

After measurement, the rheological parameters of TT and CPB were evaluated using built-in rheological data analysis software. The flow curves (shear stress versus shear rate plots) were fitted with different well known rheological models by applying the best fitting option available in the software. The software then generated the model parameters (including the yield stress, consistency and index flow rate) of a suitable model by matching experimental data as closely as possible with the calculated flow curve for model selection. The performance of the different models was compared using the standard error.

3 RESULTS AND DISCUSSION

3.1 Calibration tests on Newtonian and non-Newtonian fluids

Different certified Newtonian fluids with viscosity from 1 Pa.s for RT1000 to 100 Pa.s for RT100000 were used. Only results for the fluid RT60000 with a viscosity of 60 Pa.s at 20℃ are presented in Figure 2 for shear rates from 0 to 1.4 (and shear stresses from 0 to 85 Pa). The measured viscosity closely approaches the reference value. The deviation (or the difference between the measured result and the reference data) is in the range of 1.9%.

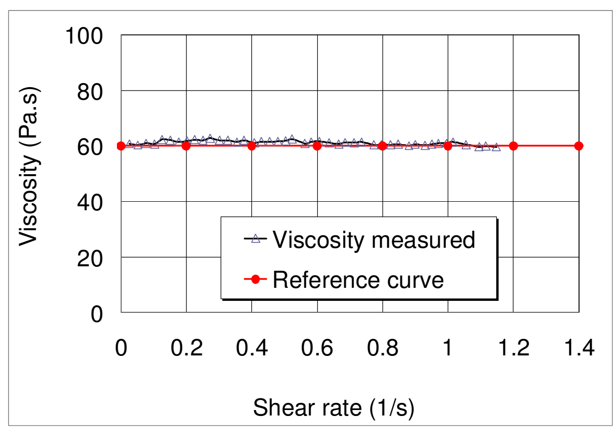

Figure 2. Viscosity curves of certified Newtonian fluid RT 60000 fluid

The certified non-Newtonian fluid NNTF1 shows Bingham behaviour for shear rates up to 10 s^{-1} with a yield stress of 10.7 Pa and power low behaviour for shear rates beyond 10 s^{-1}. The flow curve obtained with this fluid is compared with the reference flow curve (Figure 3), where the maximum and minimum reference flow curves are also given, showing a quasi-perfect match. Deviation for the fluid NNTF1 is about ±0.8% for the yield stress and ±1.5% for the viscosity. These differences can be attributed to an artefact in the sample placement, including sample over- and under-filling on the geometry gap.

In general, the calibration test results are in good agreement with reference data from fluid manufacturers. We may conclude that the measurement procedures were appropriate.

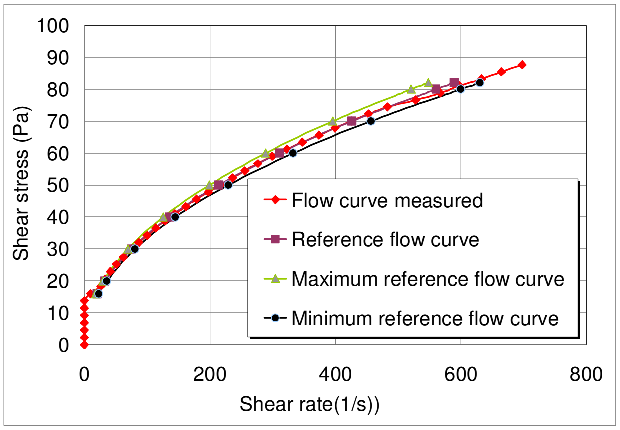

Figure 3. Reference and measured flow curves of certified non-Newtonian fluid NNTF1

3.2 Up and down flow curves: effect of shear history and thixotropic behaviour

The shear behaviour of fluid material is typically investigated using a rheometer with up and down flow procedures. The resulting flow curve presents an ascending part with increasing shear stress and a descending part with decreasing shear stress.

Figures 4 and 5 present the up and down flow curves in linear scale for the CPB sample (at 70 wt% solid mass concentration) containing 2 wt% and 6 wt% binder content of CP and CP-Slag@20:80, respectively. A difference between the up and down flow curves can be observed. The up and down flow curves give yield stress known as static and dynamic yield stress, respectively According to Barnes and Nguyen (2001), the static yield stress is taken as “the departure from linearity of stress response in a plastic solid as the strain is increased.” The dynamic yield stress is “taken as the cessation of flow in a structured liquid as the stress is decreased.” These definitions are assumed in this study.

The flow curves in Figures 4 and 5 are presented in semi-logarithmic scale in Figures 6 and Figure 7, showing clearly different behaviour. This behaviour is explained by the fact that the CPB material at rest develops a structure, which is destroyed when submitted to shearing and then rebuilt once the stress is removed. In fact, during the increasing stress ramp, the applied stress was increased gradually, which progressively destroyed the flocculation state due to electrostatic attractive forces between particles.

Hence, the up flow curves present an initial part at low shear rate, which usually corresponds to the viscoelastic solid regime of the material (Coussot 2005). When the shear stress is sufficiently higher than the yield stress, the sample reaches steady state flow and is in a fully dispersed state at the end of the up flow. Particles are in fully dispersed state and cannot flocculate because the shear stress exceeds the electrostatic forces. Afterwards, structuration is recovered with decreasing shear stress.

The decreasing curves appear to pass from viscous to solid state without transition, and the yield stress is better indicated than in the up flow curves. Ragouilliaux et al. (2006) found the same behaviour when investigating drilling mud rheological properties. The up flow curves show poor fit to the flow models due to the viscoelastic phase in the curve beginning. In fact, the standard errors obtained on the up flow curve fitting are far higher than for the down flow curves. This can be explained by the fact that this curve depicts the slurry shear history, including the mixing process before pre-shear loading (Goudoulas et al. 2003).

In order to obtain a good fit with a relatively low standard error, the fitting of the up flow curves must be limited to the steady flow part of the curve. In contrast, the down curves are relatively smooth, and the fitting models can consider the entire flow curve with a relatively low standard error. The calculated yield stresses are quite accurate. The difference between these two yield stresses depicts a hysteresis area between the up and down flow curves indicating the structure build-up process.

This behaviour is known as the thixotropic effect, which is time-dependent, and has been noted in some cemented materials. Figures 4 to 7 show increased hysteresis areas with increasing binder content (for a given binder type). Thus, for 0% binder (uncemented tailings or TT), the up and down flow curves are almost similar.

In the following sections, the reported rheological parameters are based on down flow curves as representative of the material rheological behaviour.

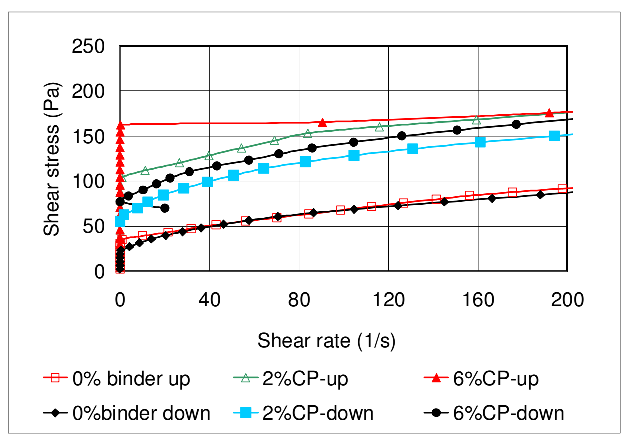

Figure 4. Up and down flow curves for CPB samples with 2 wt% and 6 wt% of CP binder type using a linear scale at 0 h curing time.

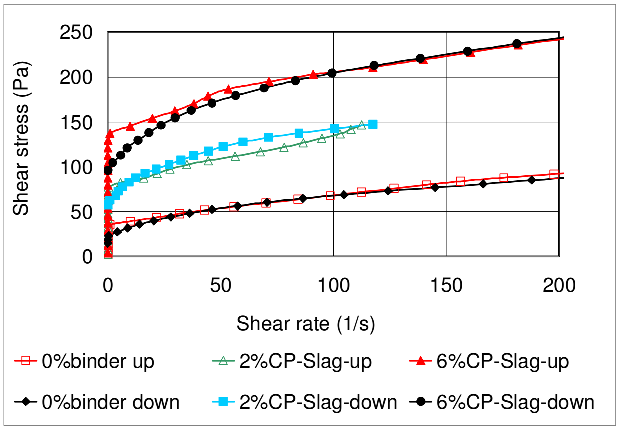

Figure 5: Up and down flow curves of CPB with 2 wt% and 6 wt% of GU-Slag binder type using a linear plotting scale at 0 h curing time.

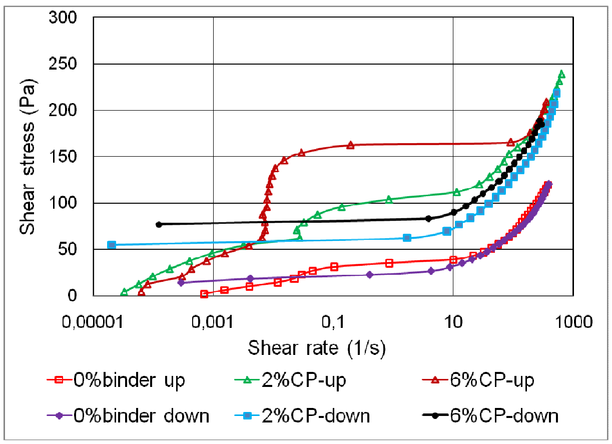

Figure 6. Up and down flow curves of CPB with 2 wt% and 6 wt% of CP binder type using a semi-logarithmic scale at 0 h curing time.

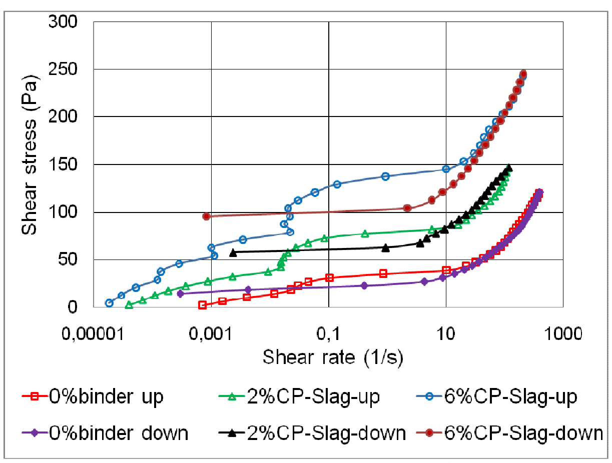

Figure 7: Up and down flow curves of CPB with 2 wt% and 6 wt% CP-Slag binder type on semi-logarithmic scale at 0 curing time.

3.3 Effect of binder type and content

The rheological behaviour of uncemented thickened tailings is typically different from that of CPB due to binder addition (as explained above). As mentioned in the introduction, adding cement to tailings mixtures enhances some CPB properties. In this section, the effect of adding CP binder and CP-Slag@1:4 binder on CPB rheological behaviour was examined. In this purpose, CPB flow curves immediately after adding binder (0 hour curing time) are compared with TT flow curves. Furthermore, to evaluate the effect of binder hydration on the CPB rheological parameters, the flow curves are compared at two curing times.

Figures 8 to 10 show the flow curves plotted in a linear scale, and Figures 11 to 13 present the viscosity curves in a semi-logarithmic scale for TT and CPB (binder content of 2 wt%, 4.5 wt% and 6 wt%) considering the two added binder types at 0, 1, 2 and 4 hours curing time.

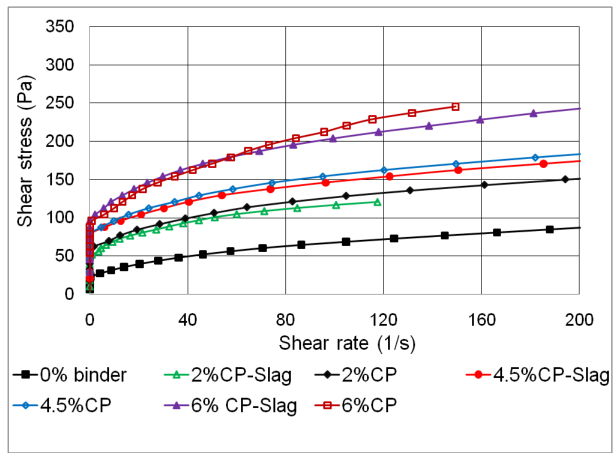

Figure 8. Flow curves for TT and CPB samples at 0 hour curing time for different contents of binder types CP and CP-Slag.

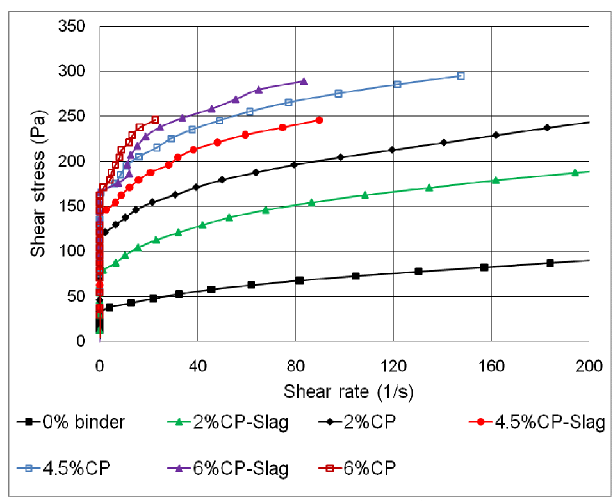

Figure 9. Flow curves for TT and CPB samples at 2 hours curing time for different contents of binder types CP and CP-Slag.

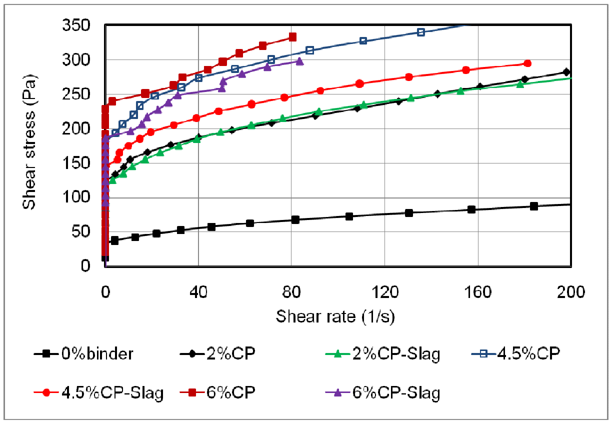

Figure 10. Flow curves for CPB samples at 4 hours curing time for different contents of binder types CP and CP-Slag.

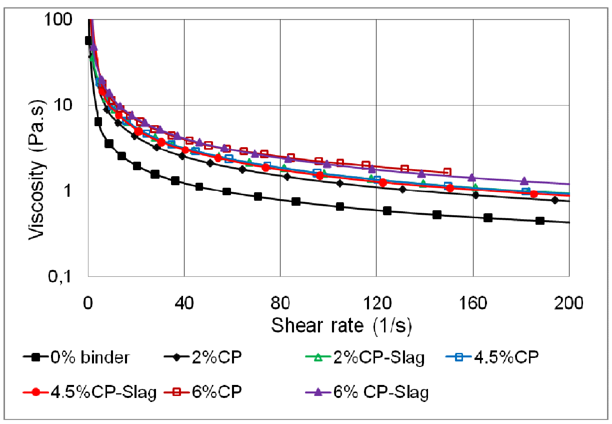

Figure 11. Viscosity curves for TT and CPB samples at 0 hour curing time for different contents of binder types CP and CP-Slag.

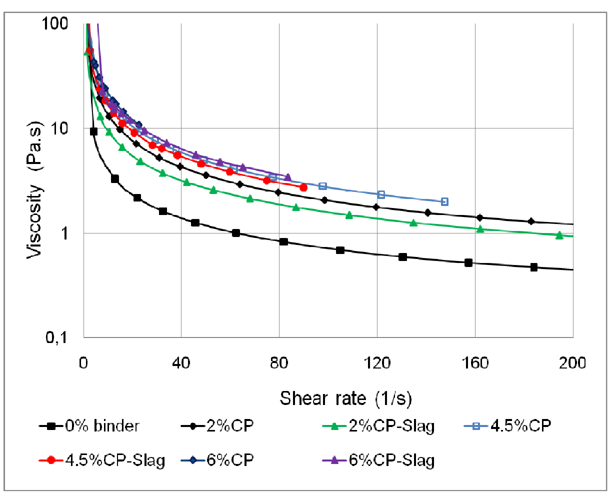

Figure 12. Viscosity curves for TT and CPB samples at 2 hours curing time for different contents of binder types CP and CP-Slag.

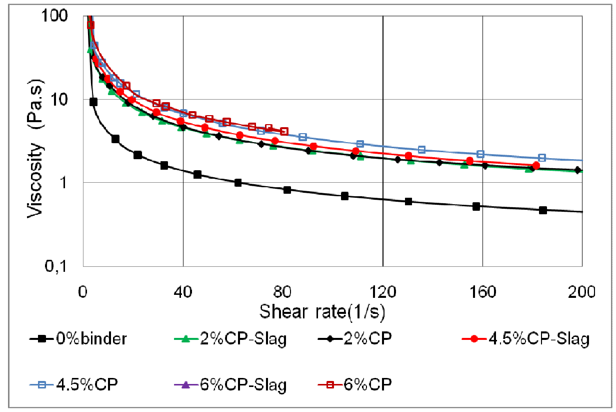

Figure 13. Viscosity curves for CPB at 4 hours curing time for different contents of binder types CP and CP-Slag.

The data analysis of these curves gives best fitting results with the Herschell–Bulkley model, written as follows:

$$\tau = \tau_{HB} + K\dot{\gamma}^{n} \qquad [1]$$

In this equation, τ_{HB} is the yield stress of Herschell–Bulkley model (Pa), K is the consistency (Pa.s^n) and n the index of shear rate (-). Table 2 summarizes the model fitting parameters (τ_{HB}, K and n) for the tested TT and CPB samples.

Table 2. Herschell–Bulkley rheological model parameters for CPB with CP and CP-Slag binder types

CT (h)	wt% B	CPB (CP binder)			CPB (CP-Slag binder)		
		τ_{HB} (Pa)	K (Pa.s^n)	n	τ_{HB} (Pa)	K (Pa.s^n)	n
0h	0 (TT)	20.5	3.6	0.55	20.5	3.60	0.55
	2	53.6	7.42	0.48	53.1	10.60	0.46
	4.5	74.9	8.1	0.49	75.6	5.90	0.53
	6	85.2	8.05	0.60	89.0	12.87	0.47
1h	0	31.4	1.17	0.73	31.4	1.17	0.73
	2	76.5	4.64	0.56	61.7	13.21	0.43
	4.5	105.1	5.18	0.58	89.6	10.36	0.54
	6	109.4	11.36	0.48	107.1	12.61	0.49
2h	0	32.9	5.13	0.52	32.9	5.13	0.52
	2	108.7	9.83	0.49	91.1	13.25	0.47
	4.5	151	15.09	0.45	132.7	10.36	0.54
	6	159.2	10.21	0.71	156.8	11.2	0.57
4h	0	33.4	4.96	0.52	33.4	4.96	0.52
	2	122.8	4.83	0.64	106.3	12.98	0.48
	4.5	169.8	17.36	0.46	133.7	14.70	0.46
	6	220.9	2.22	0.90	181.4	3.98	0.77

(CT: Curing time)

The yield stress value for TT is about 20.5 Pa at 0 hour curing time, whereas the yield stress for CPB with 2 wt%, 4.5 wt% and 6 wt% of CP binder contents is 53.6 Pa, 74.9 Pa, and 85.0 Pa, respectively. The yield stress for samples with CP-Slag at the same binder content is close to that of samples with CP binder (i.e., 53.1 Pa, 75.6 Pa and 89 Pa, respectively). Compared to TT, the yield stress of CPB increases about 2 times for the 2 wt% binder content and as much as 4 times for 6 wt% binder content right after binder adding and mixing. It can be concluded that for a given binder content, both binder types induce an increase in the yield stress and viscosity of CPB.

For 2 wt% binder content, the yield stress increased from 53.6 Pa at 0 hour curing time to 108.7 Pa at 2 hours curing time for CP binder and from 53.1 to 91.1 Pa for CP-Slag binder. With 4.5 wt% binder, the yield stress increased from 74.9 Pa to 151 Pa for CPB with CP binder and from 75.6 to 132.7 Pa for CPB with CP-Slag. With 6 wt% binder content, the yield stress increased from 85.2 Pa at 0 hour curing time to 159.2 Pa (at 2 hours curing time) for CPB with CP binder and from 89.0 to 156.8 Pa with CP-Slag binder. For all binder contents, i.e., 2%, 4.5% and 6 wt%, the yield stress for CPB with CP binder is higher than that for CPB with CP-Slag binder. Moreover, Figures 11 to 13 show that the viscosity curves for CPB with CP-Slag binder are consistently below the CPB with CP binder only viscosity curves for a same curing time.

3.4 Effect of curing time

From the results presented in Figures 8 to 13 (and Table 2), it can be seen that both down flow and viscosity curves vary significantly with both binder proportion and curing time. The change in yield stress obtained from the Herschell–Bulkley model as a function of curing time is presented in Figure 14 for different binder contents, showing that the yield stress increases with curing time. The yield stress change in TT is lower than for CPB, and appears to be constant after 1 hour curing time. For fresh CPB (containing either CP or CP-Slag binder type) after mixing (0 hour) and for a given binder content, the yield stresses are practically identical. However, after a curing time of 2 hours, an increase in yield stress is seen for both binder types for all binder contents. This increase in yield stress is low for from 0 to 1 hour curing time, and becomes greater for from 1h to 2h curing time.

According to Xu and Stark (2005), the Portland cement hydration process comprises four steps: initial, dormant or induction, acceleration and deceleration. The tests performed here fall into the induction period (0–2h) and the beginning of the acceleration period (2–4 h). This increase in yield stress (structure breakdown) during the induction period is questionable, as it does not correspond to normal hydration. However, Yang and Jennings (1993) showed that the increase in yield stress during the first 2 hours appears to depend strongly on the microstructure produced at rest, which can be broken down by the mixing effort and shearing. Here again the yield stress increase is higher for the CP-only binder type than the CP-Slag binder type for a same binder content.

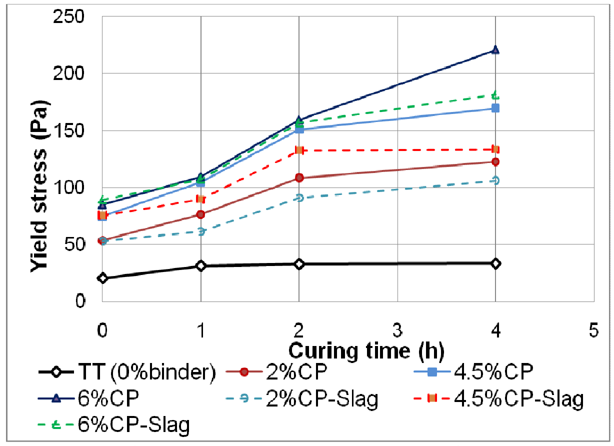

Figure 14. Change in the yield stress of CPB with curing time.

4 CONCLUSION

This study examined the shear behaviour of thickened tailings (TT) and cemented paste backfill (CPB) in order to understand the effect of binder addition to filtered tailings cake on rheological properties. Two types of binder were tested: a general use Portland cement (CP) and a blend of 20% CP and 80% Slag (CP-Slag@1:4). Four binder contents by dry mass of tailings were tested: 0, 2, 4.5 and 6 wt%. Shear tests were performed on samples after curing at 0, 1, 2 and 4 hours. The solid proportion for all recipes was 70 wt%.

The flow of these materials was characterized using parallel plate geometry (serrated stainless steel rotor and stator) attached to a torsional rheometer. The continuous ramp shear path was determined as the best shear flow path. The difference between up and down flow curves was used to evaluate the thixotropic behaviour of the tested samples, which was found to increase with increasing binder content. CPB samples with CP binder were found to behave more thixotropically than those with CP-Slag binder. It was observed that the Herschell–Bulkley model was more suited for fitting the flow curves obtained with the different CPB mix recipes.

This study also confirmed the increase trend of rheological parameters (yield stress and viscosity) of CPB with binder addition. This increase depends on binder type and content as well as curing time. The yield stresses of CPBs containing CP binder and CP-Slag binder are almost identical at 0 hour curing time (TT), but differ at 1 hour, 2 hours and 4 hours curing.

ACKNOWLEDGEMENTS

This research was supported by the *Fond institutionel pour la recherche* (FIR) of UQAT, and the NSERC Discovery Grant Program. The equipment used was mostly financed by the CFI. The authors gratefully acknowledge their support.

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