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Effects of chelant (EDTA) addition on properties of cement-solidified municipal incinerator fly ash

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Abstract

A preliminary study was conducted to investigate the effects of EDTA (a chelant) addition on properties of cement-solidified municipal incinerator fly ash (MIFA) due to its increasing use for the removal of metals from soil and wastes. MIFA specimens (designated MIFA-A and MIFA-B, respectively) from two different municipal incineration plants were solidified by a cement-based technique. Properties studied included unconfined compressive strength (UCS) and leaching toxicity by Toxicity Characteristic Leaching Procedure (TCLP). Regardless of an addition of EDTA, experimental results have shown that UCS values of all the solidified MIFA specimens are greater than the R.O.C. EPA regulatory requirement for landfilling (i.e. 10 kg/cm^2). The effect of EDTA addition on UCS was found to be trivial. Results of the Analysis of Variance (ANOVA) for UCS values also showed that the amount of EDTA added was not a controlling parameter in this work. It was found that solidification of MIFA by ordinary portland cement (OPC) alone is able to yield solidified monoliths of satisfactory properties. TCLP results of EDTA-added MIFA specimens also showed a limited effect due to EDTA addition. Whether an addition of other chelants would yield a significant effect on properties of solidified MIFA specimens needs further studies. © 1998 Elsevier Science B.V.

Keywords: Municipal incinerator fly ash; Solidification; Chelant; Statistical analysis

1. Introduction

The health and environmental hazards of MIFA have brought much concern to the public worldwide. In Taiwan, according to the statistics of R.O.C. Environmental Protection Administration (R.O.C. EPA) [1], the generation rate of municipal solid waste

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(MSW) is 1.14 kg per capita per day in 1995. That is, a total of 8 400 000 metric tons MSW was generated in 1995. Of which, only 14.98% was treated by incineration and the rest were disposed of by landfilling (79.24%) and other methods (5.78%). Because a majority of landfills will come to the end of their lives within a year or two, R.O.C. EPA has planned to construct 22 large-scale MSW incineration plants by the end of year 2000. (Currently, five MSW incineration plants are in continuous operation.) It was estimated that at least 1700 metric tons per day of municipal incinerator ashes will be generated in the future [2], of which 15–20% will be MIFA. MIFA has been reported to be hazardous due to its high leaching toxicity of heavy metals and high toxicity equivalent quantity of dioxin-like compounds [3–13]. Thus, MIFA has been categorized as a special waste or hazardous waste in many countries. A proper treatment and/or disposal of MIFA is thus needed.

The cement-based solidification technology, regardless of its disadvantages, is a popular method widely used for the treatment of many hazardous wastes including MIFA [9,11,12,14–19]. To the knowledge of the present authors, many MSW incineration plants to be built in Taiwan have planned to adopt the employment of chelant(s) in their semi-dry scrubbers for air pollution control. The chelant(s) used might end up in the MIFA fraction, which will then be solidified. The information regarding the effects of chelant addition on solidification of MIFA is lacking in the literature. Therefore, it is worth studying the role of chelant(s) in solidifying MIFA.

This investigation is aimed at studying the effects of the presence of a chelant (e.g. EDTA) with MIFA on UCS and TCLP leaching toxicity of solidified MIFA. This is because that chelants are likely to be used increasingly for the removal of heavy metals from soil and wastes. To simulate the chelant-containing MIFA, in this study EDTA was mixed with MIFA and OPC while preparing the solidified MIFA specimens. As a reference experiment, specimens of MIFA mixed with EDTA solution without OPC were also prepared. This is to determine whether EDTA alone would be significant in reducing the leaching toxicity of MIFA. For solidified MIFA specimens, their UCS values were analyzed statistically to determine the role of each experimental factor in solidification.

2. Experimental

2.1. Materials

MIFA specimens from two different municipal incineration plants were solidified by OPC (i.e. ASTM Type I portland cement). Plant A employs cyclones, semi-dry scrubber, and fabric filters for air pollution control, whereas Plant B uses cyclones, dry scrubber, and fabric filters. Normally, in either semi-dry scrubber or dry scrubber system a large amount of lime is used for neutralizing acid gases such as HCl and SO_x. Thus, particulates collected by fabric filters would contain a substantial amount of lime and diatomaceous earth. In this study, these two MIFA specimens are designated MIFA-A and MIFA-B, respectively. However, MIFA-A contained both the larger size fraction collected by cyclones and the finer size fraction collected by fabric filters; whereas

MIFA-B contained only the finer size fraction collected by fabric filters. Ethylenedinitrilo tetraacetic acid disodium salt dihydrate (designated EDTA in this study; 99% in purity; Merck) was the chelant used in solidification. Water/mixing water used is ASTM Type I deionized water. All chemicals used are reagent grade.

2.2. Experimental design

In this work, the solidification recipes employed followed the L_9 orthogonal arrays [20,21]. The experimental factors adopted included the weights of EDTA, OPC, mixing water, and MIFA. Each experimental factor had three levels of variation. The detailed experimental design of solidification of MIFA is shown in Table 1.

2.3. Methods

Each MIFA specimen was first characterized using various standard methods adopted by Republic of China and United States [22,23]. The determined properties included the moisture content, pH and total contents and TCLP leaching toxicity of heavy metals. In addition, the particle size distribution was determined by a Coulter LS100 Particle Size Analyzer; and loss on ignition, by ASTM C 311-90 [24].

Making and curing of solidified specimens were conducted according to CNS 1230 A3043 [25]. In this study, PVC molds of cylindrical shape (50 mm $\times$ 100 mm; $d \times h$) were used for all the test specimens. The mixing of MIFA, OPC, and EDTA solution was conducted in an electrically driven mechanical mixer of the epicyclic type, which

Table 1
Experimental design of solidification of municipal incinerator fly ash using the L_9 orthogonal arrays

Solidified monolith No.	Experimental factor for solidification			
	EDTA	OPC	WATER	MIFA
1	1	1	1	1
2	1	2	2	2
3	1	3	3	3
4	2	1	2	3
5	2	2	3	1
6	2	3	1	2
7	3	1	3	2
8	3	2	1	3
9	3	3	2	1

Level of variation	EDTA (g)	OPC (g)	WATER (g)	MIFA (g)
1	0.372	300	450	550
2	1.86	350	500	600
3	3.72	400	550	800

EDTA denotes the weight of the chelant used.
OPC denotes the weight of ordinary portland cement used (as received).
WATER denotes the weight of mixing water used.
MIFA denotes the weight of municipal incinerator fly ash treated (as received).

Table 2
Various physical and chemical properties of MIFA-A and MIFA-B

MIFA specimen	Moisture content (%)	pH (in 0.01 M $CaCl_2$)	Loss on ignition (%)	Particle size (μm)	
				Mean	Median
MIFA-A	9.5	11.38	10.39	97.61	22.09
MIFA-B	2.17	10.24	20.70	12.60	7.12

imparts both a planetary and revolving motion to the mixer paddle. After curing, UCS and leaching toxicity of the solidified specimens were evaluated. UCS was measured according to CNS 1232 A3045 [26]. The leaching toxicity was determined through the employment of the TCLP test for the extraction of heavy metals, followed by the analysis of heavy metal concentrations using a flame atomic absorption spectrophotometer.

3. Results and discussion

3.1. Characteristics of municipal incinerator fly ashes

Various physical and chemical properties of MIFA-A and MIFA-B studied in this work were presented in Tables 2 and 3. Since Plant A employs a semi-dry scrubber and Plant B employs a dry scrubber, it is reasonable for MIFA-A to have a greater moisture content. As indicated above, MIFA-B contained only the finer size fraction of particulates, it is reasonable to have a much smaller mean particle size than that of MIFA-A. Rather high values of loss on ignition for both MIFA-A and MIFA-B might be due to the presence of a large quantity of diatomaceous earth, which has been employed for the protection of fabric filters. From the TCLP leaching toxicity of heavy metals shown in Table 3, the leached lead concentrations for both MIFA specimens are greater than the current R.O.C. EPA regulatory threshold (i.e. 5 mg/l). Therefore, both the MIFA-A and MIFA-B specimens studied in this work were identified as hazardous wastes. Solidification or other treatment of these wastes was inevitably needed before their final disposal.

3.2. Unconfined compressive strengths of solidified MIFA specimens

Results of UCS measurements are shown in Tables 4 and 5. In these tables, the term ‘control group’ denotes the specimens solidified only using OPC; whereas ‘sample

Table 3
Total content and TCLP leaching toxicity of MIFA-A and MIFA-B

Type of metal	Total content (mg/kg)		TCLP (mg/l)	
	MIFA-A	MIFA-B	MIFA-A	MIFA-B
Pb	4073	6230	75.2	112.4
Cd	212	219	0.08	0.09
Cr	94	53	0.99	1.34
Zn	7245	14 263	10.65	6.15
Cu	733	828	0.56	1.30

Table 4
Unconfined compressive strengths of solidified monoliths of MIFA-A at an age of 28 days

Solidified monolith No.	Unconfined compressive strength (kg/cm^2)		
	Control group	Sample group	Difference
1	35 ± 0	38 ± 1	3
2	46 ± 3	46 ± 5	0
3	37 ± 0	33 ± 1	− 4
4	34 ± 1	26 ± 1	− 8
5	48 ± 1	34 ± 2	− 14
6	47 ± 4	86 ± 10	39
7	30 ± 2	28 ± 1	− 2
8	52 ± 2	55 ± 1	3
9	49 ± 2	53 ± 2	4

For each solidified specimen, regardless of the control group or sample group, the amount of binder used is OPC as shown in Table 1.
Control group denotes specimens solidified only by Type I portland cement.
Sample group denotes specimens solidified by cement with an addition of EDTA.
Difference = UCS of sample group − UCS of control group.
Three solidified specimens were made for each solidification recipe.
Each unconfined compressive strength value is expressed as follows: average value ± standard deviation.

group' denotes the specimens solidified by OPC with an addition of EDTA. Results of previous studies have indicated that an addition of a superplasticizer to cement paste yielded a greater UCS value of the solidified MIFA specimen than its corresponding specimen in the control group [9,11]. However, this does not hold true for the case of EDTA addition. From Tables 4 and 5, it is evident that an addition of EDTA to cement not necessarily results in greater UCS values for the sample group. In many cases the

Table 5
Unconfined compressive strengths of solidified monoliths of MIFA-B at an age of 28 days

Solidified monolith No.	Unconfined compressive strength (kg/cm^2)		
	Control group	Sample group	Difference
1	31 ± 3	34 ± 6	3
2	35 ± 3	38 ± 8	3
3	36 ± 3	42 ± 9	6
4	27 ± 3	25 ± 2	− 2
5	26 ± 2	29 ± 0	3
6	46 ± 2	52 ± 6	6
7	15 ± 4	10 ± 0	− 5
8	66 ± 6	59 ± 0	− 7
9	56 ± 1	47 ± 2	− 9

For each solidified specimen, regardless of the control group or sample group, the amount of binder used is OPC as shown in Table 1.
Control group denotes specimens solidified only by Type I portland cement.
Sample group denotes specimens solidified by cement with an addition of EDTA.
Difference = UCS of sample group − UCS of control group.
Three solidified specimens were made for each solidification recipe.
Each unconfined compressive strength value is expressed as follows: average value ± standard deviation.

UCS differences are negative values. It was also observed that all the UCS values in both the sample group and control group were rather low. Perhaps this is due to a high content of lime within the MIFA specimens, resulting in a swelling effect during solidification. This point has to be verified by further studies. Nonetheless, all the UCS values obtained were found to be greater than the current R.O.C. EPA regulatory requirement for landfilling solidified wastes (i.e. 10 kg/cm^2).

Tables 6–8 show the results of statistical analyses of the UCS values with respect to each experimental factor for solidified MIFA-A and MIFA-B specimens. Here, the degree of contribution was determined by ANOVA; whereas response values were determined by the regular analysis [20]. In Table 6, the weight of OPC used was determined to be a controlling parameter for the solidification of MIFA-A when EDTA was not added. No controlling parameter was found in the case of solidifying MIFA-B. Although the weight of mixing water has the largest degree of contribution for UCS values of the sample group of solidified MIFA-A (see Table 7), it is not a controlling parameter in this case of solidification from a statistical point of view. So is the weight of EDTA. This holds true for UCS differences between the sample group and control group of solidified MIFA-A. From Table 8, however, it is clear that the weights of OPC and mixing water are identified as two controlling parameters for UCS values of the sample group of solidified MIFA-B. Each of these two experimental factors has a significance level of 5% according to the *F*-test. Again, the weight of EDTA is not a

Table 6
Degree of contribution and response values for unconfined compressive strengths of solidified monoliths of MIFA-A and MIFA-B at an age of 28 days (control group)

UCS values of the control group							
MIFA-A				MIFA-B			
a	b	c	d	a	b	c	d
		1	39.33			1	34.00
(e)	2.89	2	43.00	(e)	5.72	2	33.00
		3	43.67			3	45.67
		1	33.00			1	24.33
OPC	73.75[a]	2	48.67	OPC	30.86	2	42.33
		3	44.33			3	46.00
		1	44.67			1	47.67
Water	9.19	2	43.00	Water	27.57	2	39.33
		3	38.33			3	25.67
		1	44.00			1	37.67
MIFA	14.17	2	41.00	MIFA	35.84	2	32.00
		3	41.00			3	43.00

a; the experimental factor.
b; the degree of contribution (%).
c; the level of variation.
d; the response value.
(e); the error term.
OPC; the weight of ordinary portland cement used.
Water; the weight of mixing water used.
MIFA; the weight of municipal incinerator fly ash treated.
[a]A level of significance of 5%.

Table 7
Degree of contribution and response values for unconfined compressive strengths of solidified monolith on MIFA-A at an age of 28 days

UCS values of the sample group				UCS differences between the sample group and control group			
a	b	c	d	a	b	c	d
		1	39.00			1	−0.67
EDTA	20.62	2	48.67	EDTA	15.50	2	6.00
		3	45.33			3	1.67
		1	30.67			1	−2.00
OPC	32.93	2	45.00	OPC	25.36	2	−4.00
		3	57.33			3	13.00
		1	59.67			1	15.00
Water	37.89	2	41.67	Water	39.28	2	−1.33
		3	31.67			3	−6.67
		1	41.67			1	−2.23
MIFA	8.55	2	53.33	MIFA	19.86	2	12.00
		3	38.00			3	−2.67

a; the experimental factor.
b; the degree of contribution (%).
c; the level of variation.
d; the response value.
EDTA; the weight of the chelant used.
OPC; the weight of ordinary portland cement used.
Water; the weight of mixing water used.
MIFA; the weight of municipal incinerator fly ash treated.

significant experimental factor in this case. But, for UCS differences between the sample group and control group of solidified MIFA-B, the weight of EDTA was determined to be a controlling parameter. It has the largest degree of contribution (i.e. 78.69%) and its corresponding significance level is 5%. Response values indicate that, under the experimental conditions used, a smaller amount of EDTA added would be favorable to UCS differences between the sample group and control group of solidified MIFA-B.

3.3. *TCLP leaching toxicity of solidified MIFA specimens*

As indicated above, the TCLP leached lead concentrations of MIFA-A and MIFA-B are greater than the current R.O.C. EPA regulatory threshold. Therefore, lead is the only heavy metal of concern in this study.

The TCLP leached lead concentrations of the control groups and sample groups for the solidified MIFA-A and MIFA-B are shown in Tables 9 and 10. All the leached concentrations of lead were found to be lower than the regulatory limit of 5 mg/l. It was noted that solidification treatment of MIFA-A and MIFA-B specimens by OPC alone was sufficient to greatly reduce the leaching of lead and other metals from the MIFA specimens studied. More importantly, an addition of EDTA to cement did not result in greater leached concentrations of other heavy metals (e.g. cadmium) from solidified MIFA specimens. In other words, the effects of EDTA addition to cement were not noticeable in this work. Whether this phenomenon will be observed for other chelants is

Table 8
Degree of contribution and response values for unconfined compressive strengths of solidified monolith on MIFA-B at an age of 28 days

UCS values of the sample group				UCS differences between the sample group and control group			
a	b	c	d	a	b	c	d
		1	38.00			1	4.00
EDTA	4.19	2	35.33	EDTA	78.69[a]	2	2.33
		3	38.67			3	−7.00
		1	23.00			1	−1.33
OPC	53.00[a]	2	42.00	OPC	12.77	2	−0.33
		3	47.00			3	1.00
		1	48.33			1	0.67
Water	37.42[a]	2	36.67	Water	7.51	2	−2.67
		3	27.00			3	1.33
		1	36.67			1	−1.00
MIFA	5.39	2	33.33	MIFA	1.04	2	1.33
		3	42.00			3	−1.00

a; the experimental factor.
b; the degree of contribution (%).
c; the level of variation.
d; the response value.
EDTA; the weight of the chelant used.
OPC; the weight of ordinary portland cement used.
Water; the weight of mixing water used.
MIFA; the weight of municipal incinerator fly ash treated.
[a]A level of significance of 5%.

not clear at this point. It is well known that chelants have different complexing capabilities at different pHs. The pH effects of chelants on UCS values of solidified MIFA specimens are not included in the work. Therefore, they are worth studying in the future.

3.4. Leaching toxicity of EDTA-added MIFA specimens

Since the effects of EDTA addition to cement were not significant to UCS of solidified MIFA, EDTA solution alone was mixed with MIFA-A and MIFA-B, respectively. Here, the EDTA-added MIFA specimens were also placed in the PVC molds. They were cured at ambient temperature for 28 days before the TCLP test was conducted. Table 11 shows the mix formulation employed in this practice. Results of TCLP leaching toxicity for EDTA-added MIFA-A and EDTA-added MIFA-B are shown in Figs. 1 and 2, respectively.

Experimental results have indicated that an addition of EDTA to the MIFA specimens are capable of reducing the leached concentrations of heavy metals, particularly for lead (see Table 3 and Figs. 1 and 2). However, EDTA alone is not able to reduce the leached lead concentrations to values lower than the current R.O.C. EPA regulatory requirement with the mix formulation used. Figs. 1 and 2 have shown a trend of a lower leached lead concentration using a greater amount of EDTA added. Since EDTA itself is very costly,

Table 9
Heavy-metal concentrations of TCLP leachates of solidified MIFA-A specimens at an age of 28 days

Solidified monolith No.	Control group		Sample group	
	Pb concentration (mg/l)	Cd concentration (mg/l)	Pb concentration i(mg/l)	Cd concentration (mg/l)
1	1.42	0.05	1.42	0.04
2	1.48	0.04	1.42	0.04
3	1.65	0.04	1.42	0.03
4	1.76	0.04	1.89	0.03
5	1.19	0.03	1.31	0.03
6	1.08	0.03	1.19	0.03
7	0.88	0.04	0.88	< 0.02
8	1.57	0.04	1.37	< 0.02
9	0.39	0.04	0.69	< 0.02

For each solidified specimen, regardless of the control group or sample group, the amount of binder used is OPC as shown in Table 1.
Control group denotes specimens solidified only by Type I portland cement.
Sample group denotes specimens solidified by cement with an addition of EDTA.

Table 10
Heavy-metal concentrations of TCLP leachates of solidified MIFA-B specimens at an age of 28 days

Solidified monolith No.	Control group		Sample group	
	Pb concentration (mg/l)	Cd concentration (mg/l)	Pb concentration (mg/l)	Cd concentration (mg/l)
1	1.58	0.04	1.56	0.04
2	1.27	0.02	1.27	0.02
3	1.66	0.03	1.93	0.03
4	1.78	0.03	1.75	0.03
5	2.23	0.03	1.64	0.03
6	1.83	< 0.02	1.42	< 0.02
7	1.10	0.02	1.00	0.02
8	1.12	0.03	1.19	0.03
9	1.11	0.04	0.93	0.04

For each solidified specimen, regardless of the control group or sample group, the amount of binder used is OPC as shown in Table 1.
Control group denotes specimens solidified only by Type I portland cement.
Sample group denotes specimens solidified by cement with an addition of EDTA.

Table 11
The mix formulation for EDTA-added MIFA specimens

Sample No.	EDTA (g)	WATER (g)	MIFA (g)
1	0.372	450	600
2	1.860	450	600
3	3.720	450	600

Notes: 1. EDTA denotes the weight of the chelant used.
2. WATER denotes the weight of mixing water used.
3. MIFA denotes the weight of municipal incinerator fly ash treated (as received).

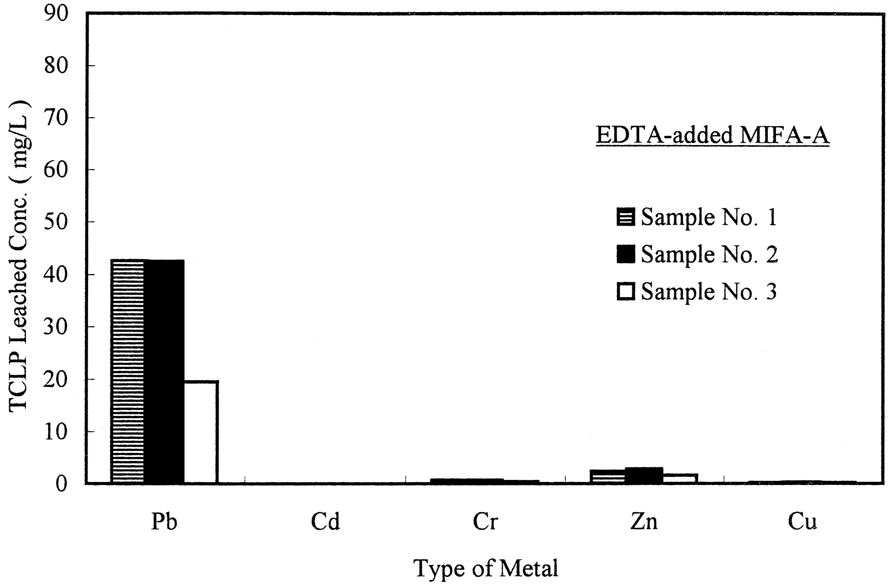

Fig. 1. Heavy-metal concentrations of TCLP leachates of EDTA-added MIFA-A specimens at an age of 28 days.

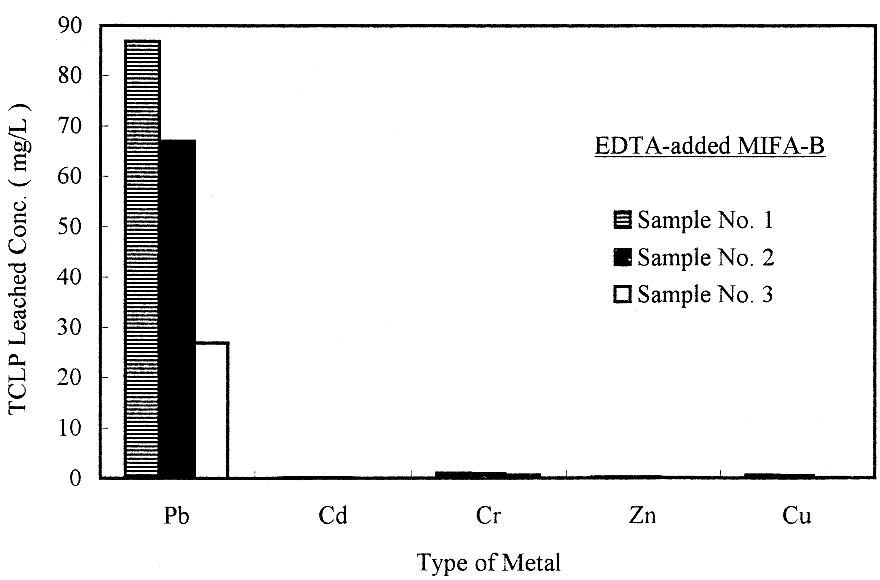

Fig. 2. Heavy-metal concentrations of TCLP leachates of EDTA-added MIFA-B specimens at an age of 28 days.

an addition of EDTA with a quantity greater than 3.72 g (see Table 11) has not been tested further to verify this trend in this study. From Table 3 and Figs. 1 and 2, it is not able to conclude that EDTA addition has a greater effect on reducing the leached Pb concentration for MIFA-A or MIFA-B.

4. Conclusions

In this investigation, the effects of EDTA (a chelant) addition on properties of cement-solidified monoliths of two municipal incinerator fly ashes, both containing a great quantity of lime, were evaluated. The MIFA specimens were found to be hazardous due to their high leached concentrations of lead. The properties of solidified monoliths studied included unconfined compressive strength and TCLP leaching toxicity. Based on the experimental results obtained, the following conclusions can be drawn: (1) In the case of EDTA addition to cement for solidifying MIFA, results of UCS measurements and their corresponding statistical analysis have shown that the weight of EDTA is not a significant parameter in solidification. OPC alone is capable of immobilizing heavy metals sorbed on MIFA. (2) An addition of EDTA alone to MIFA without OPC resulted in a limited contribution in reducing the leaching toxicity of heavy metals. Perhaps a much greater quantity of EDTA is needed for the leaching toxicity of MIFA to meet the regulatory requirement.

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[26] R.O.C. National Bureau of Standards, Method of Test for Compressive Strength of Cylindrical Concrete Specimens, CNS 1232 A3045, 1982 (in Chinese).