Cement Stabilization/Solidification of Heavy Metal-Contaminated Sediments Aided by Coal Fly Ash

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Abstract: Discharge of various wastewater containing heavy metals from metallurgical, chemical engineering industries, etc. into water body results in the accumulation of heavy metals in sediments of rivers and lakes, and further severe sediment contamination. When the severely contaminated sediments are dredged, they must be handled before disposal. In this paper, the dredged sediments were stabilized/solidified with cement aided by coal fly ash. The compressive strength of stabilization/solidification specimens decreased with the increase of sediment/binder ratio. All specimens showed a satisfactory compressive strength except that the compressive strength of the specimen with the high sediment/binder ration of 3.33 was lower than 0.35 MPa, the requirement of US EPA. Coal fly ash effectively acted as the binder, but the excessive addition of coal fly ash resulted in the reduction of specimen compressive strength. The pH of TCLP leachates for the specimens, which met the requirement of the compressive strength, was distributed between 11.07 and 12.50, and the heavy metal concentration of the TCLP leachates was lower than 0.029 mg/L. A similar trend of acid neutralization capacity of the stabilization/solidification specimens was observed as that of the compressive strength. Curing in a hermetically sealed plastic bag at ambient temperature and a longer curing period improved the stabilization/solidification effect. Humidity and temperature played an important role during curing period. Under an aggressive condition with a fixed leachate pH of 4, the stabilization/solidification technology increased the heavy metals fixed to 88.47%, 93.30% and 87.71% for zinc, lead and cadmium, respectively, and the concentration of zinc, lead and cadmium in the leachates was lower than the limits of the identification standards for hazardous wastes-identification for extraction toxicity (GB 5085.3-2007). So cement stabilization/solidification aided by coal fly ash can be an alternative disposal technology for dredged sediments contaminated by heavy metals.

Keywords: Stabilization/solidification, dredged sediments, coal fly ash, compressive strength, leaching test, curing condition.

1. INTRODUCTION

Wastewater containing heavy metals was discharged into rivers and canals in China, where the heavy metals precipitated or were combined with particles to join the sediments, leading to sediment contamination. Due to environment change, such as oxidation condition during a dry summer, pH reduction resulted from acid rain, etc., the heavy metals in sediments may be dissolved into water again. When the sediments are disturbed by floods and huge flows, the heavy metals can also be resuspended in water; subsequently, the heavy metals may be hydrodynamically transported to downstream [1]. Heavy metal pollution degree increases and heavy metal pollution range expands.

In recent years, frequency of water pollution accidents has greatly increased and drinking water safety has been threatened. To solve the problem of sediment contamination, the seriously polluted sediments will be or have been dredged from rivers and canals. Accordingly, sometimes large quantities of seriously contaminated sediments have been deposited on the banks or close to the canal. Stephens *et al*. [2] and Liu *et al*. [3] reported that zinc, cadmium and lead in dredged sediments are readily mobilizable to different degrees.

The contaminated sediments must be handled as a hazardous material before disposal. Stabilization/solidification is, according to the US Environmental Protection Agency (EPA), one of the best available demonstrated technologies to treat certain industrial wastes and contaminated dredged sediments [4, 5]. Parsa *et al*. [6] reported that stabilization/solidification technologies can not remove heavy metals from the polluted wastes but have an important role in fixing the heavy metals in the solid matrix physically as well as chemically to reduce their mobility, to minimize the threat to environment, and to ensure the compliance with the existing regulatory standards. Previous studies have conducted to stabilize/solidify the heavy metals with ordinary Portland cement, and a large quantity of cement was used as binder. Boura *et al*. [7] reported a solids/binder ratio from 0.30 to 0.55 for sludge stabilization/solidification. However, about one ton of green house gas CO_2 is released to the atmosphere during manufacturing one ton of Portland cement as a result of decarbonation of lime, which causes major problem of global warming [8, 9]. Part of Portland

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cement can be replaced by coal fly ash to reduce the CO_2 emission. Coal fly ash is one type of binding supplement materials in waste stabilization, which mainly used in two kinds of mixtures: Portland cement plus coal fly ash or lime plus coal fly ash. The coal fly ash exhibits pozzolanic properties, and reactive CaO, SiO_2 and Al_2O_3 contained in the Portland cement/coal fly ash mixture can react with each other in the hydration process to form calcium silicate hydrate (C–S–H) and $6CaO{\cdot}Al_2O_3{\cdot}3SO_3{\cdot}32H_2O$, etc. Through the formation of well bonded and low porosity network, the heavy metals can be immobilized [10-12]. At present, a number of studies have been focused on the solidification/stabilization of sludge and other hazardous wastes containing heavy metals using cement, lime, and coal fly ash as the binders [6, 8, 9, 13, 14]. However, there are little reports on the solidification/stabilization of contaminated dredged sediment.

A local stream in Hunan, China, flows through industry zone, and various wastewater containing heavy metals from metallurgical, chemical engineering industry, etc. was admitted to be discharged for over 50 years, leading to severe sediment contamination. The severely contaminated sediments have been planned to be dredged from the stream. The objective of the present work is to stabilize/solidify the heavy metals in the dredged sediments with ordinary Portland cement and coal fly ash. The treatability of dredged sediments by the stabilization/solidification process at different curing conditions and different leaching conditions was studied in detailed.

2. MATERIALS AND METHODS

2.1. Materials

For all analysis, chemicals used were of analytic grade and used as received, and water used was deioned water.

Sediment samples were taken from the local contaminated Stream. The sediments from 5 sampling sites along the this stream were mixed as the experimental materials. The sediments showed alkaline with a moisture content of 47.67%. Because of the long-term accumulation of pollutants, the sediments contain high content of heavy metals as follows: zinc, 3124.5 mg/kg dry solid (DS); lead 100.5 mg/kg DS and cadmium 105 mg/kg DS.

Coal fly ash and commercially available ordinary Portland cement were used as the binder for sediment stabilization/solidification. Coal fly ash was collected from a local thermal power station and its chemical composition is listed in Table **1**. Portland cement of P.O 42.5 MPa was commercially purchased.

Table 1: Chemical Composition of Coal Fly Ash

Component	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	SO_3	K_2O	Na_2O	CaO	MgO	MnO_2
Content (%)	54.83	28.79	3.77	1.71	1.23	4.94	1.30	1.22	0.92	0.024

2.2. Methods

2.2.1. Stabilization/Solidification Procedure

Sediments, cement, coal fly ash, sand and suitable water were sequentially added into an artificial concrete mixing container, and the mixture was stirred and vibrated for about 5 min to ensure the uniform distribution and complex mixing of all the materials and to remove the entrapped air. The stabilization/ solidification specimens were unmolded after 24-hour hardening, and then cured under different conditions and for different periods.

2.2.2. Compressive Strength Test

Compressive strength was selected as the benchmark parameter to simplify the specimen screening process, which has an intrinsic importance in the structural design of concrete structures [15]. The mixture of sediments, cement, coal fly ash, sand and water with different sediment/binder ratios was inserted in a cubic mold of 5 cm × 5 cm × 5 cm, compacted, vibrated to drive all the entrapped air away. The cubes were unmolded after 24-hour harding, and then the stabilization/solidification specimens were cured at room temperature for 3 d. Compressive strength test of the specimens was performed on a Universal Testing Machine (WDW-100, China)

2.2.3. Metal Leaching Test

Toxicity characteristic leaching procedure (TCLP) test and general acid neutralization capacity (GANC) test were used to directly or indirectly evaluate heavy metal stabilization/solidification effect. For TCLP test

[16], the stabilization/solidification specimens were crashed and grinded into particles smaller than 9.5 mm in diameter, then 2.5 g of grinded particles were placed in a polyethylene bottle, followed by the addition of 50 ml of acetic acid buffer solution with a pH of 2.8. The bottle was placed on a gyratory rotator (SHZ-82, China) and rotated at 180 r/min for 16±2 h, in order to prevent from stratification and further to reach the equilibrium rapidly. After rotation, the samples were filtered through a quantitative filter paper with slow flow rate, then the pH of leachates was measured with a pH meter (ORP-412, China) and the heavy metal concentration in leachates was measured with an atomic adsorption spectrometry (Perkin Elmer AA700, USA).

Environment Canada published the acid neutralization capacity (ANC) test to measure the capacity of different binders and wastes to resist pH reduction by exposure to a dilute nitric acid solution. The GANC test modified by Isenburg *et al*. [17] is a single-batch procedure that uses an acetic acid buffer solution with a pH of 2.8 (the same acetic acid buffer solution used for TCLP test). The acetic acid/solid ratio ranged from 0 to 6 mol/kg. With a liquid/solid ratio of 20 ml/g the mixture of grinded particles and the acetic acid buffer solution was rotated at 180 r/min on the gyratory rotator (SHZ-82, China) for 48 h. After rotation, samples were filtered through a quantitative filter paper with slow flow rate, and the pH of leachates was determined with the pH meter (ORP-412, China).

2.2.4. Metal Leaching Test Under Aggressive Condition

In order to prevent the potential risk of specimens for heavy metal stabilization/solidification, we carried out the metal leaching test under aggressive condition at a fixed pH of 4. With a liquid/solid ratio of 20 ml/g, the mixture of grinded particles and acetic acid solution with a pH of 4 was rotated at 180 r/min on the gyratory rotator (SHZ-82, China) for 16±2 h. During rotation, the pH of solutions was kept fixed at 4 by adding acetic acid. After rotation, samples were filtered through a quantitative filter paper with slow flow rate, and the heavy metal concentration in leachates was measured with the atomic adsorption spectrometry (Perkin Elmer AA700, USA).

3. RESULTS AND DISCUSSION

3.1. Stabilization/Solidification of Sediments with Different Sediment/Binder Ratios

Compressive strength can be used to provide a baseline comparison between unstabilized and stabilized wastes. Unstabilized wastes generally do not exhibit good compressive strength; however, if the waste is stabilized into a concrete-like form, the compressive strength characteristics can be expected to increase significantly [18, 19]. Minimum compressive strength required for a stabilization/solidification material should be evaluated on the basis of the design loads. U.S. EPA considers a compressive strength of 0.35 MPa to be satisfactory for a stabilization/ solidify-cation material cured for 28 d. Prepared specimens with different sediment/binder ratios were cured for 3 d at room temperature and then subjected to compressive strength test. The experiment arrangement and the compressive strength of the specimens are shown in Table **2**. It was observed that the compressive strength for all specimens generally decreased with the increase of sediment/binder ratio. The compressive strength of the specimen M6 was 0.28 MPa, lower than the requirement of U.S. EPA, and all the other specimens show a satisfactory compressive strength. The low binder addition may affect the C-S-H hydration. For the specimen M1, M2 and M3, which were all prepared with a sediment/binder ratio of 1, the compressive strength reduced with the increase of coal fly ash addition and the simultaneous decrease of cement dosage, indicating that the cement played a

Table 2: Arrangement of Specimens and Their Compressive Strength (Compressive Strength of 0.35 MPa, and Curing Time of 3d)

Specimen	Sediments (wt.%)	Cement (wt.%)	Fly Ash (wt.%)	Sand (wt.%)	Compressive Strength (MPa)
M1	25.0	12.5	0	25.0	2.30
M2	25.0	17.5	7.5	25.0	1.77
M3	25.0	10.0	15.0	25.0	0.94
M4	25.0	10.0	2.5	12.5	1.47
M5	25.0	5.0	5.0	5.0	0.49
M6	25.0	5.0	2.5	7.5	0.28

more important role than coal fly ash as the binder [4, 20]. Between the specimen M5 and M6, the compressive strength of M6 was lower than that of M5 because of the less addition of coal fly ash with the same quantity of sediments and cement, demonstrating that the coal fly ash acted actually as a binder. Between the specimen M3 and M4, the compressive strength of M3 was lower than that of M4 because of the excessive addition of coal fly ash with the same quantity of sediments and cement. Although the coal fly ash exhibits pozzolanic properties, it showed obvious weaker capacity as a binder to bind and harden the aggregates into a rocklike mass than cement. So the suitable proportion between cement and coal fly ash is important.

3.2. Heavy Metal Leaching after Sediment Stabilization/Solidification

3.2.1. Influence of Sediment/Binder Ratio

Leaching tests were carried out to display the effectiveness of heavy metal immobilization within cement-based matrixes. One part of the grinded samples of the stabilized specimens M1-M5 cured for 3 d at room temperature and the non-stabilized dredged sediments were subjected to TCLP test. Another part of the same samples was then subjected to GANC test. The results of TCLP test are shown in Table **3**. The pH of leachates for the stabilization/solidification matrixes slowly decreased with the increase of the sediment/binder ratio, and was observed between 11.07 and

Table 3: Characteristics of TCLP Leachates (Rotation Speed of 180 r/min, Rotation Time of 16±2 h)

Specimen	pH	Zn (mg/L)	Pb (mg/L)	Cd (mg/L)
Sediments	6.29	2.558	0.090	0.297
M1	12.50	0.021	0.020	0.046
M2	12.08	0.019	0.020	0.045
M3	11.49	0.019	0.018	0.053
M4	11.78	0.016	0.007	0.054
M5	11.07	0.029	0.028	0.064

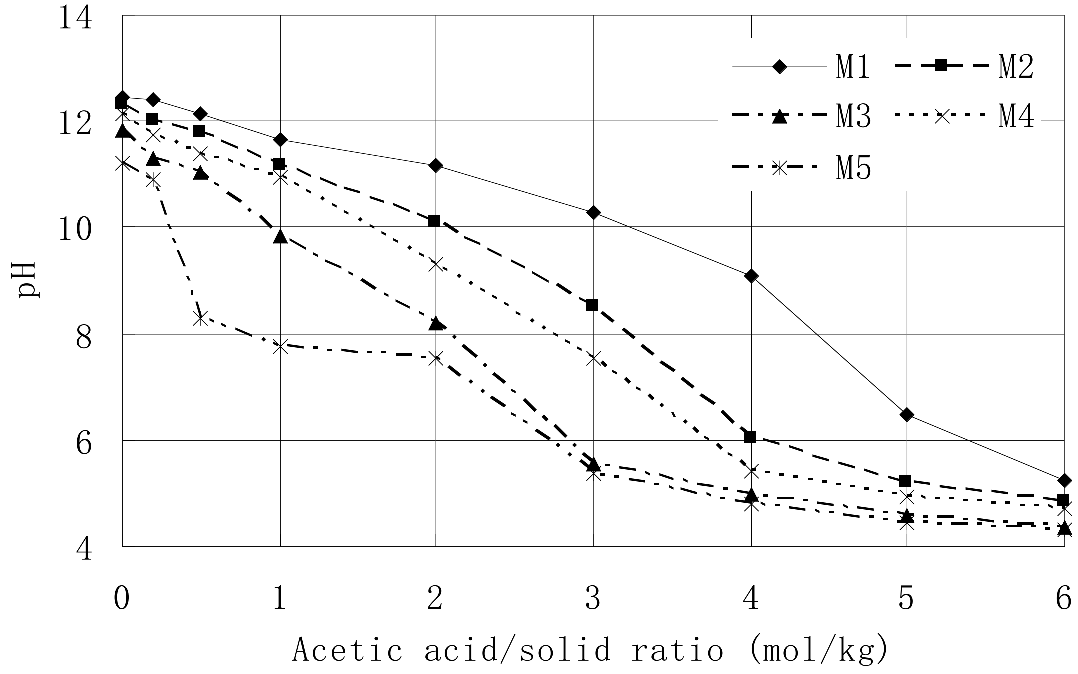

Figure 1: GANC curves of tested specimens cured for 3 d (Rotation speed of 180 r/min, rotation time of 48 h).

12.50 throughout the TCLP test. However, the pH of leachates for the raw sediments was very low, only 6.29.

The availability levels of heavy metals in TCLP leachates for the stabilization/solidification specimens were very low, showing a very low extraction toxicity of the stabilization/solidification products. Zinc was found in the highest concentration of 2.558 mg/L in the TCLP leachate of the non-stabilized dredged sediments. Although zinc was not completely immobilized in the concrete-like matrixes, its concentration in leachates of the stabilized specimens was low, ranging from 0.016 to 0.029 mg/L. The concentration of zinc in leachates decreased by over 98% after stabilization/solidification processing. The concentration of more toxic cadmium in leachates of the non-stabilized dredged sediments was 0.297 mg/L, and its concentration in leachates of the stabilized specimens at least decreased by 91%. It should be pointed out that main reasons of heavy metal immobilized in the stabilization/solidification matrixes were alkaline nature and buffer capacity provided by calcium hydroxide and C–S–H. The leaching of metal contaminants in these matrixes can be considered as pH dependent and metal hydroxide solubility controlled process [21]. Although zinc, lead and cadmium possess amphoteric properties, the solubility of their hydroxides is lowest at a pH 9.4, 10.6 and 11.6, respectively [22]. During the hydration process not only the highly alkaline environment results in the formation of metal hydrated phases and metal hydroxides in the stabilization/solidification matrixes, but also the metal hydroxide precipitates may reacts with hydrating cement phase to form complex hydrated compound and be incorporated in hydrating C–S–H [14, 19, 23].

Figure **1** shows the results of GANC test for the stabilization/solidification specimens cured for 3 d at room temperature. The results of GANC test gave more evidences that the specimens with lower sediment/binder ratio had stronger acid neutralization capacity. A similar trend of the acid neutralization capacity of the stabilization/solidification specimens was observed with that of the compressive strength (Table **2**). With the increase of coal fly ash addition, the acid neutralization capacity of the specimens reduced, following the order of M1 > M2 > M3. The acid neutralization capacity is related with the kind and the quantity of binders, and the stabilization capacity of stabilization/solidification products. The acid neutralization capacity of the specimen M4 was stronger than that of M3, which was probably caused by the negative influence of the excessive coal fly ash addition on the formation of C-S-H and the further incomplete hydration between the sediments and the alkaline binders, so the stabilization/solidification matrixes may be easily destroyed by the acetic acid buffer solution, leading to more rapid pH reduction. When the binder content further reduced, the acid neutralization capacity of specimen M5 rapidly decreased.

3.2.2. Influence of Environmental Curing Condition

In order to study the influence of environmental conditions during curing, the stabilization/solidification specimens M2, M3 and M4 were selected to test under different environmental curing conditions: A, at ambient temperature in laboratory, B, in a hermetically sealed plastic bag at ambient temperature and C, in a stove at a temperature of 60°C. The curing period of the stabilization/solidification specimens was 1 3 and 7 d. The results of TCLP test of these specimens are shown in Table **4** and Table **5**. The results of TCLP test from

Table 4: Influence of Stabilization/Solidification Curing Condition on Characteristics of TCLP Leachates

Specimen	Condition	Period (d)	pH	Zn (mg/L)	Pb (mg/L)	Cd (mg/L)
M2	A	1	12.5	0.060	0.015	0.083
M2	B	1	12.95	0.013	0.021	0.084
M2	C	1	12.03	0.020	0.036	0.070
M2	A	3	12.37	0.009	0.019	0.036
M2	B	3	12.53	0.017	0.013	0.040
M2	C	3	11.74	0.003	0.019	0.038
M2	A	7	11.52	0.004	0.015	0.017
M2	B	7	12.1	0.011	0.006	0.017
M2	C	7	11.2	0.003	0.017	0.032

Curing conditions: A, at ambient temperature in laboratory; B: in a hermetically sealed plastic bag at ambient temperature; C: in a stove at a temperature of 60°C.

Table 5: Influence of Stabilization/Solidification Curing Period on Characteristics of TCLP Leachates

Specimen	Condition	Period (d)	pH	Zn (mg/L)	Pb (mg/L)	Cd (mg/L)
M3	A	1	12.61	0.055	0.035	0.081
M3	A	3	11.78	0.003	0.017	0.041
M3	A	7	11.20	0.002	0.012	0.036
M3	B	1	12.31	0.047	0.034	0.080
M3	B	3	11.93	0.013	0.024	0.044
M3	B	7	11.20	0.004	0.023	0.036
M4	A	1	11.90	0.046	0.055	0.067
M4	A	3	11.62	0.012	0.046	0.064
M4	A	7	11.37	0.016	0.041	0.072
M4	B	1	12.03	0.056	0.059	0.071
M4	B	3	11.78	0.015	0.045	0.063
M4	B	7	11.39	0.021	0.045	0.088

Curing conditions: A, at ambient temperature in laboratory; B: in a hermetically sealed plastic bag at ambient temperature; C: in a stove at a temperature of 60°C.

Table **4** show that the curing conditions have an influence on the characteristics of leachates. As can be seen, the pH of specimens cured under the condition B was obvious higher than that cured under the condition A and the condition C. The similar situation appeared for the specimen M3 and M4. This showed that humidity and temperature during curing period played an important role, not only on the physical properties but also on the chemical stability of the final stabilization/solidification specimens [24, 25].

From Table **5** we can observe that a general decrease of the leachate pH and the concentration of zinc and lead in leachates with the increase of the curing period under all three conditions, it may be due to the further hydration reaction of Portland cement and the further curing of calcium silicate hydrate (C-S-H) in the stabilization/solidification matrixes, thus the heavy metals both chemically fixed in the lattice of hydration products and physically encapsulated in the matrixes [26, 27]. However, an unlike change of the cadmium concentration in leachates was observed, because the leachte pH was very close to 11.6, at which the solubility of cadmium hydroxide is lowest. In addition the concentration of zinc, lead and cadmium decreased greatly from the specimens cured for 1 d to that cured for 3 d, while the concentration of heavy metals was nearly same between the specimens cured for 3 d and that cured for 7 d. This indicated that the fixation effect may be mainly reached in the beginning phase. Because of the high alkaline environment of the stabilization/solidification matrixes, the leachated concentration of zinc, lead and cadmium was low, and the fixation rate of heavy metals was higher than 98.32%.

In the case of specimen M4 cured for 1 d and 3 d, a GANC test was carried out. The results are shown in Figure **2**. As can be seen, the acid neutralization capacity of the specimens cured for 3 d was obviously stronger than that cured for 1 d, particularly under condition A. Under condition B the sealed plastic bag prevented the moisture form evaporation, and the higher moisture content enhanced the hydration within the stabilization/solidification products to form more stable crystal lattice matrixes and to maintain their alkalinity.

3.2.2. Metal Fixation Under Aggressive Leaching Condition

Metal leaching test was carried out under a fixed final pH of leachates of 4.0 to simulate the effects of aggressive environmental condition. The leachable portion of the heavy metals from raw sediments was available with a zinc concentration of 39.50 mg/L, a lead concentration of 0.39 mg/L and a cadmium concentration of 1.30 mg/L, and correspondingly the percentage of immobilized heavy metals was 74.72%, 92.24% and 75.16%, respectively. The concentration of cadmium exceeded 1 mg/L, the limit of the identification standards for hazardous wastes-identification for extraction toxicity (GB 5085.3-2007, China) [28].

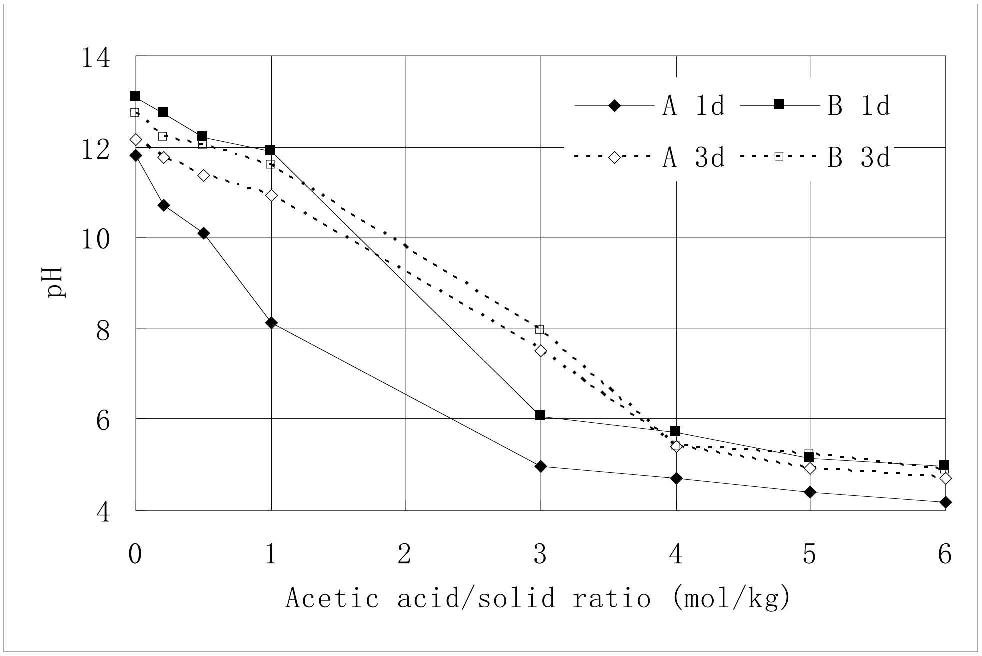

Figure 2: GANC curves of M4 specimen under different conditions and curing periods (Rotation speed of 180 r/min, rotation time of 48 h).

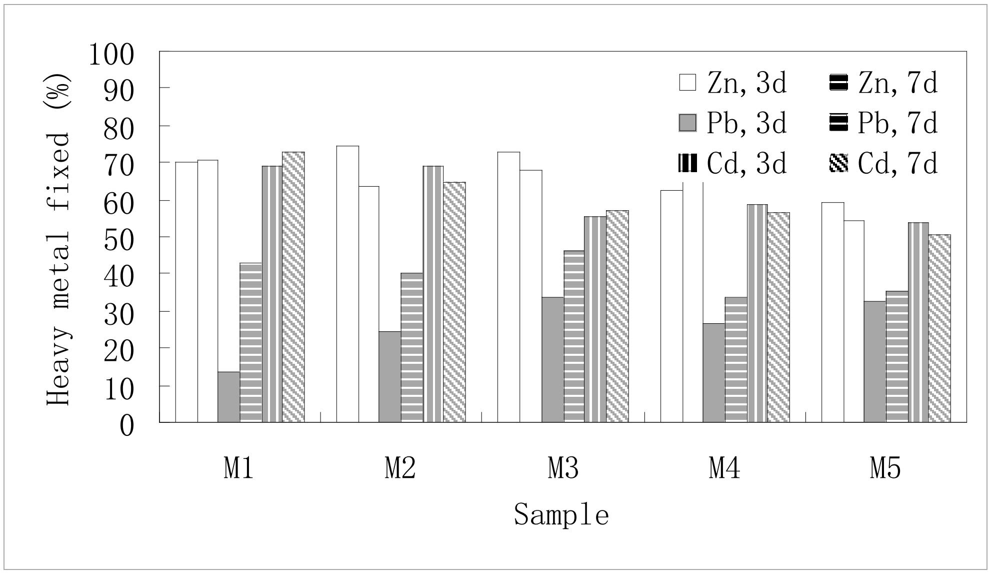

Figure 3: Heavy metal fixation at a pH of 4 and a curing period of 3 and 7 d (Rotation speed of 180 r/min, rotation time of 16±2 h).

Figure **3** presents the heavy metals fixed through stabilization/solidification processing. The stabilization/solidification technology showed a high retention of the heavy metals. Zinc immobilization was distributed between 59.49% and 74.25%. A better immobilization of zinc was observed with a higher sediment/binder ratio of 1 (the specimen M1, M2 and M3), but there was no significant difference between the specimens cured for 3 d and cured for 7 d. The maximum concentration of zinc in the leachates was 18.02 mg/L, which was much higher than that in the normal TCLP test. For cadmium, the heavy metal immobilization situation was very similar with that of zinc. The cadmium immobilization was distributed between 53.60% and 70.48%, and the highest cadmium concentration in the leachates was 0.645 mg/L, which may leads to some hidden dangers. The lead immobilization was low for the specimens cured for 3 d, which probably resulted from the low solubility and low quantity of lead in the raw sediments. The lead immobilization was comparatively improved when the specimens were cured for 7

d. The lead immobilization was distributed between 33.85% and 42.67%, and the highest lead concentration in the leachates was 0.336 mg/L after 7-day curing. Dermatas *et al*. [29] and Jirka *et al*. [30] reported that zinc and lead have the lowest leaching at a pH range of 9-11. When the pH decreased or increased beyond this range, the leaching rate increased. However, the concentration of zinc, lead and cadmium in the leachates was still lower than the limits (100, 50 and 1 mg/L, respectively) of the identification standards for hazardous wastes-identification for extraction toxicity (GB 5085.3-2007, China) [28]. The percentage of the heavy metals fixed to the total heavy metal content after stabilization/ solidification processing increased to 88.47%, 93.30% and 87.71% for zinc, lead and cadmium, respectively, and the stabilization/solidification products were safe for the environment.

CONCLUSIONS

The results of the study indicated that with the increase of sediment/binder ratio, the compressive strength was considerably decreased, and the compressive strength of specimen with a sediment/binder ratio of 3.33 was lower than 0.35 MPa, which was considered as the minimum for waste stabilization/solidification. Coal fly ash can substitute for part of cement as the binder, but the excessive addition of coal fly ash showed a negative influence on the compressive strength of the specimens. With the satisfactory compressive strength, the pH of the TCLP leachates for all specimens was distributed between 11.07 and 12.50, and all the heavy metal concentration of the TCLP leachates was lower than 0.029 mg/L. The acid neutralization capacity of the specimens decreased with the increase of the sediment/binder ratio. The curing conditions had an influence on the leaching potential of heavy metals, and curing in a hermetically sealed plastic bag at ambient temperature and for a longer period improved the stabilization/solidification effect. The aggressive leaching condition with a fixed pH of 4 significantly enhanced the leaching of the heavy metals, however, the concentration of the heavy metals was still lower than the limits of the identification standards for hazardous wastes-identification for extraction toxicity (GB 5085.3-2007, China).

The reasonable sediment/binder ratio and the addition quantity of coal fly ash are very important for cement stabilization/solidification of the heavy metal-contaminated sediments or wastes aided by coal fly ash. By sanitary landfill numerous interacting factors can simultaneously affect the complexation of the heavy metals with the binders and the stabilization/ solidification matrixes. In addition, the regulations concerning waste stabilization should have to be considered in more detail and the potential risk assessment of the stabilization/solidification products for a long-term storage (landfill) should be emphasized.

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