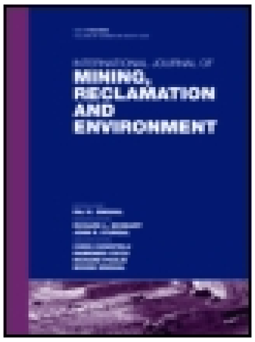

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Evaluation of geochemical behaviour of flooded cemented paste backfill of sulphide-rich tailings by dynamic-tank leaching test

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ABSTRACT

This research elucidates the geochemical behaviour of cemented paste backfill (CPB) of sulphide-rich tailings (S-rT). The dynamic-tank leaching test was performed on the CPBs for up to 360 days. The CPBs with the alkaline industrial by-products (AIPs) produced relatively lower acidity, Ec and SO_4^{2-} than the Control. The AIPs replacement appeared to considerably mitigate the release of heavy metals (HMs) including As. Only Mo and As in CPBs of AIPs exceeded the limit-values for ground-water. These findings demonstrated that a suitable CPB mixture design to control the release of all HMs in CPB is of practical importance for ground-water quality.

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KEYWORDS
Hazardous heavy metals; alkaline industrial by-products; ground-water quality

1. Introduction

Over the years, the amount of fine-sized (<100 μm) mineral processing plants tailings and mine waste in mining and minerals industry has dramatically increased worldwide. Plant tailings are often stored in the tailings dam and/or anoxic deep-sea zones. In recent years, there has been an increasing trend for depositing the plant tailings in underground mine openings as cemented paste backfill (CPB) due to i) the lack of adequate land for tailings/waste dam, ii) the potential long-term stability problems in tailings dams iii) strict environmental regulations with regard to surface disposal of tailings [1–3]. In addition, CPB, which is basically composed of dewatered-tailings, cement and water, offers some significant benefits for the management of tailings. These include i) the increased neutralisation potential and decreased porosity of tailings as a result of cement addition, ii) reduction of sulphide oxidation/acid mine drainage (AMD) generation in long-term under flooding conditions after mine closure owing to low permeability of CPB and iii) stabilisation of the pollutant heavy and toxic metals (Cu, Pb, As, Zn, etc.) due to the encapsulating effect of CPB [1,4,5].

During the ore extraction process, the mine water forms through the water seepage from surface and/or underground into underground mine from which is drained with pumps. The mine water may contain potentially harmful metal ions in high concentrations. CPB is usually placed into the mined-out openings. The oxidation of CPB produced from sulphide-rich tailings (S-rT) may occur in long term leading eventually to the solubilisation/mobilisation of various potential contaminants present in the CPB matrix in contact with mine water. After the termination of ore extraction operations, i.e. mine closure, the underground mine (and hence CBP) may be completely flooded with ground-water. In fact, the adequate water saturation within CPB can mitigate the sulphide

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oxidation and the resultant AMD formation essentially due to the creation of anaerobic, i.e. oxygen-deficient conditions given the limited solubility and diffusion of oxygen in water [4,6–8].

The dynamic-tank leaching (DTL) test reported to show more representative chemical release results to assess the long-term environmental behaviour of the CPB [9], is carried out to understand the sequential leaching behaviours and leaching mechanism of monolithic cement-based materials (concrete, CPB, etc.) in a diffusion-controlled environment. In this test, the leachate is periodically changed with the fresh distilled water to simulate the leaching behaviour of the low permeability-CPBs [10] and the amounts of release depending on surface area of the monolithic CPBs are determined by DTL test under a continuous contact with water. In recent years, there have been some studies designed to evaluate the geochemical behaviour of flooded CPB body in underground mines using DTL test [5,6,8,11–13]. To study the behaviour of arsenic (As) in the CPB, Coussy et al. [5] produced three different CPB samples from the arsenopyrite-tailings and using different binder materials (OPC: ordinary Portland cement, OPC-FA: OPC with fly ash and OPC-S: OPC with slag). They found that the CPBs of OPC-FA released higher As than those CPBs of OPC and OPC-S. The authors also observed that the pH of all CPBs remained highly alkaline (10.4–11.9) over the leaching time and the high pHs promoted the As-release. A similar research was conducted by Jiao et al. [11] who stated that the most accurate method for the determination of the effect of CPB on the surface or ground-water was to carry out the geochemical analyses of CPBs by soaking the monolithic CPB samples in distilled-water during long leaching times. The authors monitored the release of hazardous heavy metals (i.e. As, Pb, Cd and Zn) from five cubic CPBs (tailings+slag+binder+water) soaked in distilled-water for the specified times (8, 30, 45, 60 and 75 days). They concluded that the CPB does not adversely affect the ground-water quality, and hence, this technology can be suitably utilised in underground mines [11]. Similarly, Hamberg et al. [12] assessed the evolution of leachate pH and leaching behaviour of some constituents (i.e. As, Ca, Fe, S) in tailings and monolithic CPBs containing OPC and OPC-FA by using DTL test over the test period of 0.25–-64 days. The results demonstrated that pH of monolithic CPBs remained at alkaline levels (7.9–10.4) over the test periods, in contrast to cyanidation tailings (CT) samples for which the acidic pHs (i.e. 3.9–6.0) were recorded over the same test periods. Correspondingly, the sulphate release from CT was higher than those from CPBs. On the other hand, CPB containing OPC produced considerably higher As-release than CT and CPB containing OPC-FA. In a different study, Hamberg et al. [6] performed DTL tests on the 31-day and 446-day cured CPBs of OPC and OPC-FA for up to 64 days. They monitored the variations in pH and leachability of various metals (Ca, Cr, Fe, Ni, S, Zn and Cu) from CPBs under normal flooding (after 31 days) and delayed flooding (after 446 days) conditions. Based on their findings, the authors suggested that the rapid flooding of CPB body mitigates sulphide oxidation and mobility of trace metals. Furthermore, the binder content of CPB mixtures can contribute to the neutralisation potential with the concomitant reduction in the generation of AMD and thus, in the release of contaminants present in the tailings [8,14]. In this regard, Hamberg et al. [8] prepared low-strength (0.2 MPa) CPB samples to examine the influence of strength on the leaching of some metals (Zn, Cu and Ni) in the DTL test during 0.25–64 days. They reported that the CPB body having low-strength is not environmentally beneficial (i.e. the potential for ground-water pollution) due to the high Cu-release. In another research, Seipel et al. [13] conducted the DTL test for 2–288 h on CPBs with 4 wt.% binder to evaluate CPB stability and influence of the post-closure release of metals on the ground-water quality. They found that the pH-values in CPBs ranged from 6.5 to 9.5 with relatively high As-release, above the regulatory level for ground-waters. It is pertinent to note that the sulphur content of tailings utilised by various researchers in many studies reported [5,6,11,12] is limited to 0.84–2.0% with the geochemical measurements of up to 64 and 75 days. In Turkey, S-rT containing 15.82 up to 37.40% pyritic sulphur (S^{2-}) are utilised in CPB operations [15,16]. When the tailings contain high amount of sulphide minerals, the oxidation of some sulphides, e.g. pyrite in particular that occur upon exposure to air and moisture leads to the generation of SO_4^{2-} and H^+ as the oxidation products. They cause the internal stress by reacting with the hydration products, which eventually lose their binding properties leading to the formation of a porous and cracked CPB structure. Therefore, the strength and durability of CPB can be negatively affected [17–19]. Furthermore, the buffering capacity of CPB and the mobility of heavy metals are affected by the increasing acidity within the CPB environment. Previous researchers reported that i) the utilisation of binder only is insufficient to mitigate the acid/sulphate attack, ii) increasing the cement dosage and/or utilisation of an alkaline additive (fly ash, lime, etc.) as a replacement for binder could not prevent the generation of cracks and loss of strength in CPB of S-rT [20,21]. For this reason, AIPs were tested as replacement for S-rT to make most of their binding as well as buffering effects so as to mitigate potential problems associated with the S-rT of CPB. In this respect, a comprehensive evaluation of long-term geochemical behaviour of CPB produced from S-rT is essential under flooding conditions.

This study was therefore developed under laboratory conditions with the objective of evaluating the effect of flooding on the leachability of metals from CPB of S-rT (15.82% S^{2-}). The study in fact examines the mine closure scenario that the underground mine and hence CPB having been placed into the mined workings are completely flooded on closure. The impact of the utilisation of different alkaline industrial by-products (AIPs: blast furnace slag (BFS) and C-type fly ash (C-FA)) as an additive on the environmental parameters (acidity (pH), conductivity (Ec), sulphate (SO_4^{2-}) and hazardous heavy metals (HMs: Cu, As, Zn, Pb, Cd, etc.)) of flooded CPB were evaluated by using dynamic-tank leaching (DTL) tests over the specified leaching times (30, 60, 90, 180, 270 and 360 days). The release of HMs was compared with the limit-values (L-V) for ground-water [22]. Moreover, the effect of mineralogical and microstructural properties on the geochemical parameters of CPB in the DTL test were discussed using XRD and MIP data.

2. Materials and experimental studies

2.1. Alkaline industrial by-products

Alkaline industrial by-products (AIPs: BFS (Blast furnace slag) and C-FA (C-type fly ash)) utilised as replacement to S-rT were procured from Kardemir iron-steel plant and Afşin-Elbistan thermal power plant in Turkey, respectively. Before the grinding operation, BFS and C-FA were dried in ventilated oven at 50°C/36 h. The BFS and C-FA were granulated to obtain the materials with very similar particle size distribution to S-rT since both were utilised as replacement for S-rT, and the grinding time was determined as 240 and 105 min for BFS and C-FA, respectively, and the AIPs were then granulated to −100 µm in a laboratory ball mill. Approximately 10 kg of BFS and C-FA was prepared for use in experimental studies.

A Malvern Mastersizer Hydro 2000 MU size analyser was utilised to determine the grain size distributions of ground BFS and C-FA samples (Figure 1). BFS and C-FA were found to have a 80% passing size of 54.5 µm and 50 µm with a specific surface area of 4640 and 6360 cm^2/g (Table 1), respectively. These AIPs were also analysed using XRF (X-ray fluorescence) and ICP-AES (inductively coupled plasma atomic emission spectroscopy) to determine the chemical characterisations of AIPs, and contain predominantly SiO_2 and CaO (30.62–37.02%) as an indication of their alkaline nature. The specific gravity of BFS and C-FA was measured as 2.42 and 2.91 g/cm^3, respectively (Table 1). XRD analysis has indicated that the BFS appeared to be

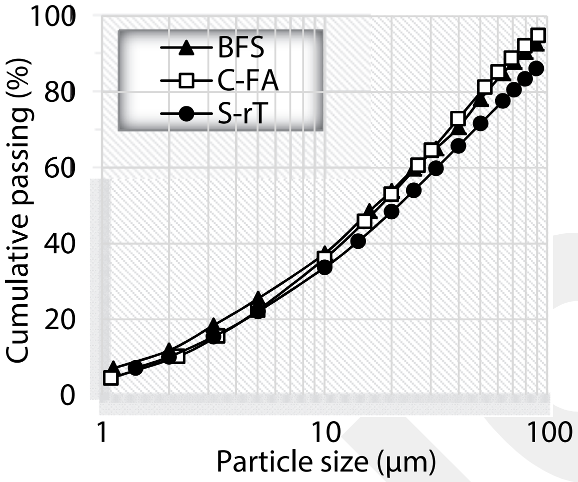

Figure 1. Grain size distributions of AIPs and S-rT [3].

Table 1. Chemical, physical and mineralogical properties of the AIPs, S-rT and OPC [3].

Characteristics	BFS (%)	C-FA (%)	S-rT (%)	OPC (%)
Chemical composition				
SiO_2	39.85	34.96	31.89	21.02
Al_2O_3	10.58	13.92	8.97	5.27
Fe_2O_3	1.21	6.42	33.09	3.06
MgO	6.26	2.32	4.08	2.19
CaO	37.02	30.62	3.48	62.91
Na_2O	0.30	0.27	0.68	0.38
K_2O	0.97	0.91	0.36	0.81
TiO_2	0.77	0.71	0.66	0.30
P_2O_5	0.01	0.40	0.04	0.11
MnO	2.54	0.04	0.10	0.09
Cr_2O_3	0.019	0.054	0.032	0.035
Free CaO	-	8.67	-	1.19
Loss–on–ignition (LOI)	−0.5	3.4	16.1	3.6
Total	99.85	94.16	99.48	99.92
Sulphide content (S^{-2}) (%)	-	-	15.82	-
Pyrite content (FeS_2) (%)	-	-	29.66	-
Physical properties				
Specific gravity (g/cm^3)	2.91	2.42	3.37	3.12
Specific surface area (cm^2/g)	4640	6360	4440	4330
Mineralogical composition	*BFS*	*C-FA*		*S-rT*
	Glassy phase	Calcite, Portlandite Anhydrite, Quartz		Calcite, Quartz Albite, Pyrite Chlorite

essentially amorphous in form, while, portlandite and anhydrite are identified to be present in the C-FA (Figure 2(a,b)).

2.2. Binder and sulphide-rich tailings

The binder (OPC: CEM I 42.5 R) was supplied by Aşkale Cement Industry in Trabzon, Turkey, and a sample of sulphide-rich tailings (S-rT) was obtained from Kure Copper-Mine of Etibakır A.Ş in Kastamonu, Turkey. As can be seen from Figure 1 and Table 1, the S-rT contains 48.4% fines

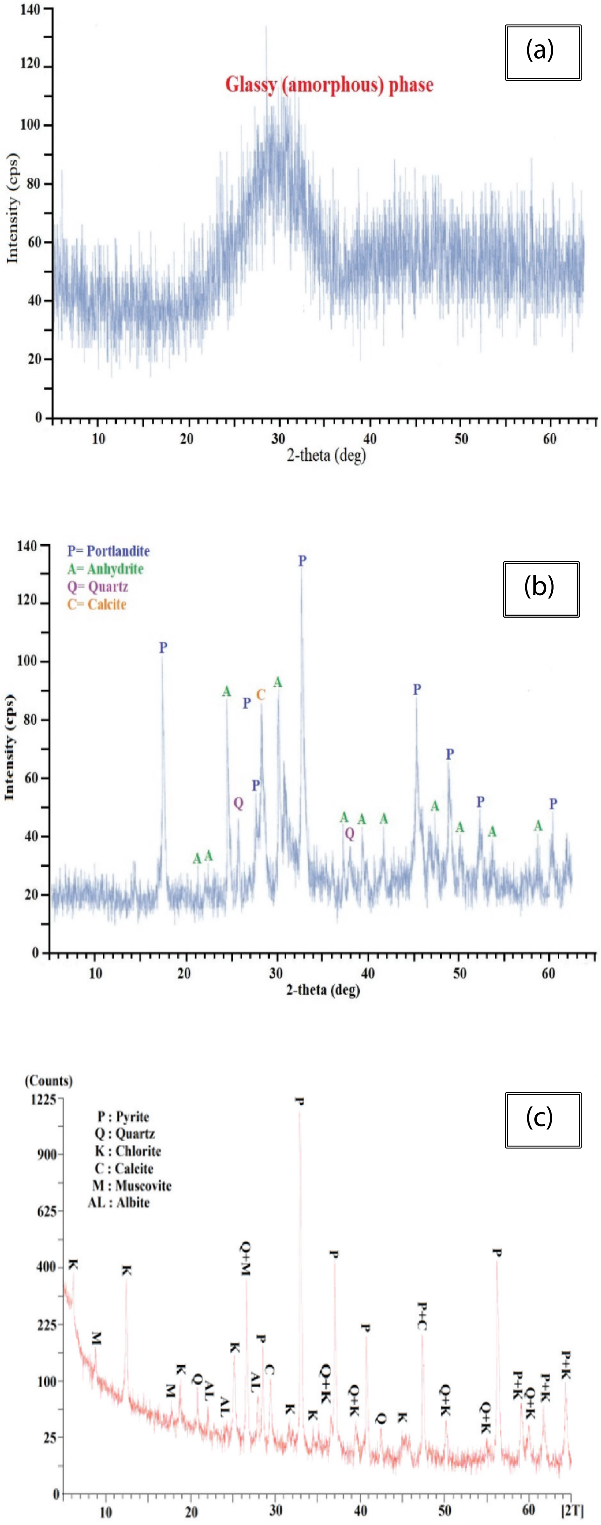

Figure 2. XRD profiles of BFS (a), C-FA (b) and, S-rT (c) [3].

Table 2. Acid-base accounting (ABA) test results of the S-rT and AIPs [3].

Properties	S-rT	BFS	C-FA
Sulphide (S^{2-}) content (%)	15.82	-	-
CaO content (%)	3.48	37.02	30.62
Free CaO content (%)	-	-	8.67
Fizz rating of materials	None	Moderate	Slight
Acid normality (N) and volume (ml)	0.1/20	0.5/40	0.1/40
Acid potential (AP)*	494.38	0	0
Neutralisation potential (NP)*	8.58	532.25	318.84
Net Neutralisation potential (NNP)*	−485.80	532.25	318.84

*: kg $CaCO_3$/ton waste

(−20 µm) and 15.82% sulphur. Figure 2© also indicates that the dominant mineral phase in the S-rT is pyrite, consistent with its chemical composition as presented in Table 1.

The acid potential (AP) of S-rT was primarily determined by multiplying the total sulphur value (%S_{total}) by 31.25. The fizz and titration (acid-base accounting: ABA) tests designed by Sobek et al. [23] were then implemented for calculation of neutralisation potential (NP) and net neutralisation potential (NNP) of S-rT and AIPs (Table 2). Further details of these tests can be found in Yılmaz et al. [3].

2.3. *Mixture design of CPB samples*

Three types of CPB samples (15 CPBs in total) at a fixed OPC dosage (8.5 wt.%) were produced using only S-rT (Control) and AIPs (BFS or C-FA) as 10 wt.% substitute for S-rT in the mixture. These CPBs were therefore coded as Control, 10BFS and 10 C-FA (Table 3). The CPB mixtures consisting from S-rT, OPC, AIPs and tap water were prepared by mixing and homogenising using a mechanical blender (Figure 3(a)) at 19.05 cm consistency. A total of 15 CPBs were fabricated for DTL test, MIP, XRD and metalloids content (Table 3). The as-received S-rT was determined to contain 73% process water of which the initial pH and sulphate were 6.85 and 5616 ppm, respectively.

The cylindrical moulds with dimensions of 5 cm (diameter) to 10 cm (height) were utilised to cast the CPB mixtures. The open-top and perforated bottom CPB moulds were initially placed on a stand to allow the drainage of excess water for 24 h (Figure 3(b)). Thereafter, CPB moulds were allowed to cure for up to 180 days in the curing cabinet in which humidity and temperature were controlled at ~80% and 20°C, respectively (Figure 3(c)). The CPB moulds were exposed to air from top, which was kept open over the curing period. Following 180 days of open-top curing, the CPBs were subjected to chemical and mineralogical characterisation prior to the DTL tests (Table 4).

Table 3. The mixture design parameters and number of CPBs used in the tests.

Characteristics		Control	10BFS	10 C-FA
Mixture Properties				
Materials (wt.%)	S-rT	100	90	90
	AIP	-	10	10
Solids content (SC) (wt.%)		73.70	74.14	73.44
Cement dosage (CD) (wt.%)			8.5	
Water to cement ratio (w/c)		4.20	4.10	4.25
Slump (cm)			19.05	
Strength at 180 days (MPa)		1.56	4.89	4.40
Test Name			*Number of Samples*	
XRD	Before DTL	1	1	1
Metalloids Content				
DTL		3	3	3
XRD	After DTL			
MIP		1	1	1

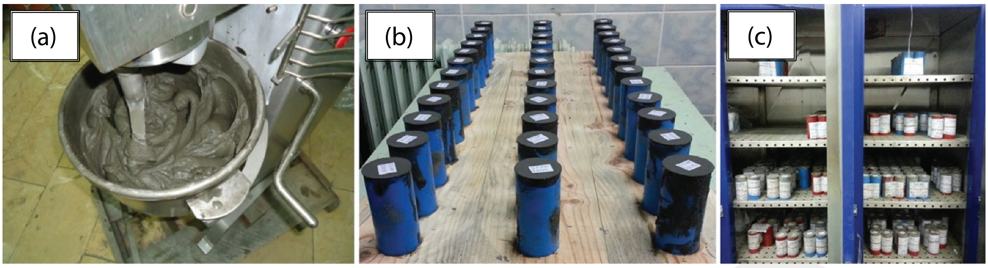

Figure 3. Fabrication of CPBs: mixing (a), draining (b) and curing (c).

Table 4. The concentrations of major and trace elements of 180-day CPBs.

Characteristics	Control	10BFS	10 C-FA
Major Elements (wt.%)			
Fe	16.88	16.25	15.39
Ca	4.99	5.23	5.11
Al	2.84	3.26	2.93
Mg	1.79	2.05	1.72
Trace Elements (ppm)			
Cu	1153	1130	1022
Zn	903	843	825
Mn	587	2273	527
Co	582	558	507
As	144	136	126
Ni	54	51	60
Pb	88	80	75
Cr	126	146	130
Mo	9.6	8.6	9.6
Cd	3.1	2.9	2.8

2.4. Dynamic-tank leaching (DTL) test

The laboratory scale DTL tests were conducted to evaluate the influence of CPBs on surface or ground-water quality in underground applications. In these tests, the 180-day cured monolithic CPBs (9 samples in total) were demoulded and soaked in de-ionised water within closed plastic containers to determine the leachability of potential contaminants over the specified test periods (30, 60, 90, 180, 270 and 360 days) according to the methods suggested by US EPA 1315–1 [24] and ASTM C1308-08 [25] (Figure 4). Before the DTL test, the length of the CPBs was measured with calipers to calculate its geometric surface area (GSA) and leachant volume (LV) using the equations (1) and (2).

$$GSA = 2\pi r \times (r+h) \quad (1)$$

$$LV = GSA \times 9 \pm 1\ cm^3 \quad (2)$$

where;

“r and h” are the radius and length of sample, respectively,

After the calculation of GSA and LV for each CPB, CPBs were placed into the high-density polyethylene (HDPE) containers (Figure 4). There is a grating platform located 5 cm above the bottom of the container on which the CPB sample was placed to ensure the exposure of all the surfaces of the CPB to water. The calculated amount of distilled-water (2.27–2.29 L) was added into the containers to completely soak the CPB sample in water for the predetermined leaching times (30, 60, 90, 180, 270 and 360 days). Each DTL test was performed in triplicate and the mean values were calculated and presented in the results.

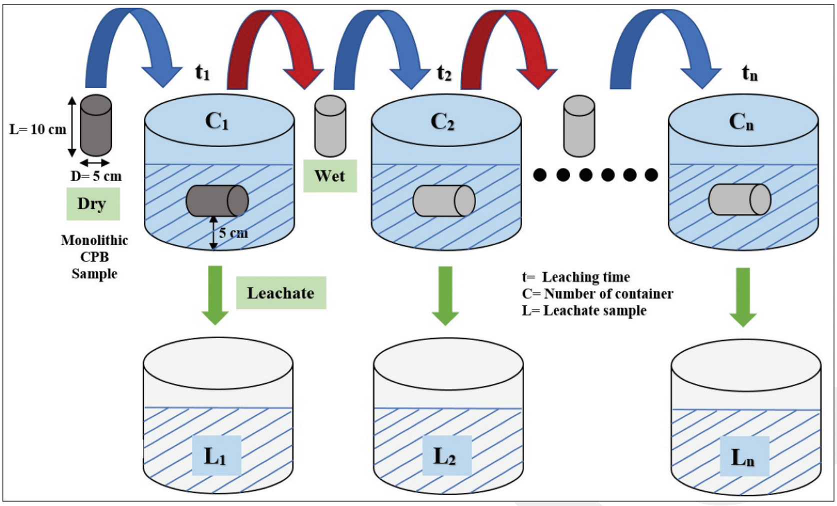

Figure 4. Schematic presentation of DTL test for monolithic samples [24].

The fresh-leachate samples obtained from DTL tests at the end of the specified leaching times were firstly filtered through a membrane filter (0.45 µm) to obtain a clear supernatant, which was immediately analysed for pH, Ec and SO_4^{2-} using Orion Star A111 Benchtop model pH-metre, Orion Star A321 model Ec-metre, photometer (Palintest 5000), respectively. A sample of this supernatant was also used for the determination of the HMs (Cu, Zn, Co, As, Ni, Pb, Cr, Mo and Cd) by an ICP-MS.

2.5. Studies on mineralogy and microstructure of CPBs

To evaluate the connection between the geochemical characteristics and porosity properties of CPBs, the MIP (Mercury Intrusion Porosimetry) analyses of three (Control, 10BFS and 10 C-FA) 180-day cured CPBs were carried out using a MIP apparatus (Micromeritics Autopore IV 9510) [26]. These CPBs for MIP analyses were produced to have the same mixture properties as those used in DTL test, but these samples were not subjected to any destructive test. The samples for MIP studies were also dried in a ventilated oven for 36 h at 50°C prior to these studies. Moreover, to provide a visual insight into the mineralogical changes occurred under the DTL tests, XRD analyses of dried CPBs before and after the DTL tests were also performed by using a Bruker D8 Discover XRD in the AGU Central Research Facility (AGU-CRF).

3. Results and discussion

3.1. Evolution of pH and Ec in the leachates of the CPBs

The acidity (pH) and conductivity (Ec) in the leachates of the CPBs (Control, 10BFS and 10 C-FA) over the specified leaching times (30, 60, 90, 180, 270 and 360 days) are presented in Figure 5. It was observed that pH of all leachates obtained from the flooding of monolithic CPBs in DTL tests increased during the first four leaching cycles (30, 60, 90 and 180 days) and remained alkaline levels ($pH = 7.03–8.95$). This initial trend of increase in pH was observed to reverse for Control CPBs after 180 days with a tendency of decline in pH over the following period of leaching up to 360 days. Notwithstanding this, pH of the leachates of CPBs of 10BFS and 10 C-FA continued to increase even after 180 days of leaching and reached 10.28 and 8.57, respectively, at the end of the last cycle (360 days) (Figure 5(a)). This seems to be consistent with the respective neutralisation potential

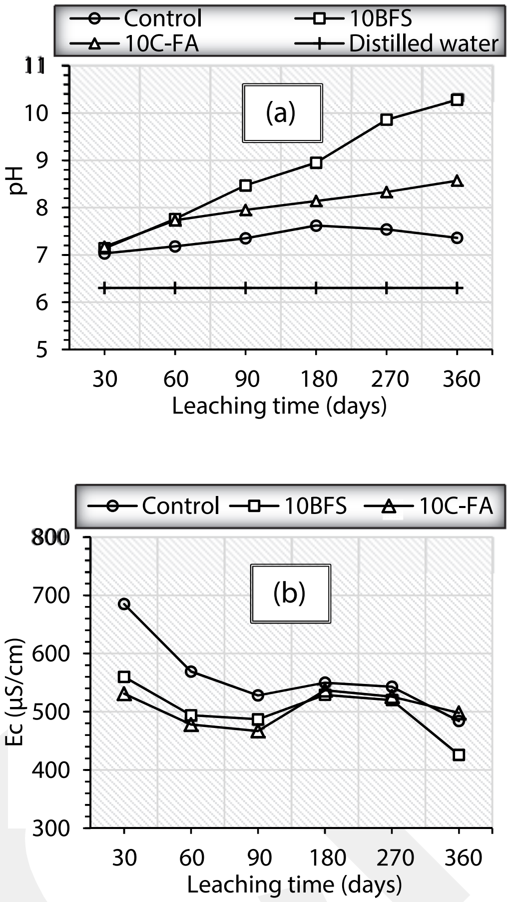

Figure 5. Evolution of pH (a) and Ec (b) of CPBs during the DTL test.

(318.84–532.25 kg $CaCO_3$*/ton)* of AIPs used in that acid formed by the oxidation of sulphide(s) such as pyrite present is neutralised by the alkaline, i.e. acid consuming phases present in the AIPs (Figure 6) [3,27]. High specific surface area *(4640–6360* $cm^2/g > 4440cm^2/g$*)* of AIPs enhances the rate of neutralisation [28]. In addition, high pH in the leachates of CPBs of 10BFS and 10 C-FA could be linked not only with the release of Ca, Mg and Mn but also with the reduced SO_4^{2-} release from CPBs, consistent with the findings reported by Benzaazoua et al. [29]. Compared with this current study, previous studies have reported relatively higher leachate pHs (i.e. up to 11.9) in the 64-day DTL tests on the CPBs of low sulphide tailings [5,6,8,12] and concretes of low sulphide waste rocks (WRs) [30]. This can be attributed to the comparatively very low sulphide content of the tailings and WRs utilised in these studies (*i.e. 0.47–0.84% c.f. 15.82% S in the current study*).

Figure 5(b) demonstrates the conductivity (Ec) (426–685 μS/cm) of the leachates of CPBs at 30–360 days of leaching times. The conductivities of all the leachates appeared to decrease during the first three cycles (30–90 days). Thereafter, Ec-values remained relatively similar levels. The decrease in conductivity could be ascribed to the reduction in the release of ions (e.g. Ca^{2+}, Mg^{2+} and SO_4^{2-}) linked with the increase in pH since these ions are expected to significantly affect the development of conductivity [2,30,31]. Annapoorna and Janardhana [32] indicated that the permissible limit value of Ec for ground-water suggested by WHO [22] is 1400 μS/cm. In this regard, Ec-values recorded in this study are consistently below the permissible limit (Figure 5(b)).

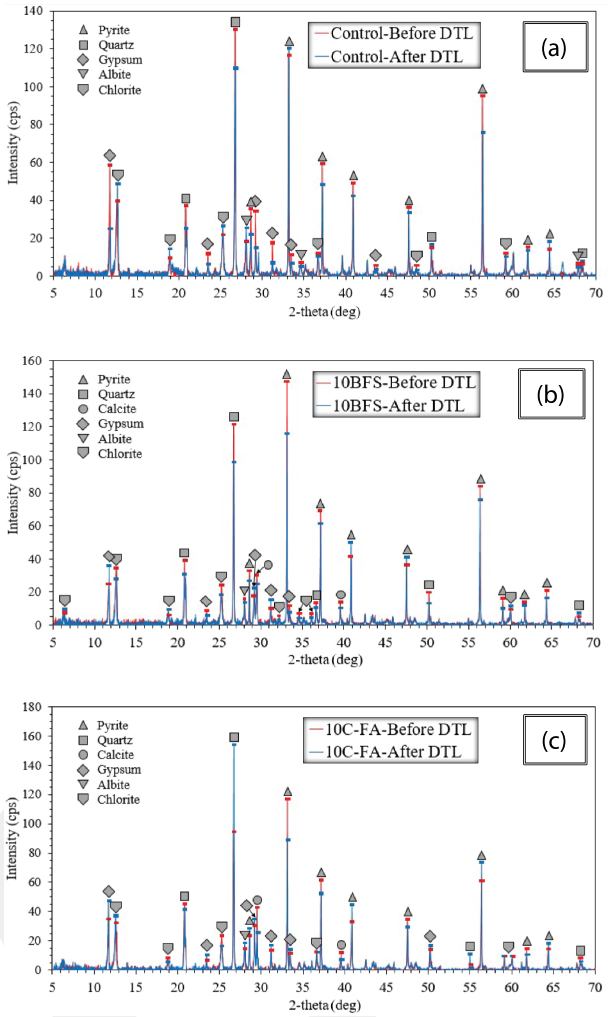

Figure 6. XRD profiles of Control (a), 10BFS (b) and 10C-FA (c) before and after the DTL tests.

3.2. Characterisation of oxidation and neutralisation products

Sulphate (SO_4^{2-}), formed by pyrite oxidation, is the principal oxidation product (OP), while, the fundamental neutralisation products (NPs) produced by dissolving carbonate and silicate minerals, and buffering OP are the secondary Ca, Mg and Mn bearing phases in the form of insoluble sulphates and hydroxides [31]. Accordingly, the levels of sulphate in the leachates and the cumulative sulphate released from the CPBs (Control, 10BFS and 10 C-FA) during the DTL tests are shown in Figure 7. The amounts of SO_4^{2-} released into the leachates varied between 47.21 and 96.33 g/m^2 (Figure 7(a)). The SO_4^{2-} release from the CPB samples (Figure 7) appeared to be linked with the evolution of the leachate pHs recorded (Figure 5(a)), i.e. the higher the leachate pH, the lower the SO_4^{2-} release. In this regard, it can be seen from Figure 7 that the leaching of SO_4^{2-} from the Control was consistently higher than those from 10BFS and 10 C-FA. The XRD profiles obtained for the CPBs before and after DTL tests suggest relatively more extensive pyrite oxidation in Control than 10BFS and 10 C-FA (Figure 6),

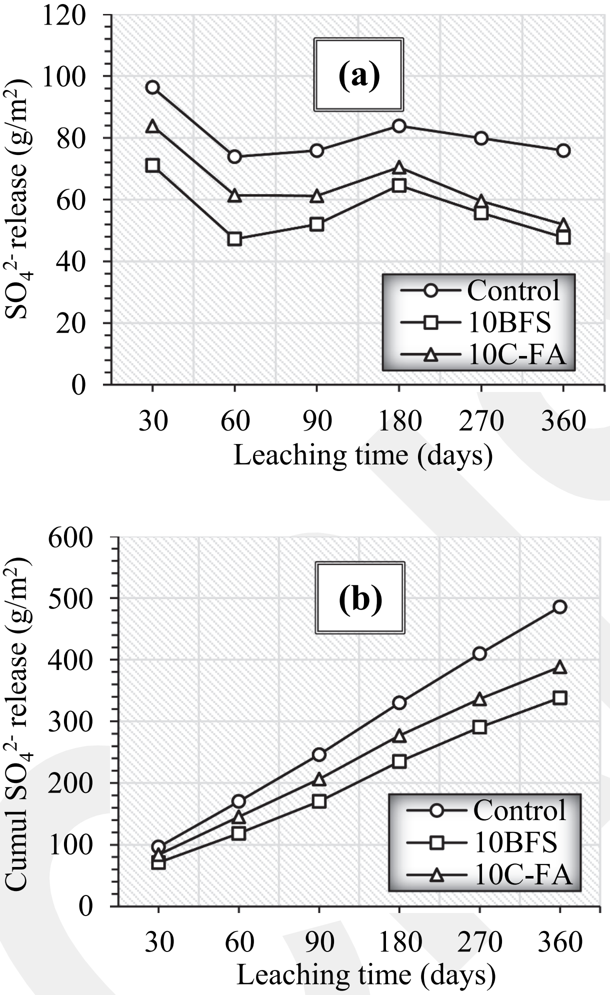

Figure 7. Normal (a) and cumulative (b) SO_4^{2-} release of CPBs during the DTL test.

consistent with their respective leachate pH profiles. The cumulative release of SO_4^{2-} for Control was determined to reach 485.65 g/m^2 at the end of the leaching time of 360 days compared with 338.22 and 388.32 g/m^2 for 10BFS and 10 C-FA, respectively (Figure 7 (b)). These findings demonstrate the mitigating effect of AIPs (BFS and C-FA) in the CPB mixture on the oxidation of pyrite presumably through the filling of voids between coarser tailings particles by fine AIPs or their hydration products leading to the denser CPB structure having low porosity and permeability for oxygen diffusion [3,19,33,34].

Figures 8 and 9 illustrate the release of elements (Ca+Mg+Mn) as neutralisation by-products and the plot of cumulative release of Ca+Mg+Mn (NPs) versus cumulative sulphate (OP) (i.e. oxidation-neutralisation plot) in the DTL tests at different (30, 60, 90, 180, 270 and 360 days) leaching times. It is evident from Figure 8 that the Control released higher amount of NPs than 10BFS and 10 C-FA at all leaching cycles except 360 days. This is consistent with higher release of sulphate (Figure 7) and lower pHs (Figure 5) as a result of higher sulphide oxidation in the Control than those in 10BFS and 10 C-FA (Figure 6).

The oxidation-neutralisation plot (Figure 9) can be utilised as a beneficial parameter for the evaluation and/or prediction of the geochemical properties of CPBs. In this regard, it suggested that

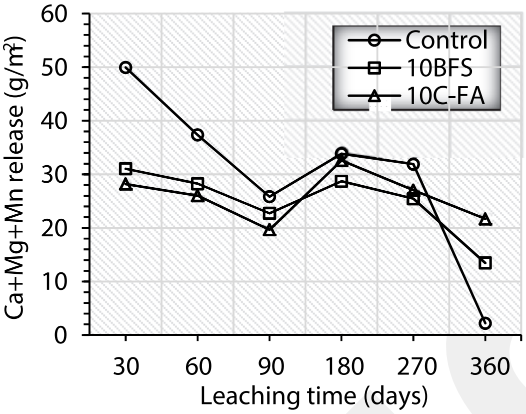

Figure 8. The NPs (Ca+Mg+Mn) release of CPBs during the DTL test.

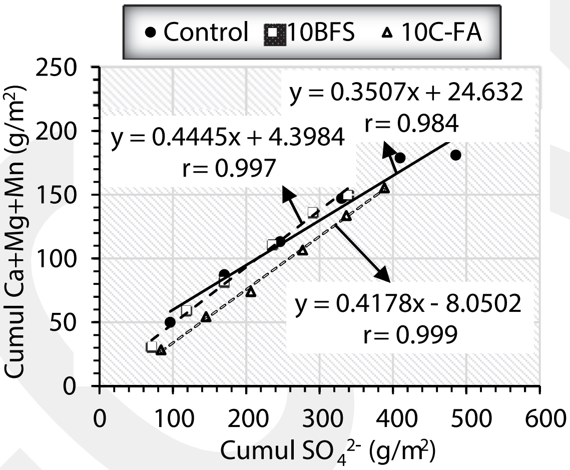

Figure 9. Oxidation-neutralization curves of CPBs during the DTL test.

the neutralisating capacity of all CPBs was sufficient to neutralise the acid generated by the oxidation of pyrite present since pHs of leachates of all the CPBs were maintained alkaline levels (Figure 5). However, the release of neutralisation by-products (Ca+Mg+Mn) from the Control sharply decreased at the last leaching cycle (360 days) (Figure 8). This could be ascribed to the exhaustion of neutralising capacity of the Control sample in agreement with the final pH levels (Figure 5) registered at this last cycle of leaching. It can be seen from Figure 9 that there is a linear relationship ($r \geq 0.98$) between cumulative release of NPs (Ca+Mg+Mn) and OP (SO_4^{2-}). Slopes of the linear trendlines obtained for Control, 10BFS and 10 C-FA were determined in Figure 9. The trendline for the Control appeared to have the lowest slope (0.35) compared with those for 10BFS and 10 C-FA (0.45 and 0.42, respectively). This is consistent with the earlier findings (Figures 5–8) indicating its higher vulnerability to the oxidation and hence to the release of hazardous constituents, relatively lower neutralisation potential and the beneficial effect of the addition of AIPs for improving the resistance to oxidation, increasing the neutralisation potential, mitigating the potential environmental impact of CPB.

3.3. Metal release from CPBs

Figure 10 demonstrates the total release of hazardous heavy metals (HMs: Cu, Zn, Co, As, Ni, Pb, Cr, Mo and Cd) from CPBs (Control, 10BFS and 10 C-FA) during the 360-day DTL tests. The Cu-release from the Control determined to be 4.5 mg/m^2, whilst, no release of Cu from 10BFS and 10 C-FA was recorded (Figure 10). This can be explained by the reduced oxidation of the sulphide moiety (Figure 6) and the maintenance of sufficiently alkaline conditions (Figure 5) to minimise the solubility of copper in the presence of AIPs as discussed above. Compared with these current findings, relatively higher amounts of Cu (up to 17.4 mg/kg and 50.0 mg/m^2) from concretes [35] and CPBs [8], respectively, were reported during 63–64 days of leaching time. Hamberg et al. [8] attributed high Cu-release to the production of low quality (i.e. the low-strength) CPBs at the binder dosages of 1–3 wt.%. In this regard, the less porous-CPB structure particularly in the presence of AIPs was generated at a high binder dosage (8.5 wt.%) and thereby, sulphide oxidation for Cu-release and the transfer of Cu from pore water within CPBs into leachate were mitigated, in agreement with the study of Hamberg et al. [8]. In addition, Cu-release from the Control is

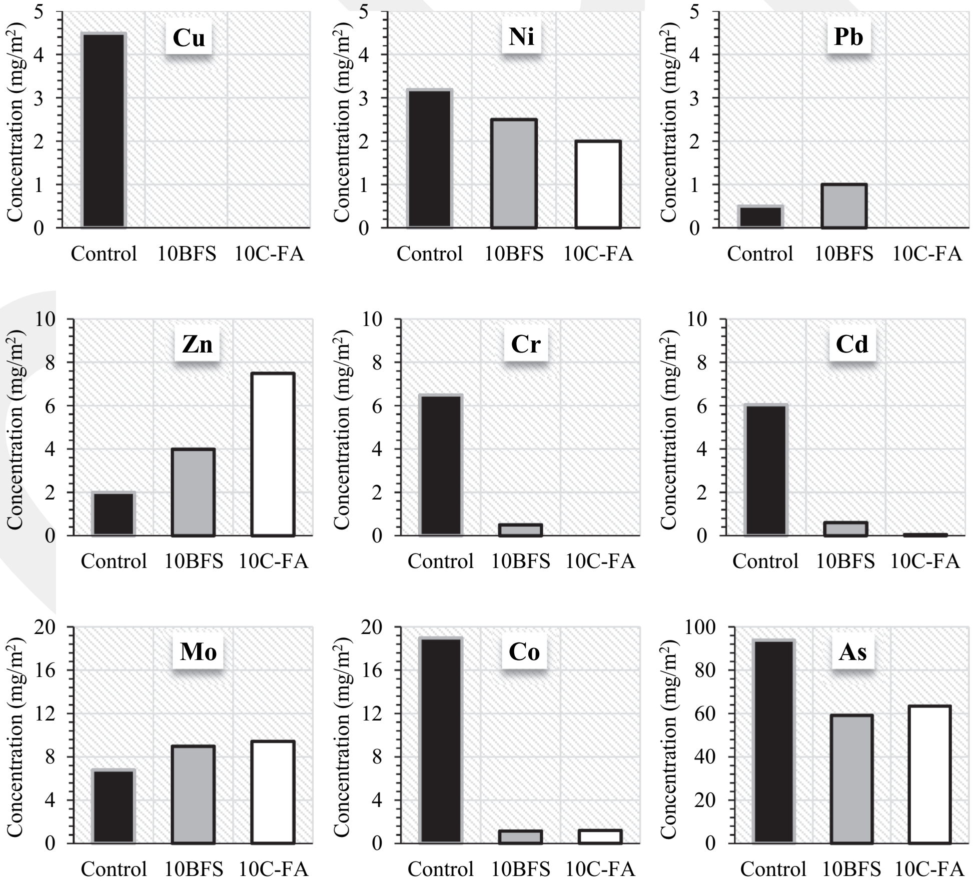

Figure 10. Total concentrations of HHMs released from CPBs during the DTL test.

Table 5. Maximum regulation limit-values for ground-water [22].

Heavy metals		Cu	Zn	Co	As	Ni	Pb	Cr	Mo	Cd
WHO	(mg/L)	2.0	3.0	-	0.01	0.07	0.01	0.05	0.07	0.003
	(mg/m^2)	201	301	-	1.0	7.0	1.0	5.0	7.0	0.3

considerably low (4.5 mg/m^2) compared with the limit-value (L-V) of Cu (201 mg/m^2) in ground-water [22] (Table 5).

The Ni-release from all the CPBs appears below the L-V (<7.0 mg/m^2) for ground-water (Table 5). Notwithstanding this, the highest release of Ni was found to occur from the Control (Figure 10). This in turn shows the beneficial effect of addition of AIPs on Ni mobilisation from CPB as also suggested by Chen et al. [36]. Hamberg et al. [8] reported relatively a high release of Ni (8.5 mg/m^2) from CPBs prepared at 1.0 wt.% cement and cured for 446 days compared with this study (i.e. 2.0–3.2 mg/m^2) (Figure 10). They also observed a significant reduction in the Ni-release (down to 3.3 mg/m^2) when the binder dosages were increased with the fly ash (3 wt.%) and the curing time was shortened to 31 days. These investigators have concluded that the slower flooding of CPB leads to high Ni-releases [8].

Figure 10 illustrates that higher mobility of Pb from the 10BFS than the Control, while, no Pb-release from the 10 C-FA was detected. This comparatively high Pb-release from the 10BFS is in fact consistent with high pH level recorded in the leachates of the 10BFS (*7.14–10.28 c.f. 7.03–7.62 for Control*) due to the increased solubility of Pb as expected with increasing the alkalinity above pH 10, in particular [8,36]. Despite the high pH in the 10 C-FA, the mobility of Pb from the 10 C-FA was not detected. This may be attributed to the amphoteric nature of lead solubility, which approaches to the minimum at around pH 9.0–9.5 [37]. In this regard, the pH of 10 C-FA sample steadily increased from 7.17 to 8.57 over the leaching period of 360 days. In addition, physical and chemical adsorption were reported to immobilise the heavy metals (i.e. Pb) in the hydration products and binder phases (CH, C-S-H, etc.) [10]. It is pertinent to note that the Pb-release from both the Control and 10BFS did not exceed the L-V (<1.0 mg/m^2) of Pb for ground-water (Figure 10 and Table 5). On the contrary to this study, the researchers [30,35] who produced different types of concrete samples to investigate the environmental behaviour of monolithic concretes reported high total Pb releases (≈9.0 mg/m^2 and 10.0–25.0 mg/kg, respectively) from all concretes in the DTL tests.

The Zn-release from all the CPBs was quantified to vary between 2.0 and 7.5 mg/m^2 which are below the L-V authorised by WHO [22] (Table 5). CPBs containing AIPs (10BFS and 10 C-FA) produced higher Zn-release than the Control in line with relatively higher alkalinity of their leachates generated [8,38]. A similar trend of increase in Zn-release (up to 10.0 mg/m^2) with increasing pH was also observed for concrete samples produced with different WRs [30]. Hamberg et al. [8] also reported the increased leaching of Zn with decreasing pH down to acidic levels. Mo also appeared to exhibit a similar leaching behaviour in character with its tendency of the increased release in the presence of AIPs (i.e. 6.8 mg/m^2 for the Control *c.f.* 8.98 and 9.42 mg/m^2 for the 10BFS and 10 C-FA). In fact, the release of Mo from the 10BFS and 10 C-FA seemed to be even higher than the L-V for ground-water (Figure 10 and Table 5).

The utilisation of BFS and C-FA in CPB mixtures appeared to remarkably reduce the Cr leaching. In this regard, the Control released 6.5 mg/m^2 Cr compared with 0.5 mg/m^2 Cr and no release from the 10BFS and 10 C-FA, respectively (Figure 10). Taha et al. [30] and Hamberg et al. [8] attributed such reduction in the leaching of metals/metalloids to the improvement in the encapsulation of metal-bearing phases within the C-S-H, which are denser in high-strength CPBs containing AIPs (*4.40–4.89 MPa c.f. 1.56 MPa for*

Control at 180 days) [3,19,34]. These findings concur with the results of Hamberg et al. [14]. They also reported low Cr-release from CPBs during the DTL test. The Cr-release from the Control appeared to be higher than the L-V (6.5 mg/m^2) in contrast to CPBs containing AIPs from which the leaching of Cr was well below the limit values (Figure 10 and Table 5).

The Cd-release from the 10BFS and 10 C-FA was limited only to 0.6 and 0.05 mg/m^2 compared with that from the Control with 6.0 mg/m^2. (Figure 10). This reduced release of Cd from the CPB samples of 10BFS and 10 C-FA could be associated with comparatively high pH levels maintained by these CPBs containing AIPs during the DTL tests, i.e. low solubility of Cd under these conditions and the expected improvement in the microstructure (total-porosity, macro-pores, meso-pores, etc.) of CPBs with the finer BFS and C-FA (Figure 11 and Table 6) as discussed below. As can be seen from Table 5, despite the significant reduction in the leaching of Cd, the 10BFS and Control (Figure 10) failed to meet the L-V for Cd release (0.3 mg/m^2). A similar trend for the leachability of Co from the CPBs was also observed with a remarkably high release of Co from the Control (19 mg/m^2) compared with 1.15 and 1.20 mg/m^2 for the 10BFS and 10 C-FA, respectively (Figure 10). Taha et al. [30] recorded a similar Co-release (≈2 mg/m^2) from concretes containing WRs during 64 day-DTL tests. It should be noted that no L-V is indicated for ground-water by WHO [22].

The release of As from the CPBs is of particular concern given its high toxicity. Figure 10 illustrates the leachability of As from the Control, 10BFS and 10 C-FA with the former releasing the highest concentration of As (93.8 mg/m^2). These observed levels of As-release (59.1–93.8 mg/m^2) appear to be well above the L-V (1.0 mg/m^2) (Figure 10 and Table 5). The addition of 10 wt.% of BFS and C-FA, respectively, to S-rT in CPB mixtures appeared to significantly mitigate the leaching of As presumably due to the beneficial effect of more alkaline pHs maintained (Figure 5). The higher As release in the Control can be also attributed to the increased formation of expansive gypsum (Figure 6(a)) leading to the loss of stability of CPB structure and the ineffective encapsulation of As bearing phases with the C-S-H at pH<9.0 [6,14]. At a high binder dosage (8.5 wt.%), the utilisation of AIPs could have improved the quality of CPB forming a dense CPB structure (Figure 11) with concomitant reduction in the diffusion of O_2 and the penetration of flooding solution into the CPB. Hamberg et al. [12] prepared CPBs utilising OPC (1 wt.%) and OPC-FA (2 wt.% OPC and 1 wt.% FA) for the evaluation of As-release from CPBs after 31 days of curing in the DTL tests up to 64 days. They showed that CPBs of OPC-FA produced considerably lower As-release than those of OPC. The findings are consistent with the current findings. However, Coussy et al. [5] produced a number of CPBs ((OPC: 100%), (OPC-FA: 50% OPC-50% Fly-ash) and (OPC-S: 20% OPC-80% Slag)) at 5 wt.% binder dosages. After 28 days of curing, these researchers studied the release of As from these CPBs in the DTL tests lasted up to 64 days. In contrast to the current findings, they observed a significantly higher release of As from the OPC-FA than those from the OPC and OPC-S (*≈540 mg/m^2 c.f. ≈140 and ≈200 mg/m^2 respectively*). They concluded that the utilisation of FA in CPB mixtures is not suitable for preventing or restricting the As-release. It is relevant to note that the BFS and C-FA used in the current study have higher CaO content (Table 1) than the FA used by Coussy et al. [5]. Therefore, the contradictory findings in the current study can be ascribed to high CaO content of the C-FA used at a high binder content (8.5 wt.%) providing higher Ca loading to mitigate As mobility. It is also pertinent to note that under these conditions the solubility/leachability of As is expected to be controlled essentially by the formation of Ca-As/Ca-Fe-As/Fe-As [39].

3.4. Effect of AIPs on CPB microstructure

To demonstrate the effect of microstructure on the geochemical behaviour of CPBs, the MIP (total- and incremental-porosity) analysis of the Control, 10BFS and 10 C-FA at 180 days was carried out

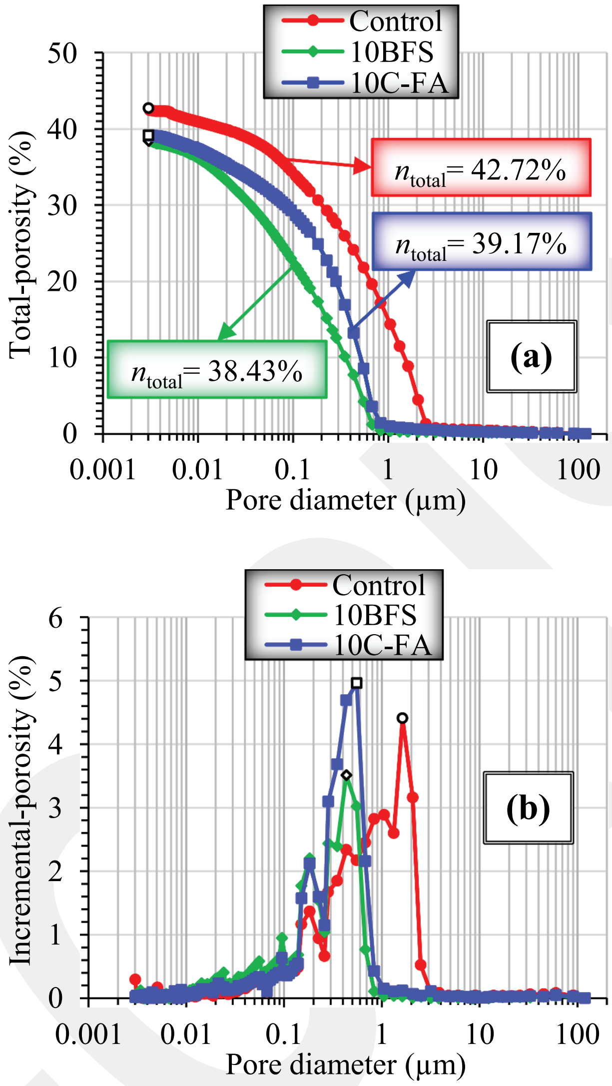

Figure 11. The total- (a) and incremental-porosity (b) values of Control, 10BFS and 10C-FA at 180 days.

Table 6. Technical parameters of the MIP curves at 180-day CPBs.

			Cumulative porosity (%)		
Sample name	Curing period (days)	n_{total} (%)	$^{a}n_{meso}$	$^{b}n_{macro}$	[c]Critical pore diameter (μm)
Control	180	42.72	5.23	37.49	1.63
10BFS		38.43	10.78	27.65	0.43
10 C-FA		39.17	7.08	32.09	0.55

[a] n_{meso} porosity (%): Mesopores range between 0.002 and 0.05 μm.
[b] n_{macro} porosity (%): Macropores have pore size larger than 0.05 μm.
[c] Critical pore diameter (μm): Critical pore size represents the steepest slope of the cumulative porosity curve [43].

(Figure 11). The total-porosity (n_{total}) of the Control was determined to be 42.72%. In comparison, the 10BFS and 10 C-FA were found to have a lower n_{total} of 38.43% and 39.17%, respectively (Figure

11(a)). It is also evident from Figure 11(b) that the BFS and C-FA developed discernably lower pore diameters than the Control.

The utilisation of BFS and C-FA at the replacement level of 10 wt.% to S-rT appeared to considerably improve the macro-pores (n_{macro}) and meso-pores (n_{meso}) of CPBs (Table 6). The macro-pores (n_{macro}) values of the 10BFS and 10 C-FA are 26.25% and 14.40%, respectively, lower than that of the Control. Similarly, about 2.06- and 1.35-fold increase in the meso-pores (n_{meso}) of the 10BFS and 10 C-FA, respectively, were also recorded in comparison with the Control. These findings are consistent with the previous studies [3,19] in which the CPBs of the BFS and C-FA, which were added as replacement (15%) of the construction and demolition waste produced lower n_{macro}-values (25–38%) and higher n_{meso}-values (2.03–2.72 fold) than those of the Control at 180 days. Furthermore, the results of critical pore diameter (d_{cr}), which has a profound effect on the hydraulic conductivity characteristics (permeability, fluid transfer, etc.) of cement-based materials (i.e. CPB) [18,40], revealed that the addition of AIPs (BFS and C-FA) considerably reduced the d_{cr}-values (73.62% and 66.26%, respectively) compared with the Control. (Table 6). These findings suggest that the beneficial effect of addition of AIPs can be attributed to the improved quality/microstructure of the CPB. In this respect, it was reported that the penetration of flooding water into pores and fluid transmission from pores in CPBs would be restricted by reducing the permeability [8,41,42].

4. Conclusion

This research aimed to develop an understanding for: (1) the geochemical behaviour of CPB in laboratory conditions, and (2) the effect of different AIPs (BFS and C-FA) on the geochemical parameters (pH, Ec, SO_4^{2-}, OP-NPs curves and HMs concentrations) of CPB. The DTL tests were carried out on the cured CPBs with and without the addition of AIPs as 10% replacement to S-rT. Furthermore, the mineralogy and microstructure of the tested CPBs were also examined by conducting the XRD and MIP analyses to provide an insight into the findings. The following conclusions can be drawn from this research;

(a) All leachates obtained from the flooding of monolithic CPBs in DTL tests appear to maintain alkaline conditions (*pH≥ 7.03*) irrespective of the leaching times. However, the 10BFS and 10 C-FA consistently produced higher pHs (e.g. 10.3 and 8.6, respectively, at the end of the last cycle (360 days)) than the Control sample (7.03), consistent with their respective neutralisation capacity.
(b) The leachability of SO_4^{2-} from the CPBs containing AIPs was lower than that of the Control at all leaching cycles. This could be attributed to the improved microstructure/quality of the CPB sample by the utilisation of AIPs limiting the oxygen access into CBP.
(c) The plot of SO_4^{2-} (i.e. primary oxidation product, OP) vs Ca+Mg+Mn (neutralisation products, NPs) suggested that the Control sample would probably continue to generate acid as indicated by a sharp decline in NPs whereas the AIPs had still adequate neutralisation potential to buffer the acid that may be generated by sulphide oxidation.
(d) The results of leachability of HMs demonstrated that the addition of AIPs which have remarkably improved the quality of CPB considerably mitigated the release of most metals/metalloids except for Mo and Zn.
(e) Only the release of Mo and As from the CPBs of AIPs were found to exceed the limit-values (L-V) for ground-water, whilst, the Control generated leachates higher in Cr, Cd and As than the L-Vs. Being well above the L-V, the release of As from all the CPB samples is of particular concern.

It can be deduced from these results that the utilisation of AIPs (BFS, C-FA, etc.) in CPB mixtures can significantly improve the quality and environmental impact of CPB. Furthermore, future work should focus on the development of high-quality CPB designs to further improve environmental performance of CPB operations with particular reference to the control of the release of more recalcitrant pollutants such as As.

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Disclosure statement

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