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Cemented paste backfill of sulphide-rich tailings: Importance of binder type and dosage

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ABSTRACT

In this study, the influence of binder type and dosage on the mechanical properties and microstructure of cemented paste backfill (CPB) was investigated using ordinary Portland cement (OPC), Portland composite cement (PCC) and sulphate resistant cement (SRC). The CPB samples of OPC and PCC were observed to lose their unconfined compressive strengths (UCSs) after 56 days. This could be associated with the sulphide moiety of the tailings, i.e. the attack on hydration products by sulphate and acid internally generated via the oxidation of pyrite present. In this respect, those CPB samples of sulphate resistant-based cements (SRC and a mix of OPC and SRC) maintained good long-term strengths and stability (i.e. no loss of strength). Increasing binder dosage (5–7 wt.%) improved the UCSs of CPB samples up to 1.9-fold with no loss of strength at >5 wt.%. Decreasing water-to-cement ratio appeared to produce a beneficial effect on the UCSs of CPB samples. SEM studies have provided further insight into the microstucture of CPB and confirmed the deleterious formation of gypsum as the expansive phase. These findings have demonstrated the practical importance of binder type/dosage and water-to-cement ratio for the short- and long-term mechanical performance of CPB.

1. Introduction

Mining activities involve the extraction of ores from the earth's crust. The underground mine openings created due to the ore extraction can be filled using waste materials with the process being known as backfilling [1]. This (i) provides ground support for the mine structures with a safe working environment, (ii) mitigates the risk of surface subsidence associated with the underground stopes, and (iii) provides a disposal site for waste rocks or mill tailings. In practice, hydraulic fill, rock fill and cemented paste backfill (CPB) are the backfilling methods; but, the latter has, in recent years, gained worldwide acceptance for use as an integral part of underground mining operations [1,2]. The most significant benefits of CPB include the alleviation of the environmental impact of potentially hazardous mill tailings (e.g. sulphide tailings) by placing them safely into underground openings and the reduction of the tailings disposal and rehabilitation costs [3–5].

CPB is a mixture of process tailings (70–85wt.% solids), a hydraulic binder (3–8wt.%) and water [6]. The inherent characteristics of these constituents essentially control the short- and long-term mechanical performance of CPB and are, hence, of practical importance [7,8]. The addition of binder is essential to achieve the desired strength and stability of CPB for which a 28-day unconfined compressive strength of 0.7–2 MPa is requisite for most applications [6]. In a typical CPB plant, binder costs (approximately US$1 per ton of CPB for each percentage point of binder) may comprise up to 80% of the operating costs [9]. Therefore, the utilization of suitable and cost-effective binders, and the optimization of binder dosage assume paramount importance in practice.

Some stability problems (i.e. loss of strength) in long-term have been reported to occur for CPB of sulphide-rich mill tailings [10–12]. Although the reasons behind these problems have yet to be unequivocally elucidated, they are often associated with the presence of sulphide minerals (pyrite in particular) in the tailings. In contact with oxygen and moisture, the oxidation of pyrite occurs and leads to the formation of acid and sulphate, which can then react with hydration products and binder phases such as calcium hydroxide (CH) and calcium aluminate (C_3A). Concomitantly, these undesirable reactions result in the formation of expansive phases such as ettringite and gypsum (i.e. referred to as sulphate attack) [11,12]. These could then culminate in the reduced backfill strength and potentially collapse of the backfill [8]. In this regard, the chemical and mineralogical characteristics of binder (i.e. binder type) and permeability contribute, to a large extent, to the sulphate resistance of CPB [8,13,14].

Calcium rich binders such as ordinary Portland cement (e.g. ASTM Type I) are particularly prone to sulphate attack [15]. Portland composite cements (PCC) containing active mineral admixtures (fly ash, slag, silica fume or natural pozzolan) or sulphate

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resistant cements (e.g. ASTM Type V) may be used in sulphate environments including CPB of sulphide-rich tailings [11,16–19]. Sulphate resistance of a binder is linked closely with its content of C_3A and C_4AF [17]. ASTM Type I (Ordinary Portland Cement) typically contains 8–12% C_3A compared with $\leqslant$8% and $\leqslant$5% for PCC and Type V cements, respectively. In addition to C_3A content, the ratio of C_3S/C_2S is also claimed to be an indication of the sulphate resistance of a binder on the ground that the hydration of C_3S produces nearly 2.2 times more CH than that of C_2S [17]. CH is often responsible for the formation of gypsum and ettringite as aforementioned. The sulphate performance of Portland composite cements is associated allegedly with the pozzolanic properties of mineral admixtures present contributing to pore refinement, dilution of C_3A and consumption of CH by pozzolanic reactions [20].

In this study, the short- and long-term performance of three different binders (ASTM Type I, V and PCC) in CPB of a sulphide-rich (pyritic) mill tailings were evaluated over a curing period of 360 days. The effects of binder dosage (5–7 wt.%) and water-to-cement ratio on the strength and durability of CPB were also examined. SEM studies were also undertaken to correlate microstructure and mineralogy of CPB samples with the mechanical performance and the inherent characteristics of binders used.

2. Materials and methods

2.1. Tailings sample

In this study, a mill tailings sample (~2 tonnes) produced from a copper–zinc flotation plant was used as representative of the feed to the CPB plant currently in operation. Following sampling, the physical, chemical and mineralogical characterisations of the tailings were performed. Particle size of the tailings was determined using a Malvern Mastersizer (S Ver. 2.15 particle size analyzer) (Fig. 1). The tailings with fines (−20 μm) content of 40% can be classified as a medium size tailings material with a relatively high potential for water retention to form a paste [21]. The tailings sample were determined to contain 26.2% S (Table 1), which appeared to be predominantly pyritic in nature as XRD analysis already confirmed the presence of pyrite as the sole sulphide phase identified (Fig. 2).

2.2. Binders used

In this study, the short- and long-term performances of ordinary Portland cement (OPC) (Type I), Portland composite cement (PCC) and sulphate resistant cements ((SRC) (Type V) alone and as a 50:50 mixture with OPC) were evaluated. It should be noted that PCC contains a natural pozzolan of volcanic origin (14%) and limestone (6%) as mineral admixtures. Binder dosage was kept constant at 5 wt.% of solids (tailings plus binder on dry basis) except in the tests where PCC was used to study the effect of binder dosage (5–7 wt.%). Prior to their use in the CPB tests, the physical and chemical properties of these binders were examined in some detail. Fig. 1 illustrates that SRC has a relatively coarser size distribution than OPC and PCC as also confirmed by its smaller surface area (Table 2). Compared with OPC and PCC, SRC was found to have the highest content of CaO and Fe_2O_3 whilst it contained Al_2O_3 at the lowest level (Table 2). Derived from the chemical composition of the binders, Bogue calculations proved that SRC had the lowest proportion of C_3A (Table 2), which was consistent with its inherent characteristic of sulphate resistance.

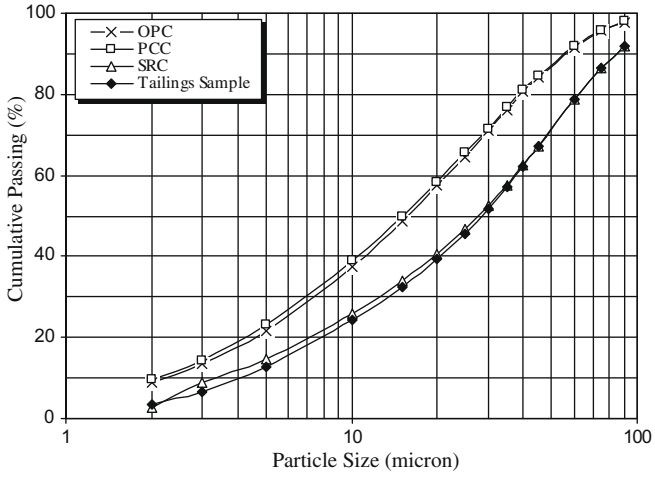

Fig. 1. Particle size distribution of the tailings sample and binders.

Table 1
Chemical composition of the sulphide tailings used.

Compound	%	Compound	%
MgO	1.19	K_2O	0.23
Al_2O_3	3.27	Na_2O	0.17
SiO_2	11.39	TiO_2	<0.01
CaO	0.95	SO_3	2.83
FeO	22.92	Cl^-	0.0025
S	26.22	Loss-on-ignition (LOI)	29.02
Total			98.2025

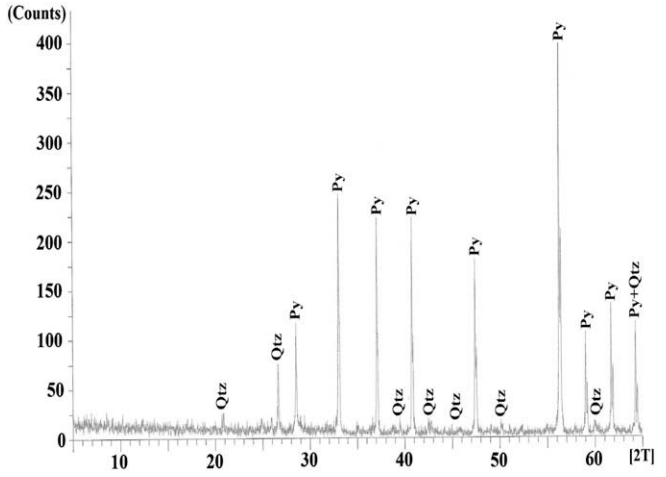

Fig. 2. XRD profile of the tailings sample (Py (Pyrite), Qtz (Quartz)).

2.3. Preparation and testing of CPB samples

A number of CPB samples (192 in total) were prepared by mixing and homogenizing the tailings, binder (OPC, PCC, and SRC) and tap water in a Univex SRMF20 Stand model blender equipped with a double spiral. The solids contents of paste mixtures were set to 77 wt.%. The effect of binder type and water-to-cement ratio was examined at a binder dosage of 5 wt.% while binder dosage was tested at 5–7 wt.% (Table 3). The chemical composition of mix water (the tailings water and tap water) with particular reference to sulphate was also determined. Having a sulphate content of 1283 mg/l, the mix water can be classified as "aggressive" towards concrete/backfill durability as suggested by DIN 4030 [22]. It should be noted that the residual water of sulphide tailings, which is inherently rich in sulphate due to the air and the sulphate con-

Table 2
Chemical, physical and mineralogical properties of the binders used in the tests.

Oxide composition	OPC (%)	PCC (%)	SRC (%)	Physical properties	OPC	PCC	SRC
SiO_2	20.31	24.51	21.31	Specific gravity	3.01	2.94	3.20
Al_2O_3	5.93	7.15	4.71	Specific surface (cm^2/g)	4345	4280	3140
Fe_2O_3	2.82	3.42	4.31	Retained on 90 μm sieve (%)	2.16	2.09	8.03
CaO	61.02	54.36	62.13	Retained on 45 μm sieve (%)	15.84	15.43	32.96
MgO	1.15	1.24	1.26				
SO_3	2.95	2.92	2.88	Compound composition			
Na_2O	0.32	0.53	0.24	C_3S	37.10	–	41.43
K_2O	1.14	1.38	0.75	C_2S	30.30	–	29.90
Free CaO	1.14	–	0.85	C_3A	10.95	–	5.20
Loss on ignition	3.78	3.75	1.54	C_4AF	8.57	–	13.10

Table 3
A summary of the experimental conditions used in the preparation of CPB samples.

	Solids content (SC)[a], wt.%	Binder dosage (BD)[b], wt.%	Water to cement ratio (w/c)[c]	Slump (in.)
Binder type	77.0	5	5.97	7.0
Binder dosage	77.0	5	5.97	7.0
		6	4.98	
		7	4.27	
Water-to-cement ratio	76.5	5	6.14	7.5
	77.0		5.97	7.0
	77.5		5.81	6.5

[a] SC : $\frac{100\times(M_{dry-tailings}+M_{dry-binder})}{(M_{dry-tailings}+M_{dry-binder}+M_{water})}$.

[b] BD : $\frac{100\times(M_{dry-binder})}{(M_{dry-binder}+M_{dry-tailings})}$.

[c] w/c : $\frac{M_{water}}{M_{dry-binder}}$; ($M$: Weight).

taining chemicals used in the flotation process and constitutes 75–80% of the mix water, contributes extensively to the sulphate content of mix water in CPB applications.

The CPB mixtures were thoroughly mixed and poured into plastic cylinders ($D \times H$: 10×20 cm) with a perforated bottom. The open-top cylinders were then allowed to cure in a room maintained at a relative humidity of ~80% i.e. to mimic the underground curing conditions. At the predetermined curing periods (up to 360 days), the CPB samples were subjected to unconfined compressive strength (UCS) tests according to ASTM C 39. UCS tests were performed using a computer-controlled mechanical press, which had a load capacity of 50 kN and a displacement speed of 0.5 mm per minute.

2.4. Studies on microstructure and mineralogy of CPB

The microstructure and texture of some 360-day CPB samples were examined under a LEO scanning electron microscope operated at an accelerating voltage of 15 kV. Energy dispersive spectrometry (EDS) of an X-ray probe coupled to the SEM was also used to aid the identification of mineral phases present. The fractured samples obtained from UCS tests were used in SEM studies.

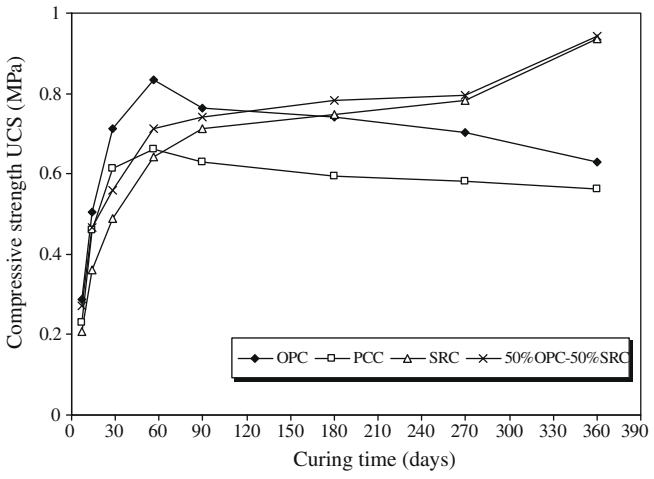

Fig. 3. The effect of binder type on the mechanical performance of CPB samples in the short and long-term (OPC: ordinary Portland cement, Type I; PCC: Portland composite cement; SRC: Sulphate resistant cement, Type V).

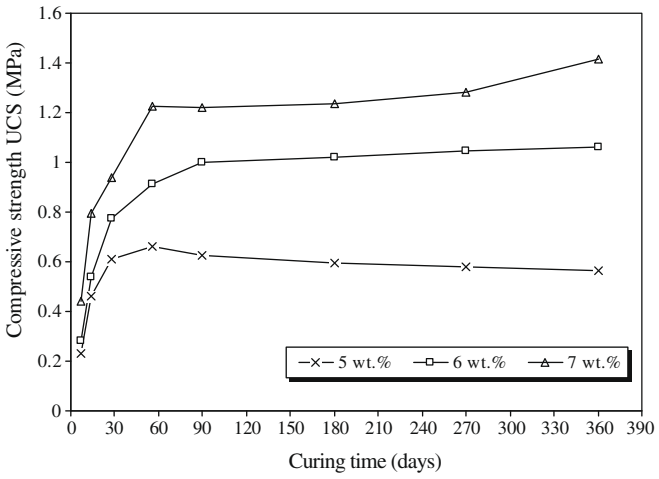

Fig. 4. The effect of binder dosage (5–7 wt.% PCC) on the mechanical performance of CPB samples in the short and long-term.

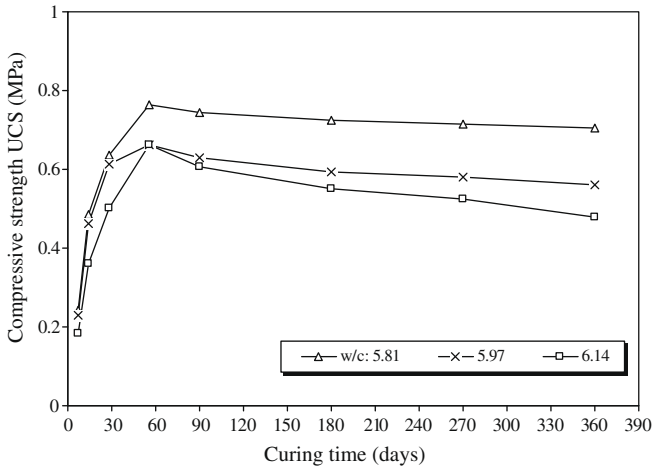

Fig. 5. The effect of water-to-cement (w/c) ratio on the mechanical performance of CPB samples in the short and long-term (at 5 wt.% PCC).

The samples were dried in an oven controlled at 50 °C and treated with acetone to cease further hydration prior to SEM analysis.

X-ray diffraction (XRD) (Philips X'pert PW 3040 Diffractometer) analyses of the acetone-treated CPB samples were also carried out to investigate the crystalline phases present with particular reference to the secondary minerals formed during the curing process. Samples were scanned over a 2-θ range of 5–70° with a 0.005° step size.

3. Results and discussion

3.1. Effect of binder type

Fig. 3 illustrates the short- and long-term (over 360 days) mechanical performances of CPB samples prepared from OPC, PCC and sulphate resistant-based cements (SRC and a 50:50 mixture of OPC and SRC) at a fixed binder dosage of 5 wt.%. Over the curing period of 56 days, a trend of increase in the strength development of the CPB samples was observed. However, after 28 days of curing, only the CPB samples prepared from OPC had the required UCS of >0.7 MPa. The rate of strength development of CPB samples of sulphate resistant cements was consistently slower than those of OPC and PCC (Fig. 3).

The strength development of CPB samples prepared from OPC peaked (0.833 MPa) at 56 days; thereafter, a trend of loss in strength (i.e. by 24.6% between 56 and 360 days) was observed. A similar (though less severe) strength loss for CPB samples of PCC also occurred. These findings suggest that OPC and PCC at 5 wt.% dosage are not particularly suitable for CPB of the sulphide-rich tailings such as that used in this study. The apparent strength losses of CPB samples observed after 56 days could be ascribed to the sulphate attack phenomenon presumably associated with high sulphide content of the mill tailings, high SO_4^{2-} content

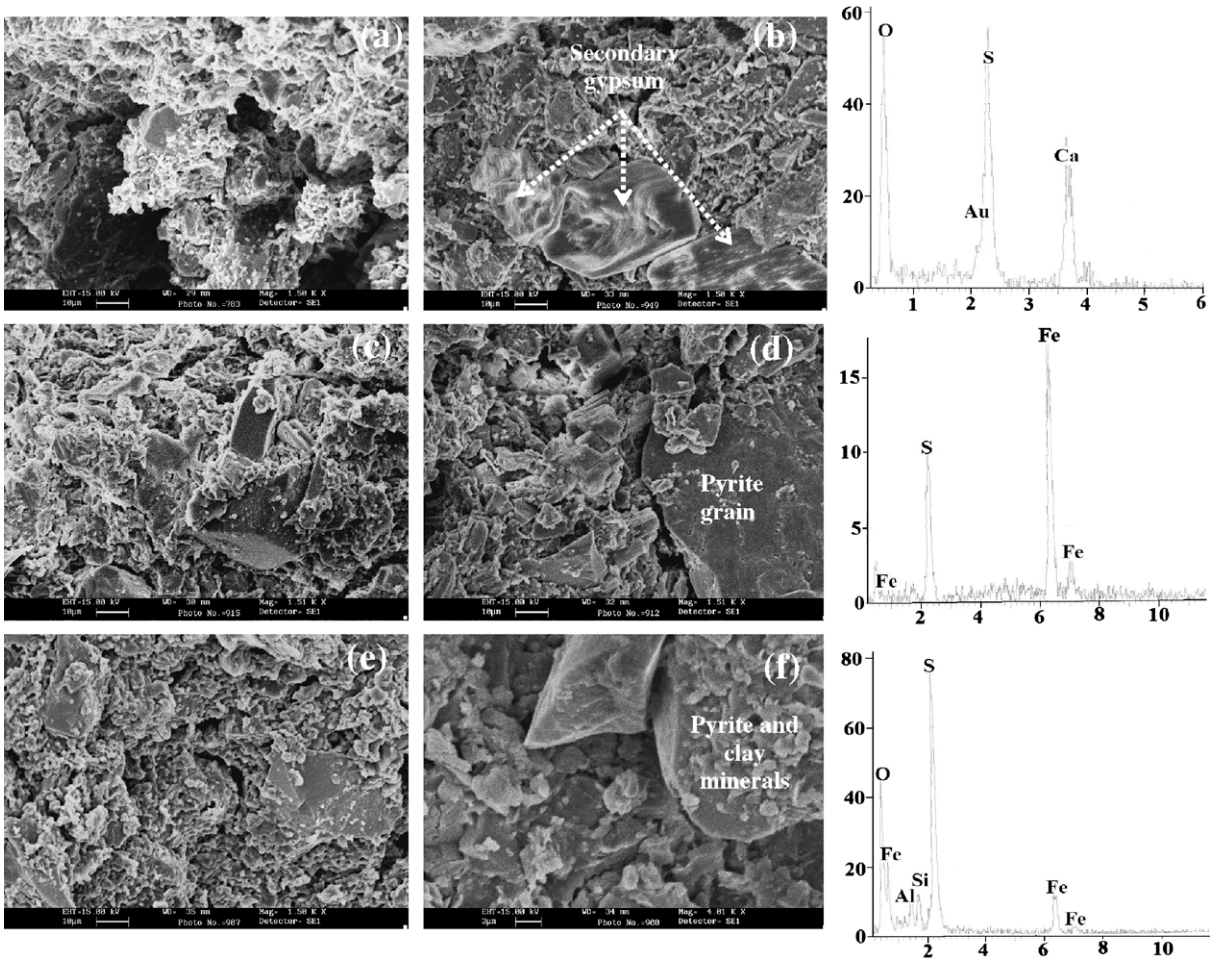

Fig. 6. SEM images of the CPB samples of OPC (a) and (b), SRC alone (c) and (d) and a blend of SRC/OPC (50–50%) (e) and (f) at a binder dosage of 5 wt.%.

of mixing water and relatively high C_3A content of these binders. The development of sulphate attack in CPB can be presumed to occur via oxidation of pyrite in the presence of air and moisture, which leads to the formation of acid and sulphate (Eq. (1)). These then react with hydration products to form expansive phases such as ettringite (Eq. (2)) and gypsum (Eq. (3)), and to decalcify/destruct C–S–H [8,14,15,23,24].

$$4FeS_2 + 15O_2 + 8H_2O \rightarrow 2Fe_2O_3 + 8SO_4^{2-} + 16H^+ \quad (1)$$

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

$$Ca(OH)_2 + SO_4^{2-} + 2H_2O \rightarrow CaSO_4 \cdot 2H_2O + 2OH^- \quad (3)$$

In contrast to OPC and PCC, the sulphate resistant binders (SRC alone and SRC/OPC mixture) maintained the strength of CPB samples in the long-term (360 days) and continued to gain strength even after 56 days (Fig. 3). It is also pertinent to note that both SRC and SRC/OPC failed to produce the required UCS of >0.7 MPa over the initial curing period of 28 days. The mixed binder (SRC/OPC) was observed to enhance the rate of development of strength of CPB samples compared with SRC alone. The SRC and OPC can be assumed to contribute to the durability and strength development, respectively, of CPB samples when used together in the binder phase. As shown in Table 2, SRC is characterised by its low C_3A content (5.2% *c.f.* 10.95% OPC), which is an important phase in intimately controlling sulphate resistance [17,25]. The incorporation of SRC into the binder phase (OPC/SRC cement) lowers the C_3A content and improves the ability of the binder to endure the sulphate attack. Conversely, the higher SRC content may also lead to an increase in the C_3S/C_2S ratio and hence, the availability of CH to react with SO_4^{2-} giving increased susceptibility to sulphate attack. It is also of pertinent to note that SRC is usually more expensive than OPC and PCC and therefore, the use of SRC blended with OPC will typically be more cost effective than SRC on its own.

3.2. Effect of binder dosage

Binder type and dosage exert a profound influence on the short- and long-term mechanical performance of CPB. Fig. 4 illustrates the beneficial effect of increasing the binder dosage (5–7 wt.% PCC) on the strength development of CPB samples over 360 days. At the binder dosages of 6 and 7 wt.%, CPB samples were able to achieve the required UCS ($\geqslant$0.7 MPa) over a curing period of 28 days. However, those CPB samples prepared at 5 wt.% PCC failed to produce a UCS of above 0.661 MPa even over a 56-day period of curing. In comparison, the mechanical strength of CPB samples containing 7 wt.% PCC was approximately 1.9 times higher than those containing 5 wt.% PCC over 56 days.

The increase in the binder dosage from 5 to 6 or 7 wt.% significantly improved the long-term performance of PCC (Fig. 4). The CPB samples at 5 wt.% PCC suffered a strength loss of 15% between 56th and 360th days. On the contrary, those samples prepared at higher binder dosages gained additional strength (i.e. by 15–17%) over the same curing period. These findings are consistent with

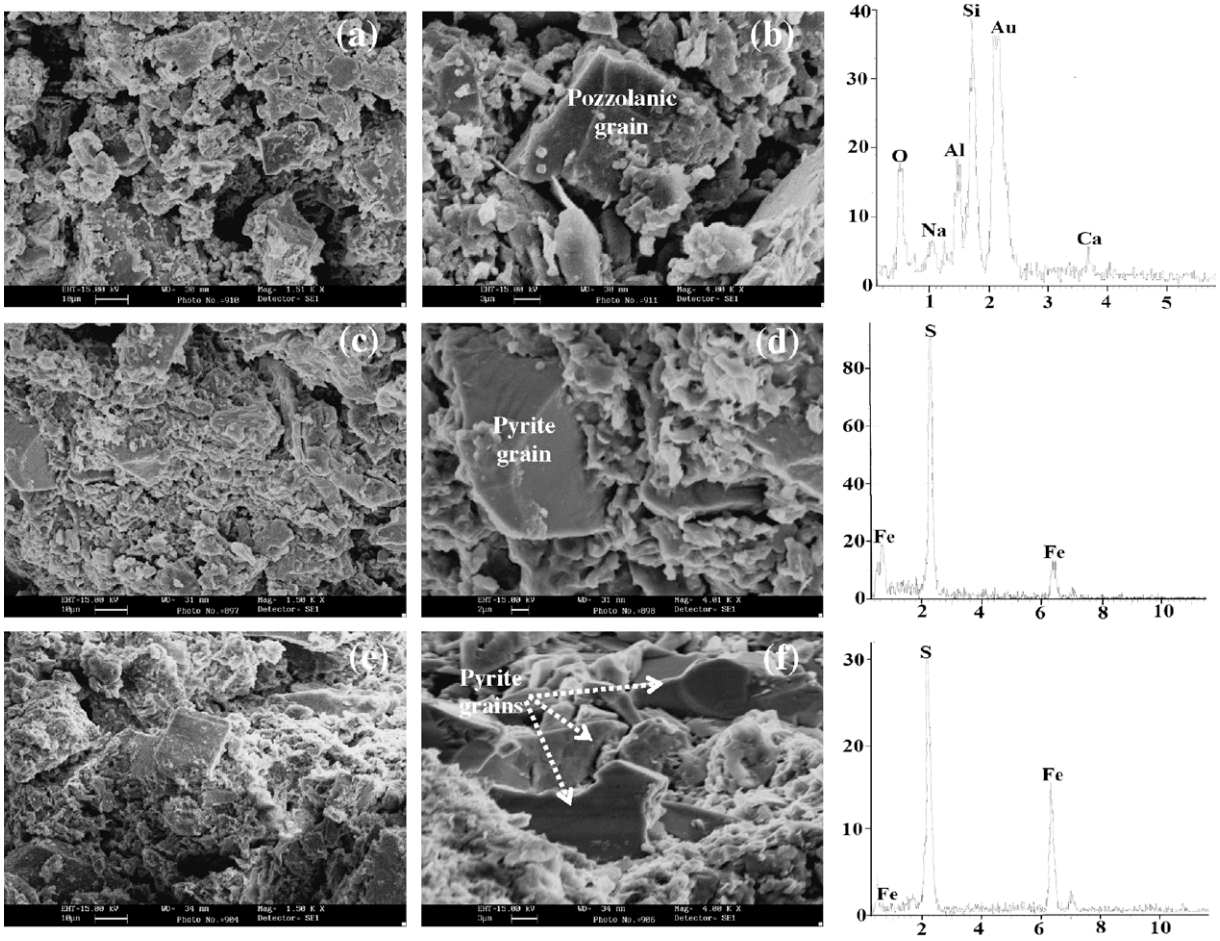

Fig. 7. SEM images of the CPB samples prepared from PCC at a binder dosage of 5 wt.% (a) and (b) and 7 wt.% (c) and (d), and at a water-to-cement (w/c) ratio of 5.81 (e) and (f).

those of Fall and Benzaazoua [12]. The beneficial effect of increasing binder content is attributed to the production of secondary C–S–H gels as a result of a pozzolanic reaction of mineral admixtures in PCC (20.7%) with CH formed during the hydration process. Providing the additional binding capacity, these C–S–H gels can also form a protective coating on the alumina-rich phases, which could otherwise participate in the formation of ettringite [25]. Furthermore, these extra C–S–H gels can improve the microstructure of CPB with the resultant reduction in permeability and probably in the oxidation pyrite and hence, the vulnerability of CPB to sulphate attack [26–29]. Despite these potential benefits, increasing the binder content will augment the operating costs of CPB.

3.3. Effect of water-to-cement ratio

The flowability of CPB mixture is significant for its transport from the paste preparation plant to the underground openings to be filled. CPB is therefore required to contain sufficient water to achieve the desired consistency. However, an increase in the water-to-cement (w/c) ratio adversely affects strength development of CPB [8]. The optimum w/c ratio is essentially a balance between maximizing the strength required and maintaining the consistency of paste for the effectiveness of pumping.

Fig. 5 presents the effect of w/c ratio on the mechanical performance of the CPB samples prepared from PCC at 5 wt.% binder dosage. Only at the lowest w/c ratio of 5.81 tested, the CPB samples were able to develop the strengths beyond the threshold level of 0.7 MPa only after a curing period of 56 days. Increasing the w/c ratio resulted in a degree of loss of strength of CPB samples in the long-term (Fig. 5). The adverse effect of increased w/c levels can be ascribed to the deterioration of the CPB microstructure due to the increased porosity and void ratio [13].

3.4. Microstructural and mineralogical assessment of CPB samples

After 360 days of curing, CPB samples were examined under SEM to provide a visual insight into the microstructure of these samples. EDS unit fitted to SEM allowed the chemical characterisation and identification of the mineral and cementitious phases within the CPB matrix. Fig. 6 illustrates the micrographs of the CPB samples produced from the binders containing only OPC (Fig. 6a and b), SRC (Fig. 6c and d) and a blend of SRC and OPC (Fig. 6e and f) at a dosage of 5 wt.%. Fig. 7 shows the influence of binder dosage (Fig. 7a–d) and w/c ratio (Fig. 7e and f) using PCC as the binder.

The microstructure of CBP samples (Figs. 6 and 7) was consistent with their respective mechanical performance for stability over 360 days of curing period (Fig. 3). The importance of microstructure for the strength and stability of mortar and CPB has been highlighted in many studies [16,24,30–32]. These studies have inferred that high strength and stability of mortar/CPB are generally concomitant with a compact or dense matrix as per the current findings in this study. To illustrate, accordant with their relatively poor performance in the long-term, CPB samples of OPC only had a higher porosity than those of PCC and SRC-based binders (Fig. 6a, c, and e). The densification of the microstructure appeared to be the most extensive for those CPB samples obtained from SRC-based binders (Fig. 6c and e).

The loose microstructure of the CPB samples such as those prepared solely from OPC (Fig. 6a) would be expected to facilitate the ingress of moisture and air, and hence, the oxidation of pyrite present in CPB. In other words, the vulnerability of CPB to sulphate attack increases with an increase in porosity of CPB matrix [31,33]. Gypsum that forms as a potentially deleterious, secondary mineral phase via sulphate attack was identified in CPB matrix of OPC only as the binder (Fig. 6b). It is relevant to note that no ettringite was detected in the SEM analysis of the samples. Fall and Benzaazoua [12] also observed the formation of secondary gypsum, which was claimed to be responsible for the reduction of strength of CPB samples after 56 days due to its expansive properties. Bing and Cohen [34] also demonstrated that formation of gypsum as a result of sulphate attack caused an expansion on concrete and consequently led to the loss of its strength and stability. However, the role of gypsum formation in the deterioration of cemented materials is controversial by some workers [35,36].

Increasing the binder dosage from 5 wt.% to 6–7 wt.% or decreasing the w/c ratio from 6.14 to 5.81 (at 5 wt.% PCC) appeared to improve the microstructure of CPB samples leading to a dense packing with the visible reduction in porosity and permeability (Fig. 7a, c, and e). The compactness of CPB matrix can be associated with the active mineral admixture (e.g. natural pozzolan) present inherently in PCC binder. These admixtures are presumed to consume $Ca(OH)_2$ liberated during the hydration process through pozzolanic reactions and to produce secondary C–S–H gels with binding properties [18,25,27–29,37–39]. In this regard, a visible C–S–H densification between pyrite grains were observed to occur with increasing binder dosage and decreasing w/c ratio (Fig. 7d and f *c.f.* Fig. 7b).

Fig. 8 illustrates XRD profiles obtained for CPB samples of OPC alone and SRC/OPC. The profiles for PCC and SRC were similar to

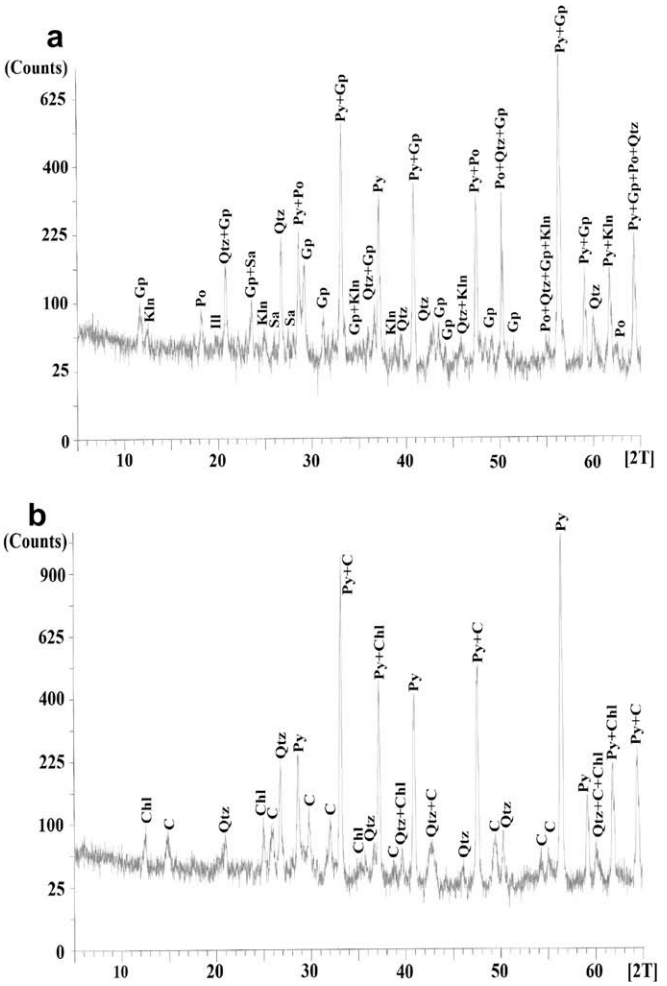

Fig. 8. XRD patterns for CPB samples prepared from OPC (a), and a blend of OPC and SRC (b) after a curing period of 360 days (Py (Pyrite), Gp (Gypsum), Qtz (Quartz), Po (Portlandite), Kln (Kaolinite), Sa (Sanidine), Ill (Illite), Chl (Chlorite), C (Plaster of Paris), Fsp (Feldspar) and Brt (Barite)).

that presented in Fig. 8b for SRC/OPC and hence not shown. XRD analysis of CPB samples (Fig. 8) revealed the presence of gypsum and portlandite only in the sample prepared from OPC alone as the binder (Fig. 8a). No portlandite was detected in the CPB samples of PCC, SRC and SRC/OPC (Fig. 8b). It should be also noted that no ettringite was identified in XRD studies.

4. Conclusions

This study highlights the potential problems associated with sulphide-rich tailings and the practical importance of binder type, dosage and water-to-cement ratio for the strength and stability of CPB material in the short- and long-term. The mechanical performance of a binder in CPB of sulphide-rich tailings is closely associated with its susceptibility to sulphate attack. The binders, ordinary Portland cement (OPC), Portland composite cement (PCC) and sulphate resistant cements (SRC and SRC/OPC) appeared to be not particularly suitable for CPB of sulphide-rich tailings at low binder dosages (e.g. 5 wt.%). The long-term mechanical performance of CPB was sustained when sulphate resistant cements were used alone (SRC) or when blended with OPC. The latter would usually be the most economic solution due to the higher cost of SRC. However, further tests at higher binder dosages (>5%) are required to ameliorate the short-term performance of SRC/OPC. The short- and long-term performances of PCC can be improved by increasing the dosage beyond 5 wt.% to achieve the desired 28-day strength of >0.7 MPa and to maintain the stability (i.e. no loss of strength) over 360 days of curing. Reducing the w/c ratio also alleviates, to some extent, the loss of strength of CPB samples of PCC. SEM observations for the microstructure of CPB samples have provided visual evidence for the comparative performances of these binders under the conditions tested in that the strength and stability of CPB are linked intimately with the compactness (i.e. permeability and porosity) of its matrix.

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