Preliminary investigation of the effect of temperature and salinity on the rheological properties of fresh cemented paste backfills

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ABSTRACT
Underground backfilling under cold climate conditions in permafrost, factors such as saline water and low temperatures must be taken into account during the design process of the hydraulic transport and distribution system of cemented paste backfill (CPB). The impact of these factors on the rheological properties of CPBs must be carefully characterized in the laboratory. This study examines the effect of temperature (2 to 45°C) and salinity of the mixing water (0 and 5 g/L) on the rheological properties of uncemented tailings and CBP with 5% HE cement prepared at a slump of 7 inches. A rotational vane rheometer equipped with a temperature control system was used. Measured flow and viscosity curves were fitted using the Herschel-Bulkley and Sisko models, respectively, providing different rheological parameters (yield stress, viscosity, consistency index, and flow index), The results illustrate the effects of binder, temperature, and salinity on the rheological behavior of CPB. For the CPB mixtures, the rheological properties (yield stress, dynamic viscosity at high shear rate) tend to increase with temperature increase. Salinity of 0.5% slightly improved these rheological properties.

RÉSUMÉ
Dans le contexte du remblayage souterrain dans le pergélisol en climat nordique, des facteurs tels que l'eau saline et les températures froides doivent être pris en compte lors de la conception d'un système de transport et de distribution hydraulique du remblai en pâte cimenté (RPC). L'impact de ces facteurs sur les propriétés rhéologiques des RPCs doit être attentivement caractérisé en laboratoire. Cette étude examine l'effet de la température (2 à 45°C) et de salinité de l'eau de gâchage (0 et 5 g/L) sur les propriétés rhéologiques des résidus de pâte et de RPC avec 5 % de ciment HE, et à un affaissement de 7 pouces. Un rhéomètre rotationnel à croisillon équipé d'un système de régulation de température a été utilisé. Les courbes d'écoulement et de viscosité mesurées ont été ajustées en utilisant respectivement les modèles de Herschel-Bulkley et de Sisko, permettant d'obtenir différents paramètres rhéologiques (seuil de cisaillement, viscosité, indice de consistance, et indice d'écoulement). Les résultats présentés permettent d'illustrer l'effet du liant, de la température et de la salinité sur le comportement rhéologique des RPCs. Pour les mélanges de RPC, les propriétés rhéologiques (seuil de cisaillement, viscosité dynamique à taux de cisaillement élevé) ont tendance à augmenter avec l'accroissement de la température. Une salinité de 0.5% améliore légèrement ces propriétés rhéologiques.

1 INTRODUCTION

Cemented paste backfill (CPB) is widely used in underground mines as secondary support to stabilize the mine workings (Belem and Benzaazoua, 2008). The CPB is a mixture of filtered tailings, water, and binder to ensure a certain cohesion. Using CPB favors maximum underground ore recovery. This technique is particularly relevant when tailings stored on surface in tailings storage facility generate acid mine drainage or contaminated neutral mine drainage. Hence, significant amount (up to 50%) of potentially problematic tailings can be placed underground instead, hence reducing surface environmental impact.

Freshly prepared CPB is usually transported through pipelines network and reticulation from the surface (backfill plant) to underground mine stopes to be filled either by gravity and/or pumping (Cooke, 2001). The CBP hydraulic pipeline transport system's design (in terms of flowability or pumpability) can be based either on the paste backfill flow behavior in pipeline from flow loop-tests (Clark et al., 1995; Cooke, 2001; Cooke, 2007; Wu et al., 2015) or on the CPB's rheological behavior (Hallbom, 2008; Senapati and Mishra, 2012). The rheological behavior is usually characterized by the yield stress and the dynamic viscosity, which can be affected by external factors (temperature, pressure, salinity, etc.) and internal factors (water content, chemistry, hydration reaction, grain size distribution, pH, solid percentage, etc.) (Wu et al., 2013).

Pipeline wall friction, backfill shearing during flowing, and external heat exchange can induce intrinsic temperature changes within the CPB material during its transportation and after its placement underground. These temperature changes can in turn affect the rheological properties of the CPB and its hardening processes. In the literature, almost all existing studies on the effects of temperature on the properties of CPB mainly targeted strength rather than the rheological properties. Most studies on the temperature's effect were conducted on pure cement pastes. In this cases, the rheological properties (yield stress and plastic viscosity) increase when the mixing and curing temperature increases, even in the dormancy period (Lei and Struble, 1997; Al-Martini and Nehdi, 2005; Petit et al., 2006) It should be mentioned that pastes without binder generally show a decrease in the yield stress and viscosity with increasing temperature (Yang et al., 2001; He et al., 2004; He et al., 2006a; He et al., 2006b, Altin et al., 2006; Senapati et al., 2009).

Only few studies focused on temperature's effect on the rheological behavior of CPB. Wu et al. (2013, 2014) established relationship between the degree of hydration of CPB and the rheological properties at a given temperature and curing time.

Concomitantly, the presence of salt ions or electrolytes in the mixing water is one of the chemical factors affecting CPB's rheology (Klein and Pawlik, 1999; Mikanovic et al., 2006; Klein and Simon, 2006). Hence, the use of saline mixing water for CBP preparation is expected in northern regions due to the natural salinity of frozen water in the permafrost (Hivon and Sego 1993) and to the added salinity resulting from the use of deicing salts on ore. These dissolved salt ions or electrolytes may affect interactions between particles, which plays an important role in the rheological properties of suspensions (Chang et al., 1993; Colic and Fisher, 1998; Johnson et al., 2000; Mikanovic et al., 2006; Huynh et al., 2006). Generally, rheological properties of suspensions are proportional to particle's attractive forces when they predominate. Conversely, when the repulsive forces are dominant, rheological parameters have relatively low values (Chang et al., 1993; Pawlik, 2005; Chen et al., 2007; Amiri et al., 2009). Depending on the type and concentration of electrolytes (salt ions) in cementitious materials (e.g., CPB, mortar, concrete, cement paste), repulsive or attractive forces can respectively increase or decrease significantly, affecting consequently the binder's hydration processes and/or their rheological properties (Klein and Simon, 2006; Mikanovic et al., 2006; Mahlaba et al., 2011a; Mahlaba et al., 2011b).

By taking temperature into account during the CPB hydraulic transport system's design becomes very important in the application of CPB in deep mines and in northern regions. In cold climate regions, it's also necessary to take into account the salinity of permafrost waters. The use of CPB in underground stopes bounded by walls of permafrost in northern regions remains poorly documented, although the technique is increasingly considered viable given the corresponding advantages. Knowing the temperature distribution's variations throughout the body of CPB confined in frozen walls of permafrost is crucial for the formulation of laboratory recipes that can develop the required mechanical strength.

The curing conditions of CPB placed in stopes in the permafrost (with walls frozen at about -10°C) may depend on the occurrence of heat exchanges, which in turn depend on different factors, including the deposition temperature of the CPB. The fresh CPB deposition temperature is therefore a key parameter to be known. Work is on progress for estimating this temperature from numerical modelling of CPB flow in pipelines. These models involve the temperature-dependent rheological parameters of CPB. Therefore, the main objective of this study is to investigate the effect of temperature and salinity on the rheological properties of CPB.

2 MATERIAL AND METHODOLOGY

2.1 Tailings characteristics

The physical and mineralogical characteristics of the tailings used in this study are shown in Table 1 where D_x, C_U, C_c and P_y represent the diameter corresponding to x% passing on the cumulative grain-size distribution curve, the coefficient of uniformity, the coefficient of curvature, and the fraction passing y µm, respectively. The tailings were obtained from dry milling of drill core samples. The relative density D_R of the tailings (measured using a Micromeretics helium pycnometer) is 2.93. The grain size distribution of the tailings was determined using a Malvern Mastersizer laser granulometer. In the light of the physical characterization results summarized in the table 1, these tailings are comparable to common Canadian mine tailings from hard rocks (Bussière, 2007). Following the USCS classification system, the tailings can be classified as low plasticity silt (ML).

The major mineral phases of the tailings consist in Quartz (40.32%), Albite (19.49), Muscovite (14.65%), Ankerite (8.77%) and Chlorite (8.07%). The Magnetite (4.58%), Calcite (2.43%), Microline (1.33%) and Pyrite (0.35%) form the minor phases.

Table 1. Physical and mineralogical characteristics of the mine tailings

Parameter	Unit	Value
Specific gravity D_R	-	2.93
D_{10}	µm	3.14
D_{30}	µm	9.38
D_{50}	µm	18.31
D_{60}	µm	24.37
D_{90}	µm	62.59
C_c	-	1.15
C_u	-	7.75
P_2	%	2
P_{20}	%	53
P_{80}	%	94
Mineral	Unit	Value
Quartz	%	40.32
Albite	%	19.49
Muscovite	%	14.65
Ankerite	%	8.77
Chlorite	%	8.07
Magnetite	%	4.58
Calcite	%	2.43
Microline	%	1.33
Pyrite	%	0.35

2.2 Experimental procedure

2.2.1 Mixture preparation

The binder used in the CPB's preparation is type HE Portland cement (formerly designated as Type III cement according to ASTM C 150-07). The binder content for the CBP samples was set to 5% (by dry mass of tailings), which corresponds to a water - binder ratio of 7. Tap water and saline water with a salt concentration of 0.5% (5 g salt per liter of water) were also used for different mixtures preparation at a fixed slump of 7 inches. Thus, the following mixtures were studied:

- uncemented paste tailings with tap water (PT);
- uncemented paste tailings with 5 g/L saline water (SPT);
- CPB with tap water,
- CPB with 5 g/L saline water (SCPB).

Based on the geochemistry of expected tailings pore water at a mine site in northern regions of Canada, different types of salts ($NaCl$, $CaCl_2$, KCl, $MgSO_4$, $MgCl_2$, $Na_2S_2O_5$, $CuSO_4$) were dissolved in appropriate concentrations in tap water to prepare the mixing saline water at a concentration of 0.5% (5 g/liter of water). The concentrations for the different salts used are listed in Table 2, with NaCl representing 60 wt% of the total salt content. The saline water pH was adjusted to 7.0 with lime.

Table 2. Types of salts and their concentration

Salt	Concentration (g/liter)
$NaCl$	3.059
$CaCl_2$	0.465
KCl	0.050
$MgSO_4$	0.351
$MgCl_2$	0.313
$Na_2S_2O_5$	0.672
$CuSO_4$	0.090
Total	5.000

The different ingredients (tailings, water and binder) were conditioned for 24 hours at the targeted testing temperatures before being mixed. For temperatures lower (2°C and 10°C) and higher (40°C) than 20°C, the conditioning was done in a cold chamber and in an oven with temperature control, respectively. The various ingredients were then mixed using a propeller mounted on a rotor at a speed of 300 rpm for 7 minutes to avoid temperature perturbation at high rotation speeds (Juilland et al., 2012). The measurement of the rheological properties was conducted right after each mixture's preparation was completed.

2.2.2 Measurement of the rheological properties

The rotational rheometer AR 2000 (from TA Instruments) equipped with a sample holder (cylinder), a vane shearing geometry and a temperature control system was used for the measurement of the rheological properties of the studied mixtures. The gap between the rotating vane and the wall of the stationary cylinder was 1 mm. This geometry gap must be at least 10 times higher than the largest grain size of the tested mixture, generally considered as D_{90}. This condition was fulfilled, as the D_{90} = 0.063 mm.

This rheometer allows shearing to be performed either under controlled shear stress or controlled shear rate. The shearing procedure adopted for the experimental program was the continuous ramp flow. This flow procedure consists in steadily increasing the shear rate $(\dot{\gamma})$ within a specified time interval (1 minute and 10 seconds in this case). Shearing stress (τ) is sampled at a fixed time interval (10 seconds, in this case). To ensure identical shear conditions, each mixture sample was sheared from 0 to 100 s^{-1} (up flow curve), and then from 100 to 0 s^{-1} (down flow curve). Up flow curves are influenced by sample placement artefacts, therefore, the use of down flow curves is generally recommended for comparison purposes because of their stability and reproducibility (Ragouilliaux and al., 2006). Data collected on the down flow curves were processed using the "Data analysis" software from TA Instruments. This operation consists in adjusting the flow curve $\tau(\dot{\gamma})$ and dynamic viscosity curve $\eta(\dot{\gamma})$ data using the «*smooth*» function to merge duplicate or triplicate data. Finally, a fitting of the merge curve was made with different rheological models. The performance of the different models was compared using the standard error (SE in ‰) defined by equation [1].

$$SE = 1000\left[\frac{\left(\Sigma\frac{(x_m - x_c)^2}{(N-2)}\right)^{1/2}}{\text{Range}}\right] \quad [1]$$

where *Range* is the difference between the maximum and minimum value measured, N is the number of points, x_m and x_c represent the measured and calculated (from the fitting model) values, respectively.

Standard error is deemed acceptable when less than 20‰ (TA Instruments Manual). In this paper, it was found that the Herschell-Bulkley and Sisko models, defined in equations [2] and [3], were more appropriate for the flow and dynamic viscosity curve fitting, respectively.

$$\tau = \tau_{0HB} + K_{HB}\dot{\gamma}^{n_{HB}} \quad [2]$$

$$\eta = \eta_{\infty} + K_S\dot{\gamma}^{n_S - 1} \quad [3]$$

where, τ_{0HB}, K_{HB} and n_{HB} represent the Herschell–Bulkley yield stress, consistency index and flow index (n_{HB} < 1 for shear-thinning, n_{HB} > 1 for shear-thickening and n_{HB} =1 for Bingham behavior), respectively; η_{∞} is the dynamic viscosity at infinite (high) shear rate ($\dot{\gamma} \rightarrow \infty$), K_s is the Sisko consistency index and n_s is the Sisko viscosity index.

Testing temperatures of 2, 10, 20 and 45°C were targeted for the mixtures without binder (PT and SPT). At the temperature of 45°C the rheological tests provided unreliable results due to repetitive sliding of CPB and SCPB sample in contact with the stationary cylinder.

Therefore, the tests were performed for temperatures between 2°C and 40°C for the CBP and between 2°C and 20 °C for the SCPB.

3 RESULTS AND DISCUSSION

3.1 Effect of temperature and salinity on flow curves

Figures 1 and 2 present typical flow curves for the PT and CPB mixtures conditioned for temperature between 2 and 45°C. The thermo-rheological behaviors of these two types of material are different in terms of relative position of the flow curves at the different temperatures.

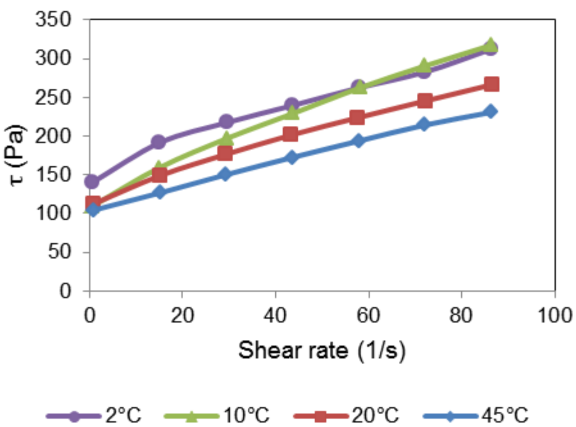

Figure 1. Flow curves for uncemented paste tailings (PT) conditioned at 2, 10, 20 and 45°C

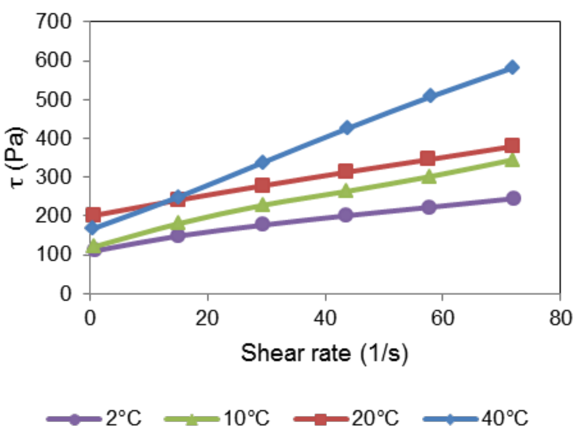

Figure 2. Flow curves for CPB samples conditioned at 2, 10, 20 and 40°C

At a given shear rate, the corresponding shear stress decreased for PT (Figure 1) and increased for CPB (Figure 2) with increasing temperature. This illustrates the behavioral differences between materials with and without binder, as explained in the introduction. These flow curves were fitted with the Herschell-Bulkley model with SE values ranging between 0.98‰ and 16.6‰, therefore providing the parameters τ_{HB}, K_{HB} and n_{HB}. This behavioral difference will also be highlighted below using the rheological parameters from the rheological models presented in Eqs. [2] and [3].

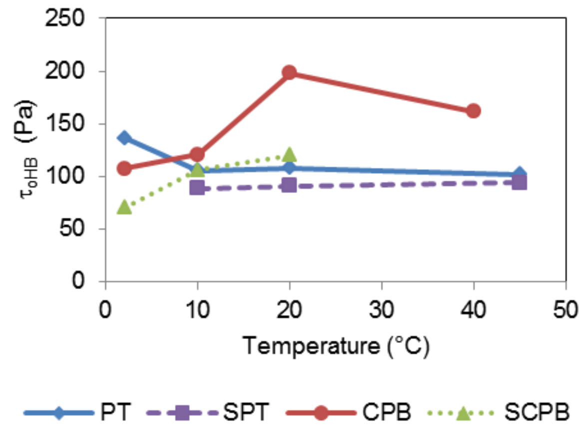

Figure 1. Variation in the yield stress as a function of temperature for PT, SPT, CPB and SCPB samples

Figure 3 presents the effect of temperature on the yield stress for all mixtures studied (PT, SPT, CPB and SCPB). The yield stress of the CPB and SCPB tends to increase with temperature increase, while it tends to decrease slightly when the temperature increases for PT and SPT. As mentioned above, this difference in terms of behaviors is related to the presence of type HE Portland cement in the CPB. Ouattara et al. (2012) investigated the effect of binder on the rheological properties of CPB and PT (same solid content) at 20°C and showed that CPB exhibited higher yield stress than PT. This observation is confirmed from Figure 3 at 20°C. At 2°C, however, the opposite seems to occur; this behavior will be deeply investigated before being confirmed. Furthermore, Figure 3 shows higher yield stress values for mixture without saline water, which indicates that the mixing water's salinity may affect rheological properties.

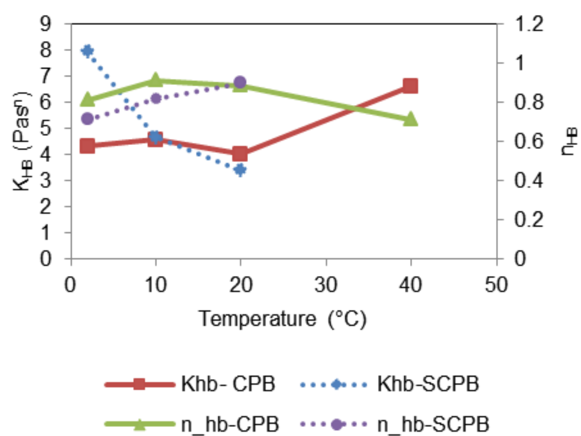

Figure 2. Variation in the consistency index and flow index as a function of temperature for CPB and SCPB

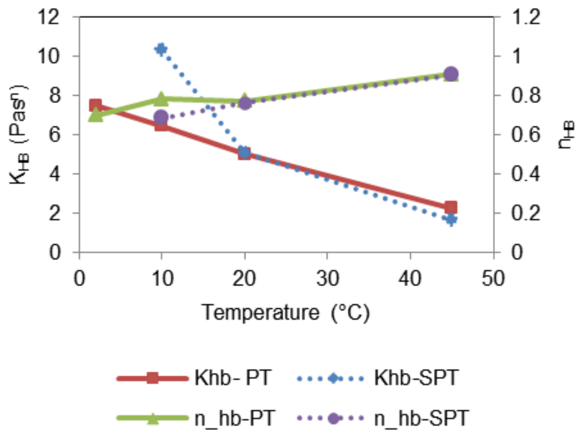

Figure 3. Variation in the consistency index and flow index as a function of temperature for PT and SPT

Figures 4 and 5 show the variation according to temperature changes, for the consistency index K_{HB} and the flow index n_{HB} of cemented paste backfill mixtures (CPB and SCPB) and of uncemented paste tailings (PT and SPT), respectively. In all cases, n_{HB} is lower than 1, i.e. the mixtures exhibit a shear-thinning behavior. The consistency and flow indexes of the CPB vary slightly for temperatures between 2 and 20°C. However, there is an increase in consistency index and decrease in the flow index from 20 to 40°C. The SCPB mixtures showed decreasing consistency index and increasing flow index when the temperature increases from 2 to 20°C. No data were available for temperatures higher than 20°C as explained previously. The consistency index K_{HB} of PT and SPT mixtures decreased when the temperature increased, while their flow index increases along with temperature. It is still difficult to explain this behavior.

3.2 Effect of temperature and salinity on viscosity curves

Figures 6 and 7 display typical viscosity curves for CPB and PT conditioned at temperatures between 2 and 45°C. These viscosity curves were fitted with the Sisko model with very low SE (ranging between 1.08‰ and 5.60‰), providing the parameters η_∞, K_S and n_S. Figure 8 presents the change in the viscosity η_∞ at high shear rate as a function of temperature. The viscosity η_∞ at infinite (high) shear rate increases for backfill mixtures (CPB and SCPB) and remained nearly constant for the uncemented paste tailings (PT and SPT). It should also be noted that CPB exhibits higher values of viscosity η_∞ than that of PT for all tested temperatures, due to the binder addition.

Figure 9 presents the effect of temperature on the Sisko viscosity index n_S for all the mixtures (PT, SPT, CPB and SCPB). In all studied cases, n_S decreased with increasing temperature. However, the decrease is not significant for CPB and PT between 2 and 10 °C.

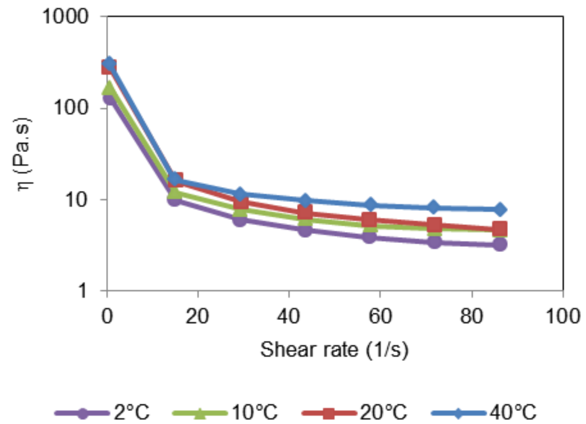

Figure 4. Viscosity curves of CPB as a function of the shear rate at temperature of 2, 10, 20 and 40°C

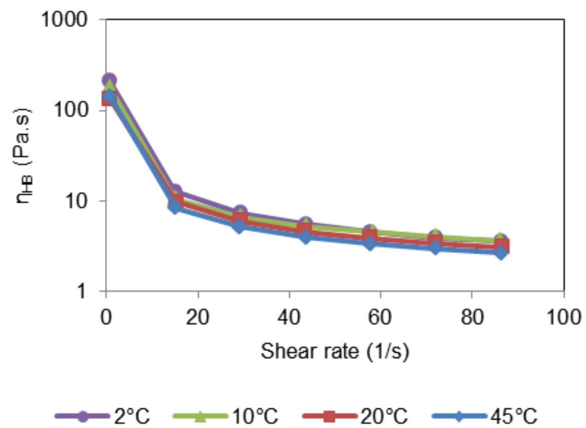

Figure 5. Viscosity curves of PT conditioned at 2, 10, 20 and 40°C

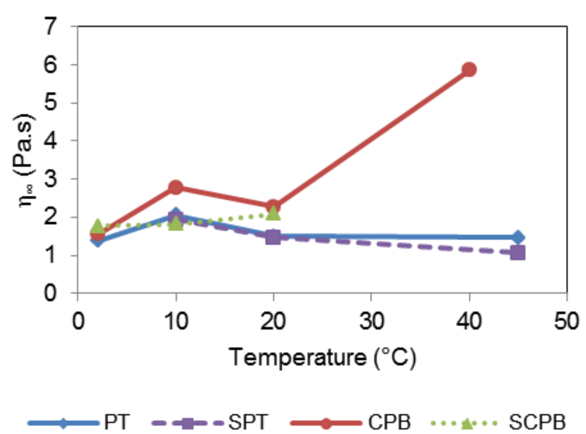

Figure 6. Effect of temperature on the viscosity at high shear rate for PT, SPT, CPB and SCPB

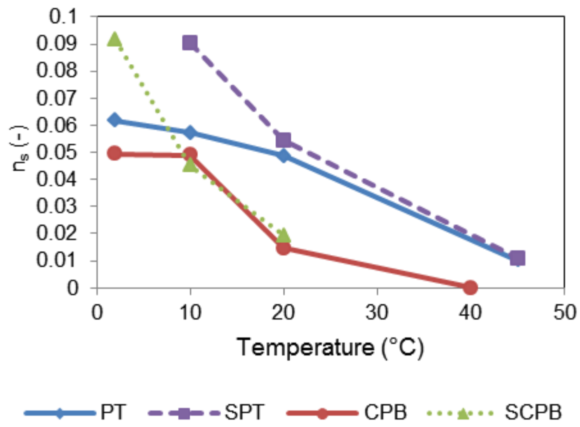

Figure 7. Effect of temperature on the Sisko viscosity index n_s for PT, SPT, CPB and SCPB mixtures

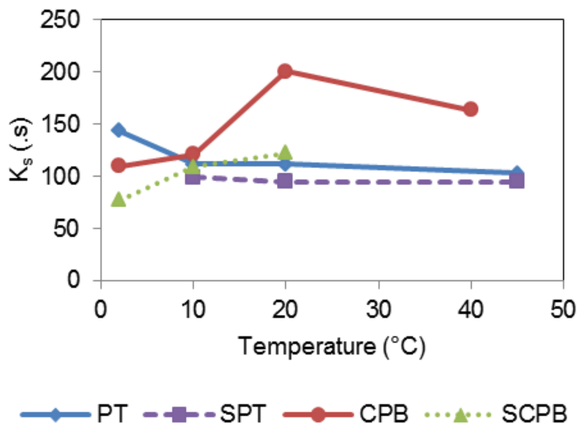

Figure 8. Variation in the Sisko consistency index K_s as a function of temperature for PT, SPT, CPB and SCPB mixtures

Figure 10 presents the variation in the Sisko consistency index K_s versus temperature for all the mixtures (PT, SPT, CPB and SCPB). This variation is similar for all the mixtures to the one of the yield stress (see Figure3). The Sisko consistency K_S of CPB and SCPB increased as temperature increases, while it decreased slightly for PT mixture. The mixtures without salts exhibit high Sisko consistencies index compared to mixtures with salts for all temperatures.

4 DISCUSSION

Results presented in Figures 3, 8 and 10 showed that the rheological properties (yield stress, viscosity at high shear rate, consistency K_S) of mixtures with saline water (SPT and SCPB) are lower than those of mixtures without saline water (PT and CPB) for all temperatures. However, SPT and SCPB exhibited higher values of consistency K_{HB} than PT and CPB at 10°C and 2°C (Figures 4 and 5). CPB and SCPB have nearly equal values of flow index n_{HB} at 20°C. Then, values of n_{HB} of SCPB become lower than those of CPB for temperatures under 20°C. As for SPT, it can be observed values of n_{HB} which are equal to those of PT at 20°C and 45°C. At 10°C, the flow index of SPT is lower than that of PT.

The Sisko viscosity index n_s seems less affected by the addition of saline water with uncemented tailings (SPT) at 20°C and 45°C and in the CPB mixture (SCPB) at 10°C and 20°C. However, there are significant differences at low temperatures of 10°C for SPT and 2°C for SCPB (Figure 9).

Figures 3 and 8 shows the effect of salinity on the rheological properties of the mixtures. Overall, the salinity (at a concentration of 5 g/liter) tends to decrease the shear yield stress and the viscosity at high shear rate. This trend is more notable at 20°C than at 10°C and 2°C, for SCPB mixtures. However, for SPT mixtures, the effect of salinity is almost unnoticeable at 10°C and 20°C (Figures 8 and 10). The yield stress of SPT is only slightly affected by temperature and its viscosity presents low values at 20°C in comparison to those obtained at 10°C and 45°C. This phenomenon is probably related to the effect of temperature on particle interaction. Further study is still required to better understand this behavior. Mikanovic et al. (2006) showed that electrolytes such as NaCl, KCl, $CaCl_2$ increased fluidity (i.e. improve the rheological parameters) of fresh cementitious materials. However, KCl and $CaCl_2$ improve the fluidity of fresh cementitious materials better than NaCl. It should be noted that NaCl, KCl, $CaCl_2$ represent fractions of 61%, 1%, and 9.3% in the saline mixing water used, respectively.

Salt ions tend to interact more directly with the cement particles and high temperatures allow their rapid adsorption on the particles in suspension (Kong et al., 2013). Repulsive forces are thereby increased, and consequently the rheological properties of the mixtures decreased. Klein and Simon (2006) have highlighted this deflocculating effect induced by NaCl and $CaCl_2$ in CPB. Precautions should be taken, as these salts can also accelerate the hydration process, causing a loss of fluidity (increase of rheological properties) of cementitious materials (Zhou et al., 1996).

5 CONCLUDING REMARKS

This study investigated the effect of temperature (between 2 and 45°C) and salinity (salt concentration of 0 and 5 g/L) on the rheological properties of uncemented paste tailings (PT and SPT) and cemented paste backfill (CPB and SCPB). The mixtures were prepared at a fixed slump of 7 inches for a single binder content of 5%. High early Portland cement (type HE) was used during the experimental program. The continuous ramp test procedure was used for the rheological characterization of the mixtures using a vane geometry rheometer equipped with a temperature control system. The flow curve model of Herschell-Bulkley and viscosity curve model of Sisko were used to obtain the different rheological parameters.

The CPB showed a different rheological behavior in accordance with the temperature, compared to PT, illustrating the binder's effect. The rheological properties (yield tress, viscosity) of CPB and SCPB tend to increase with the temperature. However, PT and SPT showed the opposite behaviour. The presence of salt translated into a positive effect (improvement) on the rheological properties of SPT and SCPB. The coupling effect of salinity and temperature showed the improvement of the rheological properties with increasing temperatures.

This study was performed on a single type of tailings; one solid concentration (75%); one type of binder (type HE cement), a single binder content (5%); and salt concentrations of 0 and 0.5% (5 g/L water). Additional tests are being performed to extend the study to other mixtures. Data gathered from the whole study will be used to develop empirical equations to estimate the rheological parameters. These equations will be implemented in a multiphasic numerical code to evaluate the flow behavior of CBP in pipelines. Furthermore, a loop test is planned for simulating the transport of the CPB in pipelines and for evaluating the impact of material shearing and the wall friction and the temperature on the rheological properties.

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