THE EFFECT OF CURING TEMPERATURE ON THE PHYSICAL PROPERTIES OF HYDRUALIC BACKFILL AND GELFILL

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ABSTRACT

This paper presents and compares the physical properties of Gelfill and cemented hydraulic fill (CHF) obtained by lab experiments. Gelfill has an alkali activator such as sodium silicate in its materials in addition to primary materials of cemented mine backfill which are deslimed tailings, water and binders. The CHF and Gelfill samples with various mixture designs were cast and cured for 28 days. Initially, the influence of sodium silicate concentration on the mechanical properties of samples was evaluated using the unconfined compressive strength (UCS) test. Consequently, the effect of curing temperature on CHF and Gelfill samples cured in various curing temperatures ranging from 5–50 °C was studied using the UCS test. Microstructure of selected samples was studied wirth the mercury intrusion porosimetry (MIP) technique.

This study concludes that: (i) The mechanical properties of CHF can be improved by the addition of appropriate amount of sodium silicate. (ii) The rate of strength acquisition in Gelfill samples is faster than CHF samples over 28 days of curing period. (iii) Curing temperature strongly influences the mechanical and microstructural properties of CHF and Gelfill samples. (iv) The microstructure analysis revealed that the addition of sodium silicate can modify the pore size distribution and total porosity of Gelfill which can contribute to the better mechanical properties of Gelfill. (v) The MIP tests suggested the microstructure of specimens were changed by adding sodium silicate in a way that the Gelfill samples had a higher percentage of fine pores than CHF samples.

KEYWORDS

Gelfill, Cemented hydraulic fill, Sodium silicate, mercury intrusion porosimetry (MIP), Mine backfill

INTRODUCTION

The increased depths of mines in the Canadian Shield and the high stress associated with these depths favour a method of mine backfill that can both meet strength requirements and help mine productivity. Mine backfill mainly consists of tailings, water and binder materials, and the last component being the most expensive. Gelfill is relatively a new mine backfill material whose binder consists of alkali activators such as sodium silicate and other cementitious materials such as blast furnace slag and normal Portland cement (Doucet & Tarr 2007; Razavi & Hassani 2007). Until very recently there have been only a few isolated publications, mostly out of McGill University, regarding mine Gelfill (Razavi & Hassani 2007; Kermani, Hassani et al., 2011a, 2011b). These papers have investigated some of the basic mechanical behaviour of Gelfill. However, the mechanical properties of Gelfill in various conditions have yet to be understood. Therefore, the main objective of this study was to investigate the influence of curing temperature and sodium silicate concentration on the mechanical performance of Gelfill and cemented hydraulic fill (CHF) through a series of laboratory experiments. Moreover, the microstructure of the Gelfill and CHF samples was studied.

MATERIALS

Tailings

Tailings are waste materials produced in ore processing plants. The materials consist primarily of finely ground host rock. The physical and chemical properties of tailings have a significant effect on the mechanical performance of mine backfill (Benzaazoua, Fall et al., 2004; Kesimal, Yilmaz et al., 2005). In this research, the tailings were delivered from one of Vale's mines in Sudbury Ontario. The particle size distribution of the tailings was determined by using the combination of sieve analysis and laser diffraction methods. The result is presented and compared with the average size of 11 mine tailings from the provinces of Quebec and Ontario (Ouellet, Bussière et al., 2007), reported in Table 1. The mineralogical content of the tailings consists of generally Quartz, Albite and slight quantity of Calcite, Muscovite, Pyrrhotite, Chalcopyrite, Anorthite, and Chlorite.

Table 1 – Physical properties of the tailings

	D10 (µm)	D50 (µm)	D60 (µm)	D90 (µm)	Cu	Cc	Specific gravity (Gs)
Tailings	4.1	82.10	52.40	116.50	28.4	2.39	2.85
Mean 11 mine tailings	2.2	20	29	102	13.2	1.24	Not Available

Binder

Binders, the most expensive part of mine backfill (up to approximately 75% of costs), are mainly used to increase the stability of fill materials (Hassani & Archibald, 1998). Normal Portland cement, fly ash and blast furnace slag have been mainly used for mine backfill. In this research, a combination of 90% blast furnace slag and 10% Type 10 Portland cement, both provided by Lafarge Canada, were used. This combination is mainly used in Vale's mines in Ontario, Canada. The densities of the slag and Portland cement used were 2.89 and 3.07 g/cm^3, respectively. The Blaine specific surface area of the slag and Portland cement was 5,998 and 3,710 cm^2/g, respectively. The chemical compositions of the blast furnace slag and Portland cement are shown in Table 2.

Table 2 – Chemical composition of the Portland cement and blast furnace slag provided by Lafarge

Chemical composition	Blast furnace slag (wt %)	Portland cement (wt %)
CaO	37.129	61.13
SiO_2	36.127	19.39
Al_2O_3	10.385	4.61
MgO	13.246	3.3
SO_3	3.362	2.27
Fe_2O_3	0.668	2.01
Na_2O	0.424	2.03
K_2O	0.489	0.71

Sodium silicate

In both civil engineering and the mining industry, sodium silicate has been used for different purposes, including as an alkali activator of slag and fly ash, a penetrating sealant, and a hydration accelerator (Razavi & Hassani, 2007). Various types of sodium silicate are manufactured from varied proportions of Na_2CO_3 and SiO_2 by smelting the silica with the sodium carbonate at temperatures around 1100–1200 °C. In this research, Type N® sodium silicate was used, provided by the PQ National Silicate Company.

Table **3** shows some properties of the sodium silicate.

Table 3 – The properties of sodium silicate (PQ National Silicate)

Sodium silicate properties	Standard	Maximum	Minimum
Na_2O (%)	8.90	9.10	8.70
SiO_2 (%)	28.66	29.00	28.20
Weight ratio (SiO_2/Na_2O)	3.22	3.27	3.15
Specific gravity @20 °C	1.394	1.401	1.388
Viscosity @ 20 °C centipoises	177	213	141
Solids (%)	37.56	38.10	36.90

Sample preparation and curing

To investigate the effect of sodium silicate concentration, 72 triplicate CHF and Gelfill specimens were prepared with 8 mixtures. The pulp density and binder dosage were maintained at 70 % and 5 wt %, respectively. CHF and Gelfill samples were made with water; mixtures prepared in small batches in a 5-L stainless steel bowl, and mixed with mixer with a stainless steel wire whip blade. Cylindrical, polyvinyl moulds 10 cm deep and 5 cm diameter were used to cast the mixtures. Specimens were then cured in a curing chamber at 90 ± 2% relative humidity and the temperature was adjusted as needed. Specimens were tested at 7, 14 and 28 days. Tables 4 and 5 show the mixture characteristics of CHF and Gelfill mixtures.

Table 4 – Binder mixtures characteristics of backfill samples

Mixture	Blast furnace slag (wt %)	Portland cement (wt %)	Sodium silicate (wt %)	Curing temperature (°C)
CHF	4.5	0.5	0.0	25
GF .1	4.5	0.5	0.1	25
GF .2	4.5	0.5	0.2	25
GF .3	4.5	0.5	0.3	25
GF .4	4.5	0.5	0.4	25
GF .5	4.5	0.5	0.5	25
GF .7	4.5	0.5	0.7	25
GF .9	4.5	0.5	0.9	25

To evaluate the effect of curing temperature on the mechanical properties of CHF and Gelfill, 90 triplicate samples of Gelfill and CHF were prepared and cured at 5 curing temperatures (5, 15, 25, 35 and 50 °C). The binder for CHF specimens was a mix of 90% blast furnace slag and 10% type 10 Portland cement. For the Gelfill specimens, 0.3 wt % sodium silicate was added to the same binder combination used to prepare the CHF specimens. For all specimens, the amount of binder was kept constant at 5 wt % , the mixing time was 5 minutes, and the pulp density was 70%. Table 5 presents the mixture designs. All the cast samples were then cured at various temperatures ranging from 5–50 °C for 7, 14 and 28 days. Finally, the uniaxial strength of samples was measured with unconfined compressive strength (UCS) tests.

Table 5 – Binder mixture characteristics of backfill samples for investigating effect of curing temperature

Mixture	Blast furnace slag (wt %)	Portland cement (wt %)	Sodium silicate (wt %)	Curing temperature (°C)	Pulp density (%)
CHF/ 5 C	4.5	0.5	0	5	70
CHF/ 15 C	4.5	0.5	0	15	70
CHF/ 25 C	4.5	0.5	0	25	70
CHF/ 35 C	4.5	0.5	0	35	70
CHF /50 C	4.5	0.5	0	50	70
GF/ 5 C	4.5	0.5	0.3	5	70
GF/ 15 C	4.5	0.5	0.3	15	70
GF/ 25 C	4.5	0.5	0.3	25	70
GF/ 35 C	4.5	0.5	0.3	35	70
GF/ 50 C	4.5	0.5	0.3	50	70

EXPERIMENTAL SETUP

Unconfined compressive strength (UCS) tests

By conducting UCS tests (ASTM D 2166-06), the mechanical strength of the cured specimens were measured. The test was conducted with a “Wykeham Farrance 100 kN” press equipped with a 50 kN load cell. A Linear Variable Differential Transformer sensor was used to measure the samples’ vertical deformation. Samples were taken out from the humidity room just prior to conducting the UCS test. A data acquisition board and a computer setup were used to record and display the data. On a given curing day for each mixture, 3 samples underwent UCS testing and the average value of the three results was recorded.

Mercury intrusion porosimetery (MIP)

Evaluating the microstructure of the cemented backfill is important in understanding the mechanical properties and durability of cemented mine backfill (Mitchell & Wong 1982; Belem, Benzaazoua et al., 2000; Fall, Benzaazoua et al., 2005). Mercury intrusion porosimetery (MIP) is a technique widely used to investigate the microstructure of cemented materials like backfill and concrete (Aligizaki, 2006). This technique can accurately determine pore size distribution and pore structure data (Ouellet, Bussiere et al., 2007). A total of 12 samples cured for 28 days were subjected to the MIP test.

RESULTS AND DISCUSSION

Effect of sodium silicate concentration on Gelfill strength

The results of UCS tests are shown in

Figure **1**. As expected, the UCS increased with increasing curing time (due to the hydration of normal Portland cement and blast furnace slag and the precipitation of hydrated sulphate phases such as Gypsum or Ettringite). The results show that for a given curing time, UCS increases by increasing the amount of sodium silicate up to 0.3% of the total dry weight (wt %). However, the UCS decreased with any further increase of sodium silicate past this 0.3 wt % point. Moreover, the UCS significantly decreases when the amount of sodium silicate surpasses 0.5 wt % and the specimens have no measurable strength within the first 14 days of curing, which could cause liquefaction during that time in mine placements. This could be due to the increase in the total porosity of samples and the amount of moisture trapped in the samples. Furthermore, Figure 1 also shows that the UCS for Gelfill with 0.3 wt % sodium silicate is considerably higher than those for CHF specimens.

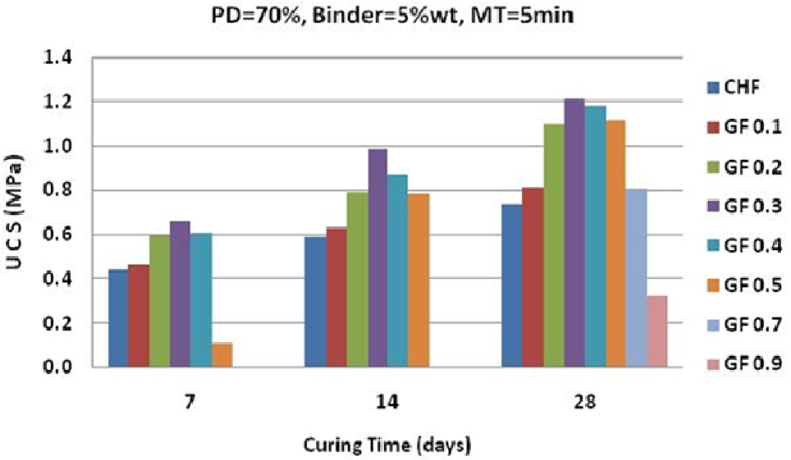

Figure 1 – Effect of sodium silicate dosage on compressive strength and UCS value evolution of CHF and Gelfill

To investigate microstructural properties and also to explain the results of UCS tests, MIP tests were performed on CHF and Gelfill samples containing 0.3 wt % sodium silicate cured for 28 days. The results are shown in

Figure **2**. The total porosity of the CHF sample (38.93%) is higher than the Gelfill sample (34.11%). Moreover, both samples have two pore size families that dominate the pore size distribution, The size of pores reported in the Gelfill samples (between 20 to 0.1 µm) were smaller than the size of pores in the CHF samples (100 to 1 µm). These two differences can explain the higher UCS values and better mechanical behaviour of the Gelfill over the CHF samples. In fact, for a given overall porosity of a sample, as pore size decreases, the distribution of an applied stress is more likely to be homogeneous and uniform (Li & Aubertin, 2003).

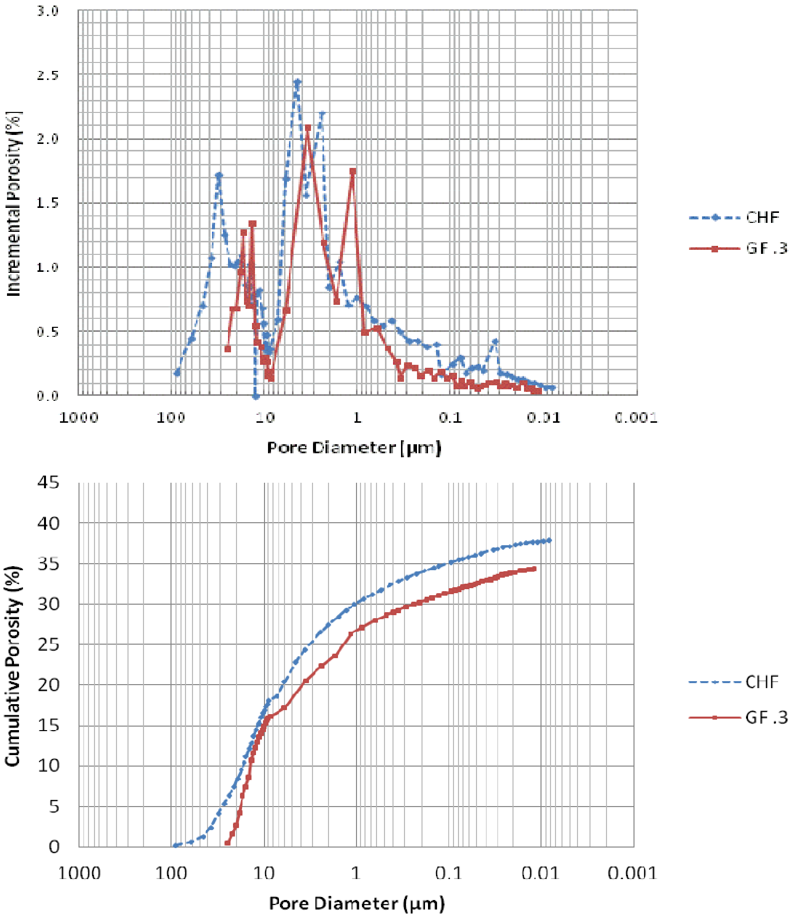

Figure 2 – Incremental pore size distribution (top) and overall porosity (bottom) of CHF and Gelfill containing 0.3 wt % sodium silicate after 28 days of curing at 25 °C

Effect of curing temperature on strength development of CHF and Gelfill

The influence of curing temperature on the hydration of various binders and concrete has been investigated by many researchers (KjeUsen, Detwiler et al., 1991; Mirza, Waleed et al., 1991; Saad, Abo-El-Eneini et al., 1996; Balendran & Martin-Buades, 2000; Wang, Shah et al., 2004; Husem & Gozutok, 2005). The results of these investigations indicated that the curing temperature plays a noticeable role in the determination of the mechanical characteristics of cemented materials. However, investigations about the influence of curing temperature on cemented backfill are very limited. For instance, Fall, Bussiere et al.

(2005) investigated the effect of curing temperature on the mechanical strength of paste backfill by conducting UCS tests on paste backfill specimens cured at various curing temperatures (0, 20, 35 and 50 °C). The results indicated that the strength development rate decreases with a reduction of curing temperature due to the diminishing of the hydration rate of binders. Therefore, the samples cured at lower curing temperatures showed lower UCS values. Moreover, it was found that the mode of strength development was different for different binder types (Fall, Bussiere et al., 2005).

Effect of curing temperature on CHF and Gelfill strength

To evaluate the effect of curing temperature on the strength of CHF and Gelfill, the results of UCS tests on CHF and Gelfill specimens are shown separately in

Figure 3 **and 4**

Figure 4, respectively. The Gelfill and CHF specimens that were cured at elevated temperatures (35 and 50 °C) rapidly developed strength for the first 14 days of curing. However, the strength of specimens cured at 35 and 50 °C remained relatively constant after 14 days of curing. This might be due to the rapid formation of a solid C-S-H gel and ettringite layer around the binder particles, which could prevent moisture from reaching the inner parts of the binder or/and the rapid precipitation of hydrated sulphate phases like gypsum. Therefore, the hydration would not be continuous. The results of an MIP test confirmed this hypothesis: the pore size distribution showed a halt at the range of a certain pore size (15 µm). After the first 14 days of curing, the strength of the CHF and Gelfill cured at 25 °C increased more rapidly than samples cured at 35 and 50 °C. Therefore, after 28 days, the strength of Gelfill samples cured at 25 °C is 35–90% higher than for the other samples. The UCS values of samples cured at 50 °C did not increase after 14 days, which could be explained by moisture being driven out and hence cessation of hydration of slag and cement in the CHF and Gelfill. Moreover, the very low UCS values of samples cured at 5 °C could be due to the low hydration rates of cement and slag at low temperature. This effect was clearly explained by Fall, Bussiere et al. (2005).

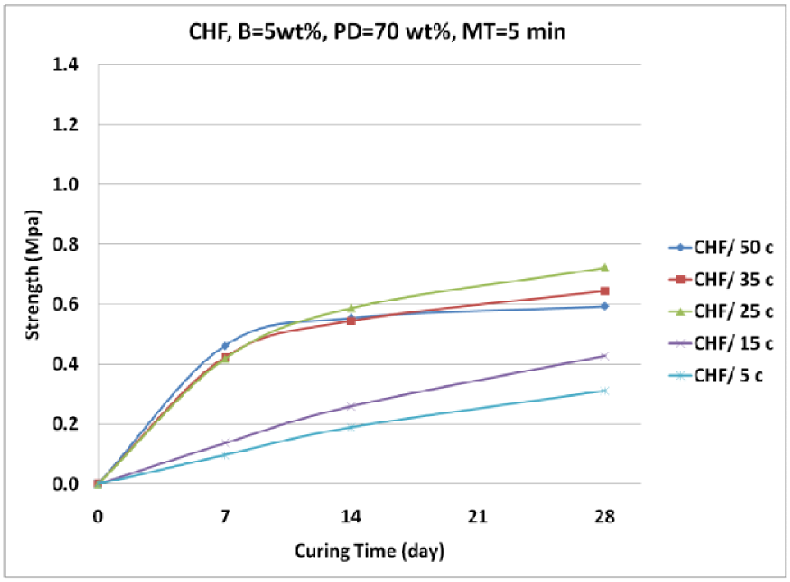

Figure 3 – Effect of curing temperature on the UCS evolution of CHF

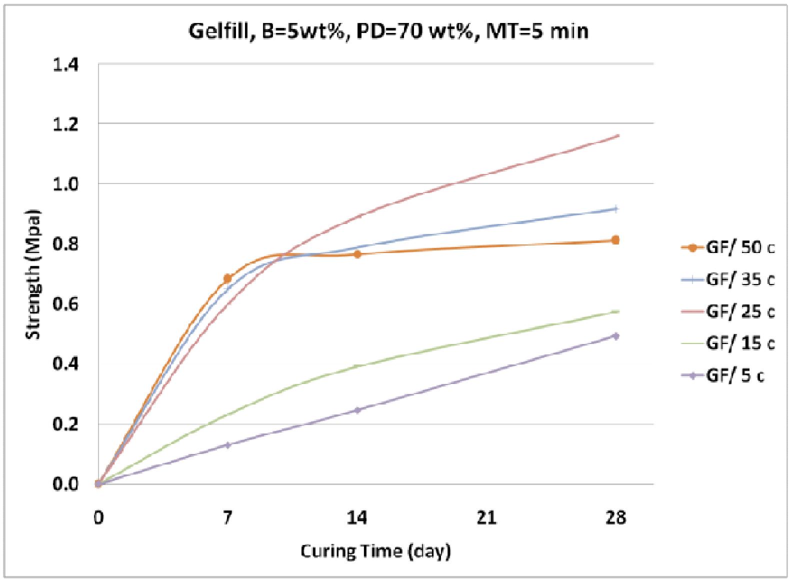

Figure 4 – Effect of curing temperature on the UCS evolution of Gelfill

To understand the behaviour of Gelfill and CHF specimens cured at various temperatures, the pore structures of three CHF and three Gelfill specimens were studied by conducting MIP tests, and the results are presented in

Figure **5** and 6. The figures show that the curing temperature can alter the pore size distribution and pore structure of the CHF and Gelfill specimens.

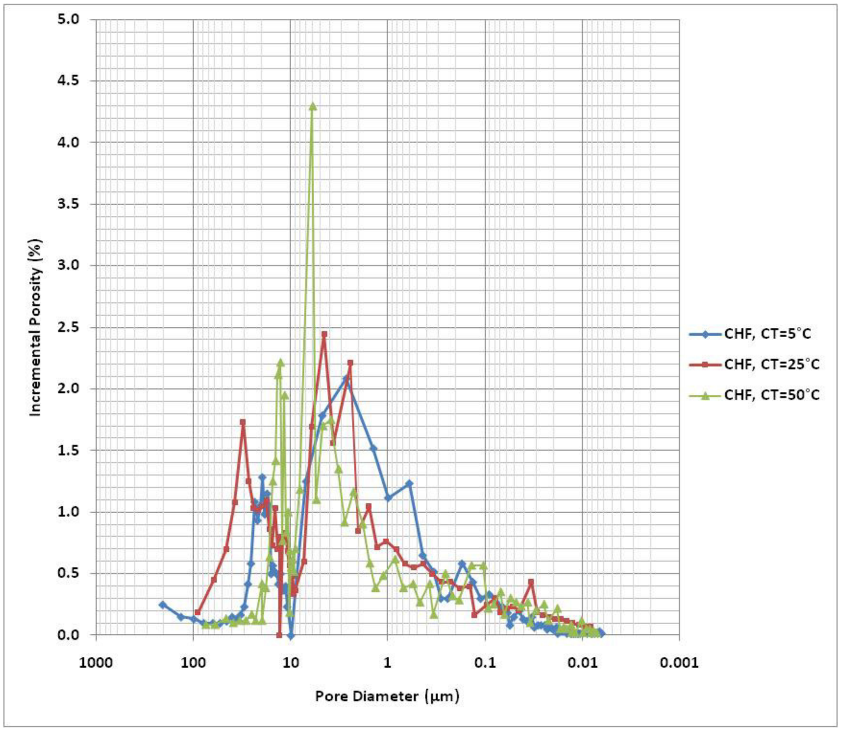

Figure 5 – Incremental pore size distribution of CHF specimens cured for 28 days

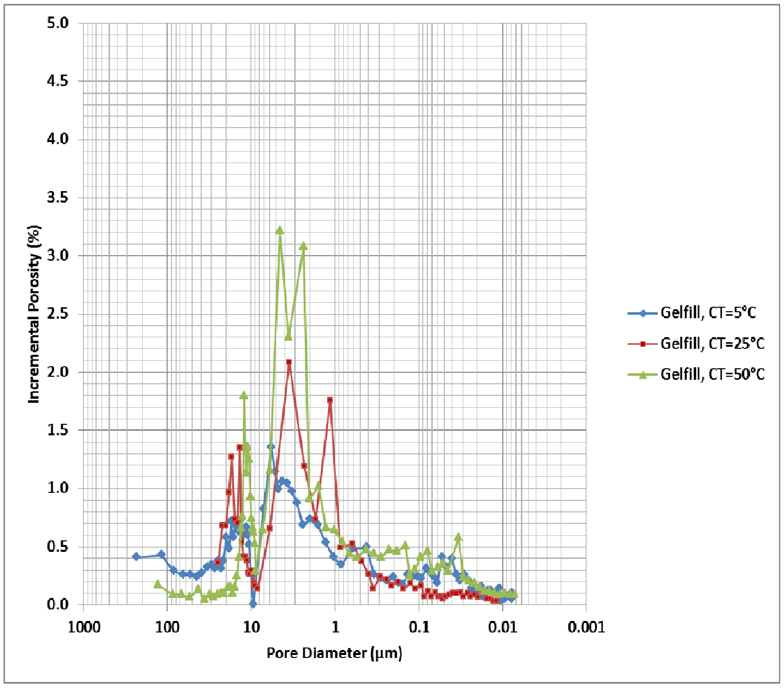

Figure 6 – Incremental pore size distribution of Gelfill specimen containing 0.3 wt % sodium silicate cured for 28 days

Results of MIP tests performed on CHF and Gelfill samples are summarized in Table 6. The higher total porosity in the samples cured at 50 °C can explain the lower UCS values in those samples. In fact, by increasing porosity, the lower UCS value can be expected (Li & Aubertin, 2003). Low UCS for the samples cured at 5 °C could be explained by the higher threshold and critical diameters. In fact, the pore size distribution of samples cured at 5 °C is dominated by larger pores than those found in other samples. It may be concluded that the combination of lower total porosities and better pore size distribution contribute to the higher UCS values obtained from samples cured at 25 °C.

Table 6 – Summary of MIP test results conducted on CHF and Gelfill specimens cured at 5, 25 and 50 °C

Specimen	Size of pores ≥ 25% (µm)	Total porosity (%)	Critical pore size (µm)	Threshold diameter (µm)
CHF-CT = 5 °C	15	34.10	6	55
CHF-CT = 25 °C	3.5	37.89	4	37
CHF-CT = 50 °C	6	41.27	6	15
Gelfill-CT = 5 °C	5	31.3	5	85
Gelfill-CT = 25 °C	2	34.42	4	23
Gelfill-CT = 50 °C	3	35.46	5	15

CONCLUSIONS

The influence of a sodium silicate concentration and curing temperature on the mechanical and microstructural properties of CHF and Gelfill is presented in this paper. The investigation confirmed that by adding an appropriate amount of sodium silicate, the mechanical properties of CHF can be significantly improved. However, Gelfill specimens with an elevated amount of sodium silicate (> 0.5 wt %) had no strength over 14 days of curing and limited strength at 28 days of curing. It was also demonstrated that curing temperature strongly influences the mechanical strength of Gelfill samples. The results also confirm that the elevated curing temperature could cease the hydration of binders in CHF and Gelfill. Finally, the research also presents a low curing temperature could cause a low UCS values in CHF and Gelfill samples.

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