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Deformation behaviors of cemented backfill using sulphide-content tailings

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Abstract

The deformation properties of cemented tailings backfill (CTB), especially prepared from sulphidic tailings, are valuable for the estimation of design parameters for underground stope filling as well as for numerical simulation. In this paper, the effect of curing time, sulphur content, cement dosage and solids concentration on the free expansion ratio of CTB specimens was investigated using an invented lab apparatus called "free expansion measuring instrument". The backfill recipe (sulphur content, binder dosage and solids concentration) and curing days had significant influences on the expansion performance of the CTB. The results indicated that with the change of sulphides, the free expansion ratio (FER) showed no obvious growth at the curing of 28 days. However, after the curing of 120 days, FER increased from 4.96% (S: 4%) to 9.61% (S: 20%). As the solids concentration increased, the FER also rised. And this growth was distinctive only when the solids concentration is relatively high (65~75%) and at the long-term curing time (120 days). The proportions of binder and the obtained FER clearly show a sub-linear and proportional relationship. Increasing the amount of binder (8~16%) could not restrict the increment of expansion ratio, and ultimately the CTB specimens would generate cracks and collapse.

1 Introduction

Cemented tailings backfill (CTB) is a heterogeneous material in which tailings are bonded together by the hardened cement. Its components (tailings, water, cement) are combined and mixed in a plant usually located on the surface and ground mine (Argane et al., 2015; Fall et al., 2008; Pokharel and Fall, 2013). CTB is potentially one of the best practical approaches for the management of process tailings since it offers significant environmental, technical and economic benefits. These include the alleviation of the environmental impact from potentially hazardous mill tailings (e.g. sulphidic tailings, in particular) by their safe disposal into the underground (up to 60~75% of the plant tailings), the support of underground stopes to provide a safe working environment and to minimize surface subsidence, as well as the reduction of the tailings disposal and rehabilitation costs (Ercikdi et al., 2015; Fall et al., 2010; Ke et al., 2015; Yin et al., 2012). A considerable number of metals (such as Cu, Pb, Au, and Zn) can be mined from sulphidic rich ore deposits. The majority of the minerals in sulphidic tailings is iron sulphides such as pyrite (FeS_2), arsenopyrite (FeAsS) and pyrrhotite ($Fe_{1-x}S$) (Kiventerä et al., 2016). The sulphides would generate acid in the presence of water and oxygen, hence resulting in the migration of heavy metals (Cu, Ni, Fe, and Zn) (Nason et al., 2014). An effective disposal of backfill materials is to place them into the underground using sulphur-bearing tailings as aggregate. The most commonly used binder material is the ordinary Portland cement (OPC), which has certain limitations when using sulphidic rich mine tailings (Kesimal et al., 2004). The presence of sulphide minerals results in sulphate attack which may decrease the strength and lead to an expansion of the CTB material. Sulphate attack can occur rapidly during curing and depends on the binding agents used. The reaction involves dissolution of hydrated calcium phases followed by the formation of expansive phases, resulting in the degradation of the backfill mechanical properties (Benzaazoua et al., 1999). Meanwhile, the accumulation of expansive phases also leads to deformations such as expansion in most cases. These issues have all affected the long-term stability of the mine (Benzaazoua et al., 2002; Kiventerä et al., 2016). The quality and performance of CTB samples are greatly affected by both intrinsic and extrinsic factors. Among them, intrinsic factors include all the parameters of tailings, cement and water, and their interactions during curing while extrinsic factors are those induced by the stope dimension, backfill-rock interaction, placement conditions, curing temperature and time, self-weight or time-dependent consolidation, and drainage or bleeding of excess water (Yilmaz et al., 2015). The unconfined compressive strength (UCS) of CTB samples is the most frequent property used to assess the mechanical performance of CTB structures because UCS testing is relatively inexpensive and quick, and can be easily incorporated into the routine quality control programs in the mine (Li and Fall, 2016; Sun et al., 2017). Nevertheless, the UCS test is a one-time destructive experiment with no repeatability. The results of the strength tests on the specimens with micro cracks are very discrete, which significantly reduces the accuracy of the experiment. Therefore, there is a lack of reliability to evaluate the mechanical properties of backfill only by UCS. Some investigation about the mechanical performance of backfill-rock interaction has been conducted by backfill direct shear property test (Koupouli et al., 2016). Arching effect defined as the transfer of pressure from a yielding mass of soil onto the adjoining stationary parts (Cui and Fall, 2016; Cui and Fall, 2017; Fahey et al., 2009) has been researched. It is clear that the expansion behavior of the filling body is an important role effecting the interfacial strength and the arching effect. Furthermore, in addition to the important role of UCS in underground stopes supporting, the deformation behavior is also a key aspect of major interest. Wu et al. (2017) propose that, in inspecting the stability of the backfill body with an empty lateral, the expansion behavior of the backfill should be seriously considered. Huang et al. (2016) have studied the deformation behavior of controlled low strength materials (CLSM) at different temperatures in the early curing time to improve the quality of backfill. Nevertheless, few studies have investigated the deformation behavior of CTB materials, especially the short- and long-term expansion. Thus, this paper describes the invented lab apparatus and presents the results of the investigation into the influence on the recipe factors (binder/cement dosage and solids concentration) and sulphur content and their interactions on the evolution of the expansion ratio of CTB in the short and long term.

2 Materials and methods

2.1 Tailings and binders

Assessment of the backfill expansion potential of the separated fractions (sulphide concentrate and low sulphidic tailings) was performed to demonstrate the expansion of the backfill specimens produced from sulphidic tailings. The sulphide concentrate was used to produce various sulphur grade CTB mixtures. The sulphide concentrate and tailings sample utilized in this study were obtained from an underground copper mine located in Central China. The binder used with the reactive tailings was the one currently used at the mine, the ordinary Portland cement (Benzaazoua et al., 2008).

Particle size analysis of the tailings indicated that the tailings can be classified as a medium-sized tailings material (Table 1) (ASTM, 2011). Chemical composition of the tailings and binders were performed according to TS EN 196-2 (EN, 2002). The sulphur content of the tailings sample determined was 3.64wt% (Table 1) and the major sulphide mineral contained was pyrite. Further detailed physical, chemical and mineralogical properties of the tailings used in this study can be found elsewhere (Ercikdi et al., 2010). The cement (P.C 42.5, according to Chinese standard: GB175-2007) was used in this study. The chemical composition of the binder can be found in Table 1.

Table 1 Chemical and physical properties of binders and tailings used

Oxide composition	SiO_2 (%)	Al_2O_3 (%)	Fe_2O_3 (%)	CaO (%)		MgO (%)	K_2O (%)	S (%)	Remarks
Tailings	21.58	8.69	24.99	0.51		3.05	0.875	3.64	6.8%FeS_2
sulphide concentrate	5.42	1.64	11.87	0.14		0.207	0.098	41.52	77.9%FeS_2
OPC	19.32	6.14	3.15	59.24		1.24	1.12	-	-
Physical properties	G_s (-)	>140 μm (%)	>95 μm (%)	D_{10} (μm)	D_{30} (μm)	D_{80} (μm)	C_u (-)	C_c (-)	%Fine (<20 μm)
Tailings	2.71	32.33	60.14	44.31	79.00	169.95	2.71	1.66	5
sulphide concentrate	4.65	8.9	36.56	40.88	65.29	115.66	2.59	1.22	2.5
OPC	2.98	0	2.33	-	-	-	-	-	-

Note: G_s: specific gravity; C_u: coefficient of uniformity (D_{60}/D_{10}); C_c: coefficient of curvature ($(D_{30})^2/(D_{10}\times D_{60})$)

2.2 Expansion rate cell apparatus

To investigate the expansion properties of CTB cured without constraints, an innovative lab apparatus called FEMI (free expansion measuring instrument) was developed, inspired by a simple set-up for obtaining the soil expansion rate in the Specification of soil test and Standard for soil test method (China, 1999; China, 1999). A schematic view of the FEMI is presented in Figure 1 (Yilmaz et al., 2015a). The apparatus consists of three main components: i) a pedestal that marks the location of specimen to guarantee the head touching the same point of specimen in each test; ii) a support rod that is equipped with a crossbeam featuring a high flexural strength to improve the reliability of the device; iii) a top displacement rod for dial gauges to obtain the CTB sample sizes over designed curing time. The dial gauge has a gauge length of 30 mm and is accurate to within 0.01 mm.

Each specimen is marked with A, B, and C on the three mutually orthogonal faces. Three sets of experimental data, H_A, H_B and H_C (the height of specimen placed at the same location and with A, B, and C face parallel to the FEMI pedestal, respectively) were obtained. Dimensions of the cubic specimens (H_d) can be gained using Equation (1).

$$H_d = \sqrt[3]{H_A \cdot H_B \cdot H_C} \tag{1}$$

In cementitious materials, the overall shrinkage is primarily attributed to the drying effect (drying shrinkage) and chemical reaction between water and cement (chemical shrinkage or non-drying shrinkage) (Chen et al., 2010). In view of a previous experimental study (Chen et al., 2010; Deng et al., 2012), the shrinkage ratio of cemented tailings materials would be stable over 7 days. In this study, the dimension of curing for 7 days (H_{7days}) was defined as initial size. The free expansive ratio (FER_d) of backfill specimens cured over design days can be obtained using Equation (2).

$$\mathrm{FER}_d = \frac{H_d - H_{7days}}{H_{7days}} \tag{2}$$

Where:

H_d = a dimension of curing some days.

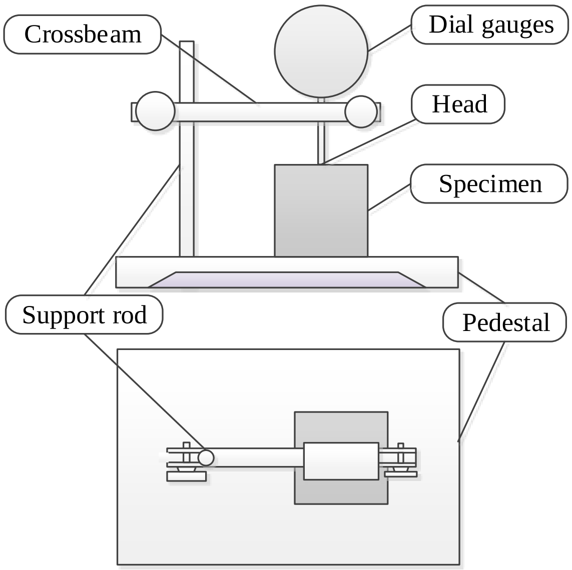

Figure 1 Schematic diagram of the expansion rate cell apparatus

2.3 Experimental design

Because of effects of sulphate on hydraulic binders, factors such as the chemistry of the cement, its proportion in the backfill and the sulphur content were considered to be the basis for the research on expansion (Fall and Benzaazoua, 2005). Instead of directly affecting the chemical reaction, solids concentration influences the concentration of sulphate in the pore water and free water of the backfill as well as determines the initial porosity of the backfill. An orthogonal design was used to research the expansion property of the cemented backfill. The measured responses were the FER after 14, 28, 56 and 120 days of curing. The experiments were run in random order. Five levels of variables were used in the experimental design. Based on a previous experimental study (Ercikdi et al., 2009) the ranges of these three factors were determined to be: 8 to 16% cement dosage, 55 to 75% solids concentration and 4 to 20% sulphur in the tailings. The variables and their levels selected for this study are shown in Table 2, in both normalized and actual units.

Table 2 Design variable and normalization

Codes	-2	-1	0	1	2
Variables					
% Cement dosage	8	10	12	14	16
% Solids concentration	55	60	65	70	75
Sulphur content	4	8	12	16	20

Note: sulphate content: Sulphur element concentration of the tailings (%).

Orthogonal table structure L_{25} (5^6) was selected according to the amount of factors and levels, and the following intuitive analysis table was drawn (Table 3) (Guo and Wei, 2016).

Table 3 Orthogonal design

Exp. *N*°	Cement dosage	Solids concentration	Sulphate content
Test 1-1-1	8	55	4
Test 1-2-2	8	60	8
Test 1-3-3	8	65	12
Test 1-4-4	8	70	16
Test 1-5-5	8	75	20
Test 2-1-2	10	55	8
Test 2-2-3	10	60	12
Test 2-3-4	10	65	16
Test 2-4-5	10	70	20
Test 2-5-1	10	75	4
Test 3-1-3	12	55	12
Test 3-2-4	12	60	16
Test 3-3-5	12	65	20
Test 3-4-1	12	70	4
Test 3-5-2	12	75	8
Test 4-1-4	14	55	16
Test 4-2-5	14	60	20
Test 4-3-1	14	65	4
Test 4-4-2	14	70	8
Test 4-5-3	14	75	12
Test 5-1-5	16	55	20
Test 5-2-1	16	60	4
Test 5-3-2	16	65	8
Test 5-3-3	16	70	12
Test 5-3-4	16	75	16

2.4 Preparation and testing of CTB samples

A number of CTB samples (more than 500 in total) were prepared from the tailings sample, sulphide concentrate, binder (OPC) and mix water (tap water). To prepare the desired consistency of CTB sulphide, the backfill ingredients (i.e. tailings, binder agent and sulphide concentrate) were thoroughly mixed under dry conditions, as presented in Figure 2. Subsequently, the dry mixture was combined with water in a planetary mortar mixer (Model No. JJ-5) for approximately 12 min. After CTB was thoroughly mixed, samples were cast into the standard mold (70.7×70.7×70.7 mm). After the molds were filled, the mixture was rammed in 25 blows using a small steel rod in order to eliminate any large trapped air bubbles within CTB, as described in the ASTM C143 standard (ASTM, 2012). Specimens were removed from the molds after 48 hours and were then left to cure for the desired days in the standard conservation box with constant temperature (20°C±1°C) and humidity (90% relative humidity) (Model No. JBY-60B) . These external curing conditions replicate underground CTB-filled mine stope conditions. Following a predetermined period (up to 120 days) of curing, the CTB samples were tested for free expansion ratio (FER) using the FEMI. Each FER value represents an average value obtained from the expansion measuring tests for more than five specimens.

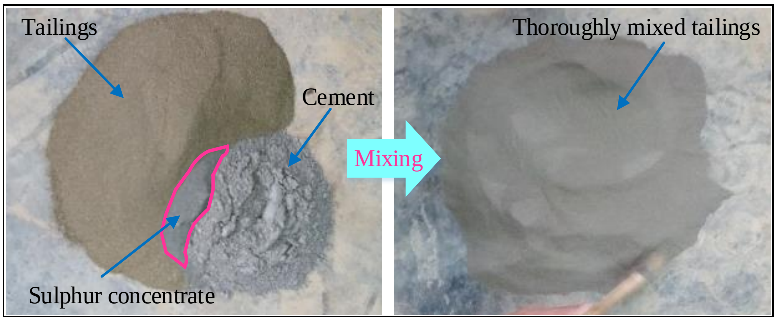

Figure 2 Tailings, binder agent and sulphur concentrate mixed in the dry regime

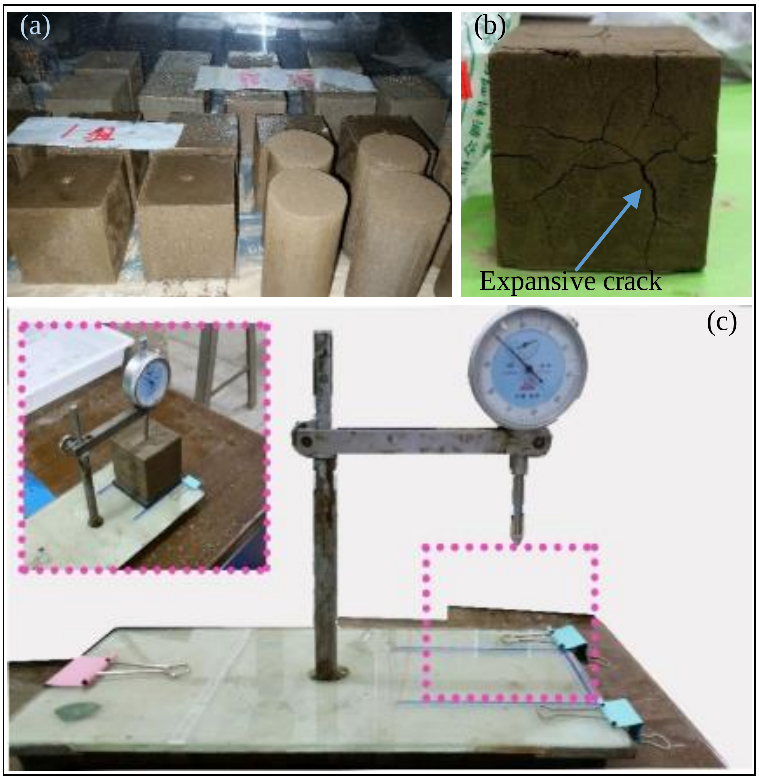

Figure 3 Photos illustrating the key steps (a) specimens cured in the standard conservation box; (b) expansive cracks occurred in specimens; (c) measuring specimens' size curing to design days

3 Results and discussion

The various levels of free expansion ratio measured by different combinations of design recipe parameters were obtained from the orthogonal table (Table 3), and K_x is the average value of the horizontal tested results corresponding to different recipes; the smaller the K_x value, the lower the average free expansion ratio (Guo and Wei, 2016). The average value of the horizontal tested results (K_x) cured over design days can be obtained using Equation (3).

	Sulphur content	**Solids concentration**	**Cement dosage**	
$K_x =$	$\left(\sum Test\ x-j-m\right)/5$	$\left(\sum Test\ i-x-m\right)/5$	$\left(\sum Test\ i-j-m\right)/5$	(3)

Where:

$Test\ x-j-m$ = a test value according to Table 3, $x,\ i,\ j\ ,m$ =1, ... , 5.

All three factors (sulphur content, cement dosage and solids concentration) have an impact on the free expansion ratio at different curing days (Table 4).

Table 4 Results of the test

	Sulphur content	Solids concentration	Cement dosage	Curing time
K_1	0.233	0.211	0.180	14 days
K_2	0.252	0.277	0.194	
K_3	0.314	0.308	0.321	
K_4	0.359	0.333	0.377	
K_5	0.364	0.393	0.449	
K_1	0.850	1.057	0.760	28 days
K_2	0.996	1.113	0.767	
K_3	1.209	1.179	1.262	
K_4	1.409	1.333	1.447	
K_5	1.659	1.441	1.886	
K_1	2.105	2.737	1.874	56 days
K_2	2.480	2.774	2.018	
K_3	2.951	2.932	2.943	
K_4	3.393	3.228	3.521	
K_5	4.157	3.415	4.729	
K_1	4.856	6.308	4.408	120 days
K_2	5.883	6.373	4.865	
K_3	6.932	6.879	7.070	
K_4	7.986	7.665	8.261	
K_5	9.613	8.045	10.666	

3.1 Effects of sulphur content on the free expansion rate (FER)

Figure 4 (according to Table 4) shows the effects of the sulphur grade of the tailings on the free expansion ratio (FER) of the different backfill samples. The amount of sulphur influenced the expansion property (i.e. FER) of the CTB specimens (Benzaazoua et al., 2004). With the sulphur content increasing from a proportion of 4% up to 20%, the free expansion ratio featured a linear growth regardless of the binder content and solids concentration. The curing periods also had a great influence on the FER. In the early ages (14~28 days), with the change of sulphur content in the mixtures, the increment of FER showed no significant improvement (0.23~0.36% and 0.85~1.66%, respectively). However, after that age, the expansion ratio and the increment of FER both improved considerably with the extension of curing period. After 120 days in particular, the free expansion ratio increased from 4.96 to 9.61% and the increment of the ratio rose to 93.7% compared with the test data at 14 days. Higher sulphur content implies the availability of sulphate, consequently leading to the precipitation of the sulphate and resulting in in the generation of the expansive species.

The sulphur content has a significant influence on the expansion performance of the CTB in different ways depending on their concentration in the pore water: sulphates will cause expansion ratio increment due to the precipitation of expansive species (Benzaazoua et al., 2004).The tailings sample used in this study is composed predominantly of pyrite. The oxidation of such sulphide phases as pyrite could occur in the presence of air and moisture which lead to the formation of acid and sulphate (Equation 4) that could react with the hydration products and three calcium aluminate (Equation 5 and 6). Meanwhile, the C-S-H bonds would be attacked and destructed, which are known as sulphate attack. The sulphate attack could culminate in the generation of expansive phases (gypsum and ettringite) and in the decalcification or destruction of C-S-H, eventually leading to deformations such as expansion (Kesimal et al., 2005)

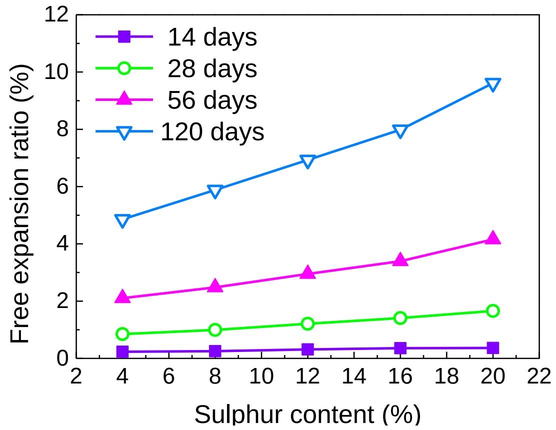

Figure 4 The effect of sulphur content on the free expansion ratio of CTB samples produced from mix tailings

$$4FeS_2 + 15O_2 + 8H_2O \rightarrow 2Fe_2O_3 + 8SO_4^{2-} + 16H^+ \quad (4)$$

$$Ca(OH)_2 + SO_4^{2-} + 2H_2O \rightarrow CaSO_4 \cdot 2H_2O + 2OH^- \quad (5)$$

$$CaO \cdot Al_2O_3 + 3CaSO_4 \cdot 2H_2O + 30H_2O \rightarrow CaO \cdot Al_2O_3 \cdot CaSO_4 \cdot 32H_2O \quad (6)$$

The formation of secondary gypsum was confirmed in CPB samples. In this regard, the deterioration of the stability of CPB samples observed followed an initial curing period of 56 days that could be ascribed to the oxidation of pyrite and, consequently, to the acid and sulphate attack. Visual observations of CPB samples after 56 days of curing indicated the development of cracks presumably due to the formation of expansive phases (Ercikdi et al., 2010).

3.2 Effect of solids concentration on the free expansion rate (FER)

Figure 5 (according to Table 4) presents the results obtained from a series of mixtures produced with the sulphidic tailings. The solids concentration played a key role in the expansion gain of CTB samples at different curing time. It can be clearly observed that an increase in the free expansion ratio (FER) is coupled with an increase in the solids concentration. Although this effect is less obvious in short term (14 days of cure) as well as in medium term (28 and 56 days of cure), it is distinctive at in long term (120 days of cure), possibly due to a certain internal weathering of the specimens. Also, it can be noted that there is an unclear increase effect of FER in the low solids concentration (55~65%) regardless of short or long-term curing periods.

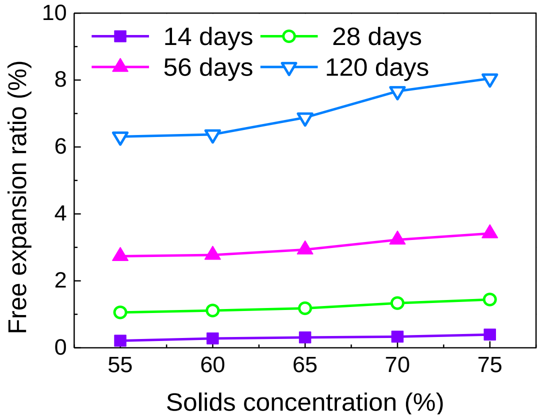

Figure 5 The effect of solids concentration on the expansion ratio of CTB samples produced from mix tailings

The mechanical properties of any backfill material are greatly affected by its physical (bulk or geotechnical index) properties, such as water content, void ratio, wet unit weight or specific gravity, and degree of saturation (Yilmaz et al., 2015). The free expansion characteristics are also critically affected by the bulk or geotechnical index. The solids concentration (or initial water content) of the CTB mixtures is undoubtedly the fundamental factor affecting its physical properties.

The solids concentration (or water-binder ratio) is very important because it controls all of the hydration and precipitation reactions, which in turn control all of the hardening processes within backfill materials. Indeed, the hardening processes of the CTB are governed by hydration reactions and the chemical solubility rates. The precipitation phenomena resulted in the formation of solid phases when they reached their saturation index (sufficient concentration). The solid phase includes not only calcium hydroxide and C-S-H but also ettringite and gypsum in the sulphidic CTB materials. Thus, increased water content from high saturation is difficult to reach due to the dilution of the soluble species (Benzaazoua et al., 2004). Therefore, the CTB specimens with low solids concentration have a low FER with the same curing days. Simultaneously, the samples with a high solids concentration (or low initial water content) have a smaller amount of porosity. This is most often due to the fact that the less excess water causes the settling of the backfill and the resultant reduction of the CTB void ratio (Ercikdi et al., 2010), which in turn leaves less space to accommodate the swelling crystals (gypsum and ettringite). Hence CTB samples with high solids concentration are more likely to produce an expansion effect at the fixed curing aging.

3.3 Effect of cement dosage on the free expansion rate (FER)

The results presented in Figure 6 (according to Table 4) are from the tests of different cement dosage (8, 10, 12, 14 and 16%) in different curing time. While it is often subjectively believed that raising the cement dosage promotes the bonding effect between tailings particles, the test results show that an increase in the amount of binder in a mixture results in a corresponding increase in the expansion ratio. Figure 6 shows clearly a sub-linear and proportional relationship between the proportions of binder and the obtained free expansion ratio. There is an unclear increase effect of FER in the low cement dosage (8~10%) regardless of short or long-term curing periods. When the cement content is 12~14%, the increment of FER is lower than that of cement with a content of 10~12% and 14~16%. Especially with curing for 120 days, this phenomenon becomes more apparent.

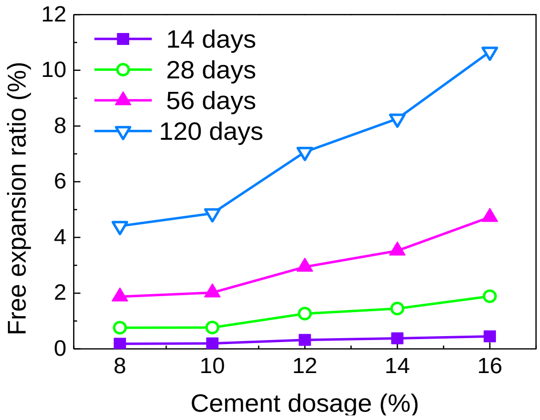

Figure 6 The effect of binder content on the expansion ratio of CTB samples produced from mix tailings

Figure 7 is a schematic diagram elucidating the occurrence of the OPC hydration process within CTB enriched with sulphide minerals (pyrite, purrhotite). The strength and stability of the concrete/backfill can be attributed mainly to the hydration of OPC through pozzolanic reactions to produce Ca $(OH)_2$ and primary/secondary C-S-H with bonding properties (Ercikdi et al., 2010) . Contingent upon the formation of primary C-S-H, the rate of hardening of CTB increases. Concomitantly, portlandite ($Ca(OH)_2$) forms which then further reacts with pozzolans to generate additional C-S-H. In the absence of pozzolanic materials, high contents of sulphate present in tailings and/or process water can react with portlandite and C_3A to form secondary gypsum and ettringite with swelling properties(Fall and Benzaazoua, 2005; Tariq and Yanful, 2013). While increasing the cement dosage could generate more hydration products ($Ca(OH)_2$ and primary/secondary C-S-H) to cement the particles of the tailings, much more portlandite and C_3A are activated in the CTB. With high contents of sulphate presenting in tailings, adequate reactants result in continuous sulphate attacks, leading to the expansion and development of microcracks in cured CTB (Benzaazoua et al., 2002; Ercikdi et al., 2009). Ultimately, increasing the amount of binder (8~16%) cannot restrict the increment of expansion ratio, and ultimately the CTB specimens would still generate cracks and collapse.

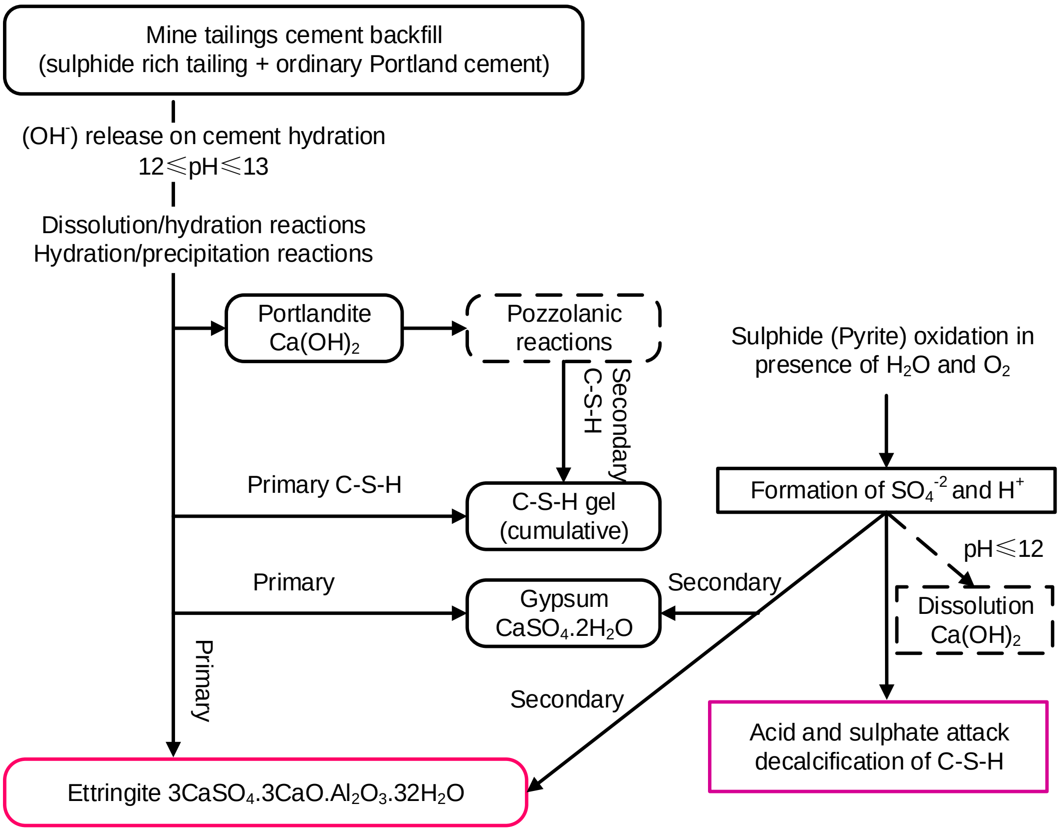

Figure 7 Schematic diagram of the decomposition process in cement tailings backfill (modified after Tariq, A., et al. (2013), Ercikdi, B., et al. (2009))

4 Conclusions

The experimental study presented in this paper aimed at examining the effects of different sulphur contents, cement dosages, curing time, and solids concentrations on the free expansion properties of CTB produced from sulphide tailings. A total of 500 CTB samples were produced in a sulphur content of 4, 8, 12, 16 and 20wt% in different recipes: a cement dosage of 8, 10, 12, 14 and 16wt% and solids concentration of 55, 60, 65, 70 and 75wt% over curing time of 7,14, 28, 56 and 112 days.

The experimental results have shown that with the increase of sulphur content, the free expansion ratio features a linear growth regardless of binder content and solids concentration. In the early ages (14~28 days), with the change of sulphides, the increment of FER was not significantly improvement (0.23~0.36% and 0.85~1.66%, respectively). After 120 days the free expansion ratio increased from 4.96 to 9.61% and the ratio increment rose to 93.7% compared with the test data at 14 days.

While the effect of solids concentration is less obvious in short-term (14 days of cure) as well as medium-term curing time (28 and 56 days of cure), it is distinctive in the long term (120 days of curing) possibly due to a certain internal weathering of the specimens. The high solids concentration of CTB mixture has less excess water that gives rise to the settling and the resultant reduction of CTB porosity. Subsequently, the low void ratio leads to less space to accommodate the swelling crystals (gypsum and ettringite).

A sub-linear and proportional relationship between the proportions of the binder and the obtained free expansion ratio was achieved. An increase in the binder dosage in a mixture leads to a corresponding increase in the expansive crystalline mineral phase. Increasing the amount of binder (8~16%) cannot restrict the increment of expansion, and ultimately the CTB specimens would generate cracks and collapse.

Acknowledgements

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