

Study of the effects of arsenic trioxide roaster waste dusts on the mechanical behaviour of cemented paste backfills

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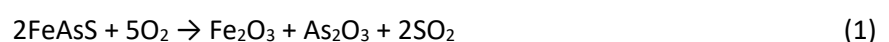
Abstract

More than 237,000 metric tonnes of arsenic trioxide roaster waste dusts have been stored underground in the abandoned Giant Mine (Yellowknife, NWT). This waste arsenic trioxide material is approximately 60% arsenic, which is hazardous to both people and the environment. Long-term management of this waste is complex due to its large quantity, physical characteristics, and current storage conditions. Currently, the frozen block method was selected for the stabilisation of the arsenic. However, because of climate change and decline in the permafrost, as well as the current toxic form of dusts, there are some critical concerns about the long-term performance of this technology. Therefore, more permanent arsenic stabilisation techniques must be considered. Among these techniques, cemented paste backfill (CPB) technology as a high-density slurry mixture of binding materials, dewatered tailings, roaster waste dusts, and mixing water, can be considered as a potential arsenic trioxide stabilisation method. In this research, the effects of the addition of the arsenic trioxide dusts (10% wt.) within CPB on the mechanical strength of the cured pastes were evaluated. The CPB samples were prepared using general use (GU) cement and a mixture of GU cement and lime kiln dust (LKD) as binding agents based on the mix designs proposed by the response surface methodology. The solid content, binder type and dosage, and curing time were selected as the variables and the unconfined compressive strength of the samples was chosen as the response of the modelling. The results of the experiments and analyses revealed that the incorporation of arsenic trioxide dusts within the CPB results in the reduction in the strength of the pastes. However, increasing binder dosage, as well as solid content, could compensate for this adverse effect. Moreover, the CPB prepared using GU cement showed higher strength than the ones incorporating GU cement/LKD.

Keywords: *arsenic trioxide dusts, arsenic stabilisation, cemented paste backfill, unconfined compressive strength, response surface methodology*

1 Introduction

The former Giant Mine is located adjacent to Great Slave Lake, about 5 km north of Yellowknife in the Northwest Territories, Canada. This mine is an abandoned gold mine that operated from 1948 until 1999. The Giant Mine ore had a complex mineralogy. Most of the gold occurred as extremely fine-grained particles that were 'refractory', i.e. mainly encased and hosted within the crystal structure of arsenopyrite (FeAsS) and to a lesser degree arsenian pyrite (Fe(As,S)₂). An oxidation process was required to convert the dense sulphide grains into porous structures and expose the gold to cyanide leaching solutions (Northwest Consulting Limited 2003). The roasting process in the Giant Mine produced SO₂ emissions, and As-rich vapours from arsenopyrite concentrates that precipitated as arsenic trioxide (As₂O₃)-rich dust from the oxidation of As (-I) to As (III) (Bromstad et al. 2017):



The collected arsenic trioxide dust was pumped underground into 10 purpose-built chambers and five mined-out stopes. Over 237,000 metric tonnes of arsenic trioxide were collected as a by-product of roasting and more than 17 million tonnes of tailings were also produced (Silke 2009, 2013).

The arsenic trioxide dust is approximately 60% arsenic, hazardous to both people and the environment and can dissolve in water (SRK 2002). Long-term management of the arsenic trioxide dust located at Giant Mine is complicated due to its large quantity, physical characteristics (dust-like), and current storage conditions (subsurface chambers and stopes). Because of this complexity, several different technologies, or methods, were assessed to determine the most promising stabilisation option (Arcadis 2017). The 'Frozen Block' method was judged to be the most appropriate management approach available at the time. It is a remedial method currently being implemented at the Giant Mine that isolates the dust by freezing the ground surrounding the dust chambers and stopes. This method was considered as an interim solution for a maximum of 100 years; however, due to the climate change and decline in the permafrost, the current toxic form of the arsenic trioxide dust requires a more permanent solution (SRK 2002; Arcadis 2017).

Significant environmental impacts of the surface storage of hazardous and sulphide-rich wastes under atmospheric conditions (i.e. AMD formation and release of the heavy and toxic metals as well as contamination of the soils and groundwaters) may be significantly attenuated by safely placing such wastes underground (Yilmaz et al. 2013), the process being known as backfilling (Ercikdi et al. 2009). Mine backfill refers to waste materials such as waste rock, deslimed, and whole mill tailings, quarried and crushed aggregates placed into underground mined voids for either disposal and/or to perform some engineering function (Benzaazoua et al. 2002). Generally, there are three types of backfilling methods: hydraulic fill, rockfill, and cemented paste backfill (CPB). In recent years, the cemented paste backfill (CPB) has gained worldwide acceptance, especially in Canada, to serve as an essential part of the underground mining operations (Ercikdi et al. 2009, 2014). CPB technology is known as a high-density slurry mixture of dewatered tailings, binding agents, and water, thickened to obtain a non-settling character for transport into mined cavities. CPB could provide environmental advantages by reducing the reactivity potential of sulphides and by stabilising pollutants. The hydraulic binders can react during hydration with metals and metalloids to form stable compounds by precipitation (Coussy et al. 2011). Mechanical strength, workability, environmental performance, hydraulic conductivity, and durability are the most important design criteria of CPBs structures. These parameters depend on the physical, chemical, and mineralogical properties of the mine tailings and other wastes, mixing waters, additives, binder types, and their proportions (Benzaazoua et al. 2002; Thompson et al. 2012). Unconfined compressive strength (UCS) can be considered the most common parameter used to determine the stability of the CPB structure due to the simplicity and low cost of the UCS test (Chen et al. 2017).

In previous studies, the CPB technology has been successfully engaged to stabilise arsenic-bearing materials (Coussy et al. 2011, 2012; Hamberg et al. 2015a, 2015b; Bull & Fall 2020a, 2020b; Zhang et al. 2019). However, there is a lack of experience in stabilising arsenic trioxide using CPBs. Based on the available knowledge on stabilisation mechanisms of arsenic in cemented paste backfills, it seems that the Giant Mine arsenic trioxide dust could be incorporated into cemented paste backfills for stabilising this hazardous waste material as well as decreasing its long-term leaching potential. So, the object of the current research is to assess the feasibility of incorporating arsenic trioxide roaster waste dust into cemented paste backfill for arsenic stabilisation. The mechanical behaviour of the CPB prepared with different binders at various binder and solid contents to obtain the most appropriate binder/s and mixing ratios was assessed and response surface methodology (RSM) was used to define the mixing design and obtain the optimal recipes. Mechanical strength was used in this study as an indicator of cementation reactions that favour contaminant stabilisation.

2 Materials and methods

2.1 Materials

The main materials for the preparation of the cemented paste backfills in this study consist of two binding agents, mine tailings, arsenic trioxide dusts, and deionised water. Tailings samples were taken from the north pond of the Giant Mine site. A trench was dug 1.8–2.1 m before taking the sample. After transferring the tailings to the laboratory, they were oven-dried at 40°C for 48 hours before carrying out the preparation, homogenisation, and characterisation processes. GU Portland cement supplied by Bomix (Quebec, Canada) was used as the main binding agent for the preparation of the cemented paste backfill. A mixture of GU cement and lime kiln dust (LKD) with a 50/50 ratio was also used as the binding agent. Arsenic trioxide dusts from the Giant Mine were used as the arsenic source. The dust samples were sent to the laboratory in eight different samples from different stopes and chambers. After some preparation and homogenisation processes on the received samples, a mixed representative sample was obtained for the characterisation and utilisation in CPB samples. Deionised water was used for the preparation of cemented paste backfill samples to eliminate the effects water chemistry on CPB behaviour.

2.2 Methods

2.2.1 Initial characterisation

The specific gravity (G_s) of tailings and dust, GU cement, and LKD was measured using an automatic gas pycnometer (Ultracyc 1200e, Quantachrome Instruments). The particle size distribution (PSD) of tailings and dust was analysed by a laser diffraction particle size analyser (Malvern Mastersizer 3000) to obtain the size corresponding to 10, 30, and 60% by weight of passing (D_{10} , D_{30} , and D_{60} , respectively) and the coefficients of uniformity (C_u) and curvature (C_c). The specific surface of tailings and dust was analysed by the Brunauer-Emmett-Teller method using liquid nitrogen (Gemini 2375, Micromeritics). The chemical composition of the GU cement and LKD, as well as the dust and tailings, were obtained by X-ray fluorescence spectrometry (S2 Ranger, Bruker AXS). Inductively coupled plasma atomic emission spectroscopy (ICP-AES) was used to determine the concentrations of the elements such as Al, As, Ca, Cd, Fe, K, Mg, Mn, Na, and S. Inductively coupled plasma mass spectrometry (ICP-MS, Agilent 7800) was used to measure the concentration of antimony (Sb) and the chemical composition of the deionised water after acidification using a 2% v/v of HNO_3 . The solid samples were digested with HNO_3 , HF, and HCl by heating in the microwave with specific care to avoid arsenic volatilisation. Boric acid (H_3BO_3) was added after microwave digestion to neutralise the residual HF. Mineralogical analysis of the samples was done with X-ray diffractometry (XRD). XRD measurements were matched with reference databases using DIFFRAC.EVA software (version 5.2.0.3) and quantification are done by Rietveld-TOPAS software (version 4.2).

2.2.2 Preparation and curing of the cemented paste backfills

The mix designs (recipes) of the CPB samples (amount of required water, tailings, binders, and arsenic trioxide dust) were prepared considering the specific gravity of the tailings, binders, arsenic trioxide dust, as well as the solids, dusts, and binder contents of the mixtures. The calculations of the required amount of each ingredient were based on the following equations:

$$\text{Solid content (\%)} = C_w = \frac{100 \times M_{\text{dry-solid}}}{M_{\text{dry-solid}} + M_{\text{water}}} \quad (2)$$

$$\text{Binder content (\%)} = B_w = \frac{100 \times M_{\text{dry-binder}}}{M_{\text{dry-tailings}} + M_{\text{dry-As}_2\text{O}_3}} \quad (3)$$

$$\text{As}_2\text{O}_3 \text{ dust content (\%)} = D_w = \frac{100 \times M_{\text{dry-As}_2\text{O}_3}}{M_{\text{dry-tailings}} + M_{\text{dry-As}_2\text{O}_3}} \quad (4)$$

where:

M_{water} = mass of water in the paste.

$$M_{dry-tailings} = \text{dry mass of tailings.}$$

$$M_{dry-binder} = \text{dry mass of binder.}$$

$$M_{dry-As_2O_3} = \text{dry mass of arsenic trioxide dusts.}$$

$$M_{dry-solid} = M_{dry-tailings} + M_{dry-As_2O_3} + M_{dry-binder}.$$

These calculations were based on the preparation of three cylindrical samples for each recipe (or run number) with the dimension of 2 × 4 inches (diameter × height, 50.8 × 101.6 mm) considering 20% additional materials to compensate for the possible losses during preparation and molding.

To decrease the number of samples, the RSM was used to define the appropriate recipes. RSM is a collection of mathematical and statistical methods that determine operating conditions and regression model equations using quantitative data obtained from relevant experiments. Central composite design based RSM was used to optimise CPB recipes with the help of the *Design-Expert* software (version 13). Table 1 shows the proposition of the RSM method by considering solid content, binder content, and curing time as the factors, and the strength (UCS) of the samples as the response. Considering two different binder combinations and three replicates for each recipe, at least 120 CPB samples were prepared and tested.

Table 1 The proposition of the CPB recipes by the RSM method

Run	Factor 1 Solid content (%)	Factor 2 Binder content (%)	Factor 3 Curing time (day)
1	70.64	5	59
2	72	4	28
3	72	4	90
4	72	6	28
5	72	6	90
6	74	3.32	59
7	74	5	7
8	74	5	59
9	74	5	59
10	74	5	59
11	74	5	59
12	74	5	59
13	74	5	59
14	74	5	111
15	74	6.68	59
16	76	4	28
17	76	4	90
18	76	6	28
19	76	6	90
20	77.36	5	59

For the preparation of cemented paste backfills, a Hobart mixer with a capacity of 4.8 L was used. All the preparations were conducted in the laboratory. Half of the water was mixed with the binder in the bowl of the mixer using a hand spatula. Then the tailings and the arsenic trioxide dust were added to the mixture, and they were mixed thoroughly in the bowl. The remaining water was added progressively to the mixture and mixing was continued for up to 5 minutes to achieve a homogenous paste. It was then cast into the three plastic molds in a single layer with minimum delay after mixing because of the rapid initial setting of the samples. After molding, to expel the air bubbles from the pastes, the cylinders were rodded 25 times using a steel rod with a diameter of 9.53 mm (3/8 in). The prepared undrained cylindrical samples were labelled, sealed by a lid, and stored in a humidity chamber (approximately 90% relative humidity and 20°C) to cure for up to 111 days before testing. The conditions remained unchanged over the curing period to ensure the comparability of tests and limit the number of influencing factors.

The strength evolution in the pastes was assessed by performing UCS tests on three separate specimens for each recipe (based on Table 1). UCS tests were carried out based on the ASTM C39 standard by a computer-controlled mechanical press with a normal loading capacity of 50 kN and a 1 mm/min displacement rate, and the stress-strain data were recorded for each sample. The samples were taken out of molds after 7, 28, 59, 90, and 111 days of curing, according to the RSM (Table 1). The base and top of the cylinders were flattened parallel to the plates of the mechanical press before the UCS tests. The UCS parameter corresponds to the maximum stress value before failure during the compression test. All the measurements were carried out in triplicate and the average values were taken as the final compressive strengths. The UCS test results were used to select the most appropriate binding agent and the mix designs with the highest strength.

3 Results and discussions

Results of the initial characterisation of the materials and mechanical strength of the CPB samples are presented in this section.

3.1 Initial characterisation

The specific gravity of the materials used for the preparation of the cemented paste backfill was 2.77, 3.45, 3.05, and 2.79 for the tailings, arsenic trioxide dusts, GU cement, and LKD, respectively. These values were used for the calculation of the required materials to obtain the CPB samples based on the mix design proposed in Table 1. Figure 1 depicts the cumulative PSD of the tailings and arsenic trioxide dusts and Table 2 presents some widely used indicators associated with PSD. The solid matrix of the CPB samples was made with 90% by mass of tailings and 10% by mass of dusts which is finer and more uniform than the tailings. The PSD results showed that more than 94% of the dusts and 53% of the tailings are less than 20 µm in diameter and most of the arsenic trioxide dusts grains are in the range of 1 to 20 µm. The specific surface (S_s) of the tailings and arsenic trioxide dusts are also shown in Table 2. The specific surface area of the tailings was about half of the specific surface area of the dusts sample. Arsenic trioxide dusts are much finer than the tailings and their specific surface is also higher.

The results of the XRF analysis on the tailings, arsenic trioxide dusts, GU cement, and LKD are shown as oxides in Table 3. Both GU cement and LKD contain high amounts of calcium oxide which is higher for the LKD (more than 75%), whereas GU has a higher amount of SiO₂. The content of other oxides in these binders is in the same range. Table 4 presents the results of the ICP analysis on the dusts and tailings sample. As can be seen, more than 57% of the dusts sample was composed of arsenic. The amounts of neutralising elements such as Ca and Mg in the tailings were significantly higher than the amount of the sulphur (about 0.5%) and the tailings can be suspected as non-acid generating tailings.

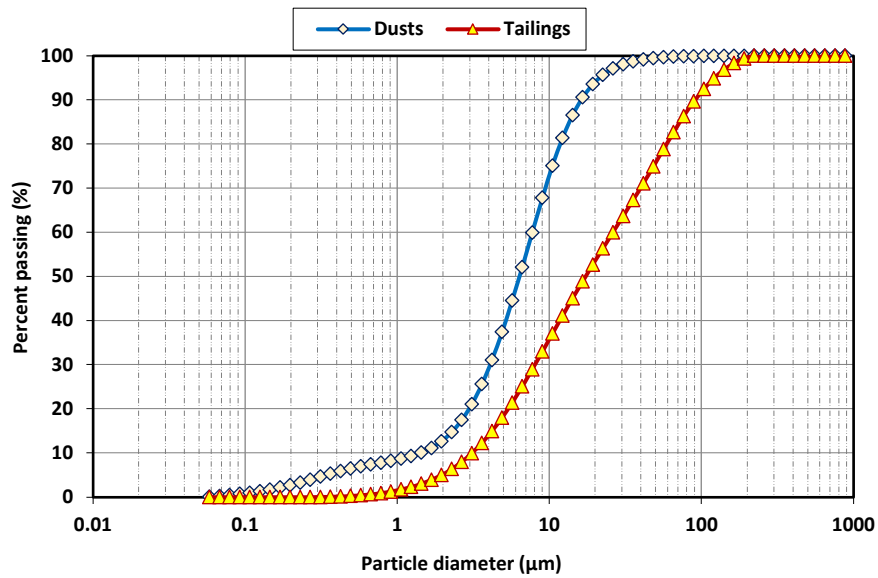


Figure 1 Particle size distribution of the dusts and tailings samples

Table 2 Results of the particle size analysis, specific gravity, and specific surface area of the representative dust and tailings samples

Parameter	Unit	Tailings sample	Dust sample
$C_u = D_{60}/D_{10}$	(-)	8.46	5.44
$C_c = D_{30}^2/(D_{60} * D_{10})$	(-)	0.80	1.51
$U = (D_{90}-D_{10})/D_{50}$	(-)	5.06	2.33
Specific surface area	m ² /g	3.59	7.04

Table 3 The results of the XRF analysis of the dusts, tailings, and binders

Sample	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	CaO	Na ₂ O	K ₂ O	TiO ₂	P ₂ O ₅	MnO	SrO	SO ₃	ZnO	V ₂ O ₅	LOI	Sum
Dusts	4.67	1.91	3.99	8.59	0.66	0.25	0.33	0.15	0.02	0.05	<0.01	<0.01	<0.01	<0.01	79.4	100.1
Tailings	48.5	9.86	10.9	5.63	8.53	0.44	1.45	0.76	0.08	0.16	<0.01	<0.01	<0.01	0.03	12.4	98.8
GU	17.2	4.84	3.26	2.30	64.3	0.30	1.80	0.13	0.32	0.03	0.29	5.15	0.03	<0.01	<0.01	99.99
LKD	7.94	4.70	1.39	0.90	75.8	0.20	1.88	0.46	0.16	<0.01	0.07	6.16	<0.01	<0.01	<0.01	99.68

Table 4 Chemical analysis results of the dusts and tailings samples by ICP-AES and ICP-MS method (mg/kg)

Element	Al	As	Ca	Cd	Fe	K	Mg	Mn	Na	S	Sb
Dusts	10730	571500	5360	7625	31720	2597	3756	128	541	2463	16782
Tailings	47000	2747	56260	25	72520	10550	29780	1092	3039	5192	118

The mineralogical analysis of the dust samples revealed that more than 86% of the dust sample was comprised of arsenolite (As₂O₃). The arsenic trioxide in the dust samples was formed during the roasting process; therefore, its mineralogical characterisation is slightly different from naturally occurring arsenic

trioxides. The tailings sample mainly includes quartz, chlorite, dolomite, and muscovite, and can be considered as typical non-acid generating tailings.

3.2 Cemented paste backfill mechanical strength results

Table 5 presents the results of UCS tests on the CPB samples prepared based on the proposed mix design using the GU and GU/LKD binders. Based on these results, it can be concluded that the samples prepared using GU binder showed much higher strength than the samples prepared by the GU/LKD mixture. The samples with the highest solid content and highest binder content showed the highest UCS. For both GU and GU/LKD binders, the optimal recipe is the CPB sample with 6% of binder and 76% of solid content, which had the highest strength after 90 days and 28 days of curing for the GU and GU/LKD binders, respectively. Note that no specific UCS target was identified for this study. Indeed, the Giant Mine will not mine near backfilled stopes nor expose backfilled faces. Currently the backfill operation aims at filling the voids and storing tailings with no target strength. However, since chemical stabilisation can be related to cementitious compounds formation, a higher UCS should hint at better contaminant stabilisation. The results are further compared in the following sections.

Table 5 Results of the UCS tests on the CPB samples prepared with GU cement and the mixture of GU and LKD

Run	<i>Factor 1</i>	<i>Factor 2</i>	<i>Factor 3</i>	<i>Responses</i>	
	Solid content (%)	Binder content (%)	Curing time (day)	UCS-GU (kPa)	UCS-GU/LKD (kPa)
1	70.64	5	59	154	24
2	72	4	28	154	42
3	72	4	90	154	42
4	72	6	28	296	100
5	72	6	90	278	105
6	74	3.32	59	155	27
7	74	5	7	174	108
8	74	5	59	268	76
9	74	5	59	282	87
10	74	5	59	264	67
11	74	5	59	268	48
12	74	5	59	276	81
13	74	5	59	296	90
14	74	5	111	232	109
15	74	6.68	59	472	175
16	76	4	28	276	92
17	76	4	90	249	90
18	76	6	28	491	214
19	76	6	90	531	213
20	77.36	5	59	478	178

3.2.1 Effect of solid content

Figure 2 shows the effects of the solid content of the CPB samples on the strength of the samples for both GU and GU/LKD binders. All these samples were prepared using a 5% of binder and cured for 59 days. In this figure, the average of the six samples with 74% solid content, which were the replicates for assessing the repeatability of the results (based on the mix design proposed by the RSM), is presented. As observed, increasing the solid content increased the strength of the samples. Due to the inverse relationship between the solid content and water content of the CPB samples (the higher the solid content, the lower the water content), it is evident that increasing the solid content resulted in an improvement in the strength of the CPB samples. The differences between the strength of the CPB samples prepared by GU and GU/LKD at the same solid and binder content could be related to the difference between the physical, chemical, and mineralogical properties of the binders.

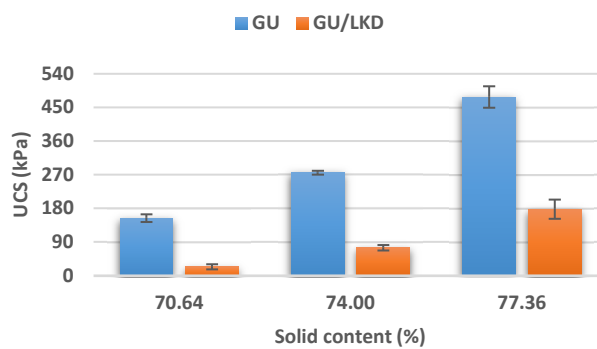
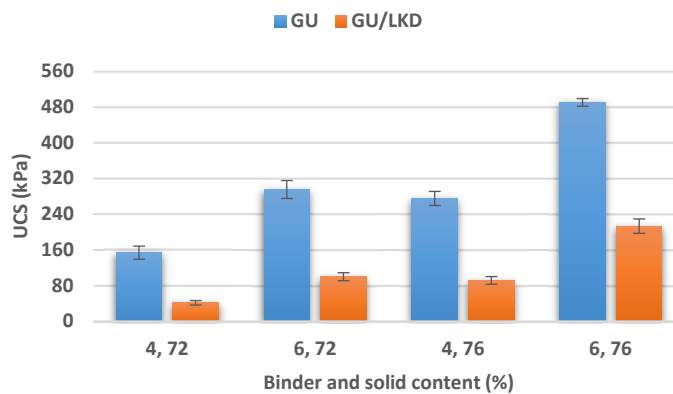


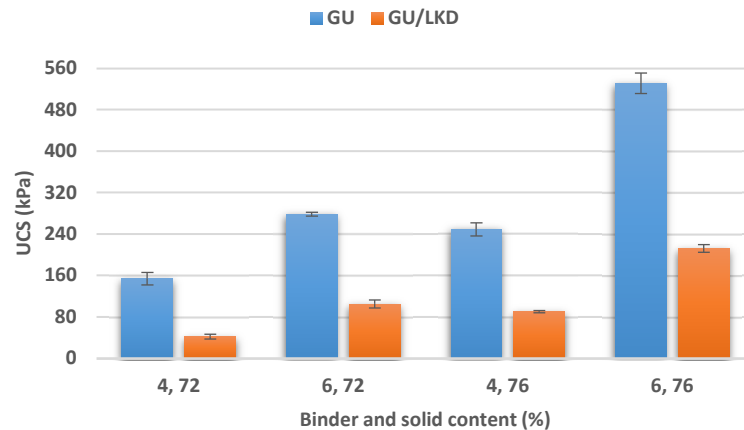
Figure 2 The effects of solid content on the UCS of the CPB samples prepared with 5% of GU and GU/LKD binders and cured for 59 days

3.2.2 Effect of binder content

Figure 3 depicts the influence of the binder content on the strength of the CPB samples. The effects of solid content on the strength of samples are also visible in this graph. Increasing the binder content increased the strength of the samples prepared by GU and GU/LKD mixtures both after 28 and 90 days. Increasing the binder content resulted in more cement hydration products, which led to the alteration of the pore structure (lower porosity), higher water consumption, and higher strength. Moreover, as expected, samples with higher solid content showed higher strength values. Changing from 4% to 6% of the binder had more effects on the strength at higher solid content (76%). Except for the 6,76 GU samples, no improvement in the strength was visible from 28 to 90 days of curing.



(a)



(b)

Figure 3 The effects of binder content (as well as solid content) on the UCS of the CPB samples prepared with GU and GU/LKD after (a) 28 days and (b) 90 days of curing

3.2.3 Effects of curing time

Figure 4 presents the influence of the curing time on the strength gain of the CPB samples. These samples were prepared using 5% of GU and GU/LKD binders at 74% solid content and cured from seven up to 111 days. Results showed that the effects of curing time are different on the GU and GU/LKD samples. For GU samples, increasing the curing time increased the strength of the samples up to 59 days and after that decreased their strength. For the GU/LKD samples, the strength was lowest at 59 days while UCS were similar at 7 and 111 days. Therefore, no trend is observable by changing the curing time. Generally, for the CPB samples without arsenic trioxide, increasing the curing time led to improvement in the strength since the binder hydration is an evolving process (Ghirian & Fall 2016).

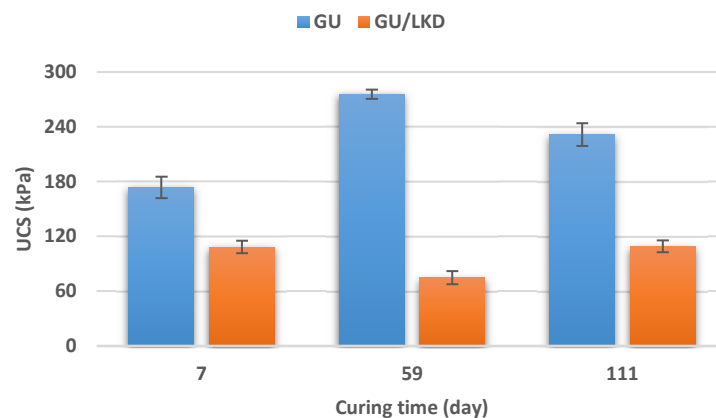


Figure 4 The effects of curing time on the UCS of the CPB samples prepared with 5% of GU and GU/LKD binders and 74% solid content

To compare the obtained results with the UCS of CPB samples without arsenic trioxide, some samples were prepared using GU cement and tailings of the Giant Mine. For the samples that were prepared using 4% and 6% GU cement at 72% solid content and cured for 28 days, the average UCS was 274 kPa and 371 kPa, respectively. The strength of the samples prepared using 6% of GU and GU/LKD binders at 76% solid content and cured for 28 days was 678 and 301 kPa, respectively. This shows that the addition of the arsenic trioxide roaster waste dusts resulted in a substantial decrease in the CPB samples strength. However, the decision about the feasibility of the incorporation of the arsenic trioxide roaster waste dust in CPB will be drawn based

on a combination of strength results and standardised leaching experiment results. Some investigations were undertaken to assess the effects of the incorporation of the arsenic trioxide roaster waste dusts in the CPB on the strength of the samples. One of the suspected reasons for this significant reduction could be the modification of the geochemical conditions of the paste due to the high solubility of the arsenic trioxide at high pH values (typical condition of the cementitious matrices). The dissolution of the arsenic trioxide in the CPB structure and strength reduction may also affect the stabilisation of arsenic within CPB, and these effects are currently under further investigation.

4 Conclusion

Based on the results of this study, it can be concluded that:

- The addition of the arsenic trioxide dusts to the CPBs substantially decreased the strength of the pastes; however, increasing the binder content and solid content compensated for this negative effect.
- Paste samples prepared using GU cement showed higher strength than the samples prepared with GU cement and LKD.
- Both binder content and solid content had a direct positive impact on the strength of the paste samples, whereas the effect of curing time on the strength was not meaningful.
- The reasons for strength reduction in the CPB samples in the presence of the arsenic trioxide, as well as the leaching characteristics of the optimal mix designs are under study. Still, one possible reason could be the reduction in the pH of the paste due to the dissolution of arsenic trioxide in the paste at higher pH values.

Acknowledgement

The authors would like to acknowledge the Giant Mine Oversight Board (GMOB) and NSERC Alliance program for the financial support.

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