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# Sulphate effect on the early age strength and self-desiccation of cemented paste backfill

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#### HIGHLIGHTS

• Sulphate can significantly decrease the strength gain rate of tailings backfill at early ages.

• Sulphate reduces the self-desiccation of Portland cement - cemented backfill.

• Sulphate content of tailings backfill is important for the barricades stability.

• Sulphate content of tailings backfill is important for mining cycle.

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#### ABSTRACT

This paper presents the results of an experimental study on the sulphate effect on the early age (1, 3, 7 and 28 days) strength and self-desiccation of cemented paste backfill (CPB). The CPB specimens have an initial sulphate concentration of 0, 5000, 15,000 and 25,000 ppm and are cured at room temperature (20 °C). Tests on the mechanical properties were conducted on the CPB, while microstructural analyses were performed on the CPB and cement paste samples. Suction and electrical conductivity monitoring were also performed on the CPB specimens with different sulphate contents. The results show that sulphate has a significant effect on the early age strength and self-desiccation of CPB. In the early ages, sulphate can have negative effects, i.e., leads to a decrease in the CPB strength and reduction in the intensity and rate of self-desiccation within the CPB. The magnitude of these effects depends on the initial sulphate concentration. The inhibition of cement hydration by sulphate ions is a key reason for the observed decrease in CPB strength and self-desiccation intensity or rate. Ettringite formation, changes in the pore structure and sulphate absorption by C-S-H are found as additional negative factors that affect CPB strength. This study has demonstrated that the effect of sulphate on the early strength and self-desiccation of CPB is an important factor for consideration in the designing of cost-effective, safe and durable CPB structures as well as for reducing the mining cycle time; in other words, increasing mining productivity.

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#### 1. Introduction

Cemented paste backfill (CPB) is a heterogeneous material made of tailings (often with a solid percentage between 70% and 85%), water (fresh and/or mine processed), and a hydraulic binder (often ordinary Portland cement) [1,2]. CPB technology is commonly and extensively used in underground mines for ground support and mine waste disposal. This emerging technology has both economic and environmental benefits [1–7].

http://dx.doi.org/10.1016/j.conbuildmat.2015.12.124 0950-0618/© 2015 Elsevier Ltd. All rights reserved. One of the most important properties of CPB is its early age mechanical strength, which controls its mechanical stability. The mechanical strength or stability of CPB is usually evaluated by the uniaxial compressive strength (UCS). UCS testing is relatively inexpensive, fast and can be easily applied in the quality control of mine backfilling [1]. The mechanical strength of CPB largely depends on the quantity of hydraulic binder used in its preparation. Generally, a small percentage (usually between 3% and 7%) of binder can provide sufficient strength that would ensure the stability of the backfill during mining operations [2]. During pillar recovery, the backfill face is exposed, and to prevent deterioration, adequate mechanical stability is required to ensure safe underground working conditions. Some previous studies [8–11] have noted that the required strength value for a free-standing wall of







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paste backfill in open stoping operations is often up to 1 MPa. However, the value can largely vary based on the function and application of the backfill. To increase mine productivity, which is associated with financial benefits, it is important to reduce the mining cycle; in other words, CPB with high early strength is required to provide sufficient early mechanical stability to CPB structures and thus increase mining productivity [1,12]. In addition, CPB with high early strength can also significantly reduce or eliminate the risk of liquefaction [1].

A large number of research work [8–18] has been carried out to investigate the factors that affect the strength of CPB. Chemical factors are one of the most important aspects. Previous studies have indicated that one common chemical factor that can affect the strength of CPB is its initial sulphate content [1,2,14,18]. The sulphate ions in CPB systems can originate from various sources [1.2] such as: (i) the oxidation of sulphide minerals in the tailings: (ii) the use of sulfur dioxide/air method for the elimination of cvanides in gold mining; (iii) the addition of gypsum (CaSO<sub>4</sub>  $\cdot$  2H<sub>2</sub>O) or anhydrite (CaSO<sub>4</sub>) to the clinker to control the setting of the cement; and (iv) the use of mine processing waters as mixing water for the preparation of CPB. Sulphate can reduce the strength of CPB in the longer term through sulphate attacks. For example, depending on the quantity of the initial sulphate content and curing temperature, sulphate can lead to positive or negative impacts on CPB strength [1,12,18]. However, the main focus of prior studies that examined the effect of sulphate on CPB was on the long-term UCS (age  $\ge$  28 days). The impact of sulphate on the early age strength of CPB and microstructure has been largely ignored, and thus, is not well understood. There is therefore a need to address this issue for safety and financial reasons as discussed above.

Besides the aforementioned early age strength, the early development of capillary pressure or suction in the CPB is also especially important for achieving early mechanical stability of the CPB [19] as well as for the opening of the barricades. Thus, this will reduce the mining cycle and increase production, i.e., increase the profitability of the mine [1]. Indeed, the critical challenge is to estimate the loads exerted onto the barricades and manage their opening time. These two issues are strongly influenced by the pore water pressure that develops behind the barricade, and suction development due to cement hydration. The latter results in the dissipation of excess pore pressure and increase in effective stress (i.e., strength gain) concurrent with transitioning from the paste phase to the hardening stage [20]. An important cause of suction development within CPB systems is self-desiccation [19,20]. Self-desiccation, a term borrowed from the concrete literature, is a phenomenon resultant of cement hydration. Cement hydration leads to a net reduction of the total volume of water and solids, thereby decreasing the pore water pressure or moisture content or leading to suction development inside cementitious materials [20–22]. Previous works [19,20] have already addressed self-desiccation in CPB systems and thus contributed to a better understanding of this issue. However, there are no studies on the effect of sulphate on the self-desiccation of CPB. Yet, there is a need to acquire sufficient knowledge on the influence of sulphate on the self-desiccation in CPB for the reasons mentioned above.

Therefore, the main objective of this paper is to experimentally study and present the results of the sulphate effect on the strength development and self-desiccation of CPB at the early ages.

#### 2. Experimental program

#### 2.1. Materials

#### 2.1.1. Tailings

Artificial tailings (silica tailings (ST)) were used in all of the CPB specimens to eliminate the uncertainties related to natural tailings. Natural tailings may contain various minerals that are problematic as well as chemical components (e.g., pyrite)

which can interact with cement hydration and thus influence the cement hydration process and products, and then affect the results obtained and their interpretation. ST are essentially made of quartz, which is one of the primary minerals found in Canadian hard rock mine tailings. The ST also have a similar particle size distribution as the average of nine types of tailings from Canadian mines (Fig. 1) [2]. With about 43 wt.% of fine particles (diameter <  $20 \,\mu$ m), the ST can be considered as medium tailings. The ST have a relative density of 2.7 and their primary chemical properties are given in Table 1.

#### 2.1.2. Binder

Portland cement type | (PCI) was used in all of the samples. Portland cement is commercially available and widely used as a binder in CPB practice. Table 2 shows the chemical properties of Portland cement as well as its relative density.

#### 2.1.3. Water

Distilled water (DW) was used as basic water in the experiments. Ferrous sulphate is the most common sulphate type found in cemented backfills. Specific amounts of sulphate salt (FeSO<sub>4</sub>  $\cdot$  7H<sub>2</sub>O) with a molecular weight of 278.01 was added to a specific volume of DW to create a mixing water with a well-known sulphate concentration (0, 5000, 15,000 and 25,000 ppm).

#### 2.2. Sample preparation and mix proportioning

The required amounts of tailings, binder and water with different sulphate concentrations (0, 5000, 15,000 and 25,000 ppm) were mixed by using a food mixer for about 7 min until a homogeneous paste was obtained. All of the CPB specimens had a constant binder content of 4.5 wt.%, and water-cement (w/c) ratio of 7.6. Then, the prepared CPB was poured into a plastic cylinder that was 5 cm in diameter and 10 cm in height. After removing the entrapped air from the moulded samples by manual vibration, the prepared samples were wrapped with plastic film to avoid the evaporation of water and then cured at room temperature for periods of 1, 3, 7 and 28 days. For the monitoring, larger plastic cylinders that were 10 cm in diameter and 20 cm in height were used.

Furthermore, samples of cemented paste (CP) for microstructural analysis were also prepared by following the procedure described above. All of the CP specimens had a constant w/c ratio of 2 (to simulate the high water content of CPB). After the required curing times, the CPB and the CP samples were subjected to various tests.

#### 2.3. Testing of specimens

#### 2.3.1. Testing of mechanical properties

In accordance with ASTMC39, UCS tests were performed on the CPB specimens with different sulphate contents after they were cured for different lengths of times. The press used has a normal loading capacity of 50 kN. The load was implemented at a relatively slow rate (1 mm/min). Each test was repeated at least twice and the average was regarded as the strength of the tested sample [1].

#### 2.3.2. Hydraulic conductivity test

Hydraulic conductivity can provide relevant information about the pore structure, such as coarseness and connectivity, and the cracking of the CPB [24]. The fluid transportability or permeation properties of CPB are significantly dependent on its pore structure. Previous studies [1,2,25] conclude that pore structure changes can be caused by sulphate attacks. Therefore, saturated hydraulic conductivity tests were conducted on the CPB samples with different sulphate concentrations to assess the effect of sulphate on the pore structure of CPB at the early ages. The



Fig. 1. Grain size distribution curve of the tailings material used.

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