Initial temperature-dependence of strength development and self-desiccation in cemented paste backfill that contains sodium silicate

CrossMark

Yong Wang [a, b], Mamadou Fall [b, *], Aixiang Wu [b]

[a] Key Laboratory of High-Efficient Mining and Safety of Metal Mines of Ministry of Education, School of Civil and Environmental Engineering, University of Science and Technology Beijing, Beijing, 100083, China
[b] Department of Civil Engineering, University Ottawa, Ottawa, Ontario, Canada

ARTICLE INFO

ABSTRACT

Advanced knowledge of the effect of the initial temperatures of cemented paste backfill (CPB) on its strength development and self-desiccation ability is needed to provide a rational basis for mixture proportioning as well as the cost-effective design of CPB structures and speeding up of the mining cycle. An experimental testing and monitoring program has been undertaken to determine the influence of various initial temperatures (2 °C, 20 °C, 35 °C and 50 °C) on the strength development and evolution of the self-desiccation (measured by the volumetric water content and suction) of the CPB that contains sodium silicate as the admixture (S-CPB) at early ages (up 28 days). The evolution of the temperature, electrical conductivity (gives information about the cement hydration progress), volumetric water content and suction of S-CPB samples with the specified initial temperatures have been monitored for 28 days. Moreover, mechanical tests and microstructural analyses are performed on these S-CPB specimens after specific curing times (6 h, 1 day, 3 days, 7 days and 28 days). The results obtained show that the initial backfill temperature has a significant influence on the curing temperature of S-CPB, its strength development and self-desiccation. Furthermore, the results reveal that the temperature-time history, cement hydration, self-desiccation and strength development of S-CPB are strongly coupled. The initial S-CPB and these couplings should be taken into account for a safe and economical design of S-CPB structures and the speeding up of mining cycles.

© 2016 Elsevier Ltd. All rights reserved.

1. Introduction

One of the most important technological innovations in the last two decades, with the aims to increase the productivity of mines, manage mine waste cost effectively and contribute to the improvement of health and safety in mine operations, is the technology of cemented paste backfill (CPB) [1]. CPB is a mix of tailings, water and binder. Its components are combined and mixed in a plant usually situated on the mine surface and transported (by gravity and/or pumping) to the underground mine openings (stopes) [2–4]. To carry fresh and hydrating CPB materials during stope filling and thereby prevent CPB from flowing into the mine working areas, retaining wall structures called barricades or bulkheads are built in each of the access ways into the stope prior to

stope filling [5–7].

The most common binder used in the preparation of CPB is Portland cement (PC). PC is not only a costly binder, but its production generates a large amount of carbon dioxide (CO_2) (CO_2 is a by-product of a chemical conversion process used in the production of clinker; CO_2 is also released during cement production by fossil fuel combustion) [8]. The cement consumption can represent up to 80% of the cost of CPB. These factors mentioned above have compelled mining companies to seek for alternatives to cement that increase the strength of the CPB, decrease the cement content and reduce the carbon footprint of the mining industry. Sodium silicate is one of the most recent chemical additives that are used to reduce the binder content in cemented backfill and increase its strength [13]. This new product, sodium silicate cemented paste backfill (S-CPB) (also named gelfill), is a mix of tailings, water, cement and sodium silicate (usually 0.3–0.4% by weight of solids).

Mechanical stability is one of the key performance properties of any CPB. Once placed, the CPB has to satisfy certain mechanical stability requirements to ensure a safe underground working

* Corresponding author. Department of Civil Engineering, University of Ottawa, 161 Colonel by, Ottawa, Ontario, K1N 6N5, Canada.
E-mail address: mfall@uottawa.ca (M. Fall).

environment for all mining personnel [9–14]. Moreover, a mechanically stable cemented backfill structure at the early ages is especially important for the opening of the barricades, thereby for reducing the mining cycle time, and thus increasing mining efficiency and improving production. So to say, the designed and built CPB structure should be capable of achieving the desired mechanical stability for ground support as early as possible.

As a structural element, the assessment of the mechanical stability of a CPB structure is mainly based on the (undrained) unconfined compressive strength (UCS). This is because the UCS test is relatively inexpensive, and can be easily incorporated into routine quality control programs at the mine [15]. The 28-day compressive strength that is required to maintain backfill stability is generally between 0.7 and 2 MPa [16]. However, the required UCS value largely varies, depending on the application and/or function of the CPB.

Moreover, the understanding and assessing of pore water pressure development in the CPB at the early ages are also critical for a safe and cost-effective design of the barricades or bulkheads. Indeed, the mechanical stability of the barricade or bulkhead walls is significantly affected by the pore water pressure developed in the CPB [7,17]. An excess of positive pore water pressure can considerably increase the loads applied onto these retaining structures [18]. It is well known that self-desiccation (induced by cement hydration) happens in the CPB. This self-desiccation results in the reduction of the positive pore water pressure or the development of negative pore pressure (suction) in the hydrating CPB structure. This means that a clear understanding of the pore water pressure changes induced by self-desiccation is critical for the proper assessment of the mechanical stability and safety of the barricades, and thus for their cost-effective design. In addition, suction has a direct impact on the strength of porous materials, and thus on CPB strength [19–21]. Moreover, pore water pressure decrease or suction development will change the effective stress in the CPB, which will affect the mechanical behavior of the CPB [9,18,20,21].

However, despite the extensive use of CPB and several past studies [e.g., 9,10,14,22–27] performed on CPB, many fundamental factors that affect its strength development and self-desiccation are still not well understood. Among these factors, the effect of the initial CPB temperature (i.e. fresh CPB temperature at time zero after its pouring into the mine stope) on its early age strength and self-desiccation is not well known. All of the past studies on the impact of temperature have only focussed on the effects of isothermal curing temperature [e.g., 8, 25] or high(fire)-temperature [(e.g. 28,29] on the strength of CPB. The influence of the initial temperature on the strength development and the self-desiccation of CPB with or without silicate sodium have been ignored. There is a need to address this issue for both economical reasons and the safety of mine workers. This is because every single CPB structure is unique with regards to differences in temperatures, and fresh CPB can have variable initial temperatures. Since CPB is a mixture of binders, water and tailings, its initial temperature is strongly affected by the initial temperatures of the mix components, such as the mixing water and tailings. The geographical location (e.g., warm/cold region) and/or the variations in temperature due to seasonal influence (e.g., winter/summer) can considerably change the temperature of the mixing waters (especially when lake or river waters supply the mixing waters for the preparation of the backfill), tailings (e.g., tailings stored outside before their use in CPB preparation) and thus that of the CPB mixtures [8]. Furthermore, an increase of the temperature in fresh CPB can occur during its transport in pipes from the surface to the underground working areas of a production section as described by Wu et al. [30]. In addition, when preparing CPB, moderate heat could be added to achieve a high early strength.

Thus, the objective of the present work is to experimentally study the influence of the various initial temperatures of CPB which contains sodium silicate on the evolution of its early age strength and self-desiccation (evaluated by the evolution of the volumetric water content (VWC) and suction in the backfill) within the S-CPB.

2. Materials and experimental program

2.1. Materials used

The materials used include water, binders, tailings and sodium silicate.

2.1.1. Water and binders

Tap water was used to mix the binders and tailings. Portland cement type I (PCI) and blast furnace slag (Slag) were used as the binders. PCI was blended with Slag. The blending ratio (weight) was 50/50. These binders are often used for CPB mixtures in mines located in eastern Canada. Table 1 shows the physical and chemical properties of the binders used.
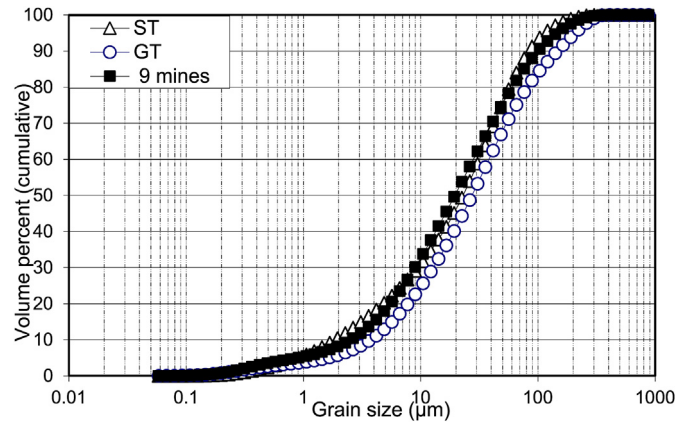
2.1.2. Tailings

The physical and chemical characteristics of tailings can vary and are mainly dependent on the parent rock properties, ore mineralogy together with the physical and chemical processes used to extract the desired product [8]. Two types of tailings are used in this study, with the aim to reveal the relevance of tailings type in the response of S-CPB at early ages. These tailings include natural gold tailings (GT) and artificial (silica) tailings (ST). GT was collected from the CPB plant of a hard rock gold mine in eastern Canada. The particle size distribution of the GT is shown in Fig. 1. This figure shows that GT has about 42 wt. % fine particles (<20 μm) and can be classified as medium tailings. ST has the advantage of allowing the accurate control of the mineralogical and chemical compositions of the tailings, and thus reducing the level of uncertainties to a minimum level. Generally, natural tailings can contain several reactive chemical elements, and often, sulphide minerals, which can interact with cement and thus, affect the interpretation of the results [8]. ST contains 99.8% SiO_2 and shows a grain size distribution close to that of GT (Fig. 1) and the average of those from nine Canadian hard rock mines. It can be observed that ST has about 45 wt. % fine particles (<20 μm) and can also be classified as medium tailings. Tables 2 and 3 show the physical and mineralogical characteristics of GT and ST, respectively. GT is mainly made of chlorite, talc, quartz, magnetite, magnesite, dolomite and sulphides (particularly pyrite and a small amount of pyrrhotite) minerals. TS is made of quartz (one of the dominant minerals in Canadian hard rock mines). Furthermore, the tailings were tested for various index properties (e.g., liquid and plastic limits) by following ASTM standards. From the results obtained, GT and ST are non-plastic and classified as sandy silts of low plasticity, and ML in the Unified Soil Classification System (UCCS; [31]). Categorization as ML is typical for tailings from hard rock mines as also determined by Vick [32].

2.1.3. Sodium silicate

Soluble sodium silicates, also named water soluble glasses, are usually produced from varied proportions of an alkali metal and silicon dioxide (SiO_2). Aside from being used as an admixture for cement, soluble sodium silicate is also used for a number of applications in various industries or fields, such as the paper industry (e.g., for binding packaging), geotechnical engineering (e.g., soil grouting, mine backfill), soap and detergent manufacturing, waterproofing, textile processing, and foundries. Soluble sodium silicates are silicate polymers. It is a polymer liquid that is clear, colorless, and viscous. It is agreed that sodium silicates activate the

Table 1
Characteristics of the binders used.

Type of binder	MgO (%)	CaO (%)	SiO$_2$ (%)	Al$_2$O$_3$ (%)	Fe$_2$O$_3$ (%)	SO$_3$ (%)	Relative density
PCI	2.65	62.82	18.03	4.53	2.70	3.82	3.10
Slag	10.98	41.14	34.23	9.54	–	3.87	3.30

Fig. 1. Grain size distribution of the tailings (ST, GT) used and the average grain size distribution of tailings from 9 Canadian mines.

Table 2
Physical characteristics of the silica tailings (ST) and gold tailings (GT).

Element	G_s	D_{10} (μm)	D_{30} (μm)	D_{50} (μm)	D_{60} (μm)	C_u	C_c
ST	2.7	1.9	9.0	22.5	31.5	16.2	1.3
GT	3.6	3.6	13.2	28.3	39.5	11.0	1.2

Table 3
Mineralogical composition of the tailings used.

Tailings/Mineral	Quartz (wt.%)	Albite (wt.%)	Dolomite (wt.%)	Calcite (wt.%)	Chlorite llb (wt.%)	Magnetite (wt.%)	Pyrite (wt.%)
GT	11.87	1.16	5.70	2.22	18.21	11.41	15.40
ST	99.8	–	–	–	–	–	–

Tailings/Mineral	Talc (wt.%)	Sphalerite (wt.%)	Magnesite (wt.%)	Actinolite (wt.%)	Stilpnomelane (wt.%)	Pyrrhotite (wt.%)	Spinel (wt.%)
GT	16.44	0.13	7.56	3.18	0.41	3.12	3.18
ST	–	–	–	–	–	–	–

tailings type (GT and ST) were prepared. The components of the backfill (GT, ST, PCI, Slag and water) were first stored in a temperature-controlled chamber until achieving the desired initial temperatures (2 °C, 20 °C, 35 °C and 50 °C). The tailings material, binders, sodium silicate and water were then mixed by using a Eurodib B20F mixer in a temperature-controlled chamber until obtaining a homogeneous paste (about 7 min). The slump or the consistency of the paste mixtures, measured by a slump test in accordance with ASTM C143, was equal to 18 cm (which is a frequently used slump value in Canadian CPB operations) for the S-CPB made of ST. For the purpose of comparing the S-CPB made of the two types of tailings (ST and GT), the samples made of GT were prepared with the same w/c ratio (7.6).

The prepared S-CPB samples with various initial temperatures (2 °C, 20 °C, 35 °C and 50 °C) were then poured into two types of thermally insulated plastic cylinders (20 × 10 cm) (Fig. 2b): (i) the first type of cylinder (test cylinder or specimen), which was not equipped with, was for testing purposes, and (ii) the second type of cylinder (monitoring cylinder or specimen) was equipped with various sensors for monitoring purposes (to monitor temperature, water volumetric content and electrical conductivity; Fig. 2). The test and monitoring cylinders were then sealed (this avoids the evaporation of water) and placed in specially designed thermally insulated boxes (Fig. 2a). The tested specimens were then cured for

hydration of Slag [13,31]. In this study, a commercial solution of sodium silicate (Type N) is used, in which the ratio of SiO$_2$ to Na$_2$O is 3:2. Table 4 shows the sodium silicate properties. Sodium silicate was added to the mix in a liquid form.

2.2. Specimen preparation, mix proportions and curing conditions

S-CPB specimens with a constant binder content (4.5% by weight), binder weight ratio (PCI/Slag: 50/50), water to cement (w/c) ratio (7.6), sodium silicate content (0.4% by weight) and different

Table 4
Sodium silicate properties (source: National Silicates Ltd.).

Property	Value
Na$_2$O% by weight	8.90
SiO$_2$ by weight	28.66
Weight ratio, %SiO$_2$/%Na$_2$O	3.22
Specific gravity @ 20 °C	1.39
% Solids	37.56

periods of 6 h, 1 day, 3 days, 7 days and 28 days. After each curing period, mechanical test was performed on each S-CPB specimen in the test cylinders. The monitoring cylinders were cured up to 28 days. The objective of the insulation was not to create adiabatic curing conditions, but to slow down the heat transfer between the backfill material and the surrounding environment (where it was room temperature, ~20 °C), in order to mimic the heat transfer between a narrow cemented backfill structure and the surrounding rockmass in the field. In the field and most cases, the CPB is surrounded by the rockmass, which cannot transfer heat very quickly. The field CPB can lose heat to the rockmass, if its temperature is higher than the rock temperature or gain heat from the rockmass, if its temperature is lower than that of the rockmass as shown in Refs. [1,27,33]. In narrow backfilled stopes, the process of heat loss and gain is faster.

In addition to the aforementioned S-CPB specimens, specimens of cement paste with a high w/c ratio (w/c = 2; to simulate the cement matrix of CPB) that contain sodium silicate (S-CP) were prepared. The prepared S-CP mixes with various initial temperatures (2 °C, 20 °C, 35 °C, and 50 °C) were poured into the test
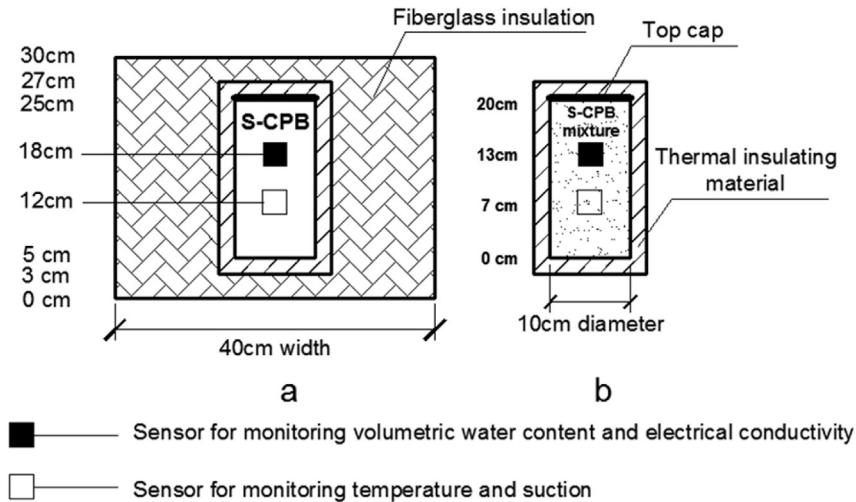
Fig. 2. Schematic diagram of monitoring set-up: (a) location of a thermally insulated monitoring cylinder in an insulation box, and (b) location of the sensors in the thermally insulated cylinder.

cylinders. The sealed cylinders were then cured in the same conditions as the S-CPB cylinders for 6 h, 1 day, 3 days, 7 days and 28 days. After the curing was completed, microstructural analyses were performed on the S-CP specimens.

2.3. Monitoring of specimens

A schematic diagram of the developed monitoring set up is presented in Fig. 2. The cylinders were continuously monitored in terms of temperature evolution, volumetric water content (VWC), suction evolution, and evolution of the electrical conductivity (EC) for a period of 28 days after the monitoring cylinders were installed in the thermal insulation boxes. The monitoring of the temperature evolution enables non-isothermal curing temperatures to be determined, to which the S-CPB specimens with various initial temperatures were subjected. The monitoring of the electrical conductivity (EC) is an effective means of assessing the progress of cement hydration, and tracking the structural changes that occur within the hydrating cementitious materials [34–36]. The monitoring of the VWC and suction provides information about the self-desiccation of the S-CPB. Sensors were connected to a data logger to record the data with time. The monitoring of suction development with time was conducted by using a dielectric water potential sensor, model MPS-2. This sensor is capable of measuring the soil water potential between −10 and −500 kPa. This sensor is also capable of monitoring the temperature evolution. In order to follow the evolution of the WVC and EC, 5 TE sensors were employed. The MPS-2 and 5 TE sensors were installed at heights of 7 and 13 cm from the bottom of the cylinder. The location of the sensors and cylinders in the insulation boxes is shown in Fig. 2.

2.4. Mechanical tests

Unconfined compressive strength (UCS) tests were performed on samples taken out from the test cylinders after a given curing time. The specimens were tested in accordance with ASTM − C 39 by using a computer-controlled mechanical press (the deformation rate was defined as 0.8 mm/min during the testing). Each test was repeated at least twice to ensure the repeatability of the results.

2.5. Microstructural analysis

The microstructure of the studied cement paste (S-CP) samples was investigated by conducting thermal analyses (TGA/DTG) as well as X-ray analyses. The thermal analyses were undertaken by using an SDT apparatus from TA Instruments, which allows for the simultaneous registration of weight loss and heat flow along the thermal treatment of the sample. The various (dried) samples (about 30 mg each) were heated in an inert nitrogen atmosphere at the rate of 10 °C per minute up to a temperature of 1000 °C. The thermal analyses allowed the binder hydration products that formed in the S-CPB system to be studied. X-ray diffraction analyses were conducted to evaluate the effect of different non-isothermal curing conditions on the mineralogical composition of the hydration products of the S-CPB samples. The X-ray diffraction analyses were performed on prepared cement pastes by using a Scintag XDS 2000 XRD.

3. Results and discussion

3.1. Temperature evolution within the backfill specimen

Fig. 3 depicts the evolution of the internal temperatures of the S-CPB samples with different initial temperatures. These different
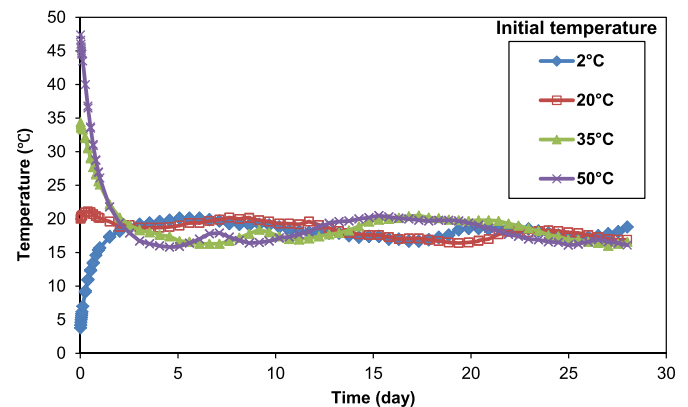

Fig. 3. Temperature evolution in the S-CPB specimens for various initial temperatures.

temperature-time histories represent the non-isothermal curing temperatures, to which the S-CPB specimens were subjected. From this figure, it is clear that the temperature-time history or curing temperature and its evolution with time strongly depends on the initial temperature of the S-CPB sample. The internal temperature of the S-CPB specimens with an initial temperature of 20 °C remains almost at the same value as that of the surrounding medium (20 °C). Only a slight increase in the backfill temperature from 20 °C to 21 °C can be observed after 8 h of curing. Then, this temperature progressively decreases until reaching the temperature of the surrounding medium after 2.5 days. This minor temperature increase is due to the heat released by binder hydration. The small increase in temperature indicates that the contribution of heat generated by binder hydration to temperature variation in all of the studied S-CPB specimens is negligible. This enables the studying of the isolated effect of the initial temperatures. Fig. 3 shows that S-CPB samples with initial temperatures (35 °C and 50 °C) higher than those of the surrounding medium (20 °C) lose heat with time until reaching temperature equilibrium (20 °C) with the surrounding medium after 2.5 days of curing. On the other hand, the S-CPB sample with an initial temperature (2 °C) lower than that of the surrounding medium gained heat. Temperature equilibrium is also reached after 2.5 days of curing. A similar behavior with regards to heat loss and gain was observed in previous field and modeling studies on CPB [e.g., 1,27,37]. This observed initial temperature dependence of the temperature-time history of S-CPB could have significant impact on the self-desiccation and early strength of S-CPB. These hypotheses are addressed in the following sections.

3.2. Effect of initial temperature on the evolution of self-desiccation of S-CPB

Figs. 4 and 5 illustrate the evolution of the volumetric water content (Fig. 4) and matrix suction (Fig. 5) of the S-CPB with various initial temperatures; in other words, the evolution of the self-desiccation. From this figure, it is clear that the self-desiccation of the S-CPB samples depends on both the curing time and initial temperature of the S-CPB.

It can be observed that, regardless of the initial temperature, the volumetric water content (VWC) decreases and the matrix suction increases as the curing time is increased. This decrease or increase
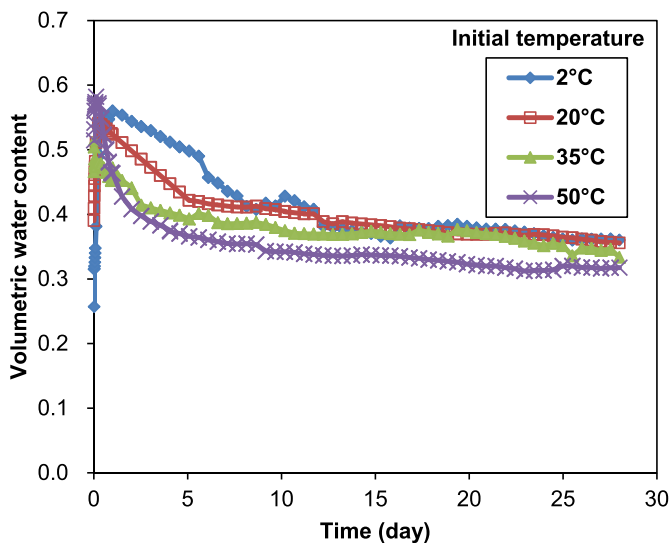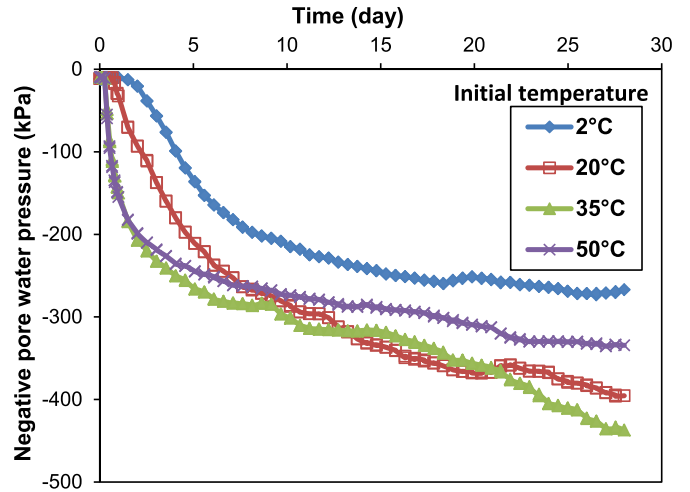

Fig. 5. Evolution of matrix suction with initial temperature and curing time for S-CPB made of silicate tailings.

is due to the progress of the cement hydration. Higher amounts of cement hydration products are generated as the curing time increases [e.g., 38—41]. This results in higher cumulative consumption of the moisture in the capillary pores of the S-CPB. This increase of the amount of hydration products formed with longer curing time is demonstrated by experimental evidence, which is shown in Fig. 6. This figure represents the results of the thermal analysis (simultaneous thermogravimetric (TG) and derivative thermogravimetric analysis (DTG)) performed on the 1 and 7 day cement pastes of the S-CPB samples with an initial temperature of 35 °C. A comparison of the TG/DTG diagrams of the cement paste cured at 1 and 7 days shows that the weight losses as well as the peaks in the 50—150 °C, 450 °C and 750 °C temperature ranges are higher for cement paste cured at 7 days. This means that higher amounts of hydration products are formed in the 7 day CPB samples.

From Figs. 4 and 5, it can be also noted that, regardless of the initial temperature, the self-desiccation is more rapid and intense at an earlier age (during the first week). At a later age (beyond 7 days), the self-desiccation slows down. For example, it can be observed that for the 2 °C S-CPB, the suction increases from 0 kPa to approximately 200 kPa from 0 to 7 days of curing time. Then, the suction slowly increases and reaches 270 kPa after 28 days. This more intense and higher rate of self-desiccation in the early stage corresponds to the period when the hydration of a Portland cement
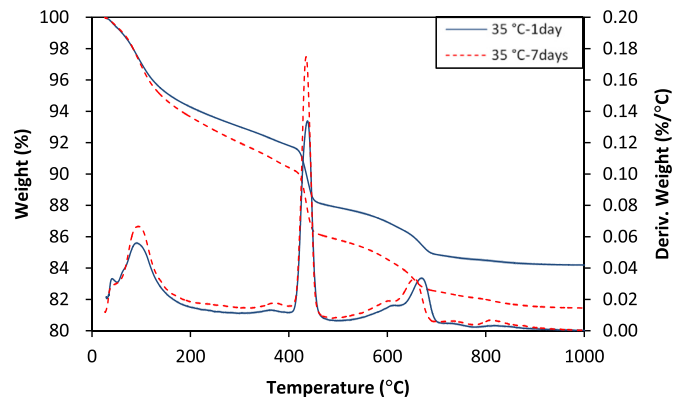

Fig. 4. Evolution of volumetric water content with initial temperature and curing time for S-CPB made of silicate tailings.

Fig. 6. Effect of curing time on the TG/DTG diagrams for cemented paste with an initial temperature of 35 °C.

system is faster [42], which is obviously associated with quicker water consumption.

However, from Figs. 4 and 5, it is also clear that the volumetric water content and matrix suction of the S-CPB samples and their evolution with time are strongly dependent on the initial temperature of the backfill, and thus on the temperature-time history to which the S-CPB samples are subjected.

In the early stages of curing (first week), the S-CPB samples with higher initial temperatures (35 °C, 50 °C) experience more rapid and intense self-desiccation than those with lower initial temperatures (20 °C, 2 °C). For example, the measured volumetric content for 3 day S-CPB samples was 0.53, 0.47, 0.39 and 0.38 for specimens with initial temperatures of 2 °C, 20 °C, 35 °C and 50 °C, respectively, whereas the recorded suction values for the 3 day S-CPB are 56, 137, 218 and 218 kPa for the specimens with initial temperatures of 2 °C, 20 °C, 35 °C and 50 °C, respectively. This more intense and quicker self-desiccation observed in the specimens with higher initial temperature is attributed to the fact that the S-CPB samples with higher initial temperatures were subjected to higher curing temperatures in the first two days of curing (Fig. 3). It is well known that at early ages, high temperature increases the rate of cement hydration, thereby resulting in more and faster consumption of the water within the capillary pores of the cemented material [e.g., 42–44]. This temperature induced increase of the cement hydration rate is explained by the fact that heat causes faster kinetics of the chemical processes (e.g., faster dissolution, nucleation or precipitation rates) and quicker diffusion through the hydrate assemblage around unreacted cement grains [e.g., 40]. This faster cement hydration with higher curing temperature is experimentally demonstrated by the results of thermal analyses (DTG diagram) performed on 7 day S-CP with initial temperatures of 2 °C and 35 °C, respectively (Fig. 7). A comparison of the DTG diagrams shows that the peaks at 400–500 °C are higher for cement paste with an initial temperature of 35 °C. These experimental observations indicate that the amount of hydration products formed increases with the curing temperature at early ages. This faster cement hydration with higher curing temperatures is also fully supported by the results of the monitoring of the electrical conductivity of the S-CPB with various initial temperatures shown in Fig. 8. This figure illustrates the effect of initial temperature on the electrical conductivity–time curves for the studied S-CPB. All of the curves show common characteristics with an increase up to the peak electrical conductivity value, followed by a gradual decrease. From Fig. 8, it can be observed that there is a clear shift of the conductivity peaks to shorter times of hydration with increasing initial temperatures or higher curing temperatures in the early
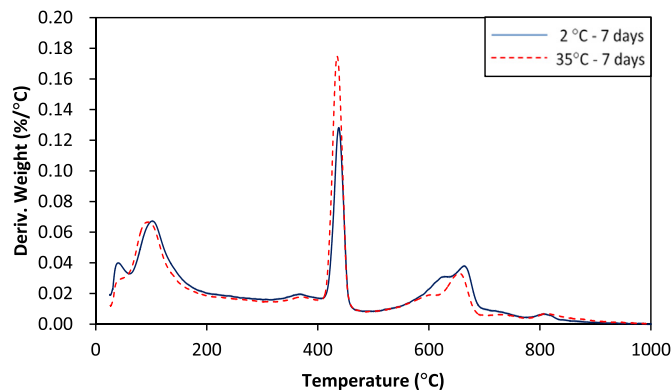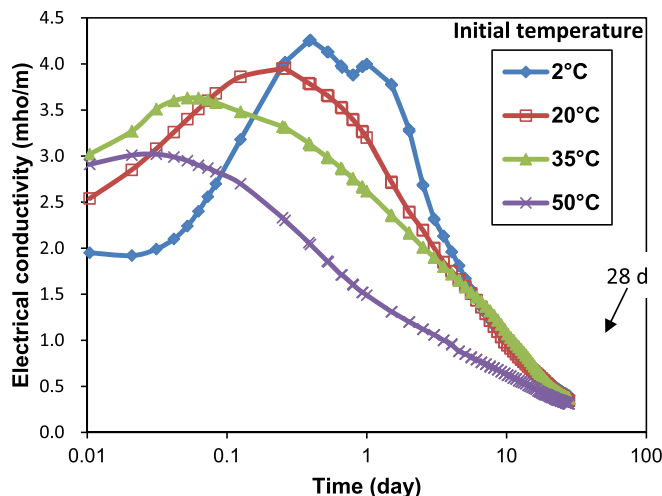

Fig. 8. Time-dependent evolution of electrical conductivity of S-CPB with different initial temperatures.

ages, which indicates that the hydration reaction rate is increased by temperature during this period [39,45–47]. Indeed, the conductivity peak takes place at 9.50 h, 4.75 h, 1.25 h and 0.75 h for the specimen with initial temperatures of 2 °C, 20 °C, 35 °C, and 50 °C, respectively.

From Figs. 4 and 5, it can be also noticed that beyond 7 days of curing, the initial backfill temperature has less effect on the volumetric water content (VWC) and suction development within the S-CPB, i.e. on its self-desiccation. For example, the measured VWC values for the 7 day S-CPB with initial temperatures of 2 °C and 50 °C are 0.41 and 0.35, respectively, whereas for the 28 day S-CPB these values are 0.37 and 0.32. This reduced impact of the initial temperature at later ages (beyond 7 days) is due to the fact that all S-CPB samples show similar temperatures after 2.5 days of curing because of the heat transfer between the backfill and the surrounding medium as illustrated in Fig. 3. Moreover, from Fig. 5, it is interesting to notice that beyond 7 days of curing, the suction of the 50 °C S-CPB is reduced compared to that of the 35 °C and 20 °C S-CPB. Differences in the internal microstructure of the S-CPB samples could probably be the reason behind this observed behavior. Detailed nano- and microstructural studies should be performed in the future to better understand the cause of the tendency of the evolution curves of suction to cross at 50 °C.

The results presented above with regards to the effect of initial backfill temperature on the self-desiccation of the cemented backfill at early ages (≤7 days) may have significant practical implications. The design of a safe and cost-effective barricade is an integral part of a successful paste backfill operation. Moreover, the early opening of a barricade is of special importance for reduction of the mining cycle time, hence increasing mining efficiency and production. This is obviously associated with financial benefit for mines [8]. The safety, cost-effectiveness and opening time of a barricade are significantly influenced by the magnitude of the pore pressure developed in the cemented backfill at early ages. The findings presented above reveal that S-CPB with a higher initial temperature demonstrates faster and more significant self-desiccation at early ages. This will obviously result in faster and more significant drop of the pore pressure within the backfill, and thus in earlier openings of the barricades. On the contrary, the rate and magnitude of self-desiccation in S-CPB with cold initial temperatures (e.g., backfill operations in cold regions) will be significantly reduced. This will have a negative impact on the stability of the barricade and the mining cycle time.

Fig. 7. Effect of initial temperature (2 °C and 35 °C) on the DTG diagrams for 7 day cemented paste.

3.3. Effect of initial temperature on the strength development of S-CPB

Fig. 9 illustrates the effect of the initial temperatures on the strength development of the studied S-CPB. It can be observed that, regardless of the type of tailings used, higher initial curing temperature results in higher strength of the S-CPB (except for samples with an initial temperature of 50 °C and older than 7 days; this will be discussed later). This is because the specimens with higher initial temperatures were subjected to higher curing temperatures in the first 2 day as graphically demonstrated in Fig. 3. A higher temperature speeds up the cement hydration. Indeed, the amount of hydration products (e.g., calcium−silicate−hydrate (C−S−H), portlandite (CH)) increases with temperature. This is beneficial for backfill strength gain, since C−S−H is considered to be the major binding phase in hardened cement [8,42]. Moreover, this higher amount of hydration results in the refinement of the pore structure, which is favorable for strength gain [8]. This greater amount of hydration products, such as C−S−H, and CH, associated with higher curing temperatures, is demonstrated by the results of different

thermal and x-ray diffraction (XRD) analyses performed on 7 day cement pastes of S-CPB with initial temperatures of 2 °C and 35 °C, respectively (Figs. 7 and 10). A comparison of the DTG diagrams of the cement pastes shows that the peaks at 400−500 °C are higher for cement paste with an initial temperature of 35 °C. This indicates that higher amounts of hydration products are formed in the 35 °C S-CPB sample. It is well recognized that peaks situated between 400 and 500 °C are due to the disintegration of CH [48−50]. These thermal analysis results are consistent with the X-ray analysis results of the cemented paste samples with initial temperatures of 2 °C and 20 °C as presented in Fig. 10. This figure shows the presence of a higher quantity of hydration products in the sample with the initial temperature of 20 °C (Fig. 10b). For example, the intensity of CH is found higher for the sample with the initial temperature of 20 °C (Fig. 10b) than that with a temperature of 2 °C (Fig. 10a). It is observed that the intensity of CH at 18° 2-theta is 210.5 CPS in the case of the sample at 2 °C whereas at 20 °C, this intensity is approximately 238.2 CPS. In the same way, the intensity of CH at 34, 47, 51° 2-theta for the sample at 20 °C is higher than that of the 2 °C samples. An additional factor should be also considered as a contributor to the initial temperature induced strength increase of S-CPB. This factor is the intensification of the self-desiccation (particularly suction increase) of S-CPB with a higher initial curing temperature as illustrated in Fig. 5. This figure depicts that the S-CPB with higher initial temperatures (35 °C, 50 °C) have higher suction at the early ages (≤7 days), which obviously results in higher strength. It is well known that suction development can lead to an increase in the strength of unsaturated porous media [19]. The rapid evolution stage of the strength (during the first
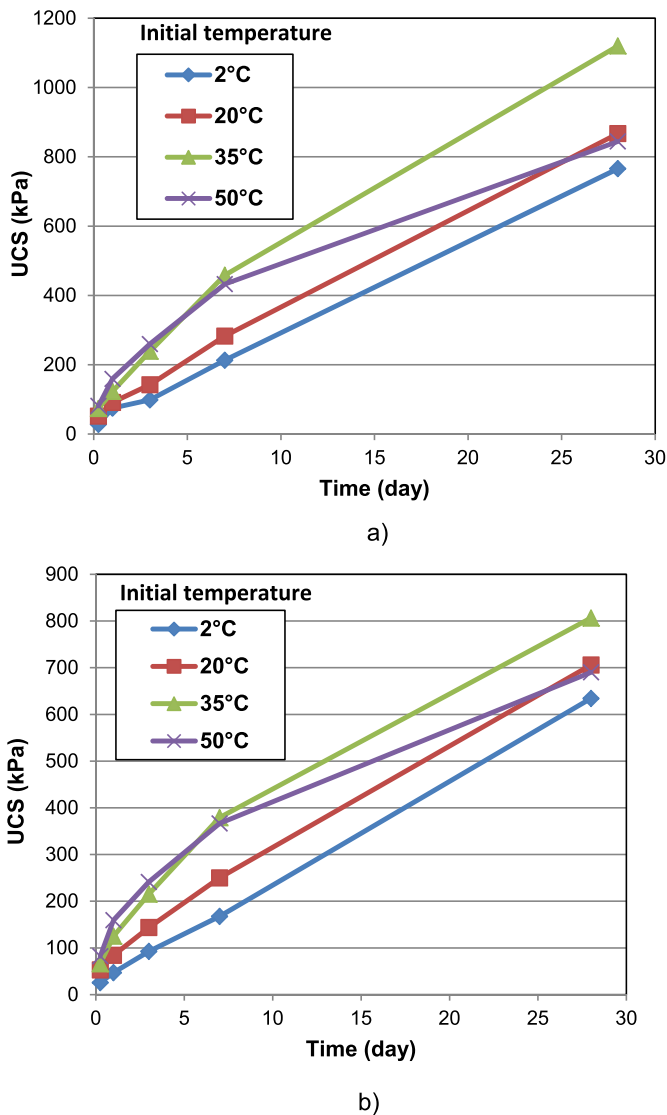

a)

b)

Fig. 9. Coupled effect of initial temperature and curing time on the UCS of S-CPB: (a) S-CPB made of silicate tailings, (b) S-CPB made of gold tailings.
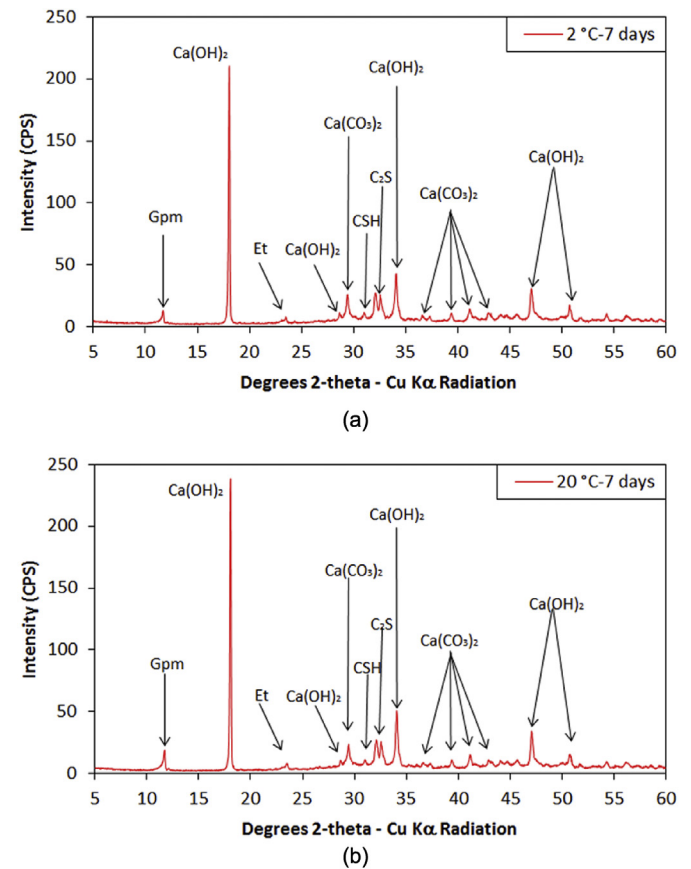

(a)

(b)

Fig. 10. Effect of initial temperature on the XRD result of 7 day cement paste made: (a) initial temperature of 2 °C; (b) initial temperature of 20 °C.

week; Fig. 9) corresponds to the time when self-desiccation is intense in the backfill material (Fig. 5).

From Fig. 9, it can be also observed that there is a decrease of the strength of the S-CPB with an initial temperature of 50 °C for curing times longer than 7 days. This phenomenon of temperature inversion in the compressive strength of cementitious materials cured at high temperatures has been the reason for many studies on the impact of temperature on cement hydration in the last decades. It is widely accepted that an overly high curing temperature slows down the kinetics of hydration (at advanced ages) after the initial acceleration [8,38,51]. This is mainly because the rapid initial rate of cement hydration causes non-uniform distribution of the hydration products across the cementitious material microstructure, with dense hydration products that form around the hydrating cement grains and retard subsequent hydration. The resulting coarser pore structure of the cementitious material has a negative effect on the strength [8,38,43,51]. This high temperature (50 °C) induced coarsening of the microstructure of cemented paste backfill at advanced ages was experimentally demonstrated by the results of a mercury intrusion porosimetry (MIP) performed by Fall et al. [8]. An additional factor which should be considered as a contributor to this temperature inversion in the strength of the 50 °C S-CPB is the inversion (drop of the suction) in the suction of the 50 °C S-CPB after 7 days of curing. Lower suction is associated with lower strength of the porous media [19].

Given the fact that a high rate of early backfill strength gain achieved in an economical manner is a key target for all in the mining industry, the results presented above with regards to the impact of initial temperature on the early age strength of S-CPB can have significant practical applications. These results show that a difference in the curing temperature in just the two first days of curing can considerably affect the rate of early age strength gain and the strength of the S-CPB, i.e. influences the stability of the S-CPB structure at early ages. This is obviously linked to the impact on mining cycle time, and thus on the mining efficiency and production. Furthermore, these results suggest that a higher initial backfill temperature would be capable of providing better S-CPB strength with respect to binder content, and potential considerable cost savings are being projected.

3.4. Coupled evolution of the temperature, cement hydration process, self-desiccation and strength of the S-CPB for various initial temperatures

The main mechanical, hydraulic (VWC, suction), chemical and thermal experimental results obtained from the monitoring and testing of the S-CPB samples are summarized in Fig. 11. This figure depicts the simultaneous evolution of the temperature (thermal factor, T), electrical conductivity (gives information about the progress of the cement hydration; chemical factor, C), self-desiccation (negative pore water pressure and VWC are hydraulic factors, H), and the strength (mechanical factor, M) of the S-CPB during its curing as well as the interactions between these factors. An understanding of the interactions among these thermal, hydraulic, chemical and mechanical factors at an early age is important in making rational decisions with regards to the cost-effective design of S-CPB structures and the opening time of the barricades.

From this figure, significant thermal effects on the cement chemical reactions as well as thermo-chemical coupling effects on the self-desiccation and the strength of S-CPB can be observed. As the curing temperature increases (i.e. the initial backfill temperature increases), the peak maxima of electrical conductivity shifts to a shorter time, which indicates an acceleration of the cement hydration process (Fig. 11). Subsequently, these thermo-chemical interactions significantly impact the time at which the suction starts

to considerably increase in the S-CPB; in other words, the time at which the self-desiccation starts or intensifies. It can be clearly seen that the time interval between the peak maxima of electrical conductivity (setting time of the cement) and the start of a significant increase in the suction (intensification of self-desiccation) is approximately 13, 10, 5 and 5 h for S-CPB samples with initial temperatures of 2 °C, 20 °C, 35 °C and 50 °C, respectively (Fig. 11). This indicates a retardation of the self-desiccation as the cement hydration slows down due to lower curing temperatures. Given the fact that self-desiccation decreases the pore water pressure behind the barricades, these findings suggest that the barricades for backfills with higher initial temperatures (35 °C, 50 °C) should be opened earlier than those for backfills with lower initial temperatures (e.g., 2 °C, 20 °C). This is naturally associated with the increase of the speed of mine cycles and thus the mine productivity.

The thermo-chemical coupling effect on the strength of S-CPB is evidenced by the fact that the S-CPB specimens with higher curing temperatures (initial temperatures of 35 °C and 50 °C) show higher strength than those with lower curing temperatures (initial temperatures of 2 °C and 20 °C), when the suction was still constant and had the same value in all of the samples (Fig. 11). From Fig. 11, it can be also noticed that there is strong coupling between the suction (self-desiccation) and the strength development of the S-CPB. It can be observed that, regardless of the initial and curing temperature, there is clear correlation between the intensification of the self-desiccation (increase of suction development) and the increase of the rate of strength gain. This is consistent with the well-known fact that higher suction generally results in higher strength in porous material [19]. These findings suggest that the higher strength and strength increase rate of cemented backfill at early ages associated with higher curing temperature is not only due to the increase of the amount of cement hydration products (e.g., C–S–H, CH) formed and the resultant refinement of the pore structure of the cemented backfill (as discussed earlier), but also to the significant increase of suction because of the intensification of self-desiccation. From the results presented above, it can be concluded that consideration of the coupled thermal (temperature-time history), chemical (cement hydration process) and hydraulic (suction induced by self-desiccation) effects on the strength development of S-CPB at early ages will play a crucial role in the design of stable backfill structures in a cost-effective way as well as in the optimization of backfill cement consumption.

4. Conclusions

In this paper, the experimental results of a study which aims to understand the influence of the various initial temperatures of CPB that contains sodium silicate as the admixture (S-CPB) on the evolution of its early age strength and self-desiccation are presented. The main conclusions derived from this study are summarized as follows.

- The initial temperature of the S-CPB has a significant influence on temperature-time history and its evolution with time; in other words, it significantly affects the curing temperature, to which the backfill structure will be subjected.
- A higher initial temperature leads to more intense and rapid self-desiccation (decrease in volumetric water content, increase in suction) in the S-CPB, particularly at very early ages (≤7 days). This more intense and quicker self-desiccation is due to the fact the S-CPB with higher initial temperatures is subjected to higher curing temperatures at early ages, which speeds up cement hydration, and thus increases the amount of capillary water consumed.
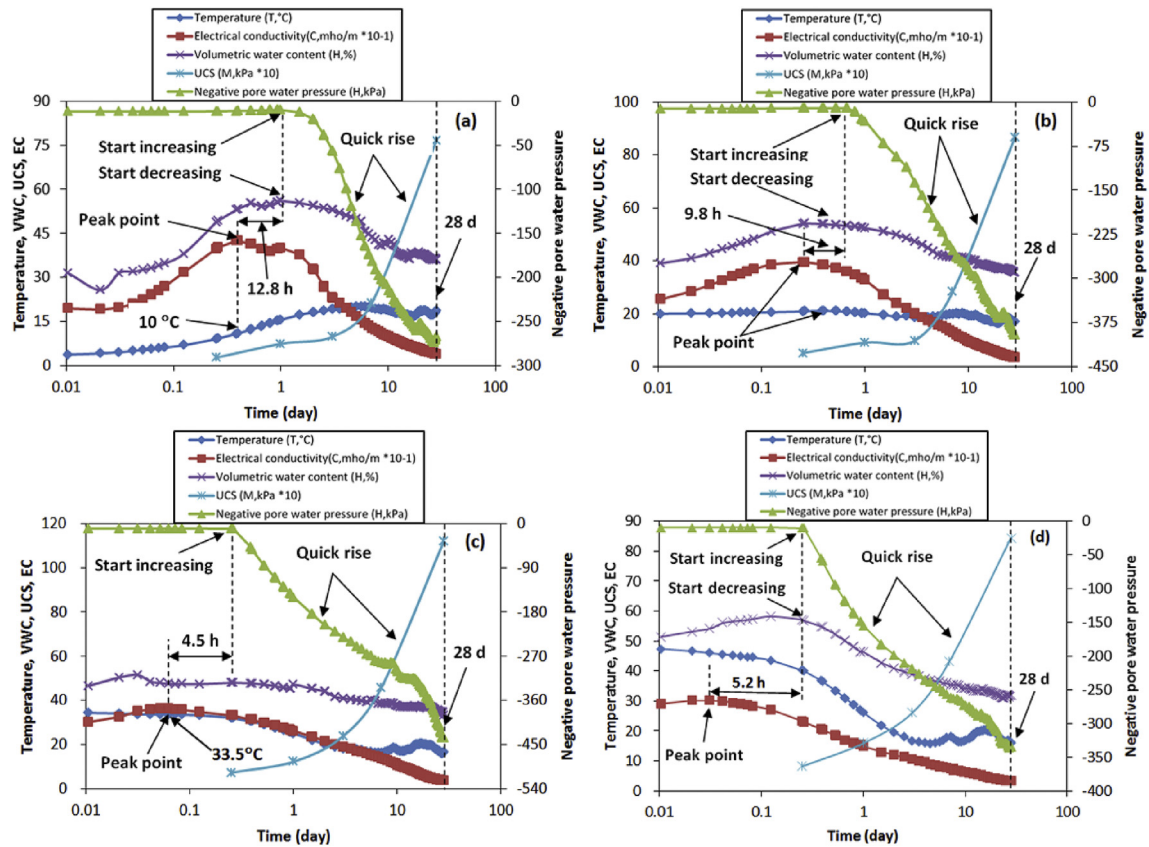
Fig. 11. Coupled evolution of the temperature, cement hydration process, self-desiccation and strength of the S-CPB for various initial temperatures (a) 2 °C, (b) 20 °C, (c) 35 °C and (d) 50 °C.

- The rate of strength gain at an early age and strength are strongly dependent on the initial temperatures of the S-CPB, and thus on its temperature-time history. The S-CPB with a higher initial temperature (35 °C, 50 °C) show higher rates of strength gain and strength at the early ages than the S-CPB with a lower initial temperature (2 °C, 20 °C). This is because a higher initial temperature results in the formation of a greater amount of cement hydration products and the resultant refinement of the pore structure. It also leads to the intensification of the self-desiccation of the CPB which results in higher suction inside the backfilled material. However, it should be emphasized that an overly high initial temperature (50 °C) can lead to the reduction in the compressive strength of S-CPB older than 7 days.
- There is strong coupling among temperature-time history, cement hydration, self-desiccation and the strength of the S-CPB. Consideration of these coupled effects is crucial for the design of stable and economical backfill structures as well as in the optimization of backfill cement consumption and the timely opening of barricades.

This study has provided valuable information for understanding the effect of initial backfill temperatures on strength development and self-desiccation ability.

Acknowledgments

References

[1] O. Nasir, M. Fall, Modeling the heat development in hydrating CPB structures, Comput. Geotech. 36 (7) (2009) 1207–1218.
[2] K.A. Klein, D. Simon, Effect of specimen composition on the strength development in cemented paste backfill, Can. Geotech. J. 43 (2006) 310–324.
[3] J.S. Mahlaba, Elsabé P. Kearsley, A. Kruger Richard, P.C. Pretorius, Evaluation of workability and strength development of fly ash pastes prepared with industrial brines rich in SO₄ and Cl⁻ to expand brine utilisation, Miner. Eng. 24 (10) (2011) 1077–1081.
[4] S. Yin, A. Wu, K. Hu, Y. Wang, Y. Zhang, The effect of solid components on the rheological and mechanical properties of cemented paste backfill, Miner. Eng. 35 (2012) 61–66.
[5] T. Grice, Underground mining with backfill, in: Proceedings of 2nd Annual Summit, Mine Tailings Disposal Systems, Brisbane, Australian, Australasian Institute of Mining and Metallurgy, Carlton South, Australia, 1998, p. 14.
[6] J.F. Archibald, F. Hassani, J. Nantel, D.O. DeGagné, Mine Backfill: An Operator' Guide. An Interactive CD-rom for Mine Operators and Engineers; 2000, 2000.
[7] M. Fall, D. Adrien, J.C. Célestin, M. Pokharel, M. Touré, Saturated hydraulic conductivity of cemented paste backfill, Miner. Eng. 22 (15) (2009) 1307–1317.
[8] M. Fall, J.C. Célestin, M. Pokharel, M. Touré, A contribution to understanding the effect of temperatures on the mechanical properties of mine cemented tailings backfill: experimental results, Eng. Geol. 114 (3–4) (2010) 397–413.
[9] R.M. Rankine, K.J. Rankine, N. Sivakugan, W. Karunasena, M.L. Bloss, Geotechnical characterisation and stability analysis of BHP Cannington paste. ll, in: Proc. of the 15th Intern. Conf. on Soil Mechanics and Geotechnical Engineering, 2001, pp. 1241–1244.
[10] E. Yilmaz, A. Kesimal, B. Ercidi, Strength development of paste backfill simples at Long term using different binders, in: Proceedings of 8th Symposium MineFill04, China; 2004, 2004, pp. 281–285.
[11] M. Fall, M. Benzaazoua, S. Ouellet, Experimental characterisation of the influence of mill tailings fineness and density on the quality of cemented paste backfill, Miner. Eng. 18 (1) (2005) 41–44.
[12] A. Kesimal, E. Yilmaz, B. Ercikdi, I. Alp, H. Deveci, Effect of properties of tailings and binder on the short and long-term strength and stability of cemented

paste backfill, Mater. Lett. 59 (28) (2005) 3703–3709.

[13] N. Abdul-Hussain, M. Fall, Thermo-hydro-mechanical behaviour of sodium silicate-cemented paste tailings in column experiments, Tunn. Undergr. Space Technol. 29 (2012) 85–93.

[14] F. Cihangir, B. Ercikdi, A. Kesimal, A. Turan, H. Deveci, Utilisation of alkali-activated blast furnace slag in paste backfill of high-sulphide mill tailings: effect of binder type and dosage, Miner. Eng. 30 (2012) 33–43.

[15] J. Vergne, Rules of thumb for the hard rock mining industry, in: Hard Rock Miner's Handbook, third ed., McIntosh Engineering Inc., USA, 2000, p. 42.

[16] 1994 Brakebusch, Basics of paste backfill systems, Min. Eng. 46 (1994) 1175–1178.

[17] L. Li, M. Aubertin, Influence of water pressure on the stress state in stopes with cohesionless backfill, Geotechn. Geol. Eng. 27 (1) (2009) 1–11.

[18] M. Helinski, A.B. Fourie, M. Fahey, M. Ismail, Assessment of the self-desiccation process in cemented mine backfills, Can. Geotech. J. 44 (2007) 1148–1156.

[19] D.G. Fredlund, H. Rahardjo, Soil Mechanics for Unsaturated Soils, John-Wiley & Sons Inc., New York, 1993.

[20] A. Ghirian, M. Fall, Coupled thermo-hydro-mechanical–chemical behaviour of cemented paste backfill in column experiments. Part I: Physical, hydraulic and thermal processes and characteristics, Eng. Geol. 164 (2013) 195–207.

[21] A. Ghirian, M. Fall, Coupled thermo-hydro-mechanical–chemical behaviour of cemented paste backfill in column experiments. Part II: Mechanical, chemical and microstructural processes and characteristics, Eng. Geol. 170 (2014) 11–23.

[22] E. Yilmaz, A. Kesimal, H. Deveci, B. Ercikdi, The Factors Affecting the Performance of Paste Backfill: Physical, Chemical and Mineralogical Characterization, First Engineering Sciences Congress for Young Researcher (MBGAK'03), Istanbul, 2003.

[23] A. Kesimal, B. Ercikdi, E. Yilmaz, The effect of desliming by sedimentation on paste backfill performance, Miner. Eng. 16 (10) (2003) 1009–1011.

[24] N. Sivakugan, K.J. Rankine, K.S. Rankin, Geotechnical consideration in mine backfilling in Australia, J. Clean. Prod. 14 (12–13) (2005) 1168–1175.

[25] M. Fall, M. Pokharel, Coupled effects of sulphate and temperature on the strength development of cemented tailings backfills: Portland cement-paste backfill, Cem. Concr. Compos. 32 (2010) 819–828.

[26] S. Huang, K. Xia, L. Qiao, Dynamic tests of cemented paste backfill: effects of strain rate, curing time, and cement content on compressive strength, J. Mater. Sci. 46 (15) (2011) 5165–5170.

[27] D. Wu, M. Fall, C. Si-jing, Coupled modeling of temperature distribution and evolution in cemented tailings backfill structures that contain mineral admixtures, Geotech. Geol. Eng. 30 (2012) 935–961.

[28] M. Fall, S.S. Samb, Effect of high temperature on strength and microstructural properties of cemented paste backfill, Fire Saf. J. 44 (4) (2009) 642–651.

[29] M. Fall, S.S. Samb, Pore structure of cemented tailings materials under natural or accidental thermal loads, Mater. Charact. 59 (5) (2008) 598–605.

[30] D. Wu, M. Fall, S.-J. Cai, Coupling temperature, cement hydration and rheological behaviour of cemented paste backfill structures, Miner. Eng. 42 (2013) 76–87.

[31] N. Abdul-Hussain, M. Fall, Unsaturated hydraulic properties of cemented tailings backfill that contains sodium silicate, Eng. Geol. 123 (2011) (2011) 288–301.

[32] S.G. Vick, Planning, Design and Analysis of Tailings Dams, BiTech Publishers Ltd, 1990.

[33] O. Nasir, M. Fall, Coupling binder hydration, temperature and compressive strength development of underground cemented paste backfill at early ages, Tunn. Undergr. Space Technol. 25 (1) (2010) 9–20.

[34] F. Farkas, F.D. Tamas, Hydration of cements studied by electrical conductivity measurements: II. Quantitative results, in: 9th Int. Baust. Silikattagung, Vol. 1, Ibausil, Weimar, Germany, P. 164, DDr. Sektion, 1985.

[35] L.H. Hansson, C.M. Hansson, Ion conduction in cement based materials, Cem. Concr. Res. 15 (1985) 201–212.

[36] L. Courard, F. Michel, S. Perkowicz, A. Garbacz, Effects of limestone fillers on surface free energy and electrical conductivity of the interstitial solution of cement mixes, Cem. Concr. Compos. 45 (2014) 111–116.

[37] T.J. Williams, D.K. Denton, M.K. Larson, R.L. Rains, J.B. Seymour, D.R. Tesarik, Geomechanics of reinforced cemented backfill in an underhand stope at the Lucky Friday Mine, Mullan, Idaho, US Department of Health and Human Services, 2001 Jul, pp. 1–18. DHHS (NIOSH) Publication No. 2001-138, Report of Investigations 9655.

[38] K.O. Kjellsen, R.J. Detwiler, Reaction kinetics of Portland cement mortars hydrated at different temperatures, Cem. Concr. Res. 22 (1992) 112–120.

[39] S. Sinthaworn, P. Nimityongskul, Effects of temperature and alkaline solution on electrical conductivity measurements of pozzolanic activity, Cem. Concr. Compos. 33 (5) (2011) 622–627.

[40] P. Termkhajornkit, R. Rémi Barbarulo, Modeling the coupled effects of temperature and fineness of Portland cement on the hydration kinetics in cement paste, Cem. Concr. Res. 42 (3) (2012) 526–538. March 2012.

[41] L. Valentini, M. Parisatto, V. Russo, G. Ferrari, J.W. Bullard, R.J. Angel, M.C. Dalconi, G. Artioli, Simulation of the hydration kinetics and elastic moduli of cement mortars by microstructural modelling, Cem. Concr. Compos. 52 (2014) 54–63.

[42] H.F.W. Taylor, The Chemistry of Cements, first ed., vol. 1, Academic Press, London, 1964.

[43] I. Elkhadiri, F. Puertas, The effect of curing temperature on sulphate-resistant cement hydration and strength, Constr. Build. Mater. 22 (7) (2008) 1331–1341.

[44] G. Sant, The influence of temperature on autogenous volume changes in cementitious materials containing shrinkage reducing admixtures, Cem. Concr. Compos. 34 (7) (2012) 855–865.

[45] T.M. Salem, S.M. Ragai, Electrical conductivity of granulated slag-cement kiln dust-silica fume pastes at different porosities, Cem. Concr. Res. 31 (2001) 781–787.

[46] M. Heikal, M.S. Morsy, I. Aiad, Effect of treatment temperature on the early hydration characteristics of superplasticized silica fume blended cement pastes, Cem. Concr. Res. 35 (2005) 680–687.

[47] İ.B. Topçu, T. Uygunoğlu, İ. Hocaoğlu, Electrical conductivity of setting cement paste with different mineral admixtures, Constr. Build. Mater. 28 (1) (2012) 414–420.

[48] A. Noumowé, Effet des hautes températures (20 °C–600 °C) sur le béton, PhD thesis, Institut National des Sciences Appliquées, 1995.

[49] Y. Anderberg, Spalling. phenomena of HPC and OC, in: Proceedings of the International Workshop on Fire Performance of High-strength Concrete, NIST, Gaithersburg 1997, 1997.

[50] Q. Zhou, F.P. Glasser, Thermal stability and decomposition mechanisms of ettringite at <120°, Cem. Concr. Res. 31 (9) (2001) 1333–1339.

[51] J.I. Escalante-Garcia, Nonevaporable water from neat OPC and replacement materials in composite cements hydrated at different temperatures, Cem. Concr. Res. 33 (11) (2003) 1883–1888.