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# Effect of Thermo-Hydro-Mechanical-Chemical Processes on The Strength Development of Cemented Paste Backfill Containing Polycarboxylate ether-based Superplasticizer

Zubaida Al-Moselly, Mamadou Fall  
*Department of Civil Engineering, University of Ottawa  
Ottawa, Ontario, Canada.*

## ABSTRACT

The interaction between thermal (T) (e.g., heat produced by the binder hydration, mine temperature), hydraulic (H) (e.g., drained/undrained conditions, self-desiccation, suction, pore pressure), mechanical (M) (e.g., stress induced by the backfill self-weight, filling rate), and chemical (C) (e.g., binder hydration, chemical interactions between cemented paste backfill (CPB) components and additives) processes or factors greatly affects the behaviour and the strength development of cemented paste backfill structure. For a reliable and optimal design of CPB structure, the coupled THMC processes must be considered during the curing of the CPB materials. Accordingly, the main objective of this experimental investigation is to assess the effects of coupled THMC factors on the strength evolution of CPB materials with polycarboxylate ether-based superplasticizer (PES). A laboratory setting curing system was designed to properly reflect various curing conditions, to which the CPB structure is subjected in the field during its curing and to assess the mechanical performance of CPB (the unconfined compressive strength). Various CPB samples with different mix components (such as superplasticizer content) were prepared, and cured under various coupled THMC conditions (stress-free, curing under stress, constant temperature, non-isothermal temperatures, and undrained drained conditions). The evolution of the pore water pressure, suction, temperature, and vertical deformation were monitored during the designed curing time. The results presented in this paper show that the THMC factors have a great influence on the strength development of CPB with superplasticizer and their impact is more pronounced in the presence of PES. The results can further aid in the design and production of a safer and more cost-effective CPB structure.

## RÉSUMÉ

L'interaction entre les processus ou facteurs thermiques (T) (par exemple, chaleur produite par l'hydratation du liant, température de la mine), hydrauliques (H) (par exemple, conditions drainées/non drainées, auto-dessiccation, aspiration, pression interstitielle), mécaniques (M) (par exemple, la contrainte induite par le poids propre du remblai, le taux de remplissage) et les facteurs chimiques (C) (par exemple, l'hydratation du liant, les interactions chimiques entre les composants du remblai en pâte cimentée (CPB) et les additifs) affectent grandement le comportement et le développement de la résistance de la structure du remblai en pâte cimentée. Pour une conception fiable et optimale de la structure CPB, les processus THMC couplés doivent être pris en compte pendant le durcissement des matériaux CPB. Par conséquent, l'objectif principal de cette étude expérimentale est d'évaluer les effets des facteurs THMC couplés sur l'évolution de la résistance des matériaux CPB avec un superplastifiant à base d'éther polycarboxylate (PES). Un système de durcissement en laboratoire a été conçu pour refléter correctement les diverses conditions de durcissement auxquelles la structure CPB est soumise sur le terrain pendant son durcissement et pour évaluer les performances mécaniques du CPB (la résistance à la compression non confinée). Plusieurs échantillons de CPB avec différents composants de mélange (tels que la teneur en superplastifiant) ont été préparés et durcis dans diverses conditions THMC couplées (sans contrainte, durcissement sous contrainte, température constante, températures non isothermes et conditions drainées non drainées). L'évolution de la pression de l'eau interstitielle, la succion, la température et la déformation verticale ont été surveillées pendant le temps de durcissement prévu. Les résultats présentés dans cet article montrent que les facteurs THMC ont une grande influence sur le développement de la résistance du CPB avec superplastifiant et leur impact est plus prononcé en présence de PES. Les résultats peuvent en outre aider à la conception et à la production d'une structure CPB plus sûre et plus rentable.

## KEYWORDS

Cemented paste backfill, tailings, superplasticizer, coupled THMC

## 1 INTRODUCTION

The concept of Cemented paste backfill (CPB) technology has become increasingly popular in underground mining backfilling methods around the world (Fall et al. 2009,

Yilmaz et al. 2004). Backfilling the underground mine stopes with CPB can prevent surface subsidence and collapse of mine stopes, thereby providing regional stability

to mine working areas. In addition, CPB minimizes the disposal of solid mine wastes (tailings) and the associated environmental challenges such as acid mine drainage and ground water pollution (Archibald et al. 2000, Ercikdi et al. 2009). CPB is an engineered mixture of dewatered tailings from the milling or processing operations of mines (70-85 wt.%), hydraulic binders (3-7 wt.%, often), and fresh and/or mine processed water (Fall and Benzaazoua 2005, Fall et al. 2008, Célestin and Fall 2009). High range water reducing chemical additives known as superplasticizer can be encompassed to the mixture of the CPB to enhance the transportation of high-density CPB materials to the underground stopes, and to avoid clogging of the pipelines systems caused by poor flowability CPB, and thereby reducing the transportation costs of CPB materials (e.g., pumping maintenance and energy cost) (Jung and Biswas 2002, Wu et al. 2013, Ercikdi et al. 2017). Several researchers have demonstrated that incorporating superplasticizers into the CPB mixture significantly improve the rheological properties of the CPB mixture and enhanced the mechanical strength of the CPB materials (Huynh et al. 2006, Ercikdi et al. 2010, Simon et al. 2011, Ouattara et al. 2017, Ouattara et al. 2018a, Mangane et al. 2018, Haruna and Fall 2020, Al-Moselly et al. 2022). Superplasticizer has the ability to adsorb and produce repulsive forces on the surface of cementitious mixtures that disperse the particles (Ouattara et al. 2018b; Yang et al. 2018).

Once the CPB is transported to the underground stopes, the CPB structure or mass is subjected to coupled thermal (T) (e.g., heat produced by binder hydration, mine temperature), hydraulic (H) (e.g., drainage conditions, self-desiccation, suction, pore pressure), mechanical (M) (e.g., stress induced by the backfill self-weight, filling rate), and chemical (C) (e.g., binder hydration, chemical interactions between CPB components and chemical additives) processes (Ghirian and Fall 2013, 2014, 2015, 2016; Cui and Fall 2017). The mechanical performance and long-term stability of the CPB structures are strongly influenced by the interactions or coupling between these THMC processes. The mechanical strength evolution of the CPB has been the subject of extensive investigations over the last two decades (Yilmaz et al. 2004; Klein and Simon 2006; Fall et al. 2007; Fall and Samb 2009; Yilmaz et al. 2009; Fall et al. 2010; Fall and Pokharel 2010; Ercikdi et al. 2013; Ghirian and Fall 2013, 2014, 2015, 2016; Cui and Fall 2016; Wang et al. 2016). However, currently no specific research has been conducted to investigate the effect of these coupled THMC processes on the strength of the CPB materials with superplasticizers, and the previous studies considered the impact of only one or two factors of the above-mentioned THMC processes. Thus, the aim of this study is to experimentally assess the effects of coupled THMC factors on the strength evolution of CPB materials with superplasticizers.

## 2 EXPERIMENTAL PROGRAM

### 2.1 Materials

Artificial silica tailings, binder agent, tap water, and polycarboxylate ether-based superplasticizer type were

utilized in this study for the preparation of the CPB specimens.

The artificial silica tailings are made of non-reactive powder of 99.8% by weight quartz ( $\text{SiO}_2$ ). The main objectives of using non-reactive silica tailings are to control the mineralogical and chemical composition (e.g., sulphate) of the tailings, and to eliminate the potential uncertainties associated to natural tailings. The presence of sulphate or other reactive chemical minerals that may be found in natural tailings affect the interpretation and analysis of the results. The chemical composition of the tailings is presented in Table 1.

Portland cement type 1 (PCI) was used as the binder agent in this study. PCI is the most common binder agent used in the CPB technology. The chemical and physical properties of the PCI are presented in Table 2.

A polycarboxylate ether-based superplasticizer (PES) was used in the preparation of the CPB mixture. The PES is a full-range water-reducing admixture that is commonly used in the concrete industry to enhance the workability and to improve the mechanical strength of concrete. The dispersing mechanism of the polycarboxylate ether-based superplasticizer is steric hindrance forces.

### 2.2 Sample Preparation and Mix Design

The influence of coupled THMC factors on the strength development of CPB materials with polycarboxylate-ether based superplasticizer (CPB-PES) were investigated using two sets of CPB specimens, a control set (i.e., samples cured under temperature of 20°C and without applying vertical pressure), and THMC set (i.e., samples cured under simulated field curing stress and temperature). For each set, the CPB mixtures were prepared using a constant binder content of 4.5%, water to cement (w/c) ratio of 7.35%, and 0.125% by weight of PES. First, the measured amounts of tailings and cement were mixed for about 2 min in a mechanical mixer. Then, the required amounts of water and PES were added and thoroughly mixed with the tailings and the binder for about 7 min to obtain a homogeneous CPB mixture. For the control test samples, the freshly prepared CPB mixtures were poured into plastic cylinders with a height of 10 cm and a diameter of 5 cm. Whereas for the THMC test samples, the prepared CPB mixtures were poured into the pressure cells described in the following section.

Table 1. Chemical compositions of the silica tailings

Mineral (symbol)	Mineral composition (% wt.)
$\text{SiO}_2$	99.8
$\text{Fe}_2\text{O}_3$	0.035
$\text{Al}_2\text{O}_3$	0.05
$\text{TiO}_2$	0.02
CaO	0.01
MgO	<0.01
$\text{Na}_2\text{O}$	<0.01
$\text{K}_2\text{O}$	0.02
Loss on ignition	0.1

### 2.3 THMC Curing System Set-Up

The effect of self-weight (overburden pressure) caused by mine stope filling processes, the evolution of cement hydration, pore water pressure, suction, temperature, and conditions on the strength of CPB structure can be investigated using the THMC curing system.

Table 2. Chemical and physical properties of the binder

Elements (units)	PCI
SO <sub>3</sub> (% wt.)	3.82
Fe <sub>2</sub> O <sub>3</sub> (% wt.)	2.70
Al <sub>2</sub> O <sub>3</sub> (% wt.)	4.53
SiO <sub>2</sub> (% wt.)	18.03
CaO (% wt.)	62.82
MgO (% wt.)	2.65
Relative density	3.20
Specific surface (m <sup>2</sup> /g)	1.30

The THMC curing system is designed to simulate the influence of various THMC factors on the strength and performance of the CPB materials with polycarboxylate-ether based superplasticizer (PES). Various modifications were applied to the pressure cell apparatus that were originally developed at the University of Ottawa by Ghirian and Fall (2013, 2015). A schematic diagram of the modified pressure cells set-up is shown in Figure 1.

The modified pressure cells comprise of a Perspex (acrylic plastic) cylinder with a diameter of 101.6 mm and a height of 304.8 mm. Two cover stainless plates were placed at the top and bottom of the cylinder with three stainless steel tie rods to hold the cylinder and cover plates together. An axial loading piston is attached to the top base end of the cylinder in to apply the required compressed air pressure. Then, the modified pressure cells were enclosed in a temperature-controlled chamber to mimic the non-isothermal curing conditions. There were several sensors attached to the pressure cells to record the development of pore water pressure, suction, and temperature.

### 2.4 THMC Curing Conditions Application and Monitoring Program

The two sets of the prepared CPB-PES samples were cured and monitored for 0.5, 1, 3, 7, and 28 days under the following curing conditions:

#### 2.4.1 Mechanical Curing Conditions

In underground mine stopes and backfill operations, the in-situ pressures applied to the CPB structure and the adopted filling rate for each specific mine significantly vary from mine to mine and even from stope to stope. These variations can largely depend on the stope geometry, cement content, depth of the stope, filling rate, and the design pressure of the barricade. Accordingly, free and field curing stress conditions were applied on the control set and the THMC set of CPB samples, respectively, to

simulate the in-situ backfilling process and to study the behaviour of the CPB structure in a stope.

The THMC set samples were cured under gradual air pressure sequences adopted from the measured field data of backfilling processes of a long hole stope of 32 m height based on Thompson et al. 2009. Following 6 days of the backfilling process, the maximum in-situ pressure was approximately 600 kPa with an average filling rate of 0.26 m/hr after 3m height. Table 3 illustrates the pressure increments and the corresponding filling rates that were adopted in this study based on Thompson et al. 2009.

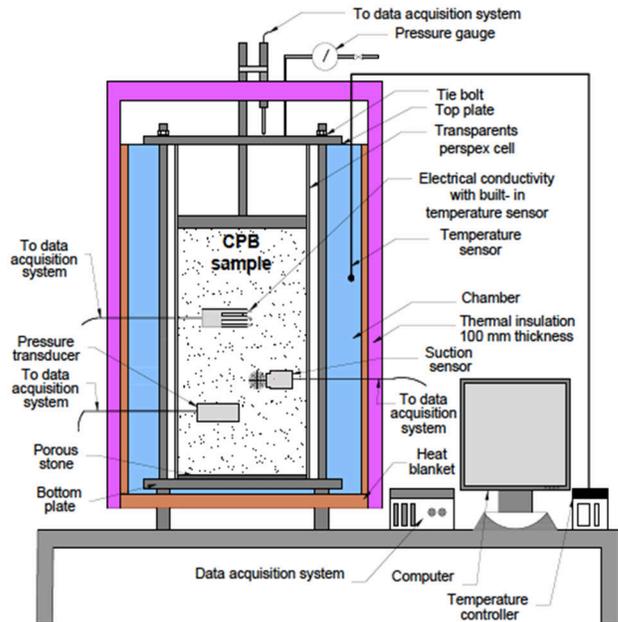


Figure 1. Schematic diagram of pressure cells set-up (modified from Ghirian and Fall 2015)

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#### 2.4.2 Hydraulic Curing Conditions

The mechanical stability of underground mines is greatly affected by the drainage conditions. Le Roux et al., 2005 showed that the mechanical strength of CPB samples in undrained backfilled conditions is lower than the mechanical strength of CPB samples in drained backfilled conditions. Therefore, considering the worst-case conditions in the field, the undrained conditions were adopted in this study for both sets. To monitor the change in the pore water pressure (PWP) in the CPB samples, Omega PX309 pressure transducers with a -15 to +15 PSI pressure range and  $\pm 0.25\%$  accuracy were utilized and attached to a data acquisition system to record the test results with time.

The negative pore water pressures were monitored with an MPS-6 dielectric water potential sensor at a range of -9 to -100,000 kPa and an accuracy of  $\pm 10\%$  of reading +2 kPa from -9 to 100 kPa. The MPS-6 dielectric water potential sensor was attached to the Decagon Em50 data logger to record the experimental data with time.

Table 3. Pressure application scheme and the corresponding equivalent CPB height

Elapsed time	Applied vertical pressure (kPa)	Equivalent height (m)	Equivalent filling rate (m/hr)
start	-	-	-
2 hr	20	1.00	0.50
4 hr	35	2.00	0.50
6 hr	55	3.00	0.50
8 hr	60	3.20	0.40
10 hr	80	4.30	0.43
12 hr	90	4.80	0.40
1 d	100	5.38	0.22
1.5 d	125	6.66	0.19
2 d	150	7.92	0.17
3 d	250	13.25	0.18
4 d	400	21.41	0.22
5 d	450	24.00	0.20
6 d	600	32.00	0.22

#### 2.4.3 Thermal Curing Conditions

Temperatures variations in underground stopes have significant impacts on the strength development of CPB materials (Fall and Pokharel 2010, Fall et al. 2010) Temperatures can considerably vary from mine to mine and during mine backfilling operations due to initial

temperatures of the CPB mixture, the heat generated by the cement hydration process, temperature of the mine, self-heating of the surrounding rocks in deep mines, the depth and geological conditions of the mine (Fall and Pokharel 2010, Fall et al. 2010, Wang et al. 2016, Cui and Fall 2016). Therefore, to simulate the in-situ backfilling process and to study the behaviour of the CPB structure in various temperatures, isothermal (temperature of 20°C) and non-isothermal curing temperatures were investigated on the control set and THMC set of CPB samples, respectively. The THMC set of CPB samples were cured under a temperature history adopted from a 30 m stope height and backfilled with CPB with binder content of 4.5% as shown in Figure 2. This field temperature–time history was predicted by using a numerical tool developed by Nasir and Fall (2009) and Wu et al. (2012).

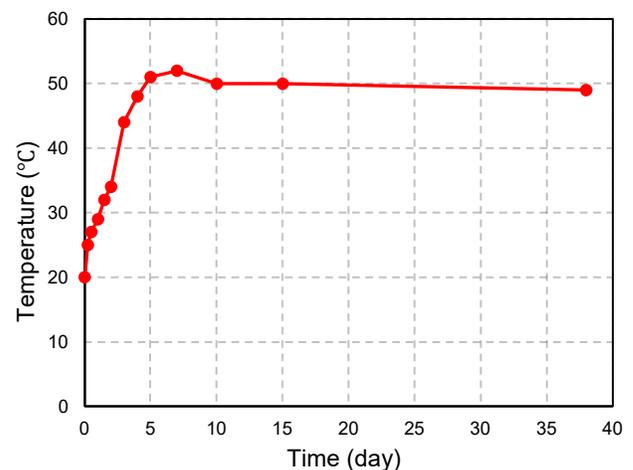


Figure 2. Temperature history for a stope size of 30 m height and binder content of 4.5%

#### 2.4.5 Chemical Curing Conditions

The two prepared CPB sets (i.e., the control set samples and the THMC set samples) were evaluated with time to assess the influence of the chemical compositions of the CPB-PES mixture on strength acquisition. The chemical processes of CPB-PES are mainly generated by the chemical interactions and the mineralogical compositions of its components including the tailings, binder, mixing water, and additives (i.e., polycarboxylate-ether based superplasticizer). The evolution of the cement hydration products and the microstructural properties of the CPB-PES were investigated by conducting void ratio ( $e$ ) and porosity ( $n$ ) tests on the CPB-PES specimens.

### 2.5 Testing Program

#### 2.5.1 Mechanical Test

Uniaxial compressive strength (UCS) tests were determined according to ASTM C39 procedure on the CPB-PES specimens at various curing times (0.5, 1, 3, 7,

and 28 days). After reaching the required curing period, the pressure cells for the THMC sets were disassembled and the CPB-PES samples were trimmed within the standard cylindrical specimens' measurement of length-to-diameter ratio (H/D) of two. A minimum of two samples were tested for each curing time to confirm the repeatability of the results. The UCS tests were conducted at a constant deformation rate of 1 mm/min. The test results data were recorded using a computer data acquisition system.

### 3 RESULTS AND DISCUSSION

#### 3.1 Effect of THMC Factors on The Strength Evolution of CPB-PES

The effects of the THMC factors on the UCS values of CPB-PES samples cured for 0.5, 1, 3, 7, and 28 days are presented in Figure 3. It can be observed that the strength values of both the control and the THMC samples increase with curing time. This is due to the precipitation of a higher amount of cement hydration products and the related pore structure refinement with time (Fall and Samb 2008, Fall and Pokharel 2010). The UCS results indicate that the compressive strength of the CPB-PES samples cured under different THMC factors (i.e., different curing conditions) is significantly greater than the control samples. For example, the 28-day UCS values of the THMC sample is 4262 kPa, whereas that of the control sample is 706 kPa. This is attributed to the combined effects of pressure application and temperature. Pressure application or curing under stress increases the packing density of the material, and this results in higher compressive strength (Ghirian and Fall 2015, Yilmaz et al. 2009).

Further, curing the CPB-PES samples in non-isothermal curing temperatures significantly increases the UCS values with time (Al-Moselly et al. 2022). Higher curing temperatures accelerate the cement hydration process and thus significantly contribute to a more refined pore structure of CPB value as well as a denser CPB structure, subsequently resulting in a higher UCS value (Fall et al. 2005, Fall and Samb 2008, Fall et al. 2010, Ghirian and Fall 2015). The increase in density of the CPB structure is experimentally demonstrated by the results of the dry density tests presented in Figure 4. Moreover, the heat generated from the cement hydration process contributes to faster chemical reactions and further precipitation of cement hydration products.

Figure 5 illustrates the evolution of the physical properties involving void ratio ( $e$ ) and porosity ( $n$ ) for both CPB-PES sets (i.e., the control set and the THMC set samples) with time. It is evident that there is an association between the evolution of the physical properties of CPB-PES samples and curing conditions. It can be seen in Figure 5 that the porosity and void ratio decrease as the curing time increases, and the THMC set samples show lower porosity and void ratio compared to the control samples. This can be explained by the impact of curing conditions on the cement hydration process and the chemical interactions between the CPB components and the polycarboxylate ether-based superplasticizer (PES) added to the mixture.

As previously mentioned, curing the CPB-PES mixture under different curing conditions (i.e., curing under stress

and non-isothermal curing temperatures) raises the degree of binder hydration with time, and this causes an increase in the precipitation of the cement hydration products. The pore voids will be filled with cement hydration products and thus, a reduction in the porosity and void ratio of the CPB-PES mixture can be observed (Ghirian and Fall 2015).

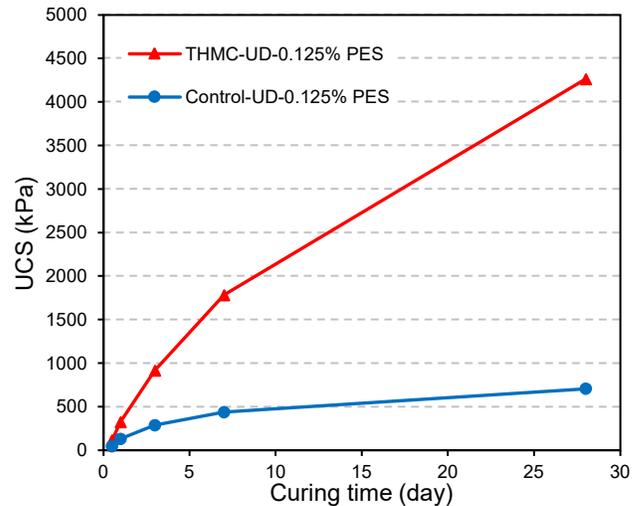


Figure 3. Influence of THMC processes on the strength of CPB-PES

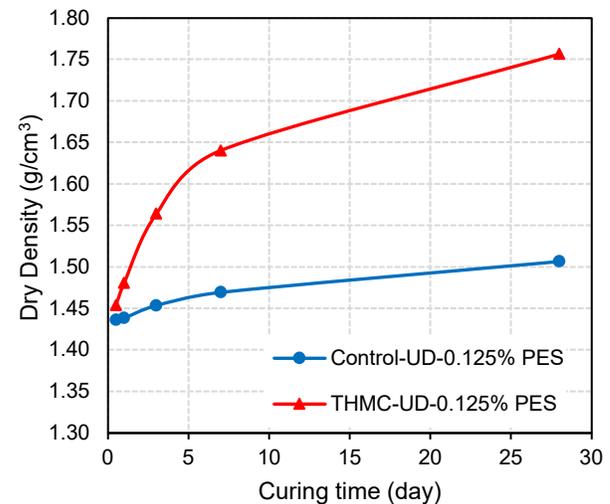


Figure 4. Dry density results of CPB-PES with time

#### 3.2 Contribution of The Chemical (C) Factor to The UCS Development

Additionally, incorporating PES to the CPB mixture significantly affects the microstructure of the CPB materials (Ercikdi et al. 2010, Yang et al. 2018). The addition of PES results in the dispersion of the solid particles (tailings and cement), and subsequently a higher rate of precipitation of

the cement hydration products as well as a decrease in porosity and void ratio can be observed (Al-Moselly et al. 2022).

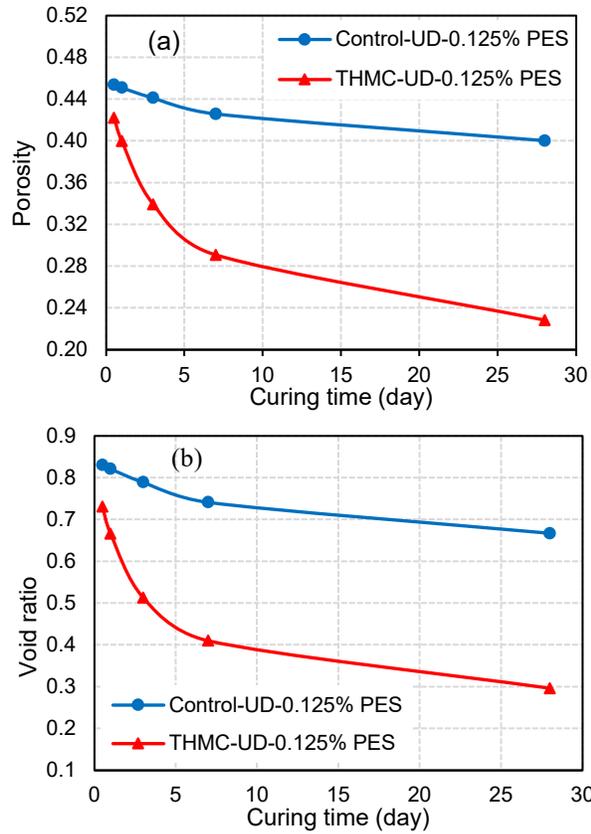


Figure 5. Evolution of physical properties: (a) porosity, (b) void ratio

### 3.3 Contribution of The Hydraulic (H) Factor to The UCS Development

The strength evolution of CPB structure is greatly affected by the development of the negative pore water pressure (suction) and the reduction of the positive pore water pressure (Ghirian and Fall 2013). Figure 6 shows the results of the suction development measured by the installed sensor for a period of 28 days. From this figure, it can be observed that the suction for both CPB-PES set samples increases with time. This can be attributed to the increased consumption of free water caused by the ongoing cement hydration process and the related chemical reactions between the CPB components (Ghirian and Fall 2014, 2015). Also, it can be seen from Figure 6 that the initial increase in suction for the CPB-PES sample cured under stress and non-isothermal curing temperatures (THMC-UD-0.125% PES) commences earlier than the control sample. This is attributed to the increased self-desiccation in the CPB-PES samples caused by the application of pressure and high curing temperatures (Fall

et al. 2010, Ghirian and Fall 2014, 2015, Al-Moselly et al. 2022).

Figure 7 presents the changes in the pore water pressure of both sets (the control set and the THMC set samples) for a period of 28 days. It can be clearly observed

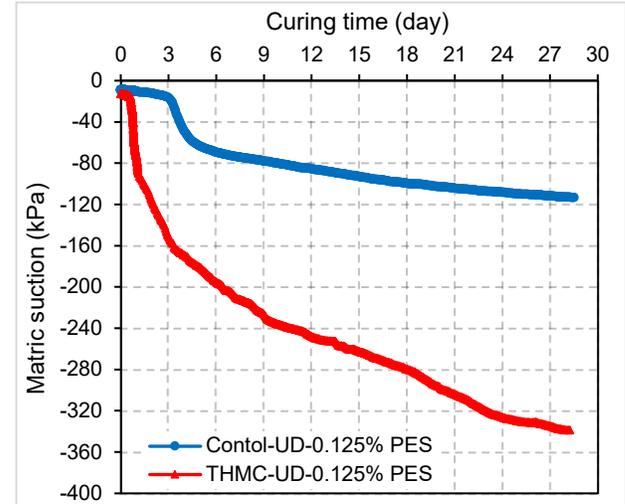


Figure 6. Changes in matric suction of CPB-PES samples with curing time

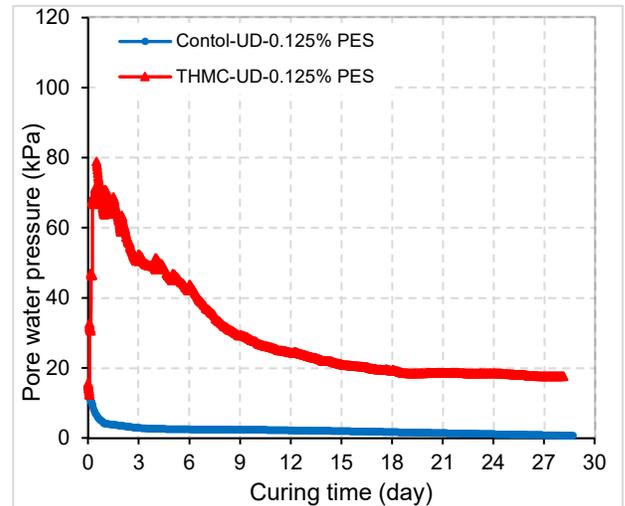


Figure 7. Evolution of the pore water pressure of CPB-PES samples with curing time

that pore water pressure for the CPB-PES sample cured under stress and non-isothermal curing temperatures (THMC-UD-0.125% PES) is greatly influenced by the increase in pressure and temperatures at the early hours of curing. Higher pressure causes the CPB-PES sample to behave like a fluid in the early hours, which in turn leads to the higher pore water pressure at the early hours of curing compared to the control sample (Ghirian and Fall 2015).

Afterwards, for both samples, there is a gradual decrease in the pore water pressure as the curing temperatures increase. This is due to the accelerated cement hydration process caused by the gradual increase of curing temperatures (see Figure 2).

#### 4 CONCLUSIONS

The main objective of this research was to investigate the effects of coupled THMC factors on the strength evolution of the CPB materials with polycarboxylate ether-based superplasticizers (PES) by simulating different curing conditions. The obtained experimental results show that THMC factors have a great influence on the strength development of CPB with superplasticizer. The THMC factors are simultaneously influenced by cement hydration processes, addition of superplasticizer, curing conditions, as well as self-desiccation. The obtained results show that non-isothermal curing conditions considerably enhance the hydro-mechanical behaviour of CPB materials with time. Also, curing under stress or the application of gradual pressure on CPB-PES structures leads to an increase in the packing density of the structure, and thus greater compressive strength. To conclude, the obtained results demonstrate the importance of considering mine conditions in evaluating the mechanical strength of the CPB structure for a safer and more affordable CPB structure.

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