# Evolution of the hydraulic and mechanical properties of gelfill



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# ABSTRACT

Hydraulic (H), and mechanical (M) properties are important performance criteria of the gelfill (GF). The understanding of these properties, their interactions and their evolution with time and/or cement hydration degree is still limited due to the fact that GF is a new cemented backfill material. Therefore, a laboratory investigation is conducted to study the hydraulic and mechanical properties of GF and their evolution with time and binder hydration degree. Over 100 GF samples, 5 cm in diameter and 10 cm in height were prepared with various binder proportions (2.0 %, 4.5 %, and 6.0 % PCI), w/c ratios (18.5, 8.2, and 6.2) respectively, and 0.4% (by weight of solids) sodium silicate type N. Samples were cured in room temperature for 3, 7, 28, 90, and 120 days. Uniaxial compressive strength (UCS) and saturated hydraulic conductivity tests were conducted. Valuable results were gained regarding the evolution of the hydraulic and mechanical properties. The study showed that hydraulic and mechanical properties are time-dependent or affected by the binder hydration degree. Mechanical properties are affected by binder content, binder types, hydration degree and curing time while the hydraulic conductivity of the GF decreases as the binder content increases and the binder hydration degree increases

# RÉSUMÉ

Les propriétés hydrauliques et mécaniques représentent d'importants critères de performance des remblais-gels (RG). La connaissance de ces propriétés, leurs interactions et leur évolution dans le temps et/ou en fonction du degré d'hydratation du ciment est toujours insuffisante; ceci est principalement dû au fait que le RG est un nouveau matériau de remblai cimenté. C'est pourquoi, une étude de laboratoire a été conduite afin d'investiguer les propriétés hydrauliques et mécaniques du RG et leur évolution dans le temps et/ou en fonction du degré d'hydratation du ciment. Plus de 100 échantillons de RG, de 5 cm en diamètre et 10 cm en hauteur, ont été préparés avec diverses proportions de ciment (2,0 %, 4,5 %, et 6,0 % CP I), rapports e/c (18.5, 8.2, et 6.2) respectivement, et avec 0,4% (en masse de solides) de silicate de sodium de type N. Les échantillons ont été soumis à différents temps de cure (3, 7, 28, 90, et 120 jours) dans des conditions de température ambiante. Des essais de résistance uniaxiale compressive et de conductivité hydraulique ont été effectués sur les échantillons. D'importants résultats ont été obtenus en ce qui concerne l'évolution des propriétés hydraulique est mécaniques sont affectées par la teneur en ciment, le type de ciment, le degré d'hydratation et le temps de cure alors que la conductivité hydraulique décroît quand la teneur en ciment et le degré d'hydratation augmentent.

# 1 INTRODUCTION

GF is a new cemented tailings backfill material, and a promising backfill technology. GF is typically made of tailings, water, binder and chemical additives (Fillset, sodium silicate gel). The chemical additives (are like a gel, therefore the name, GF) have the ability to absorb (Hassani et al., 2007; Doucet et al., 2007) a high amount of the water (cemented tailings are always prepared with excessive water to allow easy transport to underground voids). This water absorption results in a substantial increase of GF strength (Hassani et al., 2007: Doucet et al., 2007). Recycling the waste (tailings) into GF has economical and environmental advantages for the mining industry. Filling the stopes with GF results in higher ore extraction and ensuring a safe workplace for the mine worker. Furthermore, this technology reduces the quantity of mine waste on the earth surface.

Mechanical and hydraulic properties are key design parameters for GF structures. In order to safely play its role in underground mine support, GF should have adequate mechanical properties. These properties must allow the GF to withstand certain static and dynamic loads; they also have to prevent the GF structures from collapsing during adjacent mining operations. Failures of mine backfills can cause injuries, and human and financial lost. UCS is often used to evaluate the mechanical stability of the GF. This is because the UCS test is relatively quick and inexpensive. In addition to the use of the GF as underground mining support, they are used as tailings storage. In terms of waste disposal, the mechanical properties of GF should prevent it from deterioration, such as cracking (Yilmaz et al., 2003). Mechanical damage (cracking) can significantly increase hydraulic conductivity (Fall et al., 2009). Cracks can create paths for the infiltration of fluids, such as water and oxvgen, which weaken the environmental performance and durability of GF structures. The availability of oxygen and its rate of diffusion through the pore spaces of the GF are critical factors that control the rate of acid generation (Nicholson et al., 1989).

Other important parameters that affect the environmental performance and durability of the GF are transportability of fluid (e.g. permeability) and its microstructure (e.g. void ratio (e), porosity, and tortuosity. The vulnerability of the GF to AMD and capability of transport toxic elements to the mine areas and ground water (when the mine is flooded after closure) are pertinent environmental design criteria of GF structures. Exposure of the GF to AMD is dependent on the reactivity



of tailings contained in the GF. The reactivity in turn is reliant on the types and quantity of sulphide minerals present in the GF as well as hydraulic properties of the GF (seepage of water and diffusion of oxygen through the GF matrix). These properties can be evaluated by investigating the hydraulic conductivity of the GF.

The objective of this paper is to investigate the evolution of the hydraulic and mechanical properties of the GF by conducting experimental tests.

#### 2 EXPERIMENTAL PROGRAMS

#### 2.1 Materials

The materials used to prepare the GF include binder, tailings, sodium silicate, and water.

#### 2.1.1 Binder and Mixing Water

Portland cement type I (PCI) was used as the binder. It is the most common material used by the mining industry to produce cemented backfill. Tap water was used to mix the binders and tailings.

#### 2.1.2 Tailings

Silica (artificial) tailings (ground silica, TS) are used in this study. The advantage of selecting TS as the tailings material is to accurately control to the mineralogical and chemical compositions of tailings. Furthermore, natural tailings contain various amounts of sulphate and other undesirable minerals which can bring high levels of uncertainty to the obtained results and thus significantly affect the outcomes of the study. TS is made of quartz (one of the dominant minerals in Canadian hard rock mines) and shows a grain size distribution close to the average of nine Canadian mine tailings (Figure 1). It can be observed that TS has about 45 wt. % fine particles (<  $20 \mu m$ ) and can be classified as medium tailings.



Figure 1 Grain size distribution of the tailings used and the average grain size distribution of tailings from nine Canadian mines (Abdul-Hussain, 2011)

#### 2.1.3 Sodium Silicate

Sodium silicate type N was added to the mix in the liquid form. It is inorganic chemical made by combining various ratios of sand and soda ash (sodium carbonate) at high temperatures. It is described as clear, colourless, and viscous liquid.

#### 2.2 Preparation of Materials and Mix Proportions

Over 100 GF samples were prepared to test the hydraulic and mechanical properties. The degree of binder hydration was also estimated. The tailings, binder, water, and gel were mixed using a B20F food mixer. The mixing time was 10 minutes for all mixes. The tailings, binder, and water were mixed first, followed by the addition of sodium silicate gel. Preliminary UCS tests were conducted to choose the right dose of gel. Mixes with 0.1, 0.2, 0.3, and 0.4% wt sodium silicate were prepared. The best results were obtained when the sodium silicate is 0.4% wt (Figure 2). Hence, this percentage is selected for this investigation. Various binder proportions were used to prepare the mixes, including 2%, 4.5%, and 6% PCI with a solid mass concentration of 73% which correspond to w/c ratios of 18.5, 8.2, and 6.2, respectively. The slump of the prepared GF was 25 cm. The produced GF were poured into cylindrical plastic moulds that were 5 cm in diameter and 10 cm in height. The prepared samples were then sealed and cured in a laboratory environment at 20 °C ± 2 °C for periods of 3, 7, 28, 90, and 120 days.



Figure 2 Various sodium silicate proportions (0.1%, 0.2%, 0.3%, and 0.4%), 4.5% PCI, and 73% solids mass concentration

#### 2.3 Testing and Analysis Methods

#### 2.3.1 Mechanical Tests

Uniaxial compression tests (UCS) were performed on GF specimens cured at 3, 7, 28, 90, and 120 days. The specimens were tested according to the ASTM C 39 using a computer-controlled mechanical press. The press has a normal loading capacity of 50 kN. All samples were tested at a constant deformation rate of 1 mm/min. All test data were collected using a computerized data logging system. The results were saved using the computer software

LabView. For each age, at least two cylinders were tested to ensure the repeatability of the results obtained.

#### 2.3.2 Hydraulic Conductivity Tests

Saturated hydraulic conductivity tests were conducted on the GF samples. The specimens were tested according to ASTM D5084-00 using a TRI-FLEX II system. A flexible wall technique in constant head mode was employed. The samples were saturated in water for 24 hours prior to the testing. Each hydraulic conductivity test was repeated at least twice to ensure that the results always fall within an accurate limit.

#### 2.3.4 Estimation of Binder Hydration Degree

The engineering properties of cemented materials, such as strength and durability, are influenced by the microstructure of the hardened paste. The hardening of cementitious material is gradually gained through hydration of the binder, thus, the degree of binder hydration should be determined. The degree of hydration  $(\alpha)$  is defined as the ratio between the quantity of hydrated cementitious material and the original quantity of cementitious material (Schindler and Folliard, 2005). Many experimental techniques are used to measure the degree of hydration of PC. For example, measuring the heat of hydration, the non-evaporable water content, and the amount of calcium hydroxide produced in the hydration reactions. All the aforementioned methods are based on comparisons of the measured parameters with the predicted or measured parameters for a fully hydrated paste. In recent years, SEM quantitative techniques are used to estimate the degree of hydration in plain Portland cement paste. In this study, Equation 1 is used to estimate  $\alpha$ . However, this method is affected by the curing temperature, i.e. the relationship between the compressive strength and  $\alpha$  is influenced by curing temperatures. Nonetheless, this limitation is not applicable here because same curing temperatures are used for all mixes.

$$\alpha_n = \frac{UCS_n}{UCS_{final}}$$
[1]

# $\alpha_{n:} \alpha$ at n curing time

 $UCS_n$ : uniaxial compression strength at n curing time  $UCS_{final}$ : uniaxial compression strength of mature GF (in this study, it is considered that the strength of GF does not increase after 120 days)

#### 3 RESULTS and DISCUSSION

#### 3.1 Evolution of the Degree of Binder Hydration

The degree of hydration is calculated from the results of UCS tests on GF samples with different binder contents and curing times up to 120 days. The rate of hydration is almost the same for 6% and 4.5% PCI (rate for 6% is

slightly higher), while slower for 2% PCI, especially at early ages up to 7 days (Figure 3). Increasing the cement content in GF mixes speeds up the hydration reaction. More cement available in the mix means that more of the surface area is exposed to react with water. Furthermore, the hydration rate is a function of the composition of cementitious materials, the amount of cementitious materials, and water cementitious materials ratio (Schindler and Folliard, 2005). In general, higher cement content implies a higher hydration rate (e.g. Kalinski and Hippley, 2005).



Figure 3 Degree of hydration of various binder proportions

#### 3.2 Evolution of the Mechanical Properties

Figure 4 shows the UCS evolution with curing time (Figure 4a) and  $\alpha$  (Figure 4b) of the GF for different binder contents (6%, 4.5%, and 2%). It can be seen that the development of UCS values is related to the curing time, a, and binder content. A proportional relationship between the UCS values and curing time or  $\alpha$  is observed. When the hydration reaction advanced, more hydration products formed to fill the pores of the GF matrix (which enhance the UCS of the GF). This filling of the pores results in decreasing the overall porosity (Figure 5) of the hardening cement matrix and thereby, increasing the strength. Furthermore, it is noticed that increasing the binder content will increase the UCS values of the GF. Increasing the binder content leads to increases in the rate of hydration (Figure 3). A higher cement hydration rate means higher UCS values. Indeed, faster cement hydration results in precipitation of larger amounts of cement hydration products, such as C-S-H and CH. C-S-H is the major attributor of strength of cementitious material (e.g., Taylor, 1990). In addition, an increase in binder content results in the precipitation of a larger amount of binder hydration products.

The evolution of porosity with curing time for various binder contents confirms the aforementioned arguments (Figure 5). For different binder contents, the differences in the porosity are noticeable at early ages. It can be seen that as the binder content increases, the porosity decreases. Furthermore, as the hydration process proceeds, the porosity of GF samples decreases (more hydration products lead to refining of the pore structures of cementitious materials).

Figure 6 shows a comparison between two mixes of different binders. The first mix contains only PCI while the other mix contains PCI and slag, 50:50. Both mixes have the same solids concentrations (73%) and same percentage of sodium silicate (0.4%). It can be observed that the mix with slag gives significantly higher UCS results. It is shown that the UCS value for the PCI-slag (slag-GF) mix at the ages of 28 and 56 days is more than two times the UCS value for the PCI mix. This can be explained as the result of the combined effects of the following mechanisms: (i) the filler effect of the slag grains (a behaviour that PCI grains are not able to achieve), which in addition to the binder hydration products, contribute to filling the pores of the slag-GF matrix, thereby refining its pore structure (Manmohan and Mehta, 1981; Fall and Samb, 2008); (ii) besides the C-S-H from the hydration of PCI, the CH produced by the hydration of PC activates the slag reactions, and thus produces additional C-S-H, which in turn, enhances the strength of the slag-GFs; (iii) the sodium silicate acts as an alkaline activator of the slag, which in turn, results in the stimulation of the hydration of slag and thus contributes to the strength development of slag-GFs. The PCI-slag mix results obtained in this study are in good agreement with the results obtained by Doucet et al. (2007). In summary, the results presented above suggest that the partial replacement of PCI by slag can significantly increase the UCS of the GF. This could result in significant saving of binder consumption, i.e. reduction of the production costs of the GF.

Furthermore, the GF with PCI-slag (w/c = 8.2) has the same UCS values as the cemented paste backfill (slag-CPB) that was prepared by Celestin (2009) with a w/c ratio = 7.6 and the same tailings and binder proportion (4.5%) and type (PCI/slag in a ratio of 50/50). These observed similar UCS values between the GF and CPB, despite the much higher w/c ratio of the GF, is attributed to the influence of the additive used in the GF (sodium silicate on the binder hydration) the strength development of GF and the evolution of the w/c within the GF. Indeed, it is generally agreed that soluble sodium silicate doubly contributes to strength development, both by acting as an alkaline activator and giving rise to the formation of a C-S-H of low C/S ratio (Fernandes- Jiménez et al., 2009). This activation results in an acceleration of the binder hydration. Combined with the formation of additional C-S-H gel due to the reaction of SiO<sub>2</sub> (formed in the hydrolysis of sodium silicate solution) with CH (generated by the hydration of the PC), this activation contributes to the increase of the GF strength. This scenario is not possible with CPB due to the absence of sodium silicate within this material. Furthermore, Doucet et al. (2007) and Hassani et al. (2007) reported that sodium silicate has the ability to absorb a significant quantity of water, thereby resulting in a decrease of the w/c ratio of the GF. It is well known that a lower w/c ratio is associated with higher strength for a given cementitious material cured in the same conditions (e.g., Sideris and Konsta-Gdoutos, 1996).





Figure 4 UCS evolution of GF for various binder contents with (a) curing time, (b) binder hydration degree ( $\alpha$ )



Figure 5 Evolution of porosity with curing time for various binder contents



Figure 6 UCS evolution of GF for various mixes (PCI, PCI/Slag) with 4.5% binder content

#### 3.3 Evolution of the Hydraulic Properties

The effects of the binder proportion (% PCI), curing time, and degree of binder hydration on the saturated hydraulic conductivity (ksat) of the GF were examined. The results of the saturated hydraulic conductivity tests are illustrated in Figure 7. It can be seen that  $k_{sat}$  decreases when curing time (Figure 7a) and degree of binder hydration (Figure 7b) increases for the three mixes. It is obvious that the effect of curing time or degree of hydration on saturated hydraulic conductivity is significant. This behaviour is due to the increase in the degree of cement hydration with time (Figure 3). As a result, the formation of hydration products (e.g. C-S-H, CH) increases which in turn, leads to refinement of the pore structure and reduction in the porosity of the GF materials (Figure 5). This reduction occurs by filling the pores of the GF matrix and blocking the interconnected pores with hydration products.

On the other hand, it can be observed that the binder content significantly influences the saturated hydraulic conductivity of the GF materials especially at early ages. As the binder content increases, the saturated hydraulic conductivity decreases. The reason is that an increase in binder content leads to more precipitation of the hydration products (e.g. C-S-H, CH) in the GF matrix, thus causing a refinement in the pore structure and reduction in the porosity of the GF. This is confirmed by the results of mercury intrusion porosity (MIP) tests performed on two GF samples with 7 wt% and 9 wt% binder contents, respectively, and presented in Figure 8.





Figure 7 The saturated hydraulic conductivity evolution of GF for various binder contents with (a) curing time, (b) degree of binder hydration ( $\alpha$ )



Figure 8 Effect of binder content on the pore size distribution of GF paste backfill cemented with PCI/slag vs. content of fines and pore size range (data from Hassani et al., 2007)

#### 4 Conclusion

In this paper, experimental studies have been conducted on the GF samples to investigate the evolution of their hydraulic and mechanical properties. Samples are tested to obtain their saturated hydraulic conductivity and UCS. The degree of binder hydration has been estimated. Different binder contents are adopted (2%, 4.5%, and 6%) which correspond to w/c ratios of 18.5, 8.2, and 6.2, respectively. The used amount of sodium silicate is 0.4 wt %, and the samples were cured in room temperature for 3, 7, 28, 90, and 120 days.

Based on the results, it can be concluded that hydraulic and mechanical properties are strongly timedependent or affected by the degree of binder hydration. Mechanical properties are affected by binder content, binder types, degree of hydration and curing time. The evolution of UCS values is a function of curing time, degree of hydration and binder content. As the binder contents increase, the UCS values of GF samples increase with curing time or with increases in the degree of binder hydration. Increasing the cement content in GF mixes speeds up the hydration reaction. More available cement in the mix means more exposed surface area to react with water. In general, higher cement content means higher rate of hydration, and thus higher UCS. Also, using PCI-slag as a binder in the GF mix gives higher strength than a mix that contains only PCI. This is due to the fact that aside from the filler effect of slag, sodium silicate contributes to the activation of the slag and formation of additional C-S-H gel. On the other hand, the UCS values of slag-GF with w/c ratio = 8.2 are the same as slag-CPB with w/c ratio = 7.6. This is due to the ability of sodium silicate to form gel, quicken the binder hydration and reduce the w/c ratio by absorbing part of the excess water.

The saturated hydraulic conductivity of GF is influenced by binder contents especially at early ages. The hydraulic conductivity of GF decreases as the binder content increases and the degree of binder hydration increases. In addition, a direct relationship is found between the UCS and saturated hydraulic conductivity. As the UCS increases, the saturated hydraulic conductivity decreases.

This study provides essential information for understanding the evolution of the hydraulic and mechanical properties of GF. The findings of this paper will help mining operators and engineers who work with GF materials to take into consideration, the design, environmental aspects, and durability assessment of GF structures. It is also useful to predict the potential formation of AMD in GF structures.

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