The effect of microstructural evolution on the physical properties of paste Backfill

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ABSTRACT: One tailings sample from hard rock mine was used for this study. The paste backfill mixture was made with 5 wt% binder (which is made of a ratio of 80:20 blast furnace slag to ordinary Portland cement). The paste backfill cylinders were then cured in a fogroom for periods of 0, 1, 2, 5, 7, 14 and 28 days. The performed tests were SEM investigations, permeability, suction and MIP tests. The results demonstrate that paste permeability decreases rapidly from 0 to 7 days of curing time before stabilizing between 7 to 28 days. The suction increases gradually from 0 to 7 days of curing time before reaching a plateau after 14 days. The paste backfill free mercury porosity decreases gradually and becomes low from 28 days of curing time. SEM observations showed that permeability, suction and porosity results are related to increasing precipitation of hydrate minerals in the paste backfill.

1 INTRODUCTION

The use of paste backfill is currently practiced in many modern mines throughout the world, particularly in Canada and the United States because of increasingly stringent environmental regulations (Weaver & Luka 1970, Barsotti 1978, Viles & Davis 1989, Petrolito et al. 1998). Paste backfill is a hardened composite material made from a mixture of mine tailings, hydraulic binders (such as ordinary Portland cement, fly ash, blast furnace slag or a combination of these) and water. The two main benefits of paste backfill are lower operating costs and a reduction in the amounts of waste material sent to the tailings facility for disposal. This reduction of the amount of waste sent to the tailings facility decreases the environmental impact and decreases future capital expenditures related to the tailings facility (Barsotti 1978, Lundravil & Tenbergen 1995, Hassani & Archibald 1998). Because the role of hydraulic binders is to generate mechanical strength, one of the challenges is the use of cemented paste backfill as secondary pillars, to ensure the stability of the underground excavations (Thomas et al. 1979, Mitchell & Wong 1982, Mitchell 1989, Stone 1993, Amaratunga & Hein 1997, Ouellet et al. 1998, Petrolito et al. 1998).

A number of studies which have been published in the last 10 years have allowed a better understanding of the properties of paste backfill (Ouellet et al. 1998, Benzaazoua et al. 1999a & b, Bernier et al. 1999). These previous works have indicated the influence of many of the parameters on paste backfill which affect short-term and long-term strength acquisition. Some of these parameters are tailings mineralogy and particle size distribution, moisture content, binder type and proportion, the degree of saturation and finally, chemical weathering (e.g. Benzaazoua et al. 1999a & b, Benzaazoua & Belem 1999, Hassani & Hossein 2000).

The physical behaviour of a paste backfill is complex because of the continuous evolution of the paste from its mixture in the backfill plant to storage and hardening in the stope. One can consider that paste fill is similar to a soft clay at the early stage; after 28 days curing time, similar to a compacted silt and, after 91 days of curing time it can be harder than a consolidated soil and
approaches the properties of a soft rock. This change corresponds to the evolution of the backfill microstructure because of binder hydration which will alter the water flow characteristics (hydraulic conductivity and percolation rate) and mechanical strength of the paste.

The purpose of this paper is to relate the microstructure evolution of the paste backfill to its physical characteristics. To reach this goal, different tests have been performed on paste backfill samples. The backfill sample studied is a mixture containing 95% tailings and 5% binder. The tests performed are permeability tests, suction tests and mercury intrusion porosimetry after curing periods of 0, 1, 2, 5, 7, 14 and 28 days. The backfill microstructure has also been studied with a scanning electron microscope (SEM).

2 EXPERIMENTAL PROCEDURES

2.1 Samples preparation

One mill tailings from an underground hard rock mine which contains approximately 16% sulphur was sampled for the cemented paste backfill preparation. This corresponds to approximately 30% sulfides essentially pyrite and a small amount of pyrrhotite; the other significant minerals are silicates and aluminosilicates. For this study, the same backfill mixture as the one used at the mine was chosen. The hydraulic binder used consists of 80% blast furnace slag (BFS) and 20% ordinary Portland cement (OPC). The final mixture contains 95% tailings and 5% binder. The process water from the mine was used as the mixing water for sample preparation. The final mixture contains approximately 78% solids (water-to-solids ratio of 0.28) and the water-to-cement ratio (w/c) was 8.

The resulting mixtures were poured into plastic cylinders (10 cm diameter and 20 cm long) which gives a height to diameter ratio of 2. The cylinders were then sealed and cured in a fogroom maintained at approximately 70% humidity (similar to underground mine conditions) for periods of 1, 2, 5, 7, 14 and 28 days. After curing, resulting specimens were used for permeability, suction, mercury intrusion porosimetry and uniaxial compression tests.

2.2 Permeability tests

Permeability is the velocity of fluid flow through a porous medium. The permeability of the cemented paste backfill samples were evaluated with rigid-wall variable head permeameter tests. For this study, the paste backfill was directly cast in the permeameter cells and the device was assembled and conditioned in a controlled moisture room. The permeameters have an internal diameter of 8 cm, and the sample height is limited to 15 cm. The walls are made of translucent Plexiglas (cf. Aubertin et al. 1996). The permeability tests were performed under falling head conditions at different curing times, following the ASTM method. By applying the Darcy’s law, the coefficient of permeability or hydraulic conductivity, \( k \) (cm/s), is defined by the equation:

\[
k = 2.3 \frac{a}{A} \frac{L}{t} \log \frac{h_1}{h_2}
\]

where \( a \) (cm\(^2\)) is the cross-sectional area of the tube; \( A \) (cm\(^2\)) is the cross-sectional area of cemented paste backfill sample normal to direction of flow; \( h_1 \) (cm) and \( h_2 \) (cm) are the static water heads, and \( L \) (cm) is the sample height.

2.3 Suction tests

When a soil is unsaturated, the water is subjected to a negative pressure relative to atmospheric pressure. This negative pressure, called suction, is caused by the surface tension which exists between air and water in contact with the soil matrix (matrix suction) as well as the affinity between water and solids (osmotic suction). The soil-water characteristic curves, also called water retention curves, are mainly used to represent the behavior of unsaturated soil medium (e.g. Bear 1972, Kovács 1981, Fredlund and Rahardjo 1993). These water retention curves represent the relation between the volumetric water content (\( \theta \)) and suction (\( \psi \)). The main characteristic of the water retention curve is \( \psi_a \), the pressure for air entry, also called the air entry value (AEV),
which corresponds to the beginning of desaturation. In this study, the evolution of the \( \psi_a \) of the studied cemented paste backfill with respect to curing time, was investigated.

The ASTM D 3152 standard defines the test procedure used to obtain the water retention curves. The paste backfill sample was placed on a porous ceramic plate (which retains water until a pressure of 5 bars) in a pressurized cell commonly called a Tempe Cell. Then, the sample is subjected to a positive air pressure and the outflow water pressure is measured. By increasing the pressure and measuring the volume of evacuated water with each pressure change, one can establish the water retention curve, which is representative of the volumetric water content (or degree of saturation) against the suction. In this study, the \( \psi_a \) value was evaluated using the method proposed by Aubertin et al. (1998) which corresponds to the value of \( \psi_a \) at 90% degree of saturation.

2.4 Mercury Intrusion Porosimetry (MIP)

The theory of a mercury porosimeter is based on the principle that a non-reactive and non-wetting liquid will not penetrate pores until sufficient pressure is applied to force the entrance of the liquid into the pore spaces. The relationship between the applied pressure and the pore size into which the mercury will intrude is given by the Laplace's law or the Washburn equation:

\[
D = \frac{-4\gamma \cos \theta}{P} \quad \text{or} \quad r_p = \frac{-2\gamma \cos \theta}{P}
\]

where \( D \) or \( r_p \) is the opening pore diameter or radius, \( P \) is the applied pressure, \( \gamma \) is the surface tension of mercury (480 dyne/cm) and \( \theta \) is the contact angle between mercury and the pore wall (usually near 140°).

The pore diameter was calculated from applied pressure assuming cylindrical pores, to give both cumulative and incremental mercury intrusion porosity versus pore diameter or radius data for mesopores (0.002 \( \mu \)m < \( D < 0.05 \mu \)m) and macropores (\( D > 0.05 \mu \)m). The mercury intrusion porosity (\( MP \)) was evaluated using Micromeretics® AutoPore III 9420 mercury porosimeter. The MIP data allows one to determine total porosity (\( MP_{tot} \)), free access porosity (\( MP_{free} \)) and trapped porosity (\( MP_{trap} \)) of the cemented paste backfill. The calculation of the two former types of porosity requires two cycles of mercury intrusion. The backfill samples which have undergone the first cycle of mercury intrusion (\( MP_{tot} \)) are stored in a desiccator for 24 hours to drain the trapped mercury before undergoing the second cycle of mercury intrusion (\( MP_{free} \)). The difference between \( MP_{tot} \) and \( MP_{free} \) corresponds to \( MP_{trap} \). When a pore family observed after the first mercury intrusion was not observed after the second intrusion, it is because the intruded mercury was entirely trapped by that pore family.

2.5 Scanning electron microscope observations

The scanning electron microscope (SEM), a Hitachi®3500-N, was used to see the microstructure and the texture of the studied backfill samples. The SEM investigation was performed with several different magnifications. The back-scatter electron (BSE) imaging mode was chosen because it produces images with the chemical contrast superimposed on the relief contrast. The operating conditions are the following ones: 20 kV of voltage, a DC from 150 to 200 \( \mu \)A, a lowered pressure of 25 Pa to avoid the harmful electrical charge effects on image quality and a working distance of 15 \( \mu \)m. The electronic images give information on (i) the particle size distribution and grain morphology of the composite, (ii) the texture of the cement phase, (iii) the morphology of the hydrated cement phases and, (iv) the porosity of the composite.
3 RESULTS

3.1 Evolution of cemented paste backfill hydraulic conductivity

Figure 1 shows the evolution of hydraulic conductivity, \( k \), of the studied paste backfill with curing time. The overall trend of this curve is a rapid decrease of the hydraulic conductivity of the paste backfill with curing time. This evolution suggests that the hydraulic conductivity of the paste backfill decreases rapidly during the first 7 days due to the formation of the cement hydrates which gradually fill in the initial porosity. After 7 days, any change in hydraulic conductivity (near \( 10^{-8} \text{ cm/s} \)) is insignificant.

![Figure 1. Evolution of the paste backfill hydraulic conductivity with curing time](image1)

Figure 2. Evolution of the paste backfill water retention capacity through the air entry value

![Figure 2. Evolution of the paste backfill water retention capacity through the air entry value](image2)
3.2 Suction test results

The main results of the suction tests performed on the paste backfill samples at different curing times are presented on Figure 2. It should be noted that the suction test was not performed for the curing time of 28 days because the applied pressure exceeded the maximum capacity of the porous ceramic plate. This figure represents the evolution of the air entry value $\psi_a$ with curing time. Just after molding the fresh paste backfill, $\psi_a$ is at approximately 6.5 m of water pressure. After less than two days of curing time, the air entry value $\psi_a$ reaches values of between 20 and 30 m of water pressure. Beyond this date, $\psi_a$ continues to increase, but less rapidly with increasing curing time. After 14 days of curing time, $\psi_a$ reaches a value of approximately 50 m of water pressure which seems to be the maximum value obtainable for suction for this paste backfill sample.

3.3 MIP results

Figure 3 shows the MIP curves of the studied cemented paste backfill after 28 days of curing time. It should be recalled that the first MIP curve allows one to obtain the $MP_{\text{tot}}$ value, while the second MIP curve allows one to obtain the $MP_{\text{free}}$ value (Fig. 3a). The difference between the two porosity values corresponds to the trapped mercury porosity, $MP_{\text{trap}}$. The incremental porosity curves (Fig. 3b) allow one to look at the MIP results in terms of modal distribution of the pore families compared to their median diameter ($D_m$). On Figure 3b, one can notice that there are two pore families within the paste backfill. Among these two pore families, the second pore family is the more significant and the more representative of this paste backfill sample (peak of the histogram). The first pore family consists largely of macropores having median diameter ranging from 123 and 125 $\mu$m while the second family consists of mesopores having median diameter ranging from 0.2 and 1.2 $\mu$m.

![Figure 3. Typical MIP curves of the cemented paste backfill after 28 days curing time](image)

Figure 4 allows one to compare the pore distribution network of the paste backfill with that of the tailings used for its preparation. The difference between the curves of these two types of material accounts for the difference between their textures and microstructure. From this, one notices the direct influence of binder hydration on the evolution of the spatial geometry of the pore network which is much finer in the paste backfill sample than in the tailings sample. From Figure 4, the $MP_{\text{tot}}$ value is 42% for the tailings and 41% for the paste backfill; the $MP_{\text{free}}$ value is 40% for the tailings and 8% for the paste backfill and, the $MP_{\text{trap}}$ value is 2% for the tailings and 33% for the paste backfill. One can notice that the cemented paste backfill free porosity is much lower than the porosity of the tailings.
3.4 SEM investigations

The SEM observations of the studied paste backfill samples showed an even grain dispersion pattern, filled intergranular spaces, a dense matrix (Fig. 5), an absence of cracks, the presence of macropores and the presence of secondary precipitation of AlMgSiFeCaSO₄ phases. The voids between the tailings coarse particles are filled with ultra-fine particles and a cemented matrix (Fig. 5b). The paste texture has a direct effect on the hydraulic properties of the cemented paste backfill. Figure 5a shows the paste backfill matrix to be well cemented.

Figure 5. SEM images showing (a) paste backfill texture (b) details of paste backfill matrix

The SEM observations also showed the significant role of secondary precipitation in the reduction of free porosity during the course of curing time (Fig. 6). Secondary precipitation representing all the phases occurring after the hardening of backfill due to the formation of the hydrated primary phases (Fig. 6a). This contributes to the cohesion of the backfill when it fills intergranular spaces, as well as mesopores and macropores (Fig. 6b). However, these precipitates become harmful when they are expansive (example of sulfatic attack due to formation of the ettringite and the expanding secondary gypsum). For the studied samples, these secondary precipitates were especially beneficial in the acquisition of mechanical strength by thickening the matrix of the backfill and precipitating in macropores.

Figure 4. MIP curves of the tailings likened to the MIP curves of the cemented paste backfill
4 DISCUSSION

The results of the permeability tests, suction tests and SEM observations agree on the evolution of the paste backfill microstructure during the curing time. This microstructure evolution results in a reduction of the paste backfill water filtration capacity and an increase in its water retention capacity. One can note that as the microstructure evolves, the hydraulic conductivity decreases and the water retention characteristics increase. The high water retention capacity of the backfill will increase its resistance to sulphate attack or chemical weathering (Lawrence 1992) as well as reduce its self-heating potential (exothermic effect due to the extreme oxidation of sulphurous minerals present in the backfill). Moreover, a high degree of saturation decreases the ability of the backfill to acquire strong mechanical strengths (very slow hydration) and the backfill can become more susceptible to liquefaction induced by blasting or mobile equipment vibrations.

It should be noted that even if the total porosity of the paste backfill is very close to that of the tailings, these two materials are clearly distinguished by their fluid trapping capacity. A detailed and thorough study on the use of MIP results (Remy 1993) indicated that free porosity is directly related to permeability. The MIP tests carried out after 28 days curing time clearly show that the paste backfill had a low free porosity and a strong capacity for fluid trapping which is different from the properties of the tailings. This means that the cemented paste backfill becomes less permeable after 28 days of curing time. Indeed, with a $MP_{\text{trap}}$ value of 33% the cemented paste backfill can be considered as trapping the all of intruded mercury. This phenomenon is possibly caused by the irregular pore geometry in the pore network due to the formation of hydrates in the binder. SEM observations confirm that this geometry is irregular and could indeed support the fluid trapping phenomenon. The SEM imagery also demonstrates that after 28 days curing time the paste backfill matrix becomes very dense and less permeable. This agrees with the permeability test results shown in Figure 1.

Figure 1 shows that in terms of permeability, the paste backfill microstructure evolves very rapidly in the first week and remains stable afterwards. A general regression equation describing the evolution of the studied paste backfill permeability according to the curing time $k(t)$, has been found; the exponential equation is

$$ k(t) = k_0 \cdot \exp(-\alpha \cdot t) $$

(3)

where, $k_0$ (cm/s) is the initial permeability of the paste backfill or the fresh paste backfill hydraulic conductivity immediately after its mixture (without any hydration activity), $\alpha$ is backfill constant and can be considered as a proportionality factor depending on the binder type, its proportion and the physical properties of the tailings, $t$ is curing time. From the regression, $k_0 = 9.4097e-6$ cm/s and $\alpha = 0.2741$ with a coefficient of correlation $R = 0.96$. Assuming that the hydraulic properties of fresh paste backfill and tailings are similar, the following equation proposed by Aubertin et al. (1996) can be used to evaluate the initial permeability:
where, $c_0$ is tailings parameter, $\gamma_w$ is the unit weight of water (9.79 kN/m$^3$ at room temperature), $\mu$ is the viscosity of water ($\approx 9.8 \times 10^{-6}$ N.s/cm$^2$ at 20°C), $D_{10}$ (µm) is particle diameter at which 10% of the tailings passes, $C_u$ is the coefficient of uniformity, $e$ is the voids ratio and $m$ is tailings parameter. In their experiments on homogenized tailings, Aubertin et al. (1996) found that the average value of $m$ is equal to 2.16. By combining equations 3 and 4 the following general equation can be proposed for predicting the cemented paste backfill hydraulic conductivity with respect to the curing time, $k(t)$, by the following equation:

$$k(t) = c(t) \cdot \frac{\gamma_w}{\mu} \cdot D_{10}^2 \cdot C_u^{2/3} \cdot e^{3+m} \left(\frac{1}{1+e}\right) \cdot \exp(-\alpha \cdot t)$$

(5)

where $c(t)$ is backfill hydraulic constant depending on binder type, its proportion, tailings properties and curing time. From experimental results of the studied paste backfill, $c(t)$ is given by the following empirical equation:

$$c(t) = c_0 \cdot \left(0.9537 \cdot \exp(-0.2727 \cdot t)\right) \quad \text{with} \quad R = 0.97$$

(6)

where $c_0$ is tailings or fresh paste backfill hydraulic constant.

The suction test results (Fig. 2) showed that the microstructure of the studied paste backfill increased its water retention capacity during the curing period. This increase is progressive and seems to reach a plateau after 14 days of curing time. This evolution is directly related to the mode of binder hydration and its velocity. A regression equation describing the evolution of the studied paste backfill air entry value with respect to curing time, $\psi_a(t)$, has been found

$$\psi_a(t) = \psi_{max} \cdot \left(a_1 - \exp(-a_2 \cdot t)\right) \quad \text{and} \quad \psi_{max} = a_0 \cdot \psi_0$$

(7)

where $\psi_{max}$ is the maximum air entry value that the backfill can have after 28 days curing time, $a_0$ is a proportionality constant, $\psi_0$ is fresh paste backfill or tailings air entry value, $a_1$ is a constant depending on binder type and its proportion and water-to-cement ratio (w/c), $a_2$ is a constant depending on tailings physical properties, $t$ is curing time. From the regression, $\psi_0 = 6.2$ m of water pressure, $a_0 = 7.6$, $\psi_{max} = 47.1$ m of water pressure, $a_1 = 1.1$ and $a_2 = 0.235$ with a coefficient of correlation $R = 0.99$.

5 CONCLUSION AND PROSPECTS

A Canadian underground hard rock mine tailings was sampled in order to produce a cemented paste backfill with the same composition as used at this mine. The resulting mixture contains 95% tailings and 5% binder which consists of 80% blast furnace slag and 20% ordinary Portland cement. Several types of tests were performed on the paste backfill samples to observe either directly or indirectly the evolution of the texture and the microstructure of this material during the course of curing. These tests were MIP, suction and permeability tests and finally, the SEM observations.

The results of this exploratory study led to the following conclusions: (i) the paste backfill permeability decreases very rapidly in the very short term (i.e. between 0 and 7 days of curing time) before stabilizing after 7 days of curing time, (ii) the paste backfill suction increases gradually before reaching a possible plateau after 14 days of curing time, (iii) the mercury free porosity of this paste backfill is very low after 28 days curing time and, (iv) the SEM observations allow us to better understand that the decrease in the paste backfill permeability and porosity is directly related to the phenomena of minerals precipitation. From the obtained experimental results, regression models were found to predict the hydraulic conductivity, $k$, and the suction, $\psi_a$, of the studied paste backfill.

A better understanding of the evolution of pastefill properties in practice is important for different reasons: (i) the strength of pastefill in the filled stope is related to the water flow which is
a function of the paste fill properties, (ii) the self-heating potential of a sulphidic paste will depend on the water content of the paste fill in the stope which is again a function of the material properties and, (iii) the use of surface paste fill as waste management technique for sulphidic tailings will be interesting if the material has a high water retention characteristics and a low hydraulic conductivity. In short, the results of this study allowed us to better understand the evolution of the matrix of the paste backfill from the fresh paste to its hardening and opens up several interesting prospects for the continuation of this work. The next step will be to increase the variety of the binder types used to refine the proposed regression models.

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