

Use of Cemented Paste Backfill Based on Arsenic – Rich Tailings from Cyanidation

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ABSTRACT

Gold is extracted by cyanide leaching from inclusions in arsenopyrite from a mine in the north of Sweden. The major ore mineral assemblage consists of pyrrhotite and arsenopyrite-loellingite. Effluents from the gold extraction are treated with $\text{Fe}_2(\text{SO}_4)_3$, with the aim to form stable As-bearing Fe-precipitates (FEP). The use of cemented paste backfill (CPB) is one alternative suggested for management of tailings. In CPB, tailings are commonly mixed with water (typically 25%) and low proportions (3-7 %) of binders and backfilled into underground excavated areas. Tailings containing As (1000 ppm) mainly as As-bearing FEP:s were mixed with low proportions (1-3 %) of biofuel fly ash(BFA), ordinary cement (CE) and water for the formation of a monolithic CPB-mass. Two mixtures were blended; CE with 1 wt. % of cement and CE-FA consisting 2 wt. % of CE and 1 wt. % of BFA. Unmodified tailings and CPB-monoliths were submitted to a tank leaching test (TLT) for comparison of the leaching behavior of As in the tailings and CPB-masses respectively. Results from the TLT showed that the inclusion of As-rich tailings into a cementitious matrix increased leaching of As. In CPB-mixtures, small (> 1%) proportions of As desorbed from FEP:s and were subsequently incorporated into less acid-tolerant species (i.e. Ca-arsenates and As bonded to cementitious phases). CE was least effective in As-immobilization; the As-release was related to pH and the dissolution of Ca-arsenates. In CE-FA, As-release followed the behavior of Si suggesting the incorporation of As with Calcium-Silicate-Hydrates (C-S-H) in the cementitious matrix. Unmodified tailings generated an acidic environment in which As-bearing FEP: s was stable. As-leaching increased in CPB-mixtures on a short-term basis, but buffering minerals in CPB-mixtures could decrease the oxidation rate of pyrrhotite, which in turn, stabilizes pH and the As-bearing FEP: s.

Keywords: Arsenic, Cemented Paste Backfill, Cyanidation

INTRODUCTION

Gold ores and concentrates containing arsenic minerals (i.e. arsenopyrite) are often refractory in nature, meaning the minerals need to be dissolved in order to release the associated gold. Arsenopyrite usually co-exists with other sulphide minerals such as pyrrhotite and pyrite. Arsenic does not form stable complexes with cyanide, but it is very soluble during cyanidation. Consequently; the slurry produced in this process must be treated to immobilize As before discharge into tailings facilities. Cemented paste backfill (CPB) is a method where tailings are converted into a monolithic mass by the addition of low proportions (3-7 %) of binders. CPB focuses on the minimization and/or enhancement of the quality of percolating water. CPB offers moreover a geotechnical support to underground mine cavities increasing operational benefits for the mining industry (Coussy *et al.*, 2011). CPB is also used for backfilling of non-load bearing constructions or tailings dams where strength of the CPB-material is of less concern, but aims for the prevention of metal release and reduction of Acid mine drainage (AMD) remains. Immobilization of As in CPB-materials have been attributed to the formation of stable As-precipitates, the entrapment of As in the cementitious matrix by sorption onto calcium-silicate-hydrates (C-S-H) or substitution within the crystal lattice of secondary cementitious minerals (Coussy *et al.*, 2011). The most common binder material used in CPB is cement. As amounts of tailings often exceed millions of tonnes, the cost of cement becomes substantial and many studies have been done to promote the usage of alternate materials in CPB. Granulated blast furnace slag (GBFS) and biofuel fly ash (BFA) have successfully been used for partial replacement of cement in CPB due to their pozzolanic and alkaline properties (Kim & Jung, 2011; Coussy *et al.*, 2011). GBFS and BFA could neutralize acidity arising from sulphide-rich tailings and prevent trace element spread (Coussy *et al.*, 2011). However, Bertrand (1998) showed that low proportions (0.9-6.8 %) of slag and cement could be insufficient to mitigate Acid Mine Drainage (AMD)-production long-term. It is believed that alkaline materials (fly ashes, slags) increases the mobility of As, but some contradictory results have shown a reduced mobility by the possible formation of stable Ca-As complexes (Hartley *et al.*, 2004; Randall, 2012).

Cyanidation tailings slurries from As-enriched sulphide ores are often treated with lime, H₂O₂ and O₂ and Fe₂(SO₄)₃ to co-precipitate As (V) with Fe forming stable As-bearing Fe-precipitates (FEP) and to increase the pH to prevent generation of AMD. Within these treatment processes, precipitates with varying Fe/As-ratios could form. As-bearing FEPs with a Fe/As < 4 are assumed to be stable under oxidized conditions when pH 4-7 (Riveros *et al.*, 2001). The pH stability range of As-bearing FEPs is broadened with increasing Fe/As ratios and precipitates with Fe/As ratios ≥ 8 are stable over a wide pH range (3-8). Raising pH to alkaline levels may increase the risk for destabilization of As-bearing FEP. Previously conducted studies (Benzaazoua *et al.*, 2004, Coussy *et al.*, 2011; Randall, 2012; Coussy *et al.*, 2012) have focused on the stability of arsenopyrite, scorodite or spiked As in natural or synthetic CPB. The stability of As-bearing FEP:s in CPB-materials has gained less attention. The purpose of this study was therefore to investigate the behavior of As in a CPB-material using the tank leaching test (TLT).

MATERIALS AND METHODS

Tailings were collected from a gold mine in the north of Sweden. The major sulfide minerals are pyrrhotite(6.5 wt. %)and arsenopyrite-loellingite(0.2 wt. %). The gold occurred primarily as inclusions in arsenopyrite (Sciuba, 2013). Tailings were treated with Fe₂(SO₄)₃, H₂O₂, CuSO₄ and

lime in order to form stable As-bearing FEP:s, and detoxify residual cyanides. Samples were collected at depths of 0-30 cm from ten different locations on the tailings dam and mixed to form a bulk sample of approx. 15 kg. Two types of binders were tested for the preparation of various paste mixtures: Portland cement (Ordinary Portland Cement also named CE) and biofuel fly ash (FA) provided from a biofuel incineration plant located in Lycksele, Sweden. Main elements and total contents of As and S in tailings, CE and CE-FA are presented in table 1.

Table 1 Main elements, total content of S and As in tailings, CE and CE-FA (n = 3, ± SD)

	Unit	Tailings	FA	CE
SiO ₂	% of TS	55.0 ± 4.9	34.6 ± 1.3	20.6 ± 0.8
Al ₂ O ₃	"	4.69 ± 0.04	10.7 ± 0.6	5.61 ± 0.45
CaO	"	4.83 ± 0.25	14.1 ± 1.0	50.3 ± 1.8
Fe ₂ O ₃	"	16.7 ± 0.6	13.9 ± 1.0	2.81 ± 0.05
K ₂ O	"	0.92 ± 0.03	2.89 ± 0.08	0.83 ± 0.05
MgO	"	3.24 ± 0.01	2.54 ± 0.08	4.00 ± 0.17
MnO	"	0.14 ± 0.01	0.92 ± 0.03	0.10 ± 0.01
Na ₂ O	"	0.82 ± 0.03	1.24 ± 0.08	0.65 ± 0.07
P ₂ O ₅	"	0.24 ± 0.01	2.25 ± 0.05	0.06 ± 0.00
TiO ₂	"	0.18 ± 0.00	0.34 ± 0.01	0.42 ± 0.02
As	mg/kg TS	1070 ± 30	124 ± 5	10.2 ± 0.2
S	"	20933 ± 493	13700 ± 200	9960 ± 219

Speciation of As and S in tailings was assessed using the modified sequential extraction scheme described by Dold (2003) and suggested that a majority (95 %) of As in the tailings of Svartliden was associated with the Fe (III)oxy-hydroxides-fraction (Table. 2). The majority of S was associated with the water-soluble fraction and the primary sulphides fraction.

Table 2 Speciation of As and S in tailings from Svartliden, extraction scheme adopted from Dold (2003) (values in % of total content).

Fraction	Water soluble	Exchangeable	Fe(III) oxy-hydroxides	Fe(III) – oxides	Secondary sulphides	Primary sulphides	Residual
As	0.00	0.81	94.6	1.81	0.02	2.75	0.03
S	35.0	10.8	6.99	5.36	4.41	37.5	0.00

Preparation of the CPB-material

Tailings were mixed with the binders, and distilled water was slowly added to reach a solid percentage about 73-74 %. A water content of 26-27% in the CPB-mixtures is required for paste transportation through a pipe network. CPB-mixtures of CE, FA, water and tailings were poured into plastic bottles with a diameter of 5 cm and a height of 10 cm. A small hole was drilled in the bottom of each bottle before filling for the drainage of excess water. After filling, the bottles were sealed and cured for 28 days in 22°C in a moist environment. Two mixtures were prepared: CE, with the addition of 1 wt % ordinary portland cement (OPC) and CE-FA, with an addition of 2 wt % OPC and 1 wt % biofuel fly ash. Proportions of binders and tailings were adjusted to achieve required hardened strength characteristics of 200 kPa with a minimal addition of binders. Tests of unconfined compressive strength (UCS, curing time of 28 days) on the CPB-materials showed that the mechanical strength of CE and CE-FA were 207 kPa and 179 kPa respectively.

Tank leaching test (TLT)

The Dutch standard (EA NEN 7375:2004) was implemented. TLT consists of the leaching of a monolithic block. CPB-samples were taken out of the bottles after 28 days of curing and rectified into regular cylinders. CPB-monoliths were applied on plastic supports at 2 cm from the bottom of the tanks. Distilled water was thereafter filled to reach a level that ensured a 2 cm water head surrounding the CPB-sample and a liquid/solid (L/S) ratio fixed to 10cm³ of solution/cm² of exposed solid for all samples. A reference sample consisting solely of tailings was used for the comparison of cemented and unmodified tailings. Tailings (reference) were placed in paper filter bags (1µm) recessed in nylon filters. Nylon filters were then hung up in the lids of the tanks. Duplicates were realized for each CPB-sample, a total amount of 6 samples were leached at the same conditions. Leachate was removed and replaced after 6h, 1day, 2.25 days, 4 days, 9 days, 16 days, 36 days and 64 days. Leachate was stirred manually before collection and analysis. The same volume of leachate was used for each renewal. Leachates were filtered through a 0.45-µm membrane filter followed by measurement of pH, Eh and conductivity and analyzed for total content. The mass transfer of As in tailings and CPB-materials was calculated according to (EA NEN 7375:2004):

$$M_{ti} = (C_i \times V_i) / A \quad (\text{eq. 1})$$

M_{ti} (in mg/m²) is the calculated as mass of As released during a leaching period, i , C_i (mg/l) is the As concentration in the period i , V_i (L) is the leachate volume in period i and A is the specimen surface area exposed to the leachate (m²). The apparent diffusion coefficient of As was determined using the logarithm of cumulative M_t until the i -th period, plotted vs. the logarithm of time. Modelling was conducted using PHREEQC, adapting the database developed by Blanc et al., (2014). Element concentrations from the leachate in the TLT were used for the identification of arsenic species.

Sample analysis

Concentrations of 31 elements were determined for solid samples of tailings, cement and biofuel fly ash with triplicate samples. The modified EPA method 200.7 (ICP-AES) and 200.8 (ICP-SMS) (US EPA, 1991) were used and performed by an accredited laboratory (ALS Scandinavia, Luleå, Sweden). Water samples from the TLT were analyzed by an accredited laboratory (ALS Scandinavia, Luleå, Sweden) to determine their elemental composition. Elements (As, Cd, Cr, Cu, Mn, Ni, P, Pb and Zn) were determined by ICP-SFMS while Al, Ca, Fe, K, Mg, Na and S were determined by ICP-AES. ICP-SFMS and ICP-AES analyses were performed using modified versions of EPA methods 200.8 and 200.7 (US EPA, 1991), respectively.

RESULTS

In TLT, the highest As concentrations released during a leachate renewal cycle were 41.3 mg/m² observed for CE, 13.7 mg/m² for CE-FA and 1.6 mg/m² for tailings. The cumulative release of As is most pronounced in CE (101.5 mg/m²), and least in tailings (5.3 mg/m²). Leaching of As from tailings descends throughout the test but has an increasing trend in both CPB-mixtures (CE and CE-FA). The release of Fe was more than a 100-fold larger in tailings compared to that in the CPB-materials. The evolution of Si and As were closely related in CE-FA but showed less or no correlation in CE and tailings (Fig.1).

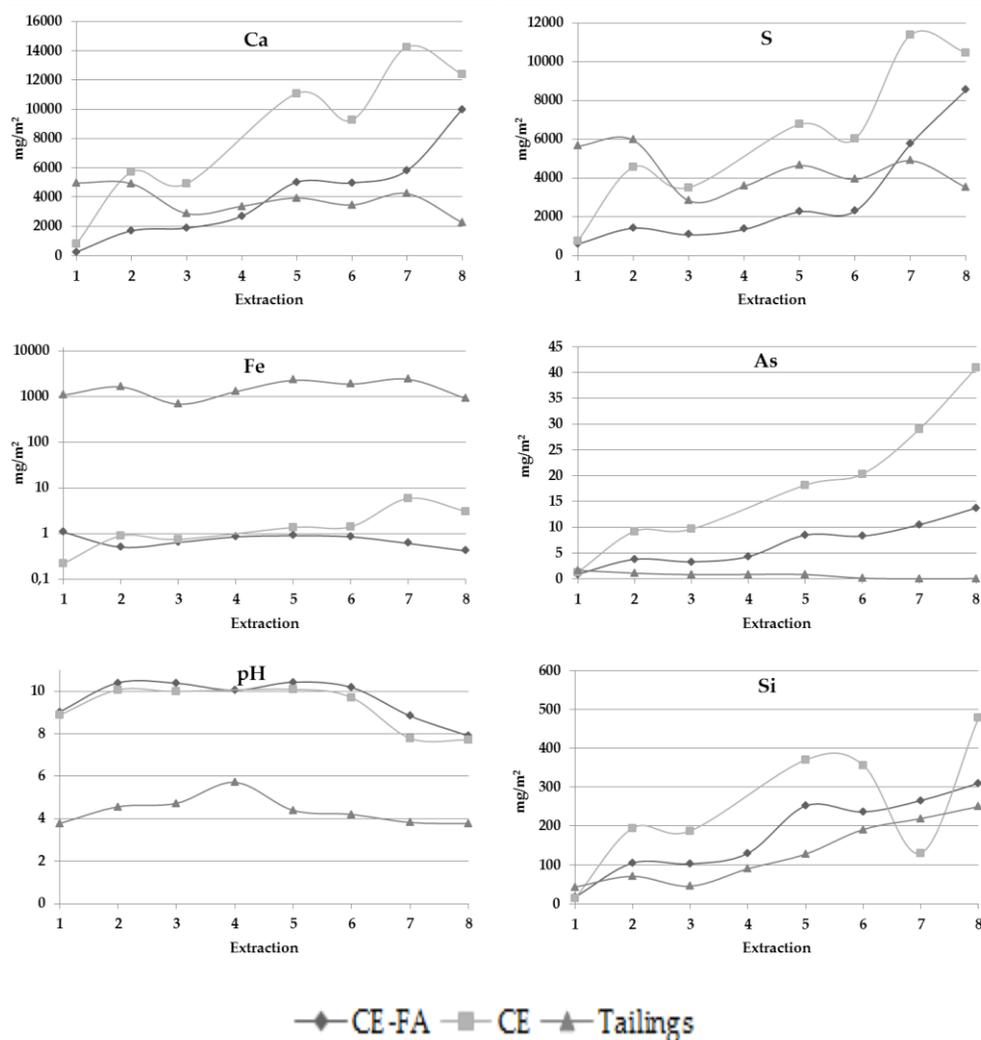


Figure 1 Evolution of pH, S, Ca, Fe, As and Si in tailings, CE and CE-FA during TLT.

For As dissolving from the surface and for the short initial release of surface-deposited As (surface wash-off), the graph slopes (r_c) are expected to be <0.4 . In the case of a diffusion control mechanism, this plot is expected to be a straight line with a slope of $0.4-0.6$ (EA NEN 7375:2004).

Table 3 Mass transfer mechanisms in tailings and CPB-materials during TLT

Mix	Cumulative As mg/m ²	r_c	Mechanism
Tailings	5.5 ± 0.7	0.21 ± 0.09	Wash-off
CE	98 ± 4	0.62 ± 0.01	Diffusion
CE-FA	53 ± 0	0.46 ± 0.13	Diffusion

According to calculations based on the standard of (EA NEN 7375:2004), the mechanisms for As-leaching was a wash-off-effect in tailings and an effect of diffusion in the CPB-materials (Table. 3).

DISCUSSION

The cumulative release of As was highest in CE ($98 \pm 4 \text{ mg/m}^2$), followed by CE-FA ($53 \pm 0 \text{ mg/m}^2$) and comparatively less in tailings ($5.5 \pm 0.7 \text{ mg/m}^2$). Oxidized conditions prevailed in CE, CE-FA and tailings throughout the test and became more oxidizing with time.

As-leaching in tailings during TLT

The As-release was low in tailings during TLT and pH varied from 3.8-5.7. Previously conducted sequential extraction tests suggest that the majority of As was associated with FEP (Table. 2). Calculations in accordance to the TLT-standard suggested that leaching of As in tailings was predominately a wash-off-effect (Voglar and Leštan, 2013). In alkaline conditions during cyanidation, As-sulfides are oxidized to arsenite and arsenate in the presence of oxygen. The proportion of each species is dependent on solution composition, redox potential and pH. Release of As was initially higher from the tailings than from the CPB-materials. This could be due to the presence of As (III) in tailings pore water that was suggested by modeling. The presence of As (III) could be due to insufficient pre-oxidation of As (III) to As (V) during treatment processes.

FEP:s could become unstable at $\text{pH} < 4$, releasing adsorbed As into solution. The As-release was descending from the 2nd extraction in the TLT when pH was 5.7 -3.8 (Fig. 1). In the middle of the TLT-test, a reddish crust appeared on the tailings surface. This crust was assumed to be a Fe-precipitate that might decrease the permeability and weathering ability. The newly formed crust of Fe-precipitates could provide additional surfaces amenable for As-adsorption obstructing further As-release. The ability of FEP for adsorbing As (V) could be reduced due to competition of sorption surfaces by high concentrations of SO_4^{2-} in the leachate (Frau et al., 2010). The negative effect of SO_4^{2-} on the adsorption of As onto FEP could potentially be counteracted by the co-adsorption of Ca^{2+} , which may function as a multi-absorbent for As at pH values of 4-6 (Jia and Demopoulos, 2005).

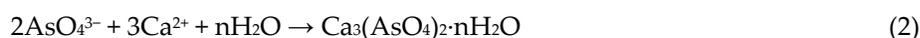
As-leaching in CPB-materials (CE and CE-FA) during TLT

Initially, the cementation has decreased leaching of Ca, S, Fe, As and Si. According to modeling, As-release in the CPB-materials was governed by As (V) and Ca-arsenates. Initial release of As (III) in tailings could have been hindered by the addition of binder materials such as cement and fly ash which promotes the oxidation of As (III) to As (V) (Moon *et al.*, 2008). Calculations in accordance to the TLT-standard suggested that leaching of As in CPB-materials is predominately an effect of diffusion (Table 3). A physical entrapment of As into the cementitious matrix could hinder the effect of As-diffusion in the CPB-materials. The hydration of pozzolanic additives under alkaline conditions forms secondary growths of calcium silicate hydrates (C-S-H). These C-S-H-products serve to improve the microstructure of CPB, leading to lower porosity and permeability mainly due to mineral precipitation (Benzaazoua *et al.* 1999; Hassani *et al.* 2001). The permeability of a CPB-material could increase due to a phenomenon called sulfate attack where sulfates react with Ca-ions forming swelling secondary phases such as gypsum. The volume of C-S-H: s could also be far in excess of the available pore volume and cause internal stress and subsequent micro-cracks increasing permeability in the CPB-material (Fall & Benzaazoua, 2005).

For CE-FA-samples, As release follows the behavior of Si. Arsenic may then have been incorporated into the matrix of (C-S-H) as suggested in Coussy *et al.*, (2011). In CE-FA where pH varies from 10.5 to 8.0, all hydrated cement phases were assumed to have been dissolved (Ayora *et al.*, 1998). The content of soluble S-species in tailings (approx. 11,000 ppm, Table. 2) increases the risk for a sulfate

attack to occur within the CPB-material (Fall & Benzaazoua, 2005). However, CE-FA includes BFA and Hassani *et al.* (2001) suggested that mixtures of fly ashes and cement in CPB-materials are more resistant to sulfate attack than cement alone.

The mechanisms of As-release in CE differed from the mixture of CE-FA. The As-release in CE was less correlated to the behavior of Si and increased more strongly as pH decreased from 10 to 8. The point of zero charge (PZC) for As-bearing FEP: s occurs at a pH≈8.3. Alkaline conditions (pH: 7.7-10.4) in CE suggested that As has been desorbed from FEP: s and re-precipitated as Ca-arsenates. In alkaline conditions observed in the CPB-materials, As (V)-species of HAsO_4^{2-} and AsO_4^{3-} are the most probable As-species (Smedley & Kinniburgh, 2002). These anions could react with calcium ions in the pore water forming Ca-arsenates according to reactions 1 and 2 (Ahn *et al.*, 2003):



Ca-arsenates formed in $\text{pH} > 10$, are highly soluble at lower pH (Benzaazoua *et al.*, 2004). The presence of Ca-ions in the pore water could however increase the surface charge density of the FEP: s increasing their capacity for As (V)-adsorption. This effect is most evident at pH: s of 8-10 (Wilkie & Hering, 1996). The addition of binders lowered Fe-leaching, alkaline environments could enhance the precipitation of FEP: s forming crusts on the sulfide surfaces. These crusts form rapidly under alkaline conditions, creating a thin permeable coating obstructing the minerals' further oxidation (Belzile *et al.*, 2004, Asta *et al.*, 2013).

Leaching of As in the CPB-mixtures was most prominent when pH fell from 10 to 8 in the end of the TLT. This is probably due to a physio-chemical effect where cementitious phases sensitive to a pH-decrease have dissolved enabling more water to percolate through the monolith. More arsenic was released from CE. Increasing the binder-proportion will not necessary increase the strength of the material, but more swelling minerals could form and decrease the permeability.

Future aspects concerning the use of CPB on As-rich tailings from cyanidation

Most of the As in the tailings were present as As-bearing FEP: s which constituted approximately 95 % of the total As. Arsenic in these precipitates is sensitive to reductive, extreme acidic or alkaline environments. Arsenic dissolutions under reductive environment may contribute to the As release into solution when the management of tailings are not adequately designed. Although, results from the tailings TLT showed that As-bearing FEPs may be stable under acidic and oxidizing conditions common to the tailings piles, more extensive pyrrhotite oxidation generating extreme acidic conditions may increase As-leaching on a long term basis.

Results from TLT suggested that the addition of binders may increase As-leaching on a short term basis. However, the addition of binders increased the tailings' acid-neutralizing capacity and introduced more Ca-ions and Fe precipitates into the tailings matrix, both of which may facilitate the immobilization of As and reduce the potential for sulfide oxidation. Another positive effect originating from the formation of a monolithic mass is that the high degree of saturation in the material reduces the intrusion of oxygen and water percolation. Pyrrhotite oxidation is not obstructed, but in alkaline conditions, Fe may precipitate on the surfaces of sulfide grains retarding its further oxidation. The addition of binders may, therefore, have a positive effect on the stability of As-bearing FEP: s on a long term basis.

CONCLUSIONS

The inclusion of As-rich tailings into a cementitious matrix increased leaching of As. Alkaline conditions in CE and CE-FA during TLT caused a small proportion of As to desorb from As-bearing FEP: s. Desorbed As could then have been incorporated into the cementitious matrix and/or re-precipitated as Ca-arsenates. Leaching of As in CE-FA followed the behavior of Si, which implies that the dissolution of cementitious phases governed the As-release. The mechanism of As-leaching in CE differed from that in CE-FA. Leaching of As in CE increased more strongly as pH decreased from 10 to 8, suggesting the formation of Ca-arsenates. As-bearing FEP: s was stable in tailings where pH was 3.8-5.7, leaching of As was low and descended during TLT. The addition of binders could have a positive effect of As-leaching long-term due to increased acid-neutralizing capacity and higher proportions of FEP: s. This, in turn, could facilitate As-adsorption and decrease the sulfide oxidation capacity.

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