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Changes in speciation and leaching behaviors of heavy metals in dredged sediment solidified/stabilized with various materials

Jianping Bao[1,2] · Liang Wang[3] · Man Xiao[4]

Abstract Solidification/stabilization (S/S) of sediments is frequently used to treat contaminants in dredged sediments. In this study, sediment collected from the Pearl River Delta (China) was solidified/stabilized with three different kinds of functional materials: cement, lime and bentonite. Lime primarily acted via induced increases in pH, while cements stabilization occurred through their silicate-based systems and the main function of bentonite was adsorption. The speciation and leaching behaviors of specific heavy metals before and after S/S were analyzed and the results showed that the residual speciation of Cd, Cr, Ni, Pb and Zn increased in all treatments except for Cu, as the exchangeable speciation, carbonate-bound speciation and Fe-Mn-oxide-bound speciation of Cu (all of which could be stabilized) were less than 2 % of the total amount. Pb leaching only decreased when pH increased, while the mobility of Cr and Ni only decreased in response to the silicate-based systems. The leached portion of the Fe-Mn-oxide-bound speciation followed the order Zn > Cu > Ni/Cd > Pb > Cr. The leached portion of organic-matter-bound species was less than 4 % for Cd, Cr, Ni and Pb, but 35.1 % and 20.6 % for Cu and Zn, respectively.

Responsible editor: Céline Guéguen

✉ Man Xiao
   xiao_man1989@163.com

1   College of Environment, Hohai University, No. 1 Xikang Road, Nanjing 210098, People's Republic of China

2   Nanjing Hohai Technology Co., Ltd., Nanjing 210098, People's Republic of China

3   South China Sea Institute of Plannning and Environmental Research, State Oceanic Administration, Guangzhou 510300, People's Republic of China

4   Australian Rivers Institute, Griffith University, Nathan, Queensland 4111, Australia

Introduction

The increasing use of materials containing heavy metals (e.g., electronic components, auto parts, fossil fuels, etc.) has resulted in a growing amount of heavy metals being released into lakes and reservoirs (Audry et al. 2004), a large proportion of which are ultimately stored in sediments (Harding and Whitton 1978). High concentrations of heavy metals in these sediments pose risks to the environment and human health. Thus, it is necessary to develop suitable remediation technologies for these heavy metal-polluted sediments.

The *ex situ* remediation of sediment has been widely applied (Peng et al. 2009; Wang et al. 2014) and shown to have the potential to meet the challenge of ever-increasing quantities of heavily polluted sediment. Among *ex situ* remediation methods, solidification/stabilization (S/S) technology has been highly applied (Song et al. 2013; Li et al. 2001). A variety of materials (e.g., cement, lime, bentonite, etc. Zhang et al. 2011) have been used to solidify/stabilize polluted sediments. Both the strength properties of these solidified sediments and concentrations of heavy metals in the leachate of sediments they were used to stabilize have been analyzed (Wang et al. 2013; Zentar et al. 2008). Strength tests have shown that sediment could be reused as building and roadbed materials after S/S (Dubois et al. 2009; Wang et al. 2012). Moreover, S/S technology is considered to be environmentally friendly (Tomasevic et al. 2013) because heavy metals in the sediment can be chemically stabilized and the environmental risk associated with the polluted sediment is dramatically reduced (Rijkenberg and Depree 2010; Samara et al. 2009). However, the effectiveness of different materials used

for S/S varies greatly (Kumpiene et al. 2008). In addition, the leaching concentrations of some types of heavy metals are still high after treatment with different materials (Alpaslan and Yukselen 2002). Thus, it is still necessary to obtain insight into the mechanism of S/S and leaching behaviors of different heavy metals in dredged sediment solidified/stabilized with various materials.

Cement, lime, and bentonite are the most common materials used for S/S because of their low price and effectiveness (Bailey et al. 1999). Increasing pH is considered to be the main function of lime during application to stabilize heavy metals. This is because leaching of heavy metals is a pH-dependent metal hydroxide solubility-controlled process (Malviya and Chaudhary 2006). Cement can also increase pH, but its effects primarily occur via the interaction of its silicate-based systems with heavy metals (Yousuf et al. 1995). The effects of bentonite on stabilization of heavy metals occur via its adsorption of heavy metal ions (Liu et al. 2006; Andini et al. 2006; Katsioti et al. 2008). Thus, it is necessary to reveal the effects of different functions (mentioned above) of various materials on stabilization of heavy metals to understand the mechanism by which stabilization occurs.

Chemical properties of different types and species of heavy metals have different strengths that affect their leaching behaviors (Lions et al. 2007). The sequential extraction procedure designed by Tessier et al. (1979) to analyze five heavy metal species has been widely applied to assess the solubility of heavy metals (Banerjee 2003; Xiong et al. 2014). This procedure divides heavy metals into five species according to the degree of difficulty in leaching. Theoretically, the leached concentration of heavy metals could be calculated by the content of different species of various heavy metals. However, most previous studies only conducted qualitative analysis and did not provide enough information to enable quantification of the leached concentration.

Therefore, the present study was conducted (i) to reveal changes in heavy metal speciation of sediment treated with cement, lime, and bentonite and (ii) to demonstrate the leaching behaviors of different species of heavy metals. In the current study, sediment was collected from the Pearl River Delta (China), and the heavy metal speciation and their leaching behaviors were analyzed before and after S/S.

Materials and methods

Sediment sampling

Sediment was collected from Dongguan Canal in Dongguan in the Pearl River Delta (South China), where a great deal of electroplating wastewater has been discharged. Five sites were fixed randomly in the middle reaches of the river. Sediment

samples were collected to a depth of 80 cm using a Piston Core Sediment Sampler XDB0204, mixed, and stored in a cold closet for later experiment. The basic properties of the sediment are shown in Table 1. The materials used for solidification were cement, lime, and bentonite. The cement was 32.5 Portland cement (Jiangnan Cement Factory, China), the composition of which is shown in Table 2. The lime, which was from Nanjing Rongjun Environment Friendly Building Materials Co., Ltd. (China), had a CaO content greater than 95 %. Sodium bentonite from Nanjing Tangshan Bentonite Co., Ltd. (China) was also used in the current study (Table 3).

Heavy metal speciation analysis

The chemical speciation of heavy metals was determined by the sequential extraction procedure described by Tessier et al. (1979). The speciation of heavy metals was divided into the following: fraction 1—exchangeable speciation; fraction 2—carbonate-bound speciation; fraction 3—Fe-Mn-oxide-bound speciation; fraction 4—organic-matter-bound speciation; and fraction 5—residual speciation. This method has been widely applied in many studies (Yuan et al. 2015). A detailed description of the method is provided below.

Step 1   Exchangeable speciation. A 1-g sample was extracted with 10 mL of 1 M $MgCl_2$ solution at pH 7.0 for 2 h at room temperature with continuous agitation. The sample was then centrifuged at $4500 \times g$ for 15 min, after which the supernatant was diluted to 50 mL for analysis.

Step 2   Carbonate-bound speciation. Residue from step 1 was continuously agitated with 10 mL of 1 M NaOAc (pH was adjusted to 5.0 with HOAc) at room temperature for 5 h. The sample was then centrifuged at $4500 \times g$ for 15 min, after which the supernatant was diluted to 50 mL for analysis.

Step 3   Fe-Mn-oxide-bound speciation. Residue from step 2 was mixed with 20 mL of 0.04 M NH_2OH-HCl in 25 % (v/v) HOAc for 6 h at 96 °C with occasional agitation. After being allowed to cool, the mixture was diluted to 20 mL with deionized water. The dilution was then centrifuged at $4500 \times g$ for 15 min, and the supernatant was diluted to 50 mL for analysis.

Step 4   Organic-matter-bound speciation. Residue from step 3 was extracted with 3 mL of 0.02 M HNO_3 and 5 mL of 30 % H_2O_2 (the pH was adjusted to 2.0 with HNO_3), heated at 85 °C for 2 h, and then extracted again with 3 mL of 30 % H_2O_2 (pH was adjusted to 2.0 with HNO_3) at 85 °C for 3 h with intermittent agitation. After being allowed to cool, 5 mL of 3.2 M NH_4OAc in 20 % (v/v) HNO_3 was added and the sample was incubated with continuous agitation

Table 1 Basic properties of the dredged sediment

pH	Moisture content/%	Organic materials/%	Cd/ mg kg^{-1}	Cr /mg kg^{-1}	Cu/ mg kg^{-1}	Ni/ mg kg^{-1}	Pb/ mg kg^{-1}	Zn/ mg kg^{-1}
7.3	61.5	12.7	12.39	396.12	1272.39	280.71	80.07	782.01

for 30 min. The mixture was then centrifuged at 4500×g for 15 min, after which the supernatant was diluted to 50 mL for analysis.

Step 5   Residual speciation. Residue from step 4 was digested with 9 mL of 12 M HCl followed by 3 mL of 15.8 M HNO$_3$ added drop by drop to reduce foaming. Next, 5 mL of 0.5 M HNO$_3$ was added and allowed to stand for 16 h at room temperature to oxidize the organic matter. The mixture was then heated and maintained for 2 h before cooling. Finally, it was centrifuged at 4500×g for 15 min and the supernatant was diluted to 50 mL for analysis.

The concentration of different heavy metals was determined by inductively coupled plasma optical emission spectroscopy (ICP-OES, PerkinElmer Optima-2000, USA).

Solidification

A total of 100 mL of sediment was mixed with 10 g cement, lime, and bentonite then stirred for 10 min using a handheld mixer (1000 CC, BMC, Taiwan) to homogenize the sample. The sediment was subsequently maintained in a 50 × 50 × 50 mm cube for 7 days (Zhu et al. 2007).

Ten-gram samples of both original sediment and solidified sediment were dried at 105 °C for 8 h, after which the moisture content was calculated. According to the moisture content, a proper weight (containing 5 g dry sediment) of both the original sediment and the solidified sediment was used to analyze the heavy metal speciation as described above.

Leaching behaviors

The toxicity characteristic leaching procedure (TCLP) was applied to assess the leaching characteristics of both the original sediment and the solidified sediment (US EPA 1986). A proper weight (containing 10 g dry sediment according to the moisture content) of sample was extracted by glacial acetic

acid solution (pH = 2.8) as medium with a liquid to solid ratio of 20. The extraction process was conducted according to Li et al. (2001).

Leaching behaviors of different species of heavy metals

The carbonate-bound speciation was considered weak acid-soluble speciation, which could be extracted with 1 M NaOAc at pH 5.0 with a solid to liquid ratio of 1:60 (Li et al. 2001). Thus, the speciation of exchangeable species and carbonate-bound species were considered unstable, 100 % of which could be leached. In addition, the residual speciation was always regarded a stable speciation that could not be extracted in an ordinary way (Zhang et al. 2014). Therefore, only the leached portion of Fe-Mn-oxide-bound species and organic-matter-bound species was analyzed in the current study. A proper weight (containing 10 g dry sediment according to the moisture content) of original sediment was prepared in sextuplicate. Three replicates were extracted by steps 1, 2, and 3 described above. The residue contained only the organic-matter-bound species and residual species because exchangeable species, carbonate-bound species, and Fe-Mn-oxide-bound species were extracted. Similarly, the other three copies were extracted by steps 1 and 2. The residue contained organic-matter-bound species, residual species, and Fe-Mn-oxide-bound species because the exchangeable speciation and carbonate-bound speciation were extracted. The six extracted samples were analyzed by TCLP, and the leached concentrations were used to calculate the leached portion of Fe-Mn-oxide-bound species and organic-matter-bound species of different heavy metals.

Data analysis

All experiments were carried out in triplicate, and the average value was used in the results and data analysis.

Table 2 Composition of cement used in the current study

SiO$_2$/%	Al$_2$O$_3$/%	Fe$_2$O$_3$/%	CaO/%	MgO/%
21.43–22.17	4.75–5.83	3.31–3.91	64.39–66.23	1.12–2.08

Table 3 Specifications of bentonite used in the current study

Swelling capacity/ mL g^{-1}	Water absorption (2 h)/%	Cation exchange capacity /mmol (100 g)$^{-1}$	Particle size (<180 mesh) /%
9.5	≥180	79.2	95

Results and discussion

Heavy metal speciation before and after S/S

The heavy metal speciation in the sediment before and after S/S is shown in Fig. 1. A small amount of heavy metals was found in exchangeable speciation, which was similar to that of soil samples collected in New York City (Cheng et al. 2011). This could have occurred because the exchangeable speciation of heavy metals in sediments and soils had been leached out under natural conditions. The residual speciation of Cd, Cr, Ni, Pb, and Zn increased obviously in all treatments, while Cu showed only a slight increase. This is because the exchangeable speciation, carbonate-bound speciation, and Fe-Mn-oxide-bound speciation of Cu (which could be stabilized) were less than 2 % of the total amount. For the same reason, the percentage of stable residual speciation of all heavy metals except for Cu increased in response to treatment with bentonite. These findings indicated that the adsorption of heavy metal ions would be

effective for all the heavy metals when the unstable speciation of heavy metals was abundant.

The residual speciation of both Pb and Zn increased in response to treatment with lime. Lime has been reported to enhance the degradation of organic matter (Singh and Kalamdhad 2013). Moreover, Aarab et al. (2006) reported that the organic matter of sewage sludge dropped from 64 to 44 % in 2 weeks when treated with lime. In addition, Weng et al. (2014) reported that decomposition of organic matter and sulfide could stabilize heavy metals such as Cu, Cd, and Pb. The organic-matter-bound speciation of Pb and Zn was also found to decrease with increasing residual species (Fig. 2). Similarly, Couvidat et al. (2015) found that Zn was primarily associated with organo-clay complexes, which could easily be affected by pH. Thus, the increase in residual species of both Pb and Zn was caused by the decomposition of organic matter induced by lime.

Excluding Cu, the portion of stable residual speciation of all heavy metals increased significantly in response to treatment with lime. However, no significant increase in the

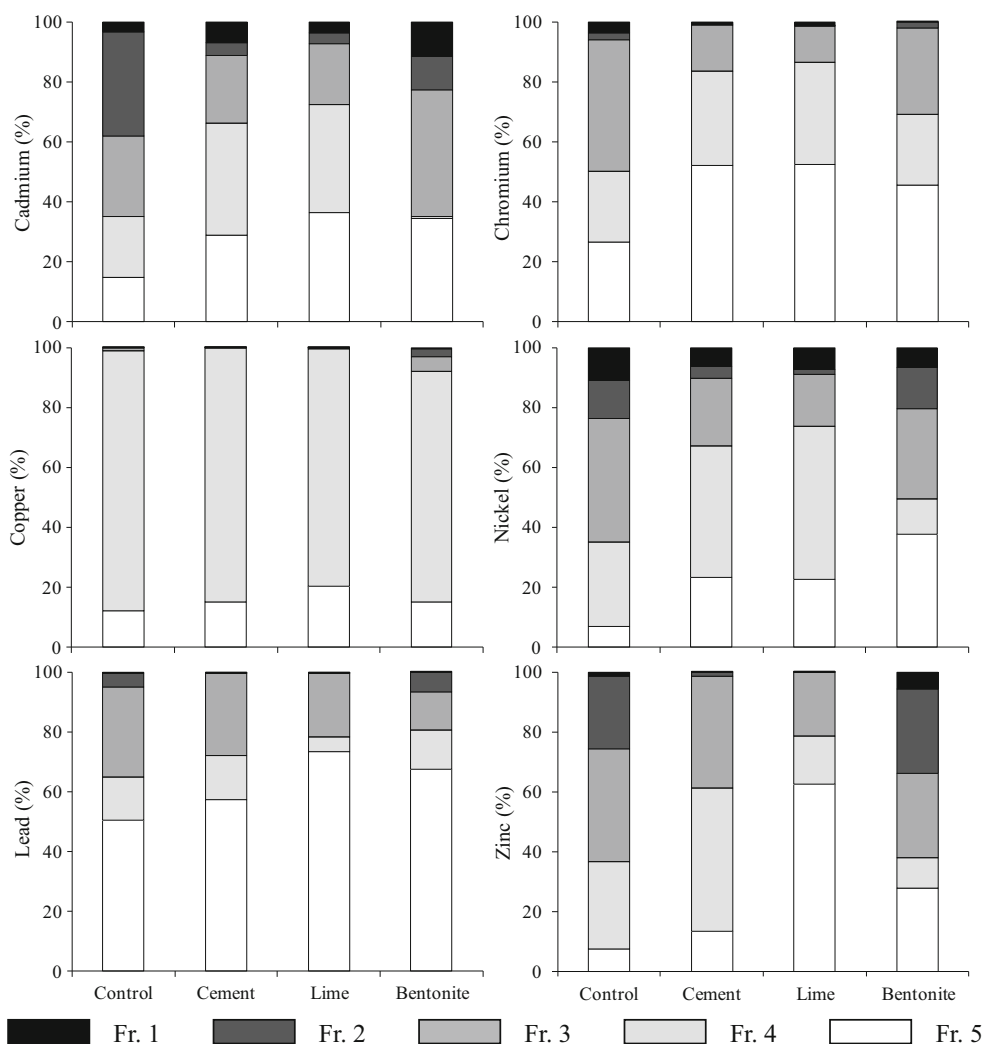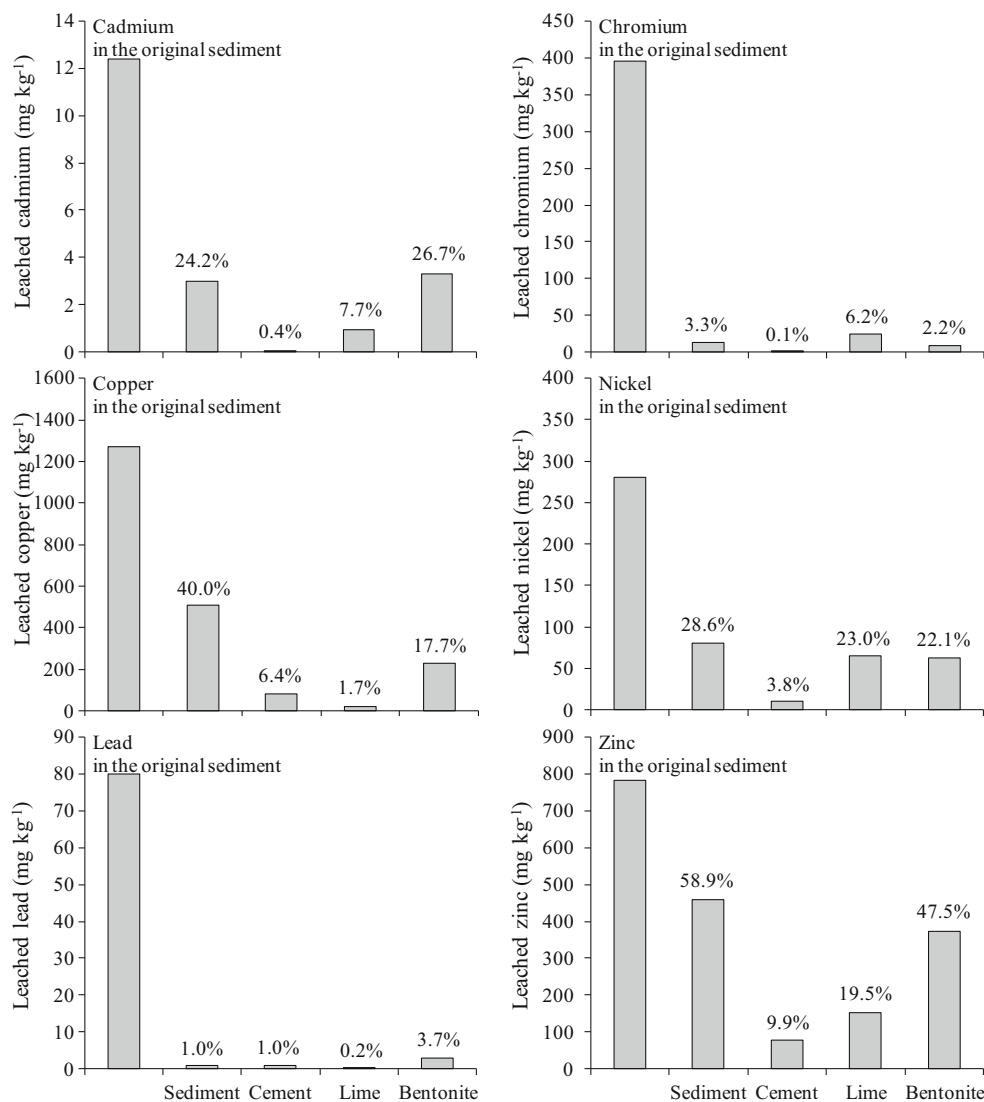**Fig. 1** Heavy metal speciation in sediment before and after S/S

Fig. 2 Leached heavy metal content calculated from the TCLP results. The *percentages* at the top of the *rectangle* indicate the proportion of the leached heavy metals to the total amount

residual speciation of Pb and Zn was observed in response to stabilization with cement. As reported by Paria and Yuet (2006), both Pb and Zn would form insoluble hydroxide at high pH (about 12), and their anionic properties preclude their adsorption onto the negative surface of C-S-H. This difference could be because the alkalinity of lime was much higher than that of cement.

Leaching behaviors of heavy metals in sediment before and after S/S

The leached heavy metal content calculated from TCLP is shown in Fig. 2. The percentage of leached heavy metals in the sediment before S/S occurred in the order $Zn > Cu > Ni/Cd > Cr > Pb$, with the percentage of leached Zn reaching about 58 % of the total Zn amount in the sediment. The leached concentration of Cu in the sediment before S/S was 64 mg L^{-1} (calculated from Fig. 2 with a liquid to solid ratio

equal to 20), which was much higher than the standard value of hazardous waste in China (50 mg L^{-1}). However, the leached concentrations of heavy metals were lower than the standard values in all treatments.

Table 4 shows the effects of various materials on the leaching concentration of heavy metals in stabilized sediment

Table 4 Effects of various materials on leaching concentration of heavy metals in stabilized sediment compared with that of the sediment before S/S

	Cd	Cr	Cu	Ni	Pb	Zn
Cement	+++	+++	++	++	/	++
Lime	+	⎯⎯	+++	/	+	+
Bentonite	−	/	+	/	⎯⎯⎯	/

+++, ++, and + indicate that the leached concentration decreased by 90, 80, and 50 %, respectively. ⎯⎯⎯, ⎯⎯, and − indicate that the leached concentration increased by 100, 50, and 10 %, respectively. / indicates no significant difference

compared with that of the sediment before S/S. After treatment with cement, the leached concentration of Cd, Cr, Cu, Ni, and Zn decreased by more than 80 %. However, the leached concentrations of all heavy metals except for Cu increased in response to treatment with bentonite. Lime decreased the leached concentration of Cd, Cu, Pb, and Zn, but increased the concentration of Cr.

These dramatic differences occurred because metal stabilization involved various mechanisms and depended on properties, species, and the interference of different heavy metals (Zhang et al. 2011). As mentioned in the "Introduction" section, the primary function of lime was increasing pH. Cement could also increase pH, but it had silicate-based systems, while adsorption was the main function of bentonite. Table 5 shows the different factors influencing the leaching behaviors of different heavy metals. Only increasing pH reduced Pb leaching. Dong et al. (2013) found that high solid anaerobic digestion increased the bioavailability of Cu, Zn, Ni, and Cr, but decreased the bioavailability of Pb. The pH value decreased with anaerobic digestion. Overall, the results provided here in conjunction with those of Dong et al. (2013) confirmed that increasing pH could reduce Pb leaching.

As shown in Table 5, only the silicate-based system effectively stabilized Cr and Ni. Roy et al. (1992) suggested that physical encapsulation of metal hydroxide was the principal mechanism of cement-based stabilization of Ni and Cr. Lin et al. (1997) indicated that both Si and Al were important to stabilization of Cr because of the formation of Ca-Cr aluminate phases and substitution of Al by Cr(III) in the C-S-H phases. Singh and Kalamdhad (2012) also found that the addition of organic matter stabilized most heavy metals, but not Cr. Many studies have reported that some epitaxial structures such as Si-NiSi$_2$ and Ni-Al were important to the stabilization of Ni (Tung et al. 1983; Ford et al. 1999). Thus, it is reasonable that the silicate-based system is more effective at stabilizing Cr and Ni than increasing pH.

Increasing pH and silicate-based systems reduced the leaching concentration of Cd, Cu, and Zn (Fig. 2).

Table 5 Factors reducing heavy metals leaching

Heavy metals	Factor reducing heavy metal leaching		
	Primary factor	Secondary factor	Other factors
Cd	Silicate-based systems	Increasing pH	
Cr	Silicate-based systems		
Cu	Increasing pH	Silicate-based systems	Adsorption
Ni	Silicate-based systems		
Pb	Increasing pH		
Zn	Silicate-based systems	Increasing pH	

Table 6 Leached portion (η) of different species of heavy metals (%)

Speciation	Exchangeable	Carbonate-bound	Fe-Mn-oxide-bound	Organic-matter-bound	Residual
Cd	100	100	33.2	2.2	0
Cr	100	100	2.4	0.4	0
Cu	100	100	73.2	35.1	0
Ni	100	100	23.6	3.4	0
Pb	100	100	17.6	0.8	0
Zn	100	100	74.4	20.6	0

Rijkenberg and Depree (2010) reported that the most important factor explaining Zn leaching was pH. Shim et al. (2014) reported that the addition of corn cob silica reduced the mobility of Zn and Cu. Su and Wong (2004) stabilized sewage sludge by fly ash and found that increasing the fly ash amendment rate significantly reduced concentrations of DTPA-extractable Cu, Zn, and Cd while increasing the pH. Qian et al. (2009) stabilized multi-contaminated sediments using apatite and ferrihydrite, both of which were alkaline. Their results showed that treatment with both alkaline materials reduced leaching Zn from 42.5 to 24.1 mg L^{-1}.

Leaching behaviors of different species of heavy metals

Table 6 showed the leached portion (η) of different species of heavy metals. The leached portion of the Fe-Mn-oxide-bound fraction followed the order of Zn > Cu > Ni/Cd > Pb > Cr. The leached portion of organic-matter-bound fraction of Cd, Cr, Ni, and Pb was less than 4 %, while it was 35.1 and 20.6 % for Cu and Zn, respectively.

The concentration of leached heavy metals could be calculated from the concentration and the leached portion of different species as follows:

$$\text{Conc}_{\text{heavy metal}} = \text{Conc}_1 \times \eta_1 + \text{Conc}_2 \times \eta_2 + \text{Conc}_3 \times \eta_3 + \text{Conc}_4 \times \eta_4 + \text{Conc}_5 \times \eta_5 \qquad (1)$$
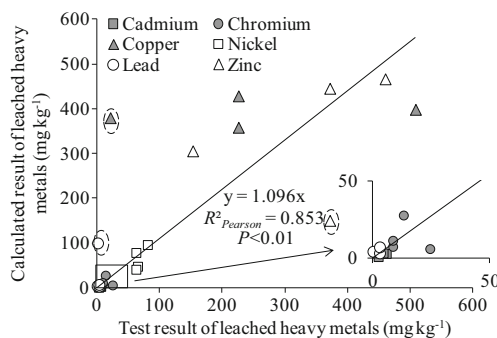

Fig. 3 Relationship between calculated and observed results of the leached concentration of different heavy metals

where $Conc_{heavy\ metal}$ is the concentration of leached heavy metals and $Conc_1$ and η_1 are the concentrations and leached portions of different species, respectively. Additionally, 1 to 5 indicates exchangeable species, carbonate-bound species, Fe-Mn-oxide-bound species, organic-matter-bound species, and residual species, respectively.

The relationship between the calculated results and the analyzed results of the leached concentration of different heavy metals is shown in Fig. 3. Most of the results fit well; thus, the leaching behaviors of different species of heavy metals could be a useful indicator to predict the concentration of heavy metals leached from soil and sediment before and after S/S.

Conclusions

1. The residual speciation of Cd, Cr, Ni, P, and Zn increased in all treatments; however, that of Cu did not. This was because the exchangeable species, carbonate-bound species, and Fe-Mn-oxide-bound species of Cu (which could be stabilized) are less than 2 % of the total.
2. Only increasing pH could reduce Pb leaching, while treatment with silicate-based systems was the only method of decreasing the mobility of Cr and Ni.
3. The leached portion of Fe-Mn-oxide-bound species occurred in the order Zn > Cu > Ni/Cd > Pb > Cr. The leached portion of organic-matter-bound species of Cd, Cr, Ni, and Pb was less than 4 %, but the portion was 35.1 and 20.6 % for Cu and Zn, respectively.
4. The leached portion of different species of heavy metals could be a useful indicator to predict the concentration of heavy metals leached from soil and sediment before and after S/S.

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