Immobilization of Cd, Zn and Pb in sewage sludge using red mud

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Abstract Sewage sludge is an inevitable end by-product of sewage treatment. Land application provides a cost-effective alternative for sewage sludge disposal. However, sewage sludge contains heavy metals that may limit its application. In this work, red mud was employed for the immobilization of heavy metals in sewage sludge. The effect of red mud amendment on heavy metal immobilization was evaluated using Toxicity Characteristic Leaching Procedure (TCLP) method. The TCLP results showed that the immobilization efficiency of Cd, Zn and Pb was 100, 92, and 82%, respectively, when sewage sludge was mixed with 10% red mud. Tests carried out in leaching columns demonstrated that heavy metal concentrations in the leachate of 10% red mud amended sludge were lower than those of the unamended sludge. Moreover, red mud decreased plant available heavy metal (Cd, Zn and Pb) content from 18.1, 17.2 and 14.6% to 6.9, 11.4 and 7.6%, respectively. Sequential chemical extraction experiments showed that after sludge was amended with 10% red mud, exchangeable fraction was reduced and iron and manganese oxides fraction was increased. Red mud amendment can effectively immobilize Cd, Zn and Pb in sewage sludge before land application.

Keywords Heavy metals · Immobilization · Red mud · Sewage sludge

Introduction

Sewage sludge is an inevitable end by-product of sewage treatment. The accumulation of sewage sludge from sewage treatment plants is of increasing concern with the development of society and economy. At present, disposal methods for sewage sludge include landfill, incineration, dumping and land application. Compared to landfill, incineration and dumping, land application is a preferable and practical option for the disposal of sewage sludge in China. Land application represents the most cost-effective way for final disposal of sewage sludge as it combines the recycling of plant nutrients and sewage sludge disposal at the same time (Gu and Wong 2004; Wong et al. 2001). The high content of organic matter and substantial nitrogen and phosphorus concentrations in sewage sludge suggest its use as a fertilizer in agriculture. However, land application of sewage sludge presents a potential threat to the environment because of the content of heavy metals in sludge, which may be aggravated if the heavy metals are mobilized in the soil to be taken up by plants or transported in drainage water (Alvarez et al. 2002; Angelidis and Gibbs 1991; Mingot et al. 1995).

Chemical immobilization has been developed for reducing mobility and solubility of heavy metal contaminants. A large number of different amendments have been proposed and tested for immobilization of heavy metals in soils. Amendments investigated for immobilization of heavy metals in soils include limestone (Basta and McGowenb 2004), phosphate rock (Hettiarachchi et al. 2000; Chen et al. 2003; Cao et al. 2003; Basta and McGowenb 2004; Scheckel and Ryan 2004; Cao et al. 2009), sewage sludge (Basta et al. 2001), natural zeolites (Gadepalle et al. 2007), beringite (Lombi et al. 2003), steel shot (Muller and Pluquet 1998) and fly ashes (Dimitris

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and Meng 2003; Querol et al. 2006; Ciccu et al. 2003; Kumpiene et al. 2007). Chemical immobilization will provide an attractive technology if the amendment is cheap and ease of application.

One industry product that has been shown to have great potential to immobilize heavy metals and reduce availability is red mud. Red mud is the residue of aluminum industry. It is alkaline and composed primarily of iron and aluminum oxides. The alkali can immobilize heavy metals by precipitation and the iron and aluminum oxides can immobilize heavy metals by sorption. It seems interesting to employ red mud, which is the cost-effective industrial residue, as an agent to immobilize heavy metals in sewage sludge.

The immobilization of heavy metals using red mud amendment in soils has been successfully demonstrated in many studies (Gray et al. 2006; Bertocchi et al. 2006; Lombi et al. 2002a, b, 2003; Brown et al. 2005; Garau et al. 2007; Liang et al. 2010). However, there have been few reports on the investigation of red mud as an amendment for immobilization of heavy metals in sewage sludge, especially in the sludge of China. Therefore, the present study focused on heavy metals immobilization in sewage sludge by red mud amendment. The objectives of this study were to: (1) explore the potential of red mud to immobilize Cd, Zn and Pb in sewage sludge; (2) investigate the DTPA extractable (plant available) heavy metals (Cd, Zn and Pb) in sewage sludge by red mud amendment; and (3) investigate the speciation of Cd, Zn and Pb after red mud immobilization.

Materials and methods

Sewage sludge and red mud

Sewage sludge samples used in this study were obtained from Xiaohongmen Sewage Treatment Plant, located at Chaoyang District of Beijing. In this plant, sewage treatment uses the typical anaerobic-anoxic-aerobic (A^2/O) process and the sewage sludge is mechanically dewatered by belt press filters. The dewatered sludge was collected and kept in a refrigerator at temperature of $4.0 \pm 0.5^\circ\text{C}$. The sewage sludge pH value was 7.13 ± 0.05 (1:2.5 soil water suspension). The electric conductivity (EC) value was $1,042 \pm 68 \mu\text{S}/\text{cm}$ (1:2.5 soil water suspension). The moisture content reached up to $83.0 \pm 0.9\%$. The total organic matter value was $497.0 \pm 0.4 \text{ mg/g}$. The total phosphorus content in the sewage sludge was $15.0 \pm 0.2 \text{ mg/g}$.

Red mud was obtained from China Aluminum Industry Shandong Branch Company, the composition and properties of which were listed in Table 1. Mineralogical constituents were determined using X-ray diffractometry

Table 1 Composition and properties of red mud

Composition	Content (w/w, %)
SiO_2	12.45
Fe_2O_3	25.68
Al_2O_3	10.98
$\text{CaO} + \text{MgO}$	13.88
Na_2O	5.76
K_2O	0.33
TiO_2	12.94
Loss on ignition	15.41
pH	12.55
Specific surface area (m^2/g)	16.48
Crystalline phases	Hematite, bayerite, calcite, cancrinite, goethite, rutile and quartz

(SmartLab, Japan). Loss of ignition was determined at 750°C in accordance with ASTM D5142-02a (Arthur 2004). The specific surface area was determined by using the N_2 BET method (Micromeritics ASAP2000, USA).

Immobilization experiments

As for the immobilization experiments, 100 g sewage sludge was thoroughly mixed with red mud to immobilize heavy metals. The dosages of red mud amendment were applied to the sewage sludge samples at mass ratios of 5, 10, 15 and 30% (w/w, dw), respectively. The mixtures were incubated in the dark at 25°C for 3 days. The mixture pH was measured at the end of the incubation, according to the standard method of International Union of Soil Sciences for soil pH measurement and the pH of the mixture was measured in de-ionized water at mixture/water ratio of 1.0:2.5 and extraction time of 30 min.

Batch leaching experiment

Batch leaching test using Toxicity Characteristic Leaching Procedure (TCLP) was carried out to investigate the efficiency of red mud to immobilize heavy metals. The TCLP extraction solution was prepared by mixing 5.7 ml acetic acid to 1 L de-ionized water, adjusting the pH to 2.88 ± 0.05 . 1 g incubated mixture was placed in a 50 ml centrifuge tube, and 20 ml of the TCLP extracting solution was added. Each sample was equilibrated for 18 h on a shaker, centrifuged, filtered. The filtrate was collected and acidified to pH = 2.0 for heavy metal concentrations measurement. The heavy metal concentrations were measured using inductively coupled plasma mass spectrometry (ICP-MS, PerkinElmer) and the immobilization efficiency was calculated as follows:

$$\eta = \left(1 - \frac{C_e}{C_0}\right) \times 100\% \quad (1)$$

where η immobilization efficiency, C_0 the leaching heavy metal concentration of sludge without red mud amendment, C_e the leaching heavy metal concentration of red mud amended sludge.

Column experiments

Based on the batch leaching experiment results, 10% red mud amended sludge was chosen for the next column experiments. Column experiments were conducted to evaluate the feasibility of the long term immobilization efficiency by red mud. Polyvinyl chloride (PVC) columns with inner diameter of 4.0 cm and length of 50.0 cm were used. 300 g unamended and amended sludge samples were placed in the columns and de-ionized water was supplied by a peristaltic pump from the top of the columns at a flow of 10 ml/h. Leachates were collected every 24 h for the measurement of heavy metal concentrations and the leaching experiments lasted for 7 days. The leaching of heavy metals from the amended sludge was compared to that of the sludge sample without red mud amendment.

Measurement of plant available heavy metals

For the measurement of plant available heavy metals, the incubated sample was dried at 60°C and milled to pass a 0.4 mm sieve. It was recognized that diethylenetriaminepentaacetic acid (DTPA) extraction provides a chemical evaluation of the amount of heavy metals that are available for plant uptake (Reyhanitabar and Gilkes 2009; Fuentes et al. 2006). According to Lindsay and Norvell's method, the DTPA extractable heavy metals were determined to be the plant available heavy metals (Lindsay and Norvell 1978). The DTPA extractable fraction was obtained by mechanical shaking 10 g of mixtures for 2 h with 20 ml of 0.05 M DTPA–0.01 M Triethanolamine (TEA) solution buffered to pH value of 7.3 (Miles and Parker 1979). The suspension was filtered through Whatman No.42 filter and heavy metal concentrations were measured.

Analysis of heavy metal speciation

In order to analyze heavy metal speciation after red mud immobilization, the incubated sludge sample was also dried at 60°C and milled to pass a 0.4 mm sieve. The conventional sequential chemical extraction method by Tessier et al. (1979), which is widely used in the investigation of heavy metal speciation in sewage sludge (Liu et al. 2007; Cai et al. 2007), was employed for the analysis of heavy

metal speciation and the extraction procedure was shown in Table 2. The extraction was carried out in 50 ml polypropylene centrifuge tubes. To each tube, 1 g samples were added. After each extraction step, the tubes were centrifuged at 10,000 rpm for 10 min. The supernatant was filtered through 0.45 µm membrane filter and the heavy metal concentrations were analyzed.

Results and discussion

Heavy metal content and speciation in sewage sludge

Table 3 shows the selected heavy metal content and speciation in the investigated sewage sludge. It can be observed that the total concentrations of Cd, Zn and Pb in the sewage sludge were much lower than the pollutant concentration limits for land application of sewage sludge recommended by the USEPA. Zn was the predominant heavy metal contaminant in the investigated sludge. Similar results have been reported by Chen et al. (2003) and Qiao et al. (2003) who reported that Zn was the predominant heavy metal in the sewage sludge. Cd and Pb are also of concern because of their potentially harmful effects on humans and animals. The solubility of heavy metals mainly depends on the heavy metal speciation in sewage sludge. Sequential chemical extraction experiments indicate that heavy metal speciation was different for Cd, Zn and Pb in the investigated sewage sludge. Zn was observed mainly in the organic matter and residual fractions, while in contrast, Cd and Pb were observed mainly in the organic matter and carbonate fractions.

Batch leaching experiment

The aim of batch leaching experiment was to determine the efficiency of red mud to immobilize heavy metals and the required percentage of red mud to add to sewage sludge. From the TCLP leaching behavior, it is clearly shown that the amended sewage sludge samples showed significant reduction in Cd, Zn and Pb leachability compared to unamended sewage sludge. Cd, Zn and Pb leachability decreases as more red mud amendments were used (Table 4). The immobilization efficiency increases as more red mud amendments were used. Red mud amendment at 10% (w/w, dw) significantly reduces the TCLP extractable heavy metals, which is comparable with that at 15% (w/w, dw) of red mud amendment. As a compromise between the immobilization efficiency and amendment cost, 10% red mud amendment was chosen as the amendment ratio for the next experiments. When sewage sludge mixed with 10% red mud, the amount of leached Cd, Zn and Pb was reduced from 0.07, 65.82, and 2.94 mg/kg to 0, 4.99, and

Table 2 Sequential chemical extraction experiments for heavy metals

Step	Treatment	Extraction conditions	Fraction
S1	1 M MgCl ₂ pH7.0	1 h shaking	Exchangeable
S2	1 M NaOAc pH5.0	5 h shaking	Bound to carbonates
S3	0.04 M NH ₂ OH-HCl in 25% (v/v) HOAc.	6 h shaking at 96°C	Bound to iron and manganese oxides
S4	(HNO ₃ 0.02 M + 30%H ₂ O ₂) pH5.0 30%H ₂ O ₂ pH2.0 3.2 M NH ₄ OAc in 20% (v/v) HNO ₃	2 h shaking at 85°C 3 h shaking at 85°C 0.5 h shaking at 85°C	Bound to organic matter
S5	HF-HClO ₄	30 min shaking at 96°C	Residual

Table 3 Heavy metal content and speciation in sewage sludge (mg/kg)

Metal	Total content	Speciation ^a					Pollution concentration limits ^b
		S1	S2	S3	S4	S5	
Cd	7 ± 1	0.85 ± 0.21	0.96 ± 0.12	0.60 ± 0.17	4.18 ± 1.04	0.70 ± 0.08	<39
Zn	1,098 ± 17	105.60 ± 0.43	14.31 ± 2.08	38.05 ± 4.29	643.08 ± 84.50	244.42 ± 1.64	<2,800
Pb	123 ± 7	19.12 ± 0.09	28.22 ± 2.38	13.44 ± 3.10	43.79 ± 1.92	19.26 ± 1.74	<300

The results are expressed as mean ± standard deviation. Mean for three replicates

^a Speciation: S1 exchangeable, S2 bound to carbonates, S3 bound to iron and manganese oxides, S4 bound to organic matter, S5 residual

^b Pollution concentration limits (USEPA. 1995. A guide to the biosolids risk assessment for the EPA Part 503 rule. EPA 832-B-93-005)

Table 4 The leachable heavy metal content in unamended and amended sewage sludge (mg/kg)

Mass ratio of red mud to sludge (%)	Cd	Zn	Pb
0	0.07 ± 0.004	65.82 ± 1.78	2.94 ± 0.21
5	ND	9.40 ± 0.64	1.11 ± 0.01
10	ND	4.99 ± 0.12	0.53 ± 0.01
15	ND	3.37 ± 0.08	0.42 ± 0.01
30	ND	2.06 ± 0.15	0.34 ± 0.02

ND Not detected (detection limit: Cd, 0.003 ppm)

0.53 mg/kg, respectively. The immobilization efficiency of Cd, Zn and Pb was 100, 92, and 82%, respectively.

Effect of amendment on pH

It can be seen from the above results that with the increase in red mud dosage, the immobilization efficiency increases. However, the increasing red mud dosage will induce an increase in the pH of the amended sludge due to the alkalinity of red mud. The pH of the amended sludge should be considered. The pH value of the mixture as determined by red mud dosage was shown in Fig. 1. It was found in Fig. 1 that, pH of the amended sludge reach 8.49 at red mud of 5% and 9.39 at red mud dosage of 10%. When red mud dosage reaches 15%, the pH was 10.97, which was high for land application. So a red mud dosage of 10% was employed in the following investigation.

It was generally believed that the leachability and bio-availability of heavy metals from sewage sludge is depended on pH. The pH of the sewage sludge affected the solubility of the heavy metals. It has been concluded that the pH of the sewage sludge has a significant effect on the immobilization efficiency, which increases with increasing pH (Schnitzer and Skinner 1966). For the elements of concern in this study, Cd, Zn and Pb leachability decreases as investigated sewage sludge pH increases.

Column experiments

A column leaching test was set up to evaluate the long term immobilization efficiency. The heavy metal concentrations in the leachate of unamended and amended sewage sludge are shown in Fig. 2. It can be seen from Fig. 2 that the concentrations of the selected heavy metals decreased over time as the more soluble forms of the heavy metals were flushed out of the columns. During the investigation period, heavy metal concentrations in the leachate of amended sludge were lower than those of the unamended sludge, which indicated that red mud amendment addition could effectively immobilize the Cd, Zn and Pb in the sludge.

As for the immobilization mechanism of heavy metals, Lombi et al. (2003) and Hofstede (1994) suggested that the major immobilization mechanism is sorption and precipitation. Bruemmer et al. (1988) and Gray et al. (2006) suggested that specific chemisorption plays an important role for the immobilization of heavy metals. As for the

Fig. 1 The pH values of mixtures

immobilization of heavy metals in sewage sludge by red mud, it seems that the immobilization mechanism is a combination of precipitation and sorption. First, the immobilization is a result of an increase in mixture pH caused by the addition of this alkaline product. The alkalinity in the red mud precipitated heavy metals through the formation of Cd, Zn and Pb hydroxides and carbonates. Second, red mud contains iron and aluminum oxides, which could adsorb heavy metals by sorption.

Effect of amendment on plant available heavy metals

Red mud amendment not only reduced the heavy metal leaching, but also reduced the content of plant available heavy metals. The changes in the content of plant available Cd, Zn and Pb of red mud amended sewage sludge were shown in Fig. 3. It can be seen that, amendment by red mud significantly reduced the content of plant available heavy metals. For Zn, the plant available percentage was decreased from 17.22 to 11.36% after the sludge was amended red mud. For Cd, the plant available percentage was decreased from 18.13 to 6.98%. For Pb, the plant available percentage was decreased from 13.64 to 7.12%.

Effect of amendment on heavy metal speciation

To identify the changes of heavy metals speciation resulting from the immobilization of the red mud amendment, unamended and amended sewage sludge samples were analyzed by sequential extraction experiments. This method provided useful evidence for explaining the heavy metals immobilization. At present, the sequential chemical extraction method by Tessier et al. (1979) and BCR (European Community Bureau of Reference) sequential extraction are the main methods for analysis the heavy

Fig. 2 Heavy metal concentrations in the leachate

metal speciation (Soylak et al. 2004; Saracoglu et al. 2009; Nemati et al. 2011; Alonso Castillo et al. 2011). In this paper, the sequential chemical extraction method by Tessier et al. (1979) was chosen. The heavy metal fractions in the sequential extraction experiments are shown in Fig. 4.

It can be seen from Fig. 4 that main Cd fraction in the sludge was bound to organic matter (55.50%), followed by

Fig. 3 Percentage of plant available heavy metals

exchangeable (13.46%), bound to carbonates (12.69%), residual (9.30%) and bound to iron and manganese oxides (9.04%). After red mud was mixed with sewage sludge, the Cd fraction pattern changed. Exchangeable fraction decreased from 13.46 to 5.32% and the carbonates and iron and manganese oxides fractions increased from 12.69 and 9.04% to 14.60 and 15.19%, respectively. The organic matter and residual fractions only decreased slightly.

It can be observed that the order for different fractions of Zn in the sludge is: Bound to organic matter (61.51%) > Residual (23.37%) > Exchangeable (10.10%) > Bound to iron and manganese oxides (3.64%) > Bound to carbonates (1.37%). The organic matter and residual fractions are high in the sewage sludge. The addition of red mud amendment did not cause marked changes of Zn speciation (Fig. 4). However, compared with the unamended sludge, the exchangeable fraction decreased from 10.10 to 0.14%. Bound to carbonates fraction increased from 1.37 to 2.97% and the iron and manganese oxides fraction increased from 3.6 to 15.01%. The organic matter and residual fractions decreased from 61.51 and 23.40% to 59.26 and 22.61%.

Pb in the sludge was associated primarily with organic matter (35.36%) and carbonates (22.78%) fractions. Red mud was efficient in reducing the exchangeable fraction. The addition of red mud amendment shifted the distribution of Pb from the exchangeable fraction to the carbonates, iron and manganese oxides and residual fractions.

From the results obtained, it seems that the increase in pH of mixture (Fig. 1), due to the alkaline of red mud amendment, resulted in an increase of heavy metals associated with the carbonate fraction. Moreover, red mud which is rich in iron and aluminum oxides that have reactive surface sites able to bind metals caused a significant portion of exchangeable heavy metals to be associated with the iron and manganese oxides fraction. This result is

Fig. 4 Heavy metal speciation in unamended and amended sewage sludge

consist with Lombi et al. (2002a) who have used chemical extraction experiments to show that amending contaminated soils with red mud results in a distribution of heavy metals from exchange fraction to iron and manganese oxides fraction.

Conclusions

1. Red mud amendment can effectively immobilize Cd, Zn and Pb in sewage sludge. When sewage sludge mixed with 10% red mud, the immobilization efficiency of Cd, Zn and Pb was 100, 92, and 82%, respectively.
2. Plant available heavy metals (Cd, Zn and Pb) were reduced after sewage sludge was amended with 10% red mud. The plant available heavy metals (Cd, Zn and Pb) were reduced from 18.13, 17.22 and 13.64% to 6.98, 11.36 and 7.12%, respectively.
3. Sequential chemical extraction experiments showed that after sewage sludge was amended with 10% red mud, exchangeable fraction was reduced and iron and manganese oxides fraction was increased.

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