



## Coupled effects of sulphate and temperature on cemented tailings materials

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### ABSTRACT

Increasing using of Cemented paste tailings (CPT) in underground mining combined to the fact that every single mine or CPT structure is unique regarding its temperatures conditions require the need to clarify and quantify the coupled effect of temperature and sulphate on the mechanical and microstructural properties of cemented paste tailings (CPT). Hence, the main objective of this study was to use an experimental approach to study the combined effect of temperature and sulphate on the strength development and microstructure (mineralogical composition of the hardened cement paste) of CPT. About 200 CPT specimens with various initial sulphate contents (0, 5000, 15 000, 25 000 ppm) and cured at different temperatures (0°C, 25°C, 20°C, 35°C, and 50°C) were tested at different curing times (28, 90, 150 days). The results show that the coupled effect of temperature and sulphate has a significant impact on the strength and microstructure of CPT. The curing temperature can have a positive or negative impact on the resistance of CPT to sulphate attack. The aforementioned coupled effect is an important that should be considered in the design of cost-effective and durable CPT structures.

### RÉSUMÉ

L'utilisation de plus en plus importante des remblais en pâte cimentés (RPC) dans les mines souterraines, combinée au fait que chaque mine ou chaque ouvrage de remblai est unique en ce qui concerne ses conditions thermiques, rendent nécessaire la clarification et la quantification de l'effet couplé de la température et du sulfate sur les propriétés mécaniques et microstructurales du RPC. C'est ainsi, le principal objectif de cette étude est, en utilisant une approche expérimentale, d'étudier l'action combinée du sulfate et de la température sur le développement de la résistance du remblai et sur sa microstructure (composition minéralogique de la matrice cimentaire). Environ 200 échantillons de RPC avec différentes teneurs en sulfate (0, 5000, 15 000, 25 000 ppm) et curés à différentes températures (0°C, 25°C, 20°C, 35°C, 50°C) ont été testés à différents temps de cure (28, 90, 150 jours). Les résultats montrent que le couplage entre la température et le sulfate a un effet significatif sur la résistance et la microstructure du RPC. La température peut avoir un effet positif ou négatif sur la résistance du RPC à l'attaque sulfatique. Le couplage mentionné ci-dessus est un important facteur à considérer dans le design optimal et durable des ouvrages de RPC.

### 1 INTRODUCTION

During the last decade, cemented paste tailings (CPT), a relatively new cemented tailings material, has become increasingly popular in underground mining operations around the world. In addition to its ground support contribution during mining operations, CPT is now an indispensable tailings management method. One of the most important quality criteria for the hardened cemented paste tailings is mechanical stability or its mechanical properties at a given time. Indeed, the CPT structure must remain stable during the extraction of adjacent stopes to ensure the safety of the mine workers and to avoid ore dilution. The most important parameters used in the practice to judge the mechanical stability of CPT structure is the uniaxial compressible strength (UCS). Another important quality criterion of the CPT is its durability, especially its resistance to internal sulphate attack. Internal sulphate attack is one of the major factors that can cause the deterioration of CPT structures. The sulphate mostly comes from the mixing water and/or from the oxidation of sulphides minerals contained in the tailings. The sulphate attack not only decreases the mechanical stability of the CPT structures, it may also

contribute to the development of cracks in the CPT; this may result in the generation of acid mine drainage, and of a potentially environment-hostile and onerous leachate.

Until now, despite the tremendous progress made in understanding the durability of CPT, especially its resistance to internal sulphate attack (e.g., Hassani et al. 2001, Fall and Benzaazoua 2005), all these studies have mostly only investigated the isolated effects of sulphate on the strength of CPT. However, it is well known that in sulphate attack on cementitious materials, the changes or deterioration of material properties and accumulation of damage are not only caused by the isolated effects of chemical loads (sulphate effect), but the coupled effects of sulphate (chemical loads) and temperature (thermal loads) play a highly significant role. These chemical and thermal factors are commonly viewed individually in CPT technology, when, in fact, they are strongly interrelated or coupled. There is a crucial need to increase our understanding of the coupled effect of sulphate and temperature on the strength of CPT as shortly explained below.

Indeed, the progressive depletion of ore available at shallow depths in a number of underground mines in Canada and around the world means that underground

mining operations are increasingly being carried out at greater depths and therefore at higher temperatures (due to the geothermal gradient). The exposed rock mass is the primary heat load source in any deep level mining operation. Rock temperatures measurements carried out (Rawlins and Phillips 2001) in a South African deep gold mine have revealed that rock temperature has the following average values with depth: 37°C at 2,000 m, 48°C at 3,000 m, 60°C at 4,000 m and 70°C at 5,000 m. Similarly, the geothermal gradients in Canada are usually such that the ambient temperature of the rock mass increases about 1°C for every increase of 100 metres of depth. In the Sudbury Basin, it has been estimated that the temperatures that will be encountered at 3,000 metres will be 46°C (Udd, 2006). These hot temperatures, associated with deep mining infer that one of the most challenging engineering tasks in deep mine ground support, is the design of stable and durable CPT. Also, taking a futuristic view where production would take place at a vertical rock breaking depth of 4,000-5,000 m, the significance of the coupled effect of deep mine temperatures and sulphate on CPT strength and durability cannot be neglected or underestimated.

Furthermore, since the CPT structures are often very large (several tens of meters in height and width), binder hydration can release (depending on the cement content) a significant quantity of heat inside the cemented backfill structure, as shown in field studies conducted by Williams et al. (2001) and modeling studies performed by Othman and Fall (2008). This is an additional heat load source in backfill operations. Williams et al. (2001) demonstrated that after only a few days (~ 4 days) of curing, the backfill temperatures increased up to approximately 50°C. These results were also confirmed by the modeling studies (Othman and Fall 2007).

Furthermore moderate heat can be added to the mixing water of CPT to increase its early strength as reported by Fall et al. (2007). Higher early age strength of CPT can have significant practical applications. Indeed, a high rate of early backfill strength gain achieved in an economical manner is a target for all in the mining industry. Early strength gain is of special importance for opening of the barricades and thus, for the reduction of the mining cycle time thus increased mining efficiency, and for speeding up production. This is obviously associated with economical benefit for mines. Furthermore, high early age strength gain may play a significant role in reducing the potential for CPT liquefaction at early age of cure and thus decreasing barricade failure risk. This is because the binder hydration product is able to create bonds between individual tailing particles of the CPT and provide strength. However, the impact of temperature on the sulphate effect on UCS of CPT at early age is unknown.

The synthesis presented above shows that the backfill or mine temperature is an important factor that has been considered in the studying of the effect of sulphate on CPT's strength or the resistance of CPT to sulphate attack (durability of CPT). These chemical (sulphate) and thermal (temperature) factors should not be studied individually, but have to be coupled in order to better evaluate the resistance of CPT to sulphate attack or the effect of sulphate on CPT's UCS in any backfill or mining thermal conditions. Consequently, a solid knowledge of

the coupled effect of sulphate and temperature on strength of CPT is vital for an economical, safe and durable design of CPT structures.

However, to the best knowledge of the authors, there is no study on the coupled effect of sulphate and temperature on the strength of CPT. For these reasons, research programs were carried out by the above authors to thoroughly investigate the sulphate effect on CPT's strength under various thermal loadings.

The main objectives of this paper are:

- to present the results of the coupled effect of sulphate and temperature on the strength of CPT at early and advanced ages;
- to develop an understanding of the performance of various types of CPT when exposed to sulphate attack under various thermal conditions.

## 2 EXPERIMENTAL PROGRAM

### 2.1 Materials

Artificial tailings and binder reagents, and waters with different amounts of sulphate were used to prepare the CPT specimens.

**Tailings:** Artificial tailings (ground silica, AT) were used. This allowed preparing CPT samples with controlled mineralogical and chemical (e.g. sulphate) composition as well as with controlled physical characteristics (grain size). The AT shows the same particle size distribution as the average of 9 Canadian mine tailings. The AT is almost exclusively made from 99.8wt% SiO<sub>2</sub>. The AT has 40% fine particles (particles with diameters lower than 20 µm) and can be classified as medium tailings. Further physical and chemical properties of the AT are given in Tables 1 and 2. The AT is well-graded (Table 1) and free of sulphide minerals (Table 2).

Table 1. Physical properties of the tailings used

Element	G <sub>s</sub>	D <sub>10</sub>	D <sub>30</sub>	D <sub>50</sub>	D <sub>60</sub>	D <sub>90</sub>	C <sub>u</sub>	C <sub>c</sub>
unit	-	µm	µm	µm	µm	µm	-	-
SI	2.7	1.9	9.0	22.5	31.5	88.9	16.2	1.3

Table 2. Main chemical elements in the tailings used

Element	Al	Ca	Si	Fe	Na	Pb	S	K
unit	wt %							
AT	0.	<0.0	99.8	<0.0	<0.0	0.0	0.0	0.0

**Mixing water:** Distilled water was used as basic waters. Specific amounts of sulphate concentrates were added to specific volume of distilled water to create mixing waters with well know sulphate concentrations. The prepared mixing waters are shown in Table 3.

Table 3. Sulphate content of the mixing waters used

Water	W <sub>0</sub>	W <sub>1</sub>	W <sub>2</sub>	W <sub>3</sub>
Sulphate (ppm)	0	5000	15000	25000

**Binder reagents:** Portland cement type I (PC I) in a weight proportion of 4.5% was used as binding agent.

## 2.2 Mixing procedures and mix proportions

A total of about 200 CPT specimens, with constant binder content (4.5%), w/c ratio (7.5), and tailing types were prepared. The tailing materials, binders and water were mixed and homogenized for about 7 min. to produce the desired CPT mixtures. The CPT mixes produced were poured into curing cylinders, 5 cm in diameter and 10 cm high. The prepared moulds were then sealed (this will avoid the evaporation of water) and cured in environmental chambers with controlled temperature at 2°C, 20°C, 35°C and 50°C for periods of 28, 90 and 150 days.

## 2.4 Testing methods

In order to evaluate the strength of the CPT specimens and the mineralogical composition of the cement paste of CPT, the following tests or analyses were performed:

- Uniaxial compressive tests, according to ASTM C 39, were performed on the CPT specimens after different curing times for the different studied temperatures. The load was applied at a slow rate (1 mm/min).
- X-Ray analyse were also conducted to evaluate the coupled effect of temperature and sulphate on the mineralogical composition of the hydration products of CPT samples. X-ray analyses were performed on cement pastes of CPT with various sulphate contents (0, 5000, 15 000, 25 000 ppm) cured at different temperatures (2°C, 20°C, 35°C, 50°C) and times (28, 90, 150 days).

## 3 RESULTS AND DISCUSSION

### 3.1 Coupled effect of sulphate and temperature on the strength of CPT at early ages

Figures 1 to 2 show the evolution of the 28 days uniaxial compressive strength (UCS) of the CPT as well as the normalized (with the UCS of CPT containing 0 ppm sulphate) UCS development of CPT for different curing temperatures and sulphate contents. From these figures, it is clear that there is a relationship between the sulphate content, the temperature and the 28 days strength of CPT as well as between the sulphate-temperature interactions and the 28 days strength of CPT.

Regardless of the sulphate content, higher curing temperature resulted in higher strength of CPT as shown in Figure 1. This is because, high temperatures accelerate the dissolution of the anhydrous clinker phases and thus the cement hydration process. For this reason, CPT cured at higher temperatures gains strength faster and is more resistant for curing times of 28 days as reported by Fall and Samb (2007) and Fall et al. (2007). Indeed, the amount of hydration products (e.g. CSH, CH, calcite) increases with increasing temperature as demonstrated by previous studies of thermal analysis performed on CPT (Fall and Samb 2006, Fall et al. 2007). This is beneficial for CPT strength gain, since CSH is considered to be the major binding phase in hardened cement (Taylor 1990, Gani 1997). Figure 1 shows also that the strength of CPT samples cured at cold temperature (2°C) is lower than that of samples cured at higher temperatures. This can be attributed to the

inhibition of the cement hydration at low temperatures as also reported by (McIntosh, 1956). At low curing temperatures, the cement hydration rate is slow, thereby leading to lower rate of precipitation of hydration products and thus to the production of lower amount of CSH gel in early age (Lothenbach, 2007). It can be also observed, that regardless of the curing temperature, CPT samples with 5000 ppm and 15 000 ppm have higher UCS values than those without sulphate. This could be attributed to the precipitation of secondary hydrated minerals (as gypsum and ettringite) within the CPT. This contributes to the hardening mechanism of CPT. These precipitates contribute to fill the void spaces or micropores within the CPT. This also results in a decrease in its internal porosity, thereby increasing the CPT strength. These results are in agreement with previous experimental investigations (SEM observations) performed on CPT by Fall and Benzaazoua (2005). However, CPT samples with higher sulphate content (25 000 ppm) show lower 28 days UCS values than those with 5000 and 15 000 ppm of sulphate, and those without sulphate regardless of the curing temperature (except at 35°C). This can be explained by the fact that the high sulphate content (25 000 ppm) may have inhibited the binder hydration, thereby resulting in lower UCS. This assumption is fully supported by the results of previous studies that revealed that high sulphate content strongly inhibits the early hydration of C<sub>3</sub>A (Fall and Benzaazoua 2005) as well as by studies performed on the hydration of cement paste containing natural anhydrite (Tzouvalas et al., 2004).

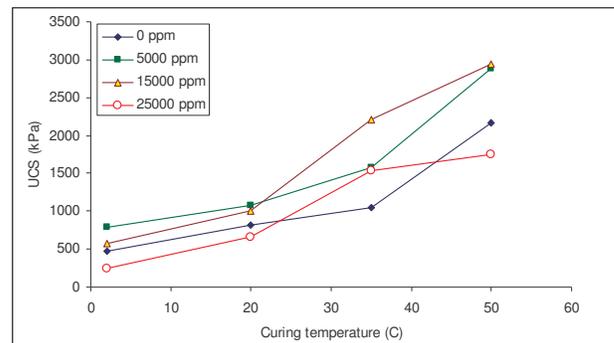


Figure 1. Coupled effect of temperature and sulphate on the 28 days UCS of CPT

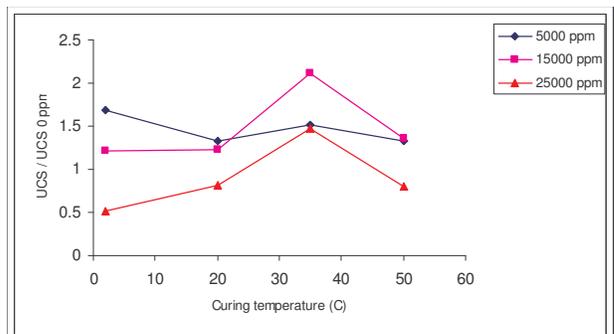


Figure 2. Coupled effect of temperature and sulphate on the relative 28 days UCS of CPT (strength normalized with the UCS of 0 ppm CPT)

From figures 1 and 2, it's also obvious that the effect of sulphate on CPT's strength at 28 days is strongly dependent on the curing temperature. It can be seen that, as the curing temperature increased, the positive effect (increase in strength) of sulphate content of 5000 and 15 000 ppm on the strength of CPT is generally higher. This may be explained by the fact that higher temperature is associated with faster binder hydration and thus a production of higher amount of calcium hydroxide (CH). Hence, more gypsum will be formed through the reaction of sulphate with CH. This gypsum will contribute to the hardening and the reduction of the porosity of the CPT, thereby increasing its strength. However, for a temperature of 50°C, CPT samples with initial sulphate content of 5000 ppm have the same strength as those with initial sulphate content of 15000 ppm (Figures 1 and 2). This may indicate a decrease of the strength gain rate with higher temperatures for an initial sulphate concentration of 15 000 ppm (comparing to sulphate concentration of 5000 ppm). This can suggest, that for an initial sulphate content of 15 000 ppm, a curing temperature of 50°C starts to have negative effect on early age strength (28 days) of CPT. This may be due to the fact that higher curing temperature leads to higher absorption of sulphate by calcium silicate hydrate (CSH), thereby leading to weaker CSH gel (the strength of Portland cemented materials is mainly controlled by CSH). This assumption is fully supported by the results of experimental studies performed by Barbarulo (2002). These studies reported that sulphate binding to C-S-H increases and is faster as the curing temperature increases. This means, it should be admitted that competition for sulphate ions exists between three reactions; (i) the fixing of sulphate ions by CSH gel; (ii) the reaction of sulphate ions with CH to form gypsum; (iii) the reactions of sulphate ions and C<sub>3</sub>A to generate ettringite. This competition for sulphate ions is experimentally demonstrated by previous studies performed on CSH samples synthesized in laboratory (Divet and Randriambolona, 1998). A higher absorption of sulphate ions by CSH will reduce the sulphate concentration in the cement paste solution and thereby limiting the amount of sulphate ions available to form gypsum as well as to generate ettringite (to increase the strength of CPT at early age through the mechanism explained above). In addition to the absorption of sulphate ions by CSH gel, the high temperature may have contributed to the destabilization of ettringite in CPT system, since the solubility of ettringite increases with increasing temperature (Perkins and Palmer 1998, Barbarulo 2002). However, the temperature induced-higher absorption of sulphate by CSH gel can not be considered as the only major parameter responsible for a decrease of the strength of CPT under coupled influence of temperature and sulphate. Indeed, from figure 1, it can be observed that the evolution of the curve of temperature vs strength of CPT specimens with 25 000 ppm is relatively different than that of CPT with lower initial sulphate content. The UCS of the 25 000 ppm CPT specimens is lower than that of CPTs with lower sulphate content at 50°C, and higher only than that of the CPT specimen without sulphate at 35°C. This may suggest that not only the temperature induced higher absorption

of sulphate of CSH gel is the cause of this low strength of 25 000 ppm CPT, but also the high concentration of sulphate should be considered as additional cause. It has also been reported (e.g. Divet and Randriambolona 1998) that the increase of sulphate concentration in cement paste solution leads to higher absorption of sulphate in CSH gel, thereby resulting in less formation of expansive mineral (e.g. gypsum). This high absorption of sulphate by CSH gel could have led to a CSH gel of lower quality, thereby affecting negatively the CPT's strength. From figures 1 and 2, it can be also seen that the inhibition of the cement hydration by high sulphate content (25 000 ppm) is severer at cold temperature. This is because of the slow binder hydration rate associated with cold curing temperature.

The results of the coupled effect of sulphate and temperature on the UCS of CPB at early age presented above, suggest that, for the studied CPTs, for a curing temperature  $\leq 35^{\circ}\text{C}$ , an initial sulphate content up to 15 000 ppm seems to have positive effect on the early age strength of CPB. This finding can have significant practical application regarding the speeding up of mining process, the reduction of liquefaction susceptibility of CPT structures and the cost-effective design of CPT structures as discussed in section 1 already. However, although the early age strength is a key parameter in cost-effective design of CPT structure, the strength of CPT at advanced ages is also highly important to judge the long term stability and durability of CPT. This makes evident the importance of answering the following question: How will the coupled effects of sulphate and temperature affect the strength of CPT at advanced ages?

### 3.2 Coupled effect of sulphate and temperature on the strength of CPT at advanced ages

Figure 3 shows the coupled effect of sulphate and temperature on the 150 days uniaxial compressive strength of the CPT. While figure 4 illustrates the normalized (with the UCS of CPT containing 0 ppm sulphate) 150 days strength of CPT for different curing temperatures and sulphate concentrations. Again, as in early age (28 days), it can be seen, that higher curing temperature is associated with higher 150 days strength of CPT (except for 25 000 ppm CPT; it will be discussed below). The reasons of this strength increase are the same as those previously discussed in section 2. Furthermore, as noticed in early age strength development of CPT, as the sulphate content increased (up to 15 000 ppm), the strength of CPT increased. This can be attributed to the precipitation of expansive minerals such as ettringite and gypsum in the pores of CPT that contribute to its hardening as explained above. Since CPT has a high porosity and large capillary pores (Fall et. al 2004), it can accommodate a large quantity of expansive mineral without any physical damage induced by the pressure developed by the expansive minerals.

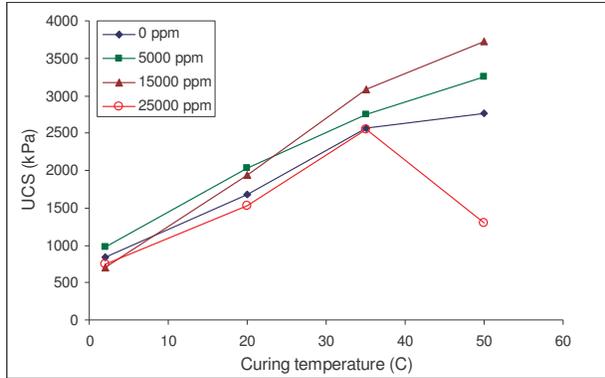


Figure 3. Coupled effect of temperature and sulphate on the relative 150 days UCS of CPT CPT (strength normalized with the UCS of 0 ppm CPT)

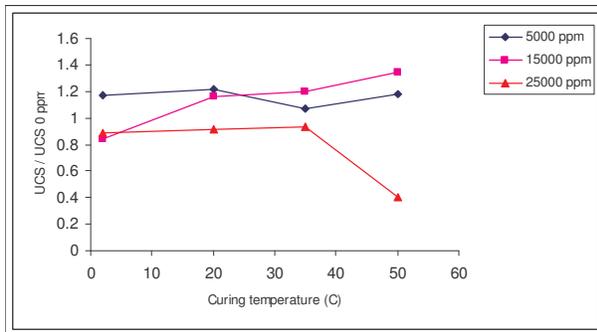


Figure 4. Coupled effect of temperature and sulphate on the 150 days UCS of CPT

From figures 3 and 4, it is also clear that the effect of sulphate on the strength of CPT at advanced ages is significantly affected by the curing temperature. It can be observed that the inhibition of the cement hydration by high sulphate contents (15 000 and 25 000 ppm) is severer at low temperature (2°C) and becomes lower with increasing curing temperature. At 2°C curing temperature, the strengths of CPTs with initial sulphate contents of 15 000 and 25 000 ppm are lower than those of the specimens with 5000 ppm sulphate content and without sulphate. At 20°C curing temperature, only the strength of CPT containing initially 25 000 ppm sulphate is lower than that of CPT without sulphate. This indicates a less severe inhibition of the cement hydration. This inhibition effect of the sulphate is also demonstrated by the results of X-ray analyses performed on CPT's cement paste presented in Figure 5. This figure shows the XRD pattern of 150 days old hardened cement paste containing initially 25000 ppm sulphate and cured at 50°C. The XRD data confirm the presence of the clinker phases  $C_3S$  and  $C_2S$  in the CPT specimen with 25000 ppm sulphate. These clinker phases were not detected by the X-ray analyses conducted on the 150 days old hardened cement paste without sulphate as shown in Figure 6.

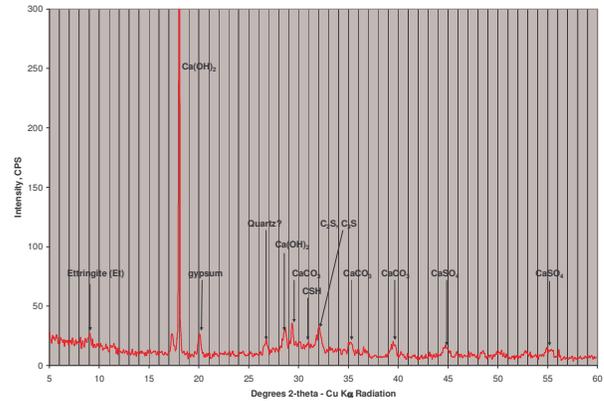


Figure 5. XRD patterns of 150-day-old hardened cement pastes with a high water-to-cement ratio ( $w/c = 2$ ) cured at 50°C and with initial sulphate content of 25 000 ppm

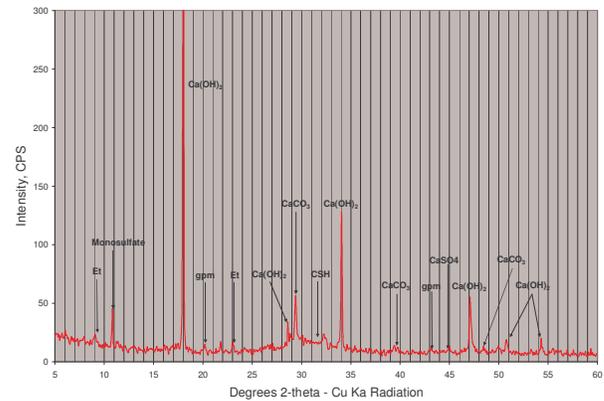


Figure 6. XRD patterns of 150-day-old hardened cement pastes with a high water-to-cement ratio ( $w/c = 2$ ) cured at 50°C and containing no sulphate

It is interesting to notice from Figures 3 and 4 that the CPT with 25 000 ppm sulphate content shows a high drop in strength at 50°C, while the specimens with 15 000 ppm and 5000 ppm show no strength reduction at the same curing temperature. The CPT specimen without sulphate is also not affected by strength loss at 50°C. This suggests that the observed strength drop is not related to the well know mechanism (cross-over effect) of high curing temperature induced strength loss of cemented materials at advanced age. The origin of this drop of the strength of CPT with 25 000 ppm and cured at 50°C could be the combined effect of following mechanisms: (i) the increase of curing temperature increases the amount of sulphate absorbed by the CSH gel; and (ii) the high sulphate concentration is also associated with higher sulphate absorption by CSH. The binding of sulphate to CSH gel lead to the formation of CSH gel of lower quality, thereby resulting in decrease of CPT's strength. The absorption of sulphate ions may have strongly limited the availability of sulphate to form gypsum or ettringite. This can indicate that the observed decrease in strength at 50°C should not be attributed to the negative effect of the expansive mineral (ettringite and gypsum) as commonly

observed in sulphate attack on conventional cemented materials cured at room temperature. This assumption is confirmed by the analyse of the figures of X-Ray performed on hardened cement paste of CPT containing 25 000 ppm sulphate (Figure 5) and 0 ppm sulphate (Figure 6) and cured at 50°C. From these figures, it can be observed that the intensity of gypsum and ettringite peak is almost the same for both figures. This indicates there is no increase of the quantity of expansive minerals formed with higher sulphate content (from 0 to 25 000 ppm). Furthermore, since the stability of ettringite decreases with higher temperatures, the destruction of ettringite phases at high temperatures (50°C) should be considered as additional cause of low amount of ettringite observed in the samples containing 25 000 ppm and cured at 50°C. This assumption is fully supported by the X-ray detection of calcium monosulphate in CPT sample presented in Figure 6. Indeed, increased temperatures promote the formation of calcium monosulphoaluminate, while the stability of ettringite decreases (e.g. Lothenbach et al. 2007). Similar observations were made by Thomas et al. (2003) on Portland cement paste. Their study revealed that ettringite is more stable than monosulfate at ambient temperatures. As the temperature increases, monosulfate eventually becomes the favored phase due to its smaller enthalpy of reaction. From the results presented above, it can be deduced that for the studied CPT materials, the sulphate will positive contribute to the strength of CPT at advance ages for initial sulphate concentrations up to 15 000 ppm and a curing temperature up to 35°C.

#### 4 SUMMARY AND CONCLUSION

This paper presents the results of a study which aims at evaluating the coupled effect of sulphate and temperature on the strength of CPT. Several CPT mixtures with various sulphate contents (0, 5000, 15000 and 25000 ppm) were cured at different temperatures (2, 20, 25, 35, and 50°C). Mechanical tests and microstructural analyses of these CPT mixtures provide valuable information on the effects of sulphate and temperature on strength and microstructure (mineralogical composition of the hardening and hardened cement paste) of CPT. It was demonstrated that the sulphate effect or attack on CPT is strongly affected by the curing temperature. Higher curing temperature can have positive or negative effect on the resistance of CPT to sulphate attack depending on the temperature value and sulphate content. The coupled effect of temperature and temperature is an important factor that has to be considered in the design of cost-effective and durable CPT structures.

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