Strength Characteristics and Heavy Metal Leaching Behavior of Contaminated Mining Sludge at Extra High Water Content Solidified/Stabilized with Lime Activated GGBS or OPC

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Abstract

Sludge management is one of the major challenges in mining activities. The direct disposal of contaminated mining sludge can bring severe damages to the environment and community. Solidification/Stabilization (S/S) is a very efficient technology for the treatment of contaminated mining sludge because it not only improves the stability of sludge dumping sites but also reduces the leachability of contaminants. Very few studies investigate the S/S of mining sludge, especially with extra high water content. This paper investigated the effectiveness of S/S for the treatment of mining sludge at extra high water content by using quick lime (CaO) activated ground granulated blast furnace slag (GGBS) in comparison to ordinary Portland cement (OPC). To evaluate the mechanical, leaching, and microstructural behavior of mining sludge at extra high water content stabilized by lime activated GGBS and OPC, a series of laboratory experiments were performed, including unconfined compressive strength (UCS), toxicity characteristics leaching procedure (TCLP), X-Ray diffraction, and scanning electron microscopy (SEM) tests, etc. Experimental results indicated that increasing the binder content led to increased strength and decreased leachability of the heavy metal. In contrast, an increase in the water content of the mixture resulted in a decrease in compressive strength and an increase in leachability of heavy metals. On the other hand, lime activated GGBS had substantially better performance than OPC in the aspect of strength development of treated mining sludge and moreover showed comparable capability of heavy metal stabilization in contrast to OPC.

Introduction

Mining activities are essential in the economic development of many countries over the world. The extraction of minerals presents opportunities, challenges, and risks to sustainable development. Mining exploitation often leads to a series of environmental and ecological challenges, for instance, soil and underwater pollution in mining areas, plant destruction and biodiversity loss, geological and land destruction. The disposal of heavy metal contaminated mining sludge (CMS) at extra high-water content could cause significant environmental and ecological damages (Wei and Virginia, 2005; Jang and Kim, 2000). Therefore, the effective remediation of extra high water content CMS has drawn the interest of researchers worldwide.

There are many remediation techniques available to treat such extra high water content CMS to avoid environmental pollution. However, the Solidification/stabilization (S/S) treatment method is attractive for many wastes, including CMS, by utilizing cement, lime, and other binders to minimize the toxicity of contaminants and enhance mechanical strength before final disposal (El-Eswed et al., 2015). Solidification/stabilization is one of the most applied technology to improve sludge stability (Zinck, 2006) and is a very recognized technique for the treatment of heavy metal contaminated soils (El-Eswed et al., 2015; Yi et al., 2016). After mixing the binder with the sludge, the binders react with heavy metal salts and form precipitations (i.e., compounds or insoluble complex hydroxides) due to their alkaline nature (Trussell and Spence, 2008). Furthermore, the heavy metals are encapsulated by hydration products such as calcium silicate hydrate (CSH), calcium aluminate hydrate (CAH) generated during the hydration process (Kogbara et al., 2011).

However, because the manufacturing of cement and lime is very often associated with enormous energy consumption and generates very high carbon dioxide emissions in the environment (Stork et al., 2014), researchers have recently been attempting to develop an eco-friendly alternative for these conventional binders in recent years with the intention of using industrial waste such as ground granulated blast furnace slag (GGBS). GGBS is a by-product of the steel industry, and its production requires low energy consumption and carbon emission (Higgins, 2007). It is an eco-friendly binder and can substitute or partially replace cement or lime for the treatment of soil in engineering projects (Keramatikerman et al., 2016; Nidzam and Kunuthia, 2010). Several researchers studied the solidification/stabilization of heavy metal contaminated soils using GGBS. The results suggested that activated GGBS could effectively improve the mechanical properties of contaminated soils and avoid the leaching of the contaminant into the environment (Goodarzi and Movahedrad, 2017; Jin and Al-Tabbaa, 2014).

Nevertheless, without an activator, GGBS cannot completely react with the soil particles. In fact, in the S/S process, the strength of the matrices containing only GGBS is generally lower than that of the samples containing activated GGBS, which indicates that the presence of an activator can considerably improve the mechanical behavior of the GGBS system (Yi et al., 2015) and reduce the leachability of heavy metal. The type of activator has a considerable impact on the resistance of treated soils (Jin and Al-Tabbaa, 2014). Generally, cement and lime are common activators used in the GGBS system (Nidzam and Kunuthia, 2010). Some recent studies also demonstrated that GGBS activated by reactive magnesia (MgO) with a perfect ratio led to a higher UCS than OPC or GGBS-CaO blends (Yi et al., 2014; Wang et al., 2015). However, the application of MgO-GGBS in soil treatment remains limited because the cost of magnesia-activated GGBS in the treatment of soil is more expensive than lime (Gu et al., 2015). In China, for example, the price of magnesia varies from US\$180 to US\$350 per ton compared to lime (i.e., US\$30 to US\$80 per ton) according to Beijing HL Consulting Company 2009 (Beijing HL Consulting Company 2009). In this context, using lime as an activator of GGBS in soil treatment can be a way to cut down the total cost of treatment.

To date, very few studies investigated the effects of lime-activated GGBS, on the Solidification/Stabilization process of mining sludge, especially with extra high water content. This study aims to gain an insight into the strength characteristics and heavy metal leaching behavior of contaminated mining sludge at extra high water content solidified/stabilized with lime-activated GGBS or OPC. It considers the leachability of 3 contaminants, Cu, Pb, and Zn, which are among the commonly encountered heavy metals in the soil (Kogbara and Al-Tabbaa, 2011). Microstructural characteristics were also investigated through X-Ray Diffraction (XRD) and scanning electron microscopy (SEM) to better understand the change in strength (constitution).

Laboratory Experiments

Materials

Contaminated sludge used in the laboratory experiments was collected from an actual copper mine site. As summarized in Table 1, basic physiochemical characteristics of the used sludge were determined according to the ASTM standard. The grain size distribution curve is shown in Fig.1, and concentrations of heavy

metals (Cu, Pb, and Zn) were tested using flame atomic absorption spectrometry according to the Environmental Protection Agency (EPA) 1311 manual.

Table 1

Physical properties of the mining sludge collected from the actual copper mine site.

Property		Value	Ref
Nature moisture content, %		50	ASTM D2216
pH		7.8	ASTM 4972-13
Specific gravity		2.61	ASTM 854
Liquid limit LL, %		41.45	ASTM D4318
Plastic limit PL, %		24.43	ASTM D4318
Plasticity Index		17.02	ASTM D4318
Particle Size Distribution	Sand fraction (0.075-2 mm), %	1.68	ASTM D6913
	Silt fraction (0.002 mm-0.075 mm), %	79.74	
	Clay and colloid fraction (<0.002 mm), %	18.58	
Soil Classification		Lean clay CL	ASTM D2487
Total Cu concentration, mg/kg		609.92	EPA 1311
Total Pb concentration, mg/kg		15.6	EPA 1311
Total Zn concentration, mg/kg		274.9	EPA 1311

In this study, ground granulated blast furnace slag (GGBS) was selected because of its environmental, technical, and economic benefits (Yi et al., 2014). The quicklime (CaO) has been used as an activator of GGBS. The reason for selecting lime is its low cost and high efficiency for heavy metal precipitation. The ordinary Portland cement was also used as the conventional binder in solidification/stabilization for comparison. The OPC used in this experiment was OPC.42.5 and is manufactured in China. The GGBS and CaO used for the experiment were obtained as a white powder from a local supplier in Wuhan. The physicochemical properties of GGBS, CaO, and OPC were determined via XRF analysis and listed in Table 2. As previously mentioned, heavy metals such as Cu, Pb, and Zn were targeted. Finally, ZnCl₂, CuCl₂ · 2H₂O, PbCl₂ were chosen to prepare the contaminated sludge and were obtained from Wuhan Xinshenshi Chemical Technology Co., Ltd.

Table 2

Chemical compositions of materials.

Composition	CaO (%)	SiO ₂ (%)	Al ₂ O ₃ (%)	Fe ₂ O ₃ (%)	MgO (%)	K ₂ O (%)	SO ₃ (%)	CO ₂ (%)	Others (%)	Loss on ignition (%)
OPC	47.81	20.33	4.26	2.54	3.41	0.70	4.02	15.30	1.3	0.33
CaO	75.51	1.46	0.68	0.101	4.79	0.017	0.19	17.10	0.152	-
GGBS	34.00	32.3	14.29	0.24	6.74	0.23	2.33	8.70	1.17	-
CMS	19.56	21.59	6.56	15.80	3.39	0.65	1.20	28.82	1.79	0.64

Preparation of contaminated mining sludge (CMS) at extra high water content

The following initial concentrations were considered: (i) Cu. 2901.53 mg/kg, (ii) Pb. 94.38 mg/kg, and (iii) Zn. 1614.73 mg/kg), corresponding to middle pollution of Pb and the high concentration degree of Zn according to the background value of soil environment in China (China National Environmental Monitoring Center, 1990). Four water contents varying from 100% to 160% have been considered in the experiments. Moreover, contaminated sludge specimens were prepared by dissolving the predetermined amount of ZnCl₂, CuCl₂·2H₂O, PbCl₂ solution in water and mixing with the sludge. The mixing was carried out through an electric agitator for 10 min following the standardized mixing procedure and braised for 14 days under standard curing conditions to allow heavy metal and sludge to reach equilibrium. The binder content was set as 10%, 12%, 15%, and 20% by weight of dry soil. For lime-activated GGBS cases, the ratio of quicklime (the activator) to GGBS is taken as 1:3 by following the recommendation in Goodarzi and Movahedrad, (2017). Furthermore, the binders were added to the contaminated sludge on predetermined dry sludge weight and mixed thoroughly for 10 minutes with an electronic mixer to obtain a homogenous mixture. The mix was then poured in cylindrical molds (50 mm in diameter and 100 mm high) and were cured in the curing box at temperatures 25 ± 1 °C.

Table 3

Testing program.

Mix type	Binder type	Group	Case No.	W (%)	A _w (%)	Curing time	No. of specimens prepared	Testing items
OPC	Ordinary Portland cement	A	A1	120	10	7, 14, 21 and 28 days	8	UCS at 7,14, 21 and 28 -day for all specimens
			A2	120	12		8	
			A3	120	15		8	
			A4	120	20		8	
		B	B1	100	12		8	TCLP at 7, 14, 21, and 28 -day for all specimens
			B2	120	12		8	
			B3	140	12		8	
			B4	160	12		8	
CG	CaO-GGBS (1:3)	C	C1	120	10		8	SEM at 28 -day for selected specimens
			C2	120	12		8	
			C3	120	15		8	
			C4	120	20		8	
		D	D1	100	12		8	
			D2	120	12		8	
			D3	140	12		8	
			D4	160	12		8	

Testing procedure

A total of 16 cases were conducted during the laboratory experiment, and the designed proportions are summarized in Table 2. The 16 cases were divided into four groups (i.e., A-D). Group A includes four CMS cases stabilized with different values of OPC content. Group B includes four CMS cases stabilized with OPC at different values of water content. Furthermore, Group C and D include four CMS cases stabilized with lime-activated GGBS at different binder content and different water content.

Four curing times were considered for each testing case (7, 14, 21, and 28 days). Specimens were extruded from the molds, and unconfined compression strength tests (UCS) were conducted to determine their crushing strength according to the ASTM standard D-1633. After UCS, the samples were crushed to reduce their particle size to less than 2mm to determine the leachability of the specimen by the toxicity characteristics leaching procedure experiment (TCLP) defined by EPA method 1311. The crushing particles were agitated in the extracting agent by dissolving 17.25 mL of glacial acetic acid in 1 L of deionized water with a pH of 2.88 ± 0.05 at a liquid to solid ratio of 1:20 for 18 hours at 30 rpm. After collection and filtration of the samples, the heavy metal concentrations in the leachates were determined by atomic absorption

spectrometer. For XRD analysis, the samples obtained from UCS were crushed and sieved through a 0.075 mm sieve to get a fine powder, and the samples were scanned in ranges from 10 to 70 (2) using a Rigaku D/Max-2500 X-ray diffractometer with a Cu-K α source to identify the crystalline phases. Scanning electron microscopy (SEM) was also used on the selected samples to analyze the microstructure properties of the stabilized soils.

Results And Discussion

Strength Characteristics of treated mining sludge at extra high water content

Figure 2 illustrates the UCS results of CMS at extra high water content stabilized by CG and OPC at different binder content and curing time (groups A and C). The CG stabilized CMS samples were not strong enough to be de-molded after 7-day of curing time. The CG stabilized CMS showed a lower 7-day UCS than OPC stabilized CMS, but the formers produced higher UCS values at later curing ages. This is attributed to the slow hydration rate of GGBS at an early age, which has nevertheless resulted in higher long-term strength once activated, as reported in previous studies (Yi et al., 2014, 2015). The 28 days UCS of the stabilized CMS specimens increases with binder content and curing time as expected due to the formation of cementitious phase. When the CG content increased from 10 to 20%, the compressive strength increased to 118.18 kPa and 1003.86 kPa at 28-day curing time, respectively. Furthermore, the UCS values of CG stabilized CMS increased significantly with curing time compared to OPC stabilized CMS. After 28-day of curing time, CG stabilized CMS showed 5.44 times higher UCS than OPC stabilized CMS at the same water content and binder content. This could be explained by the higher hydration rate of GGBS activated by lime and the increase of hydrates, such as CSH and CASH, hydrotalcite-like phases in stabilized CMS, which can increase the UCS of samples. None of the OPC stabilized CMS fulfilled the US EPA criterion (0.35 MPa) even after the 28-day curing time due to their lower strength, whereas the CG stabilized CMS meet these criteria after the 21-day curing time.

Figure 3 shows the 28-day UCS results of stabilized CMS at different water content values (cases B and D). It can be observed that, at 28-day, the UCS decreases with the increase of water content for both stabilized specimens. Indeed, the UCS decreases approximately by 3.9 times for CG stabilized specimens and 1.7 times for OPC samples when increasing the water content from 100 to 160% after 28-day of curing. However, for all of the stabilized CMS, the CG produces a higher UCS up to eight orders of magnitude than OPC stabilized samples. These results were attributed to the formation of a more voluminous hydration product in the CG stabilized CMS, which can efficiently fill the obvious pores and improve the UCS (Li et al., 2016). Additionally, stabilized high water content CMS can be used as structural backfill material because the minimum strength required is 100 KPa, as reported by (Holm et al., 2012).

Heavy Metal Leaching Behavior of treated mining sludge at extra high water content

The TCLP experiments were also carried out on stabilized CMS samples, which could represent the long-term stability of S/S material in the context of leaching. Figure 4 shows the detailed results of heavy metal leachability of Cu, Pb, and Zn stabilized by OPC and CG at the same water content and different binder content. It can be seen that leaching concentration of heavy metals such as Cu, Pb, and Zn from stabilized CMS were lower than 100 mg/L, 5 mg/L, and 100 mg/L, respectively, which are the regulatory limit specified by Chinese standard method. The leachability of heavy metals decreases with curing time and increase of binder content for both OPC and CG stabilized CMS. This shows that by incorporating binders, the leachability of heavy metals in CMS decreases, which is primarily due to their insoluble hydroxides and/or complexes, as seen in the XRD result (Fig. 6).

The leached Cu and Pb concentrations from most OPC stabilized CMS cases were on the lower side than CG stabilized CMS. More specifically, OPC stabilized CMS exhibited 6.95 % and 38.8% lower leached concentration of Cu and Pb than CG cases, respectively. This could have occurred probably because the pH of the leachate was between the range of pH acceptable for the formation of soluble Cu and Pb hydroxide, resulting in an effective immobilization for these heavy metals in OPC samples (Kumpiene et al., 2008). In contrast, CG stabilized CMS showed 15.7% lower leachability of Zn than OPC stabilized CMS. This pronounced decrease in Zn leachability in CG cases is due to the production of more voluminous hydration products such as CSH and hydrotalcite, resulting in a dense stabilized matrix that provides greater resistance to TCLP acid solution (Kogbara et al., 2013). Besides, hydrotalcite formed in CG cases (see XRD results) is an effective heavy metal absorber and reduces Zn's leachability through isomorphic substitution (Liang et al., 2013; Wang et al., 2015).

The increase in water content (Fig. 5) led to the rise in the leachability of heavy metals in both cases. Indeed the leachability increases significantly when increasing the water content from 100–160%. For all cases, OPC stabilized CMS leach out a higher concentration of Cu and Zn than CG stabilized CMS except for Pb, indicating that the increase in water content has a significant effect on the leachability of OPC stabilized CMS than CG specimens. Indeed the analysis of Fig. 3 and Fig. 5 showed that the treated CMS with a high water content present a lower UCS and higher leachability compared to those treated with lower water content. Furthermore, the CMS stabilized by CG showed better performance than OPC treated specimens, which could be responsible for better encapsulation of heavy metal (Li et al., 2016). Although the increase of water content has a significant effect on OPC stabilized CMS, the heavy metal concentrations in the leachate were below the regulatory limit according to the Chinese standard after 28 days of curing. The increase in water content did not significantly affect the immobilization of Pb because the initial concentration of Pb on the soil is not that higher. Therefore, CG-based solidification/stabilization can be used for the safe disposal of high water CMS treated at 12% binder content. The replacement of OPC with lime-activated GGBS lead to an improvement in the heavy metal retention compared to OPC stabilized CMS. The above findings demonstrated that the proposed CG binder was effective in the S/S of heavy metal contaminated sludge at extra high water content.

XRD analysis of treated mining sludge at extra high water content

The 28-day crystalline phases of OPC and CG cases determined by XRD analysis are shown in Fig. 6. Quartz has been found as the common compound of CMS, reflecting the nature of used mining sludge. Typical hydration products such as Calcium silicate hydrate (CSH), calcium aluminate silicate hydrate (CASH), and ettringite were also identified in both OPC and CG cases, suggesting that the major hydration products of CG stabilized CMS were similar to that of OPC stabilized CMS. This is in agreement with previous findings (Gu et al., 2015; Oti and Bai, 2008). However, the additional peaks of hydrotalcite were also detected in CG cases, which is the only difference between the hydration products of CG and OPC stabilized CMS. The development of these voluminous hydration products could increase the binding capability, resulting in higher strength development of stabilized CMS (Goodarzi and Movahedrad, 2017). Calcite was also detected, which is the result of the reaction between CaO and gas-phase CO₂.

Under high alkaline conditions, Pb was solidified/stabilized on the surface of CSH by an adsorption mechanism and chemical reactions to form insoluble lead silicate, as shown in Fig. 6. Trace peaks of Zinc oxide and copper oxide were identified in both specimens (Fig. 6), indicating that Cu and Zn were mainly precipitated as oxide. Zinc silicate has also been identified in XRD patterns of both cases, which is parallel with the findings of (Niu et al., 2018), who reported that Zn is usually bound to carbonate and Fe/ Mn oxide phases. The Zn tetrahedral can also be bound to the CSH tetrahedral silicate chains, leading to Zn retention. Zn was then stabilized/solidified by CSH adsorption, precipitation and incorporation into the components of hydration products such as CSH and hydrotalcite. Another complex called calcium zincate (CaZn₂(OH)₆·2H₂O) was also observed in both cases, which is supposed to form from the Ca(OH)₂ and Zn(OH)₂ reaction. In addition, due to Zn's retardant effect on cement hydration, portlandite (Ca(OH)₂) one of the major hydration products, was not detected in the OPC stabilized CMS, which is consistent with a finding previously reported in the literature (Du et al., 2014). No portlandite was also detected in the CG cases. The absence of portlandite in the CG stabilized samples is due to its consumption during the GGBS activation, which agrees with Nidzam and Kunuthia, (2010). Instead of portlandite, the trace of ettringite was detected in CG cases (Fig. 6) due to lime consumption during GGBS hydration.

SEM images for treated mining sludge at extra high water content

SEM tests were performed to examine the microstructure development on the typical 28-day OPC and CG stabilized CMS specimens, and the results are shown in Fig. 7. The OPC stabilized CMS microstructure with 12% OPC at 120% water content is shown in Fig. 7 (A), and the CG stabilized CMS with 12% CG at 120% water content is shown in Fig. 7(B). The analysis of the micrograph indicates that the soil particles were disorderly distributed with a large number of small pores. The CG and OPC hydration products of gel-like CSH, platy CASH gels, platelet hydrotalcite, and needle-like ettringite crystal have been filled into the pores of these CMS particles, leading to the disappearance of large-scale pores. This is consistent with (Pu et al., 2021), who reported that CASH appeared to be platy in soil–lime/cement reaction. Such cementation and filling ability of hydration products contributed to the strength development of stabilized CMS. The analysis

of Fig. 7(C and D) showed a large amount of pore, indicating that the increase of water content from 120–160% for OPC and CG treated specimens significantly affected the microstructure.

When increasing the water content, the hydrates products in both cases remain the same such as CSH, CASH, hydrotalcite, and ettringite. However, a large quantity of pores has been detected in Fig. 7(C and D). Indeed, the microstructure of the stabilized CMS particles changed from a dense structure to a dispersed nature with a large number of pores due to the presence of a large amount of water. Arulrajah et al. (2018) previously reported similar observations due to an increase in water content. Figure 7 (B and D) depict the microstructure of CG stabilized CMS samples with a 12% binder content produced, and the CMS particles have been strongly cemented, resulting in significant improvement in the development of strength. This agrees with the UCS results presented in Figures (3 and 4) showing that the 28 days UCS of CMS stabilized with CG were higher than OPC under the same water content.

Conclusions

In this study, lime-activated GGBS based solidification/stabilization has been proposed for the treatment of extra high water CMS. A series of tests were conducted to evaluate the effect of water content and binder content on the strength characteristics and leaching behavior of the treated material. The main conclusions drawn from the analysis include:

1. Lime-activated GGBS has substantially better performance than OPC in the aspect of strength development of treated mining sludge. At 28-day, the CG stabilized CMS showed 5.44 times higher UCS than OPC stabilized CMS at the same water content and binder content.
2. Both CG and OPC samples exhibit a decrease in the leaching concentration of heavy metal with an increase in curing time. However, CG stabilized samples show comparable capability of heavy metal stabilization in contrast to OPC.
3. XRD patterns showed that the main hydration products of both CG and OPC mixes were CSH, CASH, and ettringite. The hydrotalcite produced in the CG mix was the only difference between the hydration products of CG and OPC mixes.
4. SEM micrographs exhibited that CG mix developed dense microstructure due to the formation of more voluminous hydration products such as hydrotalcite, filling the pores between CMS particles more effectively, resulting in a dense stabilized matrix.

Declarations

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The authors declare that there is no conflict of interest.

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Figures

Figure 1

Figure 2

UCS of solidified samples with the same water content

Figure 3

UCS of solidified samples at the same binder content and different water content

Figure 4

Leaching concentration vs. binder percentage: (A) Cu in OPC samples, (B) Cu in CG samples, (C) Pb in OPC samples, (D) Pb in CG samples, (E) Zn in OPC samples, (F) Zn in CG samples

Figure 5

Leaching concentration vs. binder percentage: (A) Cu in OPC samples, (B) Cu in CG samples, (C) Pb in OPC samples, (D) Pb in CG samples, (E) Zn in OPC samples, (F) Zn in CG samples

Figure 6

XRD diffractograms of CMS samples treated by OPC and CG after 28 days of curing

Figure 7

Microstructure of contaminated sludge treated by OPC and CG after 28 days of curing: (A) case A2, (B) case C2, (C) case B4, (D) case D4.