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Long-term leaching test of incinerator bottom ash: Evaluation of Cu partition

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Abstract

Two types of leaching tests were performed on the bottom ash from municipal solid waste incinerators. A short-term batch test specified by the America Nuclear Society (ANS) and long-term column tests with acetic acid (pH 5.2) as leaching solution were used to evaluate copper leachability. The Cu leaching after the 5-d ANS test is about 1% of the original Cu content of 5300 mg/kg. Upon addition of a stabilizing agent, the Cu leaching quantity is reduced; the extent of reduction depends on the type of chemical used (phosphate, carbonate and sulfide). The 1.6% Na_2S addition showed negligible Cu leaching, and Na_2S was, therefore, used in subsequent column tests. The 30-d column test indicates a steady increase of Cu leaching amount with time and reaches about 1.5% of the original Cu content after 30 d. A 180-d column test further increased the Cu leaching to about 5.1% of the original Cu content, whereas no appreciable Cu leaching was found with the addition of 1.6% Na_2S. A sequential extraction was conducted on the raw ash, ash with the addition of Na_2S and the residue ash after 30 d of operation to characterize Cu affinity for different solid fractions. The data were used to evaluate the fate of Cu through these interactions.

1. Introduction

Large quantities of bottom ash generated and subsequent disposal of bottom ash from the incineration of municipal solid wastes (MSW) have presented a significant problem for municipalities. For example, the annual bottom ash production amounts to approximately 1.33 million tons for a total of 20 MSW incinerators in Taiwan (TEPA, 2004). A common practical management method for bottom ash is landfill disposal. Although bottom ash may meet the toxicity requirements (e.g., test for toxicity characteristic leaching procedures) regarding heavy metals, and hence, it may not be classified as hazardous waste, the eventual leaching of metals in a landfill should be a major concern in terms of affecting bioactivity in the landfill and accumulating high metal concentrations in leachate. Even with the reuse of bottom ash, the potential metal leaching always hinders its application.

There have been many studies related to metal leaching from bottom ash under different conditions with batch and column studies (e.g., Baba and Kaya, 2004; Polettini and Pomi, 2004). As well as a few investigated leaching mechanisms related to metal status, i.e., equilibrium between dissolved and immobilized metal state (Johnson et al., 1996) and the effect of the presence of dissolved organics on metal leaching (Meima et al., 1999). In general, many factors affect metal leaching, including type and quantity of leaching solution, characteristics of bottom ash, extent of metal immobilized in the bottom ashes, type of immobilized metals, and presence of organics, as well as leaching conditions (pH, oxidation reduction potential, and temperature).

To prevent metal leaching, the addition of stabilizing agents, such as sulfide, phosphate and numerous commercial proprietary products, has been demonstrated to be effective. The insoluble metal compounds formed upon addition of these stabilizing agents are generally controlling

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minerals for limiting metal release. In a study conducted by van Gerven et al. (2005), MSWI-bottom ash was placed in a CO_2 chamber (CO_2: 10% and temperature: 50 °C) and Cu leaching was reduced from 3.3 to 1.0 mg/kg. Iretskaya et al. (1999) and Reich et al. (2002) indicated that combined chemical and thermal treatments efficiently stabilize heavy metals in MSWI ash. However, long-term evaluation of such stabilized matrices, particularly under adverse conditions, has seldom been performed. Also, data on metal partition onto mineral fractions in the bottom ash (i.e., exchangeable, bound to carbonate, among others) are lacking. The use of sequential metal extraction has been applied to soils (e.g., Tao et al., 2003) for estimating bioavailability of metals to plants and sediments (e.g., Pardo et al., 1990) for potential release into water columns. Recently, Bruder-Hubscher et al. (2002) use sequential extraction to study the release of metals from bottom ash, and reported that Pb and Zn can be released under acidic conditions and Cr leached under strong acidic conditions. Unfortunately, changes in metal partition in bottom ash as a result of adding stabilizing agent and after prolong leaching have not been reported.

Consequently, the objectives of this study were to: (1) evaluate long term metal leaching with or without the addition of stabilized agents; and (2) identify the metal partition in bottom ash before and after the addition of a stabilizing agent. The tasks for meeting the first objective included the use of batch and column leaching tests with acetic acid (pH 5.2) as the leaching solution. This may simulate the worst conditions in landfill leaching with both low pH and high amount of organics. The sequential extraction procedures proposed by Tessier et al. (1979) were used for classifying metal partition into different solid fractions present in bottom ash. The macrostructure change in metal fractions upon addition of stabilizing agent should provide a direct evidence for its capability in retarding metal leaching. The metal fraction change after bottom ash has been leaching for a period of time can also shed light as to which metals are being released. In particular, Cu is our focus of metal interest, since Cu is usually much more bioavailable in soil and may exert toxicity to plants (Pshlsson, 1989), has high leaching potential (Meima et al., 1999), and is abundant in bottom ash in Taiwan (Chao, 2003).

2. Materials and methods

2.1. Materials

Bottom ash was obtained from two MSW incinerators; designated as Sample A and B. The ash samples (25 kg) were obtained at the bottom ash recycling center of each incinerator plant. The ferrous metal and non-ferrous metal are removed immediately on site, either magnetically or manually. Thereafter the bottom ash samples were air-dried for 1 week before the experiments. Bottom ash greater than 10 mm was removed (ca. 90% less than 10 mm) (is to meet the code of controlled low strength materials and road base materials). The dried samples were subject to analysis of pH, moisture content, loss of ignition, and metal content.

X-ray diffraction (XRD) was used to characterize the crystal structure of each ash sample. Raw samples were combined with different stabilizing agents to see the extent of stabilization of bottom ashes. The stabilizing agents used included H_3PO_4, Na_2CO_3, K_2HPO4 and $Na_2S \cdot H_2O$; the solubility products for $Cu_3(PO_4)_2$, $CuCO_3$ and CuS are 1.4×10^{-37}, 2.5×10^{-10} and 8.0×10^{-37}, respectively (Stumm and Morgan, 1981).

2.2. Experiments

Two types of leaching tests were performed: quasi-dynamic and continuous column tests on both raw and stabilized ash samples. The quasi-dynamic test essentially follows those described elsewhere (ANS, 1986). Briefly, a 25 g ash sample was added into 500 mL HAc leaching solution (pH 5.2 with liquid to solid ratio of 20 mL/g). The reason for using HAc as a leaching solution is that HAc is used in the Toxicity Characteristics Leaching Procedures to determine whether the waste is hazardous waste or not. Further, within the landfill matrix, HAc is the major biological byproduct. Consequently, it is felt that the use of HAc may represent realistic conditions for metal leaching studies.

Periodically (2 h, 7 h, 24 h, 2 d, 3 d, 4 d and 5 d), the leaching medium was replaced with the fresh HAc solution, and the Cu content was determined on the filtered samples (0.6 μm) from the supernatant of the centrifuged leaching solution (16,000*g* for 15 min). When the results were unexpected, duplicate experiments were conducted.

Several columns (5 cm diameter with 24 cm height) filled with about 350 g ash sample were used for observing Cu leaching of various samples. The upflow velocity was controlled at 24–36 mL/h with a pump (Cole Parmer, 7533-71). (Under this flow rate, a 30-day column test is equivalent to approximately 4.4 years of natural rainfall leaching with an average rainfall of 2500 mm/yr and both the results of modified ANS tests and column tests are to be correlated.) The same HAc solution (pH 5.2) was used as the leaching solution.

The samples with the addition of stabilized agents were initially tested for their stability in the modified ANS 16.1 method (ANS, 1986). The stabilized agents were first added into samples according to the preset weight ratio, mixed for 30 min and then subjected to the above ANS test. Once the best agent (Na_2S) was identified, more detailed studies using Na_2S were performed in both ANS and column tests.

2.3. Analysis

The pH was determined in the supernatant of 20 mL 0.01 M $CaCl_2$ solution containing a 10-g sample. Measurements of water content, ignition loss and metal content of samples were performed based on the methods outlined in the Taiwan's Standard Methods (National Institute of Experimental Laboratory, 2004). The Inductively Coupled Plasma (Perkin–Elmer SCIEX ELAN 5000) was used for determining metal content in ash samples, first digested with the mixture of H_3PO_4:HCl:HF = 5:3:1 prior to analysis. The XRD (X-pert PRO, Philips) was used for identifying potential chemical structure based on the JCPDS file data. Intensities over 2θ (20–80°) at a rate of 3°/min were monitored.

Metal concentrations from the leaching tests were determined using atomic absorption spectrometer (Perkin–Elemer 800-2-9). Sequential extraction was performed on three samples (raw bottom ash, ash with the addition of stabilizing agent and column residues) according to the procedures used by Tessier et al. (1979). The procedures and the corresponding metal fractions are illustrated in Table 1.

3. Results and discussion

3.1. Ash characterization

The mineral contents for bottom ashes are shown in Table 2 with high amounts of Ca, Si and Fe, along with other heavy metals. There are noticeable differences in Ca, Cu and Mn concentrations between the two ash samples. The comparison of metal contents present in the bottom ash from the present study with others is not possible since they are dependent on the type of MSW, incinerator operating conditions, and the nature of sample heterogeneity in the bottom ash piles. Nonetheless, elemental composition from the present study is typically in the same range of MSW bottom ashes (e.g., Speiser et al., 2000), with perhaps relatively high Cu content in two samples tested, or 5000–7000 mg/kg; for comparison, the Cu content of 2230 mg/kg was reported by Crannell et al. (2000), 1450 mg/kg by Bruder-Hubscher et al. (2002) and 280–560 mg/kg by Speiser et al. (2000). Further quantitative analysis of Cu for those samples used in subsequent leaching tests indicated the total Cu contents for sample A and B were 5300 and 6600 mg/kg, respectively. These values were used as the initial data for subsequent extraction tests.

Table 2
Mineral content for bottom ash samples

Analyte	Sample A (%)	Sample B (%)
Al	0.56	0.54
B	0.02	<0.02
Ca	8.82	5.23
Cd	<0.01	<0.01
Cr	0.14	0.03
Cu	0.51	0.66
Fe	9.01	10.63
K	0.95	1.09
Mg	0.38	0.33
Mn	0.16	0.42
Na	1.67	1.13
Ni	0.05	0.03
Pb	0.10	0.19
Si	12.04	12.65
Ti	0.89	0.75
Zn	0.41	0.42

The results of XRD for both ash samples (Fig. 1) indicate the presence of $Ca_3Al_6Si_2O_{16}$, $Ca_8Si_5O_{18}$, $CaCO_3$, $CaSO_4$, CaO, CuO, Pb_3O_4 and SiO_2; minerals typically combined with different calcium-hydrate phases (Speiser et al., 2000). For two bottom ash samples, pH ranged from 9.1 to 9.3, moisture averaged 4.5–4.8% and ignition loss about 5.1–5.3%. The ignition loss is close to the standard 5% in operating incinerators in Taiwan. The high pH, as well as high alkalinity, in ash samples certainly is the major factor controlling eventual metal leaching.

3.2. Leaching tests

The short-term 5-d ANS test results are shown in Fig. 2. The Cu leaching without addition of any stabilizing agent from Sample A at 5 d was about 55 mg/kg (ca. 1% of the

Table 1
Sequential extraction procedures (Tessier et al., 1979)

Fraction	Extraction agent	Extraction conditions	
		Mixing time at 100 rpm	Temperature
Exchangeable	10 mL $MgCl_2$ (1 M, pH = 7.0)	1 h	25 °C
Carbonate	10 mL NaOAc (1 M, pH = 5.0)	5 h	25 °C
Fe–Mn oxides	20 mL 0.04 M $NH_2OH \cdot HCl$ in 25% (v/v) HOAc	6 h	963 °C
Organic matter/Sulfides	8 mL 30% H_2O_2 (pH = 2.0 with HNO_3)	5 h	853 °C
Residual	3 mL H_3PO_4 5 mL HCl 1 mL HF	1 h	Microwave-oven (220 °C)
Water soluble	L:S = 10 ml water:1 g sample[a]	1 h	25 °C

[a] Sample weight: 100 g.

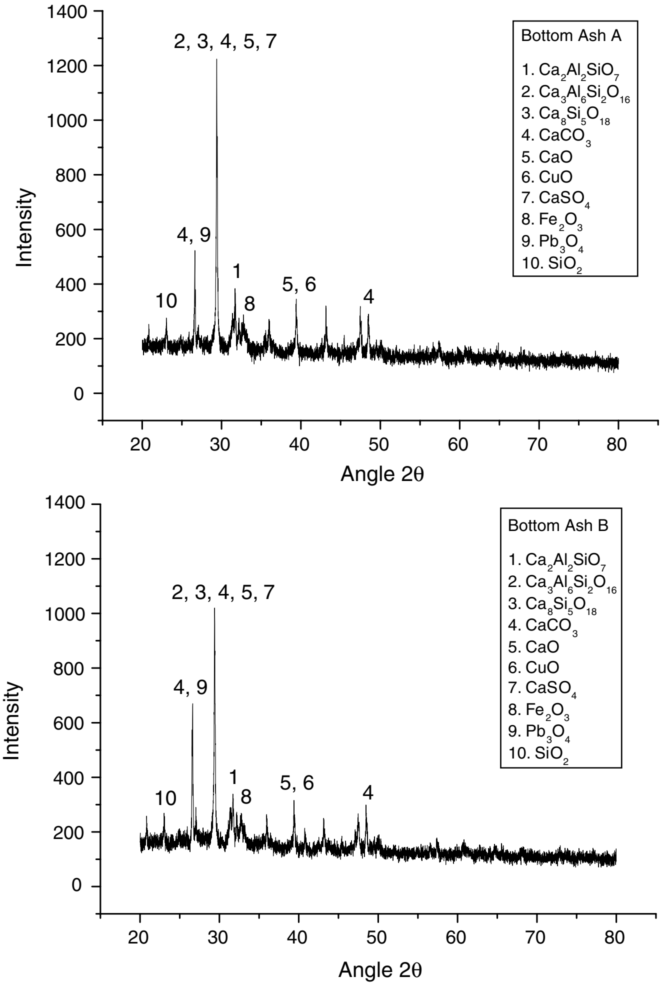

Fig. 1. XRD diagrams for two raw bottom ash samples.

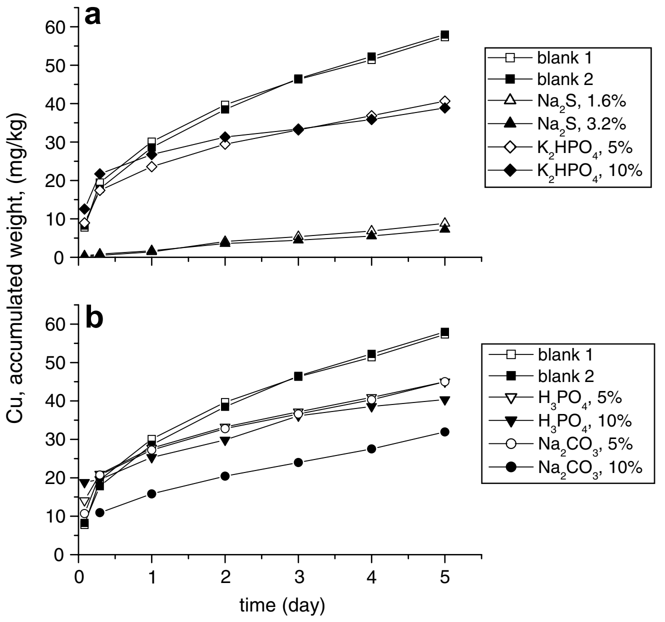

Fig. 2. Dynamic leaching tests with the addition of different stabilizing agents for bottom ash sample A.

original Cu content). The initial high pH of ash samples is responsible for a small amount of Cu release. The two duplicate runs show essentially the same results. The concentrations used in all stabilizing agents were relatively high, resulting in an insignificant difference between two dosages used for each agent, except for Na_2CO_3 in which 10% Na_2CO_3 showed a lower Cu leaching. For comparison, Meima et al. (2002) indicate 50% Cu leaching reduction with artificial carbonation with CO_2 to pH 8.3 and Crannell et al. (2000) reported 95% Cu leaching reduction with 3.6% soluble PO_4^{3-} addition. The Na_2S, among four stabilizing agents evaluated, demonstrated the lowest Cu leaching (only about 5 mg/kg), apparently due to the high stability of Cu–S precipitates. For comparison, 10% Na_2CO_3 yielded about 30 mg Cu/kg and 5–10% phosphate 35 mg/kg with 55 mg/kg without addition of any stabilizing agent. The surface oxidation products vary with pH and oxidizing conditions. Zhou et al. (1999) indicated that Cu–S bonding is stable during the oxidation treatment. In aqueous solution under alkaline conditions, Cu_2S might be oxidized to CuO, and that is the most stable oxide phase under all pH conditions (Todd and Sherman, 2003). Based on the results of this study and previous research efforts, oxidation might proceed; however, the process should be kinetically fairly slow in anoxic environments where the bottom ash is reused. In other words, even with oxidation process, Na_2S is an effective stabilizing agent for bottom ash. Consequently, Na_2S was used in the subsequent column studies.

The column test results indicate much higher cumulative Cu leaching amounts compared to Pb and Ni (Fig. 3), since Cu content in the ash sample is much higher than that of other metals. Because of the nature of the HAc leaching solution, pH values decreased with time, or dropped from the initial 8.5 to 7.2 after 30 d (Fig. 3). The Cu leached quantity after 30-d column test was only about 1.5% of the original Cu content present in the ash sample. The cumulated metal mass profiles (Fig. 3b) indicate relatively linear increases in metal leaching up to day 30. Thus a long-term metal leaching evaluation should be conducted to determine the further extent of metal leaching. As for sample B, the results are similar (data not shown) with about 1% the initial Cu content (6600 mg/kg) leaching out after the 30-d column test.

For long-term column studies, there is almost no appreciable Cu leaching for the sample with the addition of 1.6% Na_2S even after 180 d (Fig. 4). For comparison, the Cu release in the column containing sample A was about 270 mg or 5.1% of the Cu content (Fig. 4); the increased cumulative amount is about 10-fold from 1 mo to 6 mo (Figs. 3 and 4). It is noted that the amount of Cu leaching for sample B is less (about 190 mg after 6 mos; data not shown) despite the fact it contains a higher Cu content (0.66% as compared to 0.53% in sample A). Consequently, the Cu content *per se* does not necessarily determine the extent of metal leaching; other factors are also involved, including the nature of metal speciation or metal binding within the solid matrix.

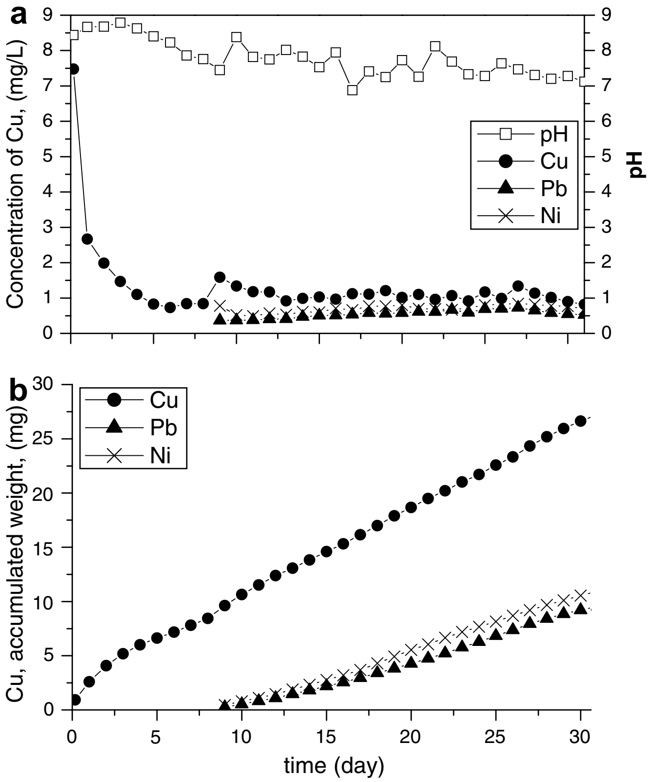

Fig. 3. Metal leaching from 30-d column tests (sample A).

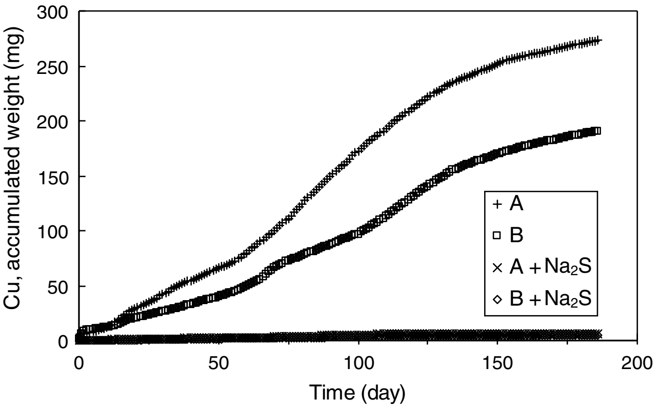

Fig. 4. Long-term 180-d cumulative Cu concentration from column test Na_2S at 1.6%.

Hirschmann and Forstner (2000) have concluded that calcite buffering capacity would last a long time between pH 7 and 8. The minimum Cu release at pHs between 8 and 9 of the bottom ash upon addition of sorbing compounds was also reported (Comans et al., 2000). Consequently, unless pH drops to a lower level, the metal leaching in general and Cu in particular would not exhibit a significant amount.

3.3. Sequential metal extraction

The sequential extraction results of Cu partition are shown in Table 3. The total Cu content in different fractions varied from 85 to 102% of the Cu content determined from the mixed acid digestion method. For the raw bottom ash, the Cu bound to oxides constitutes a major fraction (43%), with the next higher fraction being the residual (34%) and approximately the same amount bound to carbonate and associated with organics/sulfide (ca. 10% each). Since bottom ash is a product of the combustion process, the presence of high metal-oxides/hydroxides is expected, and Cu should be easily adsorbed onto Fe/Al/Mn (hydr)oxides and/or existence as CuO and $Cu(OH)_2$ (Meima et al., 1999). Since extensive complexation with dissolved organics has been reported for Cu (>95%) in bottom ash leachate (Meima et al., 1999) and the fact of the ignition loss is still about 5%, a 10% of Cu bound to organic is not unreasonable. For comparison, the Cu fractions for exchangeable, carbonate, oxides, organic and residue for soil are approximately 0.5%, 5%, 16%, 47% and 31%, respectively (Tao et al., 2003); for sediments, more than 96% of Cu are bound to organic/sulfide fraction (Pardo et al., 1990); and for bottom ash 64% of Cu bound to organic and sulfide fraction (Bruder-Hubscher et al., 2002).

After the addition of 1.6% Na_2S stabilizing agent, the Cu partition distribution is completely changed. Clearly, for whatever mechanisms, Cu previously associated with carbonate, Fe/Mn and organics now is converted into some Cu–S compounds resulting in a significant increase in the fraction bound to sulfide (21%, Table 3). Further, the residual amount also increases significantly and these two fractions (sulfide and residual) amount to 85% of the total extractable Cu; for comparison, it is only 44% with the raw sample without the addition of Na_2S. High proportions of exchangeable and carbonate fractions enable metals to be easily removed from the bottom ash. In contrast, high percentages of Fe–Mn oxides, organic-matter and residual fractions tended to fix the metals in the bottom ash, which was therefore stable, and metal might be released from those organic matter and subsequently binding with sulfide to form thermodynamically more stable species (CuS). One can be sure that some fractions of organic matter remain in the bottom ash and both dissolved and particulate organic matters may play some roles in binding metals. The high fraction of residual, as well as the increased Cu–S content, is certainly responsible for the insignificant Cu leaching even after the 180-d column with the HAc leaching solution as described before.

The ash residual present in the column after 1 mo of operation was removed and further subject to sequential extraction to observe the extent of changes in Cu partition. Many observations can be made of the Table 3 data. First, the Cu content associated with organics increased, from 420 to 610 mg/kg (50% more), apparently due to the addition of HAc – either Cu directly reacts with HAc or with other organics due to the addition of HAc. If the inorganic acid were used as the leaching solution, it would be expected that Cu bound to organics should be significantly reduced. Secondly, the amount of Cu bound to carbonate

Table 3
Results of sequential Cu extraction for sample A in Cu content (mg/kg) and percentage (%)

	Raw bottom ash		Ash with 1.6% Na_2S		Residual ash after 1 mo of operation	
	Content	Percentage	Content	Percentage	Content	Percentage
Exchangable	45	1	30	<1	5	<1
Bound to carbonate	460	11	100	2	300	9
Bound to Fe–Mn oxides	1790	43	545	12	870	27
Bound to organics/sulfide	420	10	980	21	610	19
Residual	1400	34	2975	64	1490	45
Total	4115	100	4630	100	3275	100
Acid digested	4040		4731		3855	
Recovery (%)	102		98		85	

and Fe/Mn oxides was also reduced. Specifically, the 920 mg/kg Cu reduction bound to Fe/Mn (from 1790 to 870 mg/kg) is almost 5–6-fold (160 mg/kg) that of Cu bound to carbonate (from 460 to 300 mg/kg). This implies that Cu bound to Fe/Mn oxides is more easily leaching out than that bound to carbonate. Thirdly, the residual fraction increased to 45% as compared to 34% in the original sample. In short, the sequential extraction results do provide useful information in explaining the inability of Cu leaching in the Na_2S system, and elucidate the Cu affinity for different structures after 1 mo of leaching.

Additionally, a simple water rinse (mixing for 1 h of 1 g ash sample with 10 mL water) yielded Cu leachate amount of 1.1% of total Cu content, although the L/S (liquid to solid ratio) of HAc leaching in dynamic tests (20 ml/g ash) exceeded that in the water rinse tests (10 ml/g ash). The amount of Cu leaching in fact is higher than that either from dynamic or column tests within the same time frame (1 h). More surprisingly, a significant fraction of Cu (0.32%) is also leached out with the 1.6% Na_2S stabilized sample. The effectiveness of water wash for removing metals has also been reported for MSW residues of bottom and fly ash (Kim et al., 2003) and arsenic form copper smelting solid wastes (Shih and Lin, 2003). If a simple water wash is effective, it indicates that the water dissolved organic fraction may be present in a relatively large fraction. In fact, Dugenest et al. (1999) have reported that the labile organic carbon fraction in bottom ash is relatively high (about 1500 mg/kg), which includes mainly carboxylic acids, *n*-alkanes and phthalates. This proposition apparently contradicts the Cu-fluvic complexes reported by others, since fulvic acid type components and not aliphatic and aromatic acids are responsible for Cu binding and eventually enhanced Cu leaching (van Zomeren and Comans, 2004). Nevertheless, the Cu release due to water alone and, to some extent, resultant biological activity may explain the different metal leaching between fresh and weathered bottom ashes (Meima and Comans, 1999; Meima et al., 1999; Chimenos et al., 2000). Additionally, this study suggests that Cu (or Cu oxide) in the leachate may be adsorbed again onto the ash.

4. Conclusions

Since only a few studies of the long-term metal leaching and metal fraction into different solid matrices in bottom ash had been conducted, the present study was undertaken to investigate these phenomena. The bottom ash samples were taken from two different MSW incinerators, subjected to XRD and elemental analysis and eventually used for metal leaching tests with acetic acid (pH 5.2) as the leaching solution.

As would be expected, the samples contain minerals typically with different calcium-hydrate phases, including $Ca_3Al_6Si_2O_{16}$, $Ca_8Si_5O_{18}$, $CaCO_3$, $CaSO_4$, CaO, CuO, Pb_3O_4 and SiO_2. The high pH along with these oxides is responsible for a relatively small amount of leaching, e.g., only 1% for the 5-d ANS batch test and 1.5% after 30-d column operation. This implies that only a fairly small fraction of Cu could be released from the reused ash matrix into the environment in about 4.4 years. The addition of three different types of stabilizing agent (PO_4^{3-}, CO_3^{2-} and S^{2-}) in bottom ash reduced the amount of Cu leaching, with 1.6% Na_2S yielding an insignificant amount. The formation of Cu_2S eventually might be oxidized to CuO phase. However, the transformation of Cu_2S into CuO under an anoxic condition is kinetically insignificant, thus securing the environmental risk of bottom ash reuses. In other words, whether the oxidation progressed or not, Na_2S is an effective stabilizing agent for bottom ash.

There is a definite trend of the linear Cu leaching for the 30-d column test. Therefore, a long-term of 180-d column test was conducted to see the further extent of Cu leaching. At the end of 180 d, the Cu leaching amount is about 5.1% for Sample A and the cumulative profile indicated the potential further leaching. The impact of this observation is of significance since it may present a long-term problem if there is no pretreatment to fix metals. Nevertheless, with the addition of Na_2S, the Cu leaching can be completely inhibited.

The sequential extraction of raw ash, ash with the addition of 1.6% Na_2S and residual ash after the 30-d operation shows different Cu amount towards five fractions (exchangeable, carbonate, Fe/Mn, organics/sulfide and residue). The fraction associated with sulfide increased significantly for the ash sample with addition of Na_2S, confirming the Cu fixation and thereafter the inhibition of Cu leaching was primary due to the association between Cu species and Na_2S.

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