Design of the paste backfill recipe for the Pinos Altos Mine, Mexico — influence of tailings clay mineral fraction on strength and rheology

S. Ouellet Agnico-Eagle Mines, Canada

F. Brunet Agnico-Eagle Mines, Canada

Abstract

Agnico-Eagle Mexico S.A. de C.V. (AEM) plans to commission a new paste backfill plant at the Pinos Altos mine (Mexico) in March 2010. In 2008–2009, the paste recipe was designed to develop required strength for free standing of the backfill in stope after 28 days. This recipe development was not as easy as usual because the tailings contain a significant proportion of clayey minerals and these cohesive minerals control the rheology of tailings. This paper presents the different laboratory investigations performed to achieve the required strength considering the flow characteristics of the tailings.

The paste recipe design was performed in two phases. The first one included testing of different paste recipes with varying water and binder (Chihuahua cement) content. The main investigation of the rheology (laboratory rheometer tests) of the paste was performed during this phase. The second phase was carried out to optimise the paste recipe by changing different ingredients to improve the geomechanical properties considering the clay mineral content in tailings. Tests with slag (Lafarge Canada), with deslimed tailings and with water reducing admixtures (BASF Glenium 7700TM and 7102TM) were performed (Glenium 7102 and 7700 are trademarks from BASF Construction Chemicals). In this phase, five backfill samples were also cured under 300 kPa of vertical pressure, which is similar to the one estimated at the bottom of a typical stope.

Among mixtures tested, paste with 7% Chihuahua cement/71% solids and paste with 7% Chihuahua cement and Lafarge Slag in a 20:80 proportion/71% solids gave the best results. These mixtures were selected for tests under vertical pressure and results showed significant strength increases at 28 curing days relative to the standards. Thus, significant strength increase is expected in stope due to consolidation relative to laboratory results. Tests with deslimed tailings and admixtures were not conclusive, and these options to increase strength were not retained. The rheological measurements performed on all mixtures tested showed a Bingham plastic behaviour. A clear influence of the water and binder content were observed on the yield stress of the paste.

1 Introduction

The Agnico-Eagle Mexico Pinos Altos mine is located in the Sierra Madre gold belt, 280 km west of Chihuahua, the state capital. Agnico-Eagle acquired the Pinos Altos property in 2006. In August 2007, a favourable feasibility study led to the decision to commence construction of the Pinos Altos mine. Initial production started in summer 2009, beginning with ore from the open pit. Underground mining development is ongoing. The gold is processed by conventional milling and heap leach operations. Average annual production is anticipated to be approximately 5,000 kg of gold and 737,000 kg of silver. An exploration program is in progress which includes drilling from the underground decline. The focus is on resource conversion and on expansion of the Santo Nino, Cerro Colorado, Reyna de Plata and Creston Mascota zones. Approximately 60% of the Pinos Altos mineral resource is located in the Santo Niño zone, along a regional fault zone that holds a number of other known deposits in the area. This Santo Niño zone has thicknesses of up to 45 m over a length of 2 km, and a vertical extent identified to over 750 m. It remains open to the west and at depth.

As most of modern underground operation, the Pinos Altos mine exploitation is planned with availability of a paste plant providing 3,000 T/day of paste fill. The first paste fill recipe evaluation was conducted in 2008 and revealed the significant influence of the alteration minerals contained in tailings on the strength and the

rheology. Different variations in the testing program were then introduced to increase the strength in the short time. This paper presents a summary of the studies conducted in laboratory to achieve this.

2 Materials and methodology

Most of tests were performed with the participation of the URSTM (Rouyn-Noranda, Canada). Tailings used for this paste recipe evaluation were recovered after the metallurgical tests; they were delivered to the laboratory in dry state. Tailings were analysed for chemical content by digestion in HNO₃/Br₂/HF/HCl, while sulphate content in tailings was evaluated by digestion in HCl only. Results are presented in Table 1. Tailings samples were also analysed for mineralogical composition by X-Ray diffraction (XRD) on a Bruker A.X.S. D8 Advance using a copper radiation source and scintillation detector. Samples were scanned over a 2-theta range of $5-70^{\circ}$ with a 0.005° step size. Minerals were quantified using the Rietveld method through the Bruker's TOPAS software (Table 1). The main minerals identified in the tailings are the silicate minerals quartz, microcline and albite; alteration minerals are also observed. Additional XRD analysis in the region 5–19° revealed the presence of clayey minerals that are overlapped by chlorite and/or muscovite peaks when looking at the full range XRD spectrum. This means that observed chlorite and muscovite are probably partly or completely replaced by clayey minerals such as montmorillonnite, illite and kaolinite in an amount ranging between 7 and 8.5% in the tailings. Table 1 also shows the particle size distribution (Malvern laser Mastersizer S) of the tested tailings. According to the USCS classification tailings can be classified as CL-ML (silty clay) and the cumulative volume of particles lower than 20 µm is 51%. The plasticity index of tailings has been measured between 6 and 7%, leading to an activity value of approximately 0.6. This means a low potential for expansion and suggests that the clay mineral is probably kaolinite (McCarthy, 2007).

For all paste fill mixtures tested, the Mexican binder CP030R-BRA/RS was used. Binder was analysed with Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES) for solid chemical content by digestion in Br_2 (Br_2 was recommended because calcium can be underestimated with other acids). Testing results are presented in Table 1. The solid density of the binder was also measured using an AccuPyc 1330 apparatus, resulting in a measured value 3.17 g/cm³. According to cement chemistry and density, the Mexican cement used is similar to ASTM Type 1 cement (Neville, 1981).

Water in contact with tailings was also tested three times for chemical content (see average in Table 1). The water samples were analysed using ICP-AES. The sulphate content was determined by assuming that total sulphur measured was present as $SO_4^{2^-}$ (stoichiometric conversion). Potable water from the town of Rouyn-Noranda (Quebec, Canada) was used as mixing water for paste fill samples.

	Tailings %wt/wt	Cement %wt/wt	Tails Water ppm		Tailings
Al	3.45	2.31	< 0.01	Mineralogical Composition	%
As	0.00	0.00	< 0.06	Quartz	67.65
Ba	0.06	0.02	0.04	Chlorite	2.11
Be	0.00	0.00	< 0.001	Muscovite	5.59
Bi	0.00	0.00	< 0.02	Hematite	1.31
Ca	0.38	48.50	490.00	Microcline	16.50
Cd	0.00	0.00	0.02	Albite	6.86
Co	0.00	0.00	0.08		
Cr	0.03	0.00	0.02	Specific surface (BET [*])	3.52 m²/g
Cu	0.01	0.00	8.83		
Fe	2.21	2.40	0.06	Specific gravity	2.67
Mg	0.31	1.33	37.60		
Mn	0.08	0.04	1.09	Particle sizing	μm
Мо	0.00	0.00	0.11	D10	1.76
Ni	0.01	0.00	0.16	D20	5.06
Pb	0.01	0.00	< 0.02	D30	10.38
S	0.16	1.09	1250.00	D40	16.28
Sb	0.00	0.00	< 0.09	D50	22.73
Se	0.00	0.00	0.11	D60	30.37
Si	-	-	12.25	D70	40.08
Sn	0.01	0.00	-	D80	54.00
Ti	0.14	0.20	< 0.002	D90	79.43
Zn	0.02	0.00	0.13	Cu, D60/D10	17.30
$S_{sulfate}$	0.11	-	3744.50	Cc, D30 ² /D60×D10	2.01

 Table 1
 Chemical and geotechnical properties of paste fill materials

* Brunauer, Emett and Teller method.

The evaluation of the paste fill recipe was performed in two phases. A first, exploratory phase, conducted over 180 days of curing time, was performed to test the Mexican cement in usual slump conditions. The parameters varied during this first phase are presented in the Table 2. One can see in Table 2 that the solid content was approximately 70±1%wt for paste fill mixtures. The initial project considered a value of 78%wt solids; however, this value was lowered due to a slump less than 15 mm at this solid content (Figure 1). In phase one, a total of 45 paste cylinders 15 cm long and 7.5 cm in diameter were cured at room temperature and at a relative humidity greater than 90% in a curing chamber. Three cylindrical samples of each mixture were used to evaluate the uniaxial compressive strength (UCS) after 3, 7, 28, 90 and 180 curing days using a MTS 10/GL press with a normal loading capacity of 50 kN and a displacement rate of 0.001 mm/min. The UCS corresponds to the maximum stress value (peak failure) reached during the compression test. The average of the three UCS values is used in graphic presentation in this paper. After the UCS test, the internal part of one cylinder was sampled and dried for calculation of geotechnical parameters.

Table 2Phase one paste fill mixtures

Batch Number	1	2	3	4	5
Binder content (%wt)	3	5	7	3	7
Batch solid content (%wt)	70.8	70.6	70.8	69.6	69.2
Water/cement (ratio)	14.2	8.7	6.3	15.0	6.8
Slump-ASTM C143 (mm)	135	135	127	172	169



Figure 1 Pictures of cemented paste at 78%wt, mini-slump test (left) and tails aspect (right)

Because the UCS values were low at 28 days, it was decided during the course of phase one to start a new testing phase and then test different recipes to get more strength (Table 3). Accordingly, the following three parameters were modified in the paste recipes:

- Using a different binder; the selected binder was a mix of 20% Mexican cement and 80% ground granulated blast furnace slag from Lafarge Canada.
- Removing the finest tailings particles (assumed to be clay); this was accomplished by washing tailings in a 25 µm sieve. Retained tailings were dried thereafter.
- Increasing the solid content of paste mixtures but using water reducing admixtures to maintain the slump. Two products were tested: BASF Glenium 7102 and BASF Glenium 7700.

Batch Number	6	7	8	9	10	11	12
Characteristics (see below)	1*	1	2	2	3*	3	4*
Binder content (%wt)	7	5	7	5	7	5	7
Batch solid content (%wt)	72.2	70.9	79.0	78.6	71.3	71.2	73.8
Slump (mm) (mini-cone)	60	69	9	9	67	69	60
Slump (mm) (estimated normal cone)	99	127	29	31	117	120	72
Batch Number	13	14	15	17	18	19	20
Characteristics (see below)	4	5*	5	6	6*	8	8
Binder content (%wt)	5	7	5	5	7	7	5
Batch solid content (%wt)	70.5	73.8	73.8	74.6	74.2	72.4	73.3
Slump (mm) (mini-cone)	71	34	33	32	37	38	37
Glenium added (ml)	-	15	13	12	9	-	-
Slump with Glenium (mm) (mini-cone)	-	68	70	67	70	-	-
Slump (mm) (estimated normal cone)	137	124	128	123	128	94	80

Table 3Phase two paste fill mixtures

Characteristics: 1 = Standard at 71%wt solid; similar slump as observed in phase 1 study; 2 = Standard at 78%wt solid; 3 = Paste mixed with Mexican cement (20%) and Canadian Lafarge slag (80%); 4 = Paste with deslimed tailings, particles $\leq 25 \mu m$ removed; 5 = Paste with BASF Glenium 7102; 6 = Paste with BASF Glenium 7700; 8 = Standard at 72.5%wt solid; * = Tested with CUAPS.

All batches were mixed with a Hobart mixer of 30 pints. Slumps were measured with a miniature (155 mm height) slump apparatus prior to pouring of the moulds. A total of 60 paste cylinders 10 cm long and 5 cm in diameter were cured at room temperature and at a relative humidity greater than 90% in a curing chamber. In addition to cylinders prepared with the modified recipes, three paste standards were prepared for comparison: 71%wt solids at 5 and 7%wt binder, 78%wt solids at 5 and 7%wt binder and, 72.5%wt solids at 5 and 7%wt binder. Due to the limited amount of tailings, the 155 mm slump apparatus was used. Regression between the mini slump and the normal slump was established in phase one to define the expected value in normal slump condition.

Initially, it was planned to use a solid content of 78%wt in mixtures with water reducing admixtures and reach a slump similar to the one obtained with the mix at 71%wt solids. However, after adding 33 ml of Glenium 7700, the slump became 29 mm and after adding 40 ml, the slump increased to more than 100 mm. Because this amount of admixture was huge and certainly not economical in normal operation, it was decided to change the initial solid content, prior to adding admixtures, to 72.5%wt and a new standard was mixed at this value. While not known at that moment, the mixing of these standards was to become highly important for the selection of the recipe. For each batch with water reducing admixture, the addition of Glenium in the mixture was stopped when the slump was similar to the one measured with samples at 71%wt solids. In addition to samples cured in a humidity controlled chamber, some samples were also cured under applied pressure system (CUAPS). This system (Yilmaz et al., 2008) allows assessing the strength performance of the samples under consolidated and drained conditions via the application of external pressure. The confining vertical pressure applied to the top of the samples was 300 kPa. This pressure was estimated according to what was anticipated near the bottom of a typical backfilled stope in the mine. In this study, five CUAPS tests were performed over 28 days and UCS tests were performed after dismantling of the system.

Rheologic tests were performed using the rheometer TA Instrument AR2000. For all rheologic tests performed in phase one and two of this study, the vane geometry was used and the shear rate was increased until the maximum capacity of the rheometer. In phase one, tailings and paste were tested for rheology behaviour at different water content. Tailings were tested at three solids contents (69, 71 and 73%wt) while paste mixtures were tested at two solid contents (69 and 71%wt) but with different cement content: 3%wt

and 7%wt binder for 69%wt solids and 3, 5 and 7%wt for 71%wt solids. Each sample prepared for rheological testing was manually mixed in small batches with exact proportions of tailings (from dry state), cement and water to reach the needed solids content. The rheological behaviour of each sample was tested at least twice; results are presented in Section 4.

3 UCS results

Considering a security factor of 1.5, the evaluated UCS required for the free standing (e.g. Mitchell et al., 1982) in the condition of the mine was estimated to be 470 kPa at 28 days. Figure 2 shows UCS results for phases one and two. For all samples tested in phase one, the UCS increased continuously over the testing period, and no evidence of internal cracking was observed. The strength values measured can be ordered first according to the amount of binder and second to the amount of water. However, none of the mixture tested reached the targeted strength in the required period of time. In phase two, changes made to the tailings and to the recipes induced variations on the measured strength. Figure 3 shows the ratios between the UCS measurements for batches where changes occurred and UCS measurements considered as standards, i.e. samples mixed at 71%wt solids (Batches 2 and 3). For samples tested in phase two, the following observations were made:

- *Batches 6 and 7 (repetition of Batches 2 and 3 in first testing phase):* Due to the increase in solids content for the sample with 7% wt of cement, the strength is increased by 20 and 30% at 7 days and 28 days, respectively, in comparison to the standard at 7% wt cement. The strength results obtained for the sample mixed with 5% wt of cement are closer than the standard, with an increase of 7% of the strength. This difference can be the result of slight changes in the solid content, but are probably more due to the change in the size of the samples from phase one to phase two. The CUAPS result for Batch 6 shows a strength increase of 77% relative to the standard (batch 3) at 28 days of curing.
- *Batches 8 and 9:* The strength increase is between 270 and 340% relative to the standards at 71%wt solids.
- *Batches 10 and 11:* The strength increase is between 301 and 483% relative to the standards at 71% wt solids. The CUAPS result for Batch 10 shows a strength increase of 693% comparatively to the standard (Batch 3) at 28 days of curing.
- *Batches 12 and 13:* Relative to the standards, these samples show a better strength at 7 days but this increase is not maintained at 28 day. This is particularly true for the sample containing 5%wt binder that is showing a strength decrease of -12% relative to the standard at 71%wt solids. The CUAPS result for batch 12 shows a strength increase of 74% relative to the standard (Batch 3) at 28 days of curing; this is mainly the effect of the solid content.
- *Batches 19 and 20:* The strength increase for these samples is between 34 and 84% relative to the standards at 71%wt solids.
- *Batches 14 and 15:* The strength increase for these samples is between 157 and 187% relative to the standards at 71%wt solids. In comparison to Batches 19 and 20, both samples show strength increase at 7 days (+26%) but strength decrease at 28 days (-10%). The CUAPS result for Batch 14 shows strength increases of 69% relative to the standard at 71%wt solids (Batch 3) and 13% relative to Batch 19.
- *Batches 17 and 18:* The strength increase for these samples is between 134 and 184% relative to the standards at 71%wt solids. In comparison to Batches 19 and 20, both samples show strength decrease at 7 days (-16%) but strength increase at 28 days (+33%). CUAPS result for Batch 18 shows strength increases of 128% relative to the standard at 71%wt solids (Batch 3) and 52% relative to Batch 19.

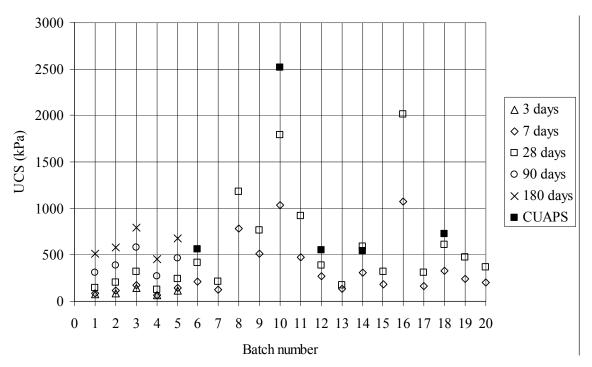


Figure 2 UCS results for phases one and two

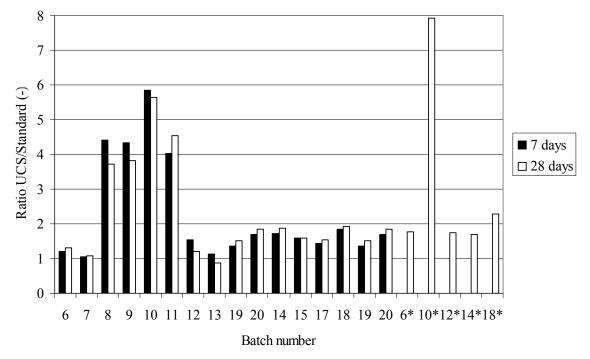


Figure 3 Ratio between UCS results and standards at 71%wt (* = CUAPS results)

4 Rheological results

Figure 4 shows rheological results obtained with the AR2000 instrument. Results are for low shear rates only because the rheometer was ineffective at higher rate; results are considered for the evaluation of the yield stress and for the viscosity at low shear rate.

Shear stress versus shear rate curves follow a Bingham plastic behaviour, which is typical for filtered tailings. The relationship for Bingham plastic fluid is given by Equation (1):

$$\tau_{\rm w} - \tau_0 = \eta \gamma \tag{1}$$

where τ_w is the shear stress at the wall, τ_0 is the yield stress, η is the non-Newtonian viscosity and γ is the shear rate. In this rheologic model, a force leading to a shear stress equal to yield stress has to be applied before the paste begins to flow; this is the shear stress at zero shear rate. In this model, the plastic viscosity remains constant with the shear rate; the viscosity is equal to the slope of the curve between the shear stress and the shear rate. In Figure 4, straight line regression relationships were calculated in the low shear rate region and parameters τ_0 and η estimated accordingly (Table 4). For the samples tested, the following observations were made:

- *Samples without binder:* from 69 to 73%wt solids, the paste becomes rapidly stiff while it was not possible to obtain more than 5 rheometer points at 73%wt curves at 69 and 71%wt are linear with the shear rate increase and Bingham behaviour is observed in the shear rate region tested. The yield stress increase is about 300 Pa from 69 to 73%wt solids, while the viscosity at 73%wt is twice the value at 69%wt.
- Samples with Mexican cement: the two samples tested at 7%wt cement are the ones at 69 and 71%wt solids. For both samples, a decrease in the yield stress is observed while the viscosity remains similar. The addition of 7%wt binder induces a yield stress decrease of 55 Pa at 69%wt solids and 38 Pa at 71%wt solids. The sample tested with 3%wt of binder at a solid content of 69%wt shows an increase of its yield stress, comparatively to the sample with no binder or with 7%wt of binder, while the viscosity is similar for all samples. Contrary to the sample at 69%wt solids, the shear rate-shear stress curve for sample with 3%wt of binder and at 71%wt solids is located between the sample with no binder and the sample with 7%wt binder.
- *Sample with slag:* the only one sample tested with blast furnace slag shows a yield stress decrease of 63 Pa relative to the sample mixed with the Mexican cement but a viscosity increase of 38 Pa·s.
- *Sample with deslimed tailings:* it was not possible to perform a test with this sample because particles and water segregated too rapidly.
- Samples with water reducing admixtures: the two samples tested with Glenium were mixed with 7%wt cement at 71%wt solids. Comparatively to the mixture at 73%wt solids and with no binder, the mixture with Glenium 7102 shows a yield stress decrease of about 45 Pa while the mixture with the Glenium 7700 shows a yield stress increase of about 39 Pa. The viscosity was also influenced by the admixture, in fact increases of 6 Pa·s and 13 Pa·s were observed by the addition of the Glenium 7102 and the Glenium 7700 respectively.

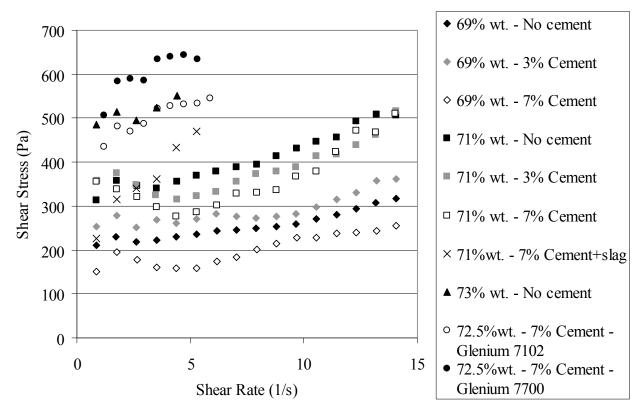


Figure 4 Shear stress versus shear rate curves for mixtures tested

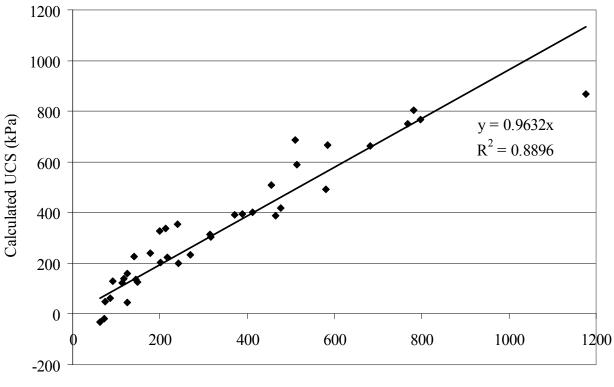
Table 4Rheo	logical characteristics o	'paste m	nixtures tested
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Batch	Characteristic	Yield Stress (Pa)	Low Shear Rate Viscosity (Pa·s)
-	69%wt solids, no cement	199.8	7.3
4	69%wt solids, 3%wt cement	236.8	7.1
5	69%wt solids, 7%wt cement	144.4	7.5
-	71%wt solids, no cement	299.5	14.2
1	71%wt solids, 3%wt cement	300.9	10.9
6	71%wt solids, 7%wt cement	261.7	13.4
10	71%wt solids, 7%wt cement+slag	198.7	51.7
-	73%wt solids, no cement	471.6	15.8
18	72.5%wt solids, 7%wt cement, Glenium 7700	510.3	28.3
14	72.5%wt solids, 7%wt cement, Glenium 7102	427.1	21.7

5 Discussion and conclusion

Figure 5 shows the calculated UCS versus the measured USC using the linear regression relationship (Equation (2)). This equation considers only the UCS results obtained for samples mixed with Mexican cement and water.

$$UCS = 3.04 \times Days + 44.31 \times \%Cement + 68.60 \times \%Solids - 4,946.48$$
 (2)



Measured UCS (kPa)

Figure 5 Calculated UCS versus measured UCS

This linear regression is indicative, as expected, that UCS is proportional to the cement quantity and to the percentage of solids in the mixture. Using Equation (2), it is possible to estimate that, with a binder proportion of 7%wt and a solid content of 73%wt, the targeted UCS is going to be obtained in laboratory within the 28 curing days wished. The use of the CUAPS method allowed the simulation of the strength development in a stope. For a paste mixture having 71%wt solids and 7%wt Mexican cement, this technique confirmed a UCS value over the required strength for free standing backfill. The paste fill recipe tested is a conservative recipe (laboratory result) that will allow the paste backfilling operation to commission the paste plant. However, the slump measured at 73%wt solids is low and special attention will be required for the distribution of the paste. A discussion of this aspect is beyond the scope of this paper.

The use of slag should be a great improvement for the optimisation of the paste backfill recipe. The use of slag is reported to usually slow down the hydration process of binders (Lumley et al., 1996); however, this was not observed in this study. UCS results with a ratio slag:cement of 80:20 show a strength increase of about four times comparatively to the standard with Mexican cement. A full understanding of the slag behaviour in paste fill is not available, however it is known that slag has a greater impact on pore refinement in cement pastes than other binders (Luo et al., 2003; Niu et al., 2002). In turn, this effect has a positive influence on the strength of the material (Ouellet, 2006). The degree of reactivity of the slag particles is proportional to the specific surface, and depends on the water availability (Escalante et al., 2001; Lumley et al., 1996). A particular aspect of slag is its interaction with water: more water is generally needed for a mixture containing both ordinary Portland cement and slag to achieve the same viscosity (Malhotra, 2001). Unfortunately, steel slag is not available in Mexico and transport costs from USA are prohibitive. However, efforts are ongoing to test different slags from different Mexican metallurgical industries.

The literature about using water reducing admixtures in paste fill is not extensive; the paper by Klein and Simon (2006) was the only one found. As reported in the literature, the behaviour of water reducing admixtures in paste fill is also observed in this study, i.e. the early strength of the sample is increased. However, although the slump was increased by the addition of the admixture, the rheological behaviour was not expected. For approximately the same slump as the one measured at 71%wt solids, the yield stresses and the viscosities at low shear rate measured for a specimen at 72.5%wt solids and containing water reducer

were significantly higher. For this reason as well as for economical reasons, the utilisation of water reducing admixture was not retained as a potential solution to increase strength.

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