



# Influence of flue gas SO<sub>2</sub> on the toxicity of heavy metals in municipal solid waste incinerator fly ash after accelerated carbonation stabilization

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## ABSTRACT

The influence of CO<sub>2</sub> content and SO<sub>2</sub> presence on the leaching toxicity of heavy metals in municipal solid waste incinerator (MSWI) fly ash was studied by examining the carbonation reaction of MSWI fly ash with different combinations of simulated incineration flue gases. Compared with raw ash, the leaching solution pH of carbonated ash decreased by almost 1 unit and the leaching concentrations of heavy metals were generally lower, with that of Pb decreasing from 19.45 mg/L (raw ash) to 4.08 mg/L (1# carbonated ash). The presence of SO<sub>2</sub> in the incineration flue gas increased the leaching concentrations of heavy metals from the fly ash to different extents after the carbonation stabilization reaction. The pH of the leaching solution was the main factor influencing the leaching concentrations of heavy metals. The increase in buffer capacity with the pH of carbonated ash caused an increase in heavy metal stability after the carbonation reaction. Accelerated carbonation stabilization of MSWI fly ash could reduce its long-term leaching concentrations (toxicity) of Cu, Pb, Se, and Zn. The leaching concentrations of heavy metals from carbonated ash also likely had better long-term stability than those from raw ash. The presence of SO<sub>2</sub> in the incineration flue gas increased the proportion of exchangeable state species of heavy metals; slightly increased the long-term leaching toxicity of Cu, Pb, Se, and Zn; and reduced the long-term stability of these metals in the fly ash after the carbonation reaction.

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## 1. Introduction

Incineration technologies are widely used to treat municipal solid waste (MSW) [1], but solid residues (i.e., bottom ash and fly ash) and atmospheric emissions are generated during municipal solid waste incineration (MSWI). The establishment of emission standards has led to improvements in air pollution control (APC) systems for flue gas treatment, primarily through more efficient separation technologies. However, these developments have increased both the amount and pollution potential of APC residues. Because of its alkalinity and the presence of leachable heavy metals, MSWI fly ash is on the list of national hazardous wastes in China [2].

Over the past three decades, major advances have been made in stabilization technologies for MSWI fly ash [3–6]. However, some of the shortcomings of conventional stabilization technologies cannot be ignored, such as concerns over long-term stabilization and the high cost of stabilization chemicals. Consequently, carbonation has

become an important topic of research regarding the long-term leaching of heavy metals.

Weathering stabilizes materials by changing their mineralogical characteristics and has some positive effects, such as reducing heavy metal leaching [7–9]. Carbonation, which has been identified as an important step in the weathering process [10], involves the dissolution of CO<sub>2</sub> in water under initially alkaline conditions. This causes pH to decrease and calcite to precipitate until the material is in equilibrium with the CO<sub>2</sub>. Moreover, heavy metals can be trapped in the newly formed minerals, with calcite being the predominant newly formed mineral during fly ash maturation [9].

Under natural weathering conditions, the atmosphere is the primary source of CO<sub>2</sub>. However, studies have found that the natural carbonation process can be accelerated using different sources of CO<sub>2</sub> and various methods [10–12]. Moreover, accelerated carbonation may represent a proper pretreatment stage prior to either recycling or landfilling fly ash [1,13,14]. The CO<sub>2</sub> source can be either pure CO<sub>2</sub> or any other CO<sub>2</sub>-rich gas, such as incinerator emissions.

Unlike other fly ash such as coal ash, which has a looser utilizing limit [15–17], MSWI fly ash must be stringently stabilized before final disposal (landfill or reuse) as a type of hazardous waste. The use of accelerated carbonation with fly ash may not only improve stabilization of fly ash but also reduce greenhouse gas emissions,

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**Table 1**  
Results of fly ash XRF analysis (mean  $\pm$  standard deviation,  $n = 3$ ).

Element	w (%)	Element	w (%)	Element	w (%)
Cl	27.310 $\pm$ 0.274	Mg	0.812 $\pm$ 0.001	Cu	0.170 $\pm$ 0.0004
Ca	25.840 $\pm$ 0.303	Al	0.694 $\pm$ 0.021	Sn	0.113 $\pm$ 0.001
O	24.511 $\pm$ 0.011	Zn	0.598 $\pm$ 0.008	Cr	0.065 $\pm$ 0.005
Na	7.457 $\pm$ 0.221	Br	0.513 $\pm$ 0.012	Mn	0.043 $\pm$ 0.0005
K	5.386 $\pm$ 0.175	P	0.286 $\pm$ 0.0003	Ba	0.043 $\pm$ 0.005
S	3.070 $\pm$ 0.014	Ti	0.263 $\pm$ 0.005	Sr	0.025 $\pm$ 0.0002
Si	1.785 $\pm$ 0.058	Pb	0.172 $\pm$ 0.008	Ni	0.011 $\pm$ 0.0001
Fe	0.825 $\pm$ 0.004				

**Table 2**  
Components of simulated reaction gases.

Reaction gas	N <sub>2</sub> (%)	O <sub>2</sub> (%)	CO <sub>2</sub> (%)	SO <sub>2</sub> (ppm)
Gas #1	–	–	100	–
Gas #2	78	10	12	–
Gas #3	78	10	12	1000

thereby achieving the goal of 'waste control by waste.' However, few studies of accelerated carbonation of MSWI fly ash using incineration flue gas have been conducted to date. Therefore, in this paper, we examined the influence of two typical components of incineration flue gas, CO<sub>2</sub> and SO<sub>2</sub>, on the leaching toxicity of heavy metals after an accelerated carbonation reaction between MSWI fly ash and simulated incineration flue gas. This study helps create a theoretical foundation for the applied use of accelerated carbonation stabilization of MSWI fly ash.

## 2. Materials and methods

### 2.1. MSWI fly ash

The MSWI fly ash used in this experiment was sampled from a specific municipal solid waste incineration power plant in Shenzhen, China. The flue gas purification treatment in this plant is a three-class disposal system consisting of a lime half-dried absorption reaction tower, active carbon insufflation absorption, and a cloth-bag dedusting system. The experimental fly ash was kept in the dark in air-emptied, sealed containers until use.

Water content of the fresh experimental fly ash was 0.92  $\pm$  0.14% (mean  $\pm$  standard deviation,  $n = 6$ ), which was measured using the drying and weight-loss method. The fly ash was grinded into smaller particles that could pass through a 200-mesh screen. The sample was then analyzed using X-ray fluorescence (XRF); the elements and their concentrations in the fly ash are shown in Table 1.

The concentrations of Cl (27.310%) and Ca (25.840%) were relatively high because the lime half-dried absorption reaction tower in this plant ejects Ca(OH)<sub>2</sub> to purify the flue gas. In addition, the decomposition of CaCO<sub>3</sub> present in the MSW (from items such as egg shells, stones, and some building wastes) makes the fly ash alkaline and contributes to the high Ca content. Chlorine, which was present in the highest concentrations, probably came from plastics and kitchen waste in the MSW, as methods of municipal solid waste sorting have not been adopted in China. This explanation is supported by the relatively high Na and K content in the MSW sample. The large amounts of Cl in the fly ash made heavy metals exist in unstable forms [18].

### 2.2. Experimental procedure

On the basis of an analysis of actual incineration flue gas from the MSWI power plant, we prepared simulated reaction gases for this experiment (Table 2). To investigate the influence of SO<sub>2</sub> on the accelerated carbonation reaction, we increased the SO<sub>2</sub> content in gas #3 above the level in the actual incineration flue gas.

Using a continuous reaction laboratory simulation system [19], the simulated reaction gas continuously flowed from the bottom of the reactor to react with the fly ash sample, and the toxicity of heavy metals in raw and carbonated fly ash was then determined. With this laboratory simulation system, the accumulated volume (accurate to 0.01 L), instantaneous flow (accurate to 0.01 L/min), temperature, and pressure were monitored online and recorded automatically.

### 2.3. Analyses

Leaching characteristics of heavy metals in raw and carbonated fly ash were investigated according to the toxicity characteristic leaching procedure (TCLP) of the United States Environmental Protection Agency (USEPA). Heavy metal contents in the leaching solution were determined by inductively coupled plasma-atomic emission spectrometry (ICP-AES).

The pH correlation-leaching characteristics of raw and carbonated fly ash were determined by a series of leaching experiments using replicate samples of ash. Five-gram samples of raw fly ash were weighed and mixed with deionized water at a liquid-to-solid ratio of 10L:1 kg, and then different concentrations of HNO<sub>3</sub> solution (0–1.5 mol/L) were added to individual mixtures. In this way, the pH of the leaching solution was kept at specific values from 3 to 13. During this process, the mixtures were continuously turned at 30  $\pm$  2 r/min and kept at 23  $\pm$  2 °C for 48 h. The mixtures were then filtered separately under vacuum through 0.65  $\mu$ m glass fiber filters and the pH and heavy metal contents of the filtrate were determined using a pH meter and ICP-AES, respectively.

The long-term stability of raw and carbonated fly ash was determined using the TCLP method. Every 100 g of ash was weighed into a 2.2 L HDPE flask, and 2 kg of extraction fluid #2 was added to each flask. The mixture in each flask was kept rotating and oscillating at 30  $\pm$  2 r/min and 22  $\pm$  3 °C. The leaching concentrations of heavy metals in each ash category were determined at 2, 7, 18, 24, 48, 72, 96, 120, and 168 h. A portion of the leaching solution was filtered through a 0.65  $\mu$ m glass fiber filter under vacuum, and the heavy metal contents in the filtrate were determined by ICP-AES.

Chemical species of heavy metals in the fly ash were determined using the modified Tessier sequential extraction procedure [20]. The extraction sequence was as follows: (1) exchangeable (NaOAc, pH 8.2), (2) bound to carbonate (NaOAc, pH 5.0), (3) bound to metal oxides (HONH<sub>3</sub>Cl), (4) bound to organic matter (HNO<sub>3</sub>, H<sub>2</sub>O<sub>2</sub>), and (5) residual (HNO<sub>3</sub>, HCl, H<sub>2</sub>O<sub>2</sub>, HF, 1:3:1:3). Heavy metal contents in extraction solutions were determined by ICP-AES.

## 3. Results and discussion

### 3.1. Leaching characteristics of heavy metals in raw and carbonated fly ash

Leaching characteristics of heavy metals in raw and carbonated fly ash were determined after the dynamic carbonation experiment

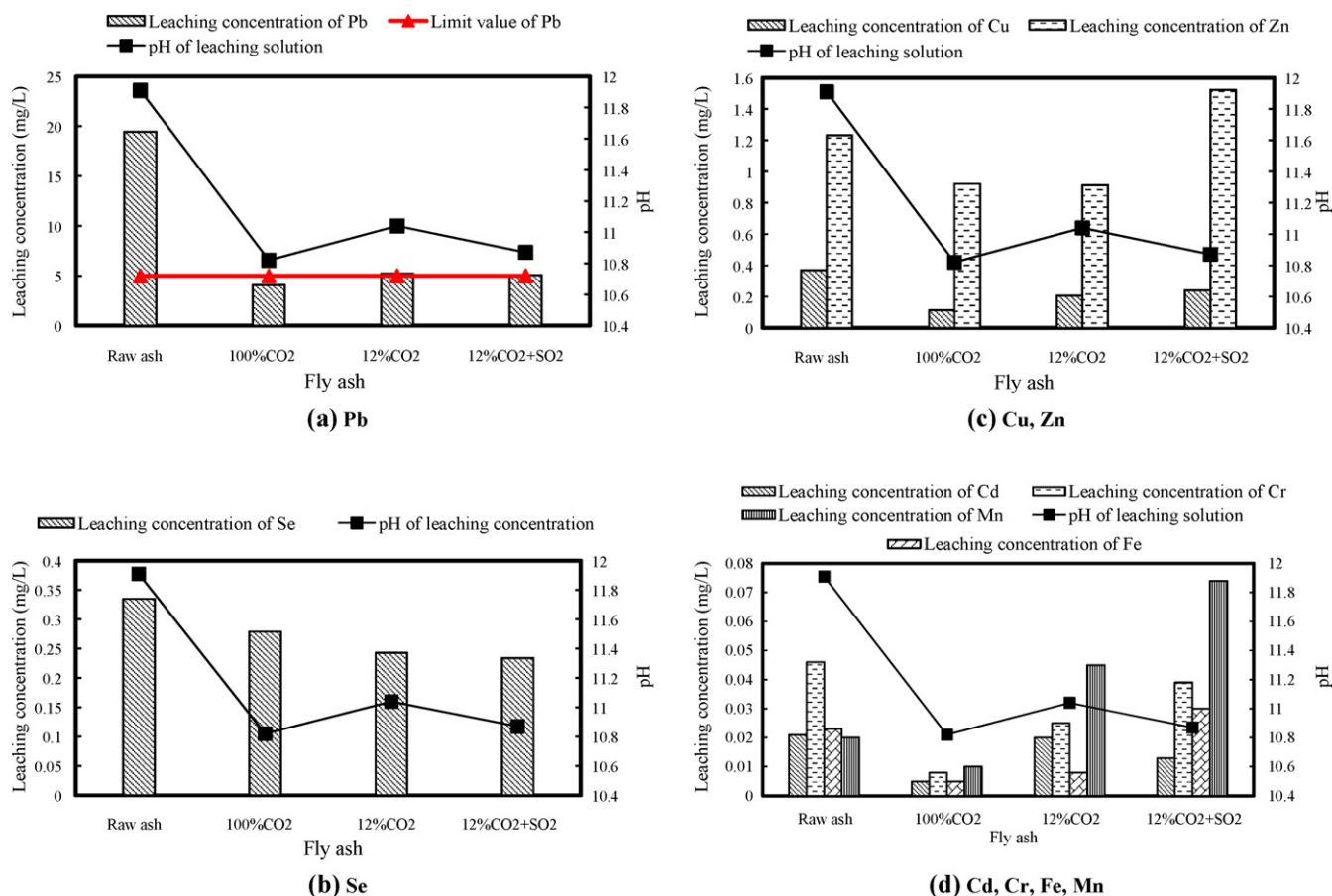


Fig. 1. TCLP leaching concentrations of heavy metals in raw and carbonated fly ash.

(Fig. 1). Only the limit value of Pb, the leaching concentration of which was above the standard in the raw ash, was marked.

The pH values of the leaching solution for raw fly ash and carbonated ashes with reactions with gas #1 (100% CO<sub>2</sub>), gas #2 (12% CO<sub>2</sub>), and gas #3 (12% CO<sub>2</sub> + 1000 ppm SO<sub>2</sub>) were 11.91 ± 0.18, 10.82 ± 0.2, 11.04 ± 0.09, and 10.87 ± 0.16, respectively. The pH of the leaching solution for carbonated ashes was almost 1 unit lower compared to the raw ash solution with the TCLP method. Leaching concentrations of heavy metals from carbonated ashes were generally lower than those from raw ash; the leaching concentrations of Pb, Se, Cu, Cd, and Cr from all of the respective carbonated ashes were lower than those from the raw ash. The leaching concentrations of all elements in Fig. 1 from the carbonated ash reacting with #1 gas (100% CO<sub>2</sub>) were clearly lower than those from the raw ash, and also lower than those from the #2 and #3 carbonated ash, except for Se. This was particularly true for Pb, whose leaching concentration was above the standard in raw ash. The leaching concentration of Pb from carbonated ash produced by a reaction with gas #1 showed a decrease from 19.45 mg/L (raw ash) to 4.08 mg/L, which was slightly below the standard of 5.0 mg/L (40 CFR §261.24). The leaching concentrations of Pb from carbonated ash from reactions with gases #2 and #3 were 5.23 and 5.07 mg/L, respectively, which were very close to the standard. Comparing #2 and #3 carbonated ash, it is noteworthy that the leaching concentrations of Cu, Zn, Cr, Fe, and Mn increased with the presence of SO<sub>2</sub> in the flue gas, and the leaching concentrations of Pb and Se were very close, while those of Zn, Fe, and Mn from #3 carbonated ash were higher than those from the raw ash. Thus, it can be inferred that the presence of SO<sub>2</sub> in the incineration flue gas increases the leaching concentrations of heavy metals from the fly ash to different extents after the carbonation stabilization reaction.

### 3.2. pH correlation-leaching characteristics of raw and carbonated fly ash

To study the degree of influence that the pH of the leaching solution has on the leaching concentrations of heavy metals, a pH correlation-leaching experiment was conducted using raw and carbonated ashes. Fig. 2 shows the results.

The pH correlation-leaching characteristic curves of carbonated fly ash were similar to that of raw ash, and the curve classifications for elements were also accordant between carbonated and raw ash. The Pb, Zn, Cu, and Se curves followed a U/V-shaped dependency on pH; namely, the leaching concentration of these metals was lowest under neutral or alkaline conditions but rapidly increased under acidic and strong alkaline conditions. In Fig. 2(c) and (f)–(h), the leaching concentrations of these metals could reach a relatively low value when the pH of the leaching solution ranged from 8 to 10. The Cr, Fe, and Mn curves followed an L-shaped dependency on pH; namely, the leaching concentration of these metals was high under acidic conditions but was sharply reduced when the pH of the leaching solution reached a certain value and then remained stable as the pH continued to increase. As shown in Fig. 2(b), the leaching concentration of Cr fell continuously until the pH of the leaching solution reached 4; then it maintained a low concentration as the pH increased to 13. Similarly, the leaching concentration of Fe fell to a relatively stable low value when the pH was 6.5 (Fig. 2(d)). The leaching concentration of Mn decreased slowly until the pH reached 10, and then remained at a low value as the pH increased (Fig. 2(e)) [21,22]. The type of leaching characteristic curve for Cd was not obvious; Cd in raw ash and #1 carbonated ash produced a U-shaped curve, while that in #2 and #3 carbonated ash showed a continually decreasing curve. Thus, it could be inferred that CO<sub>2</sub>

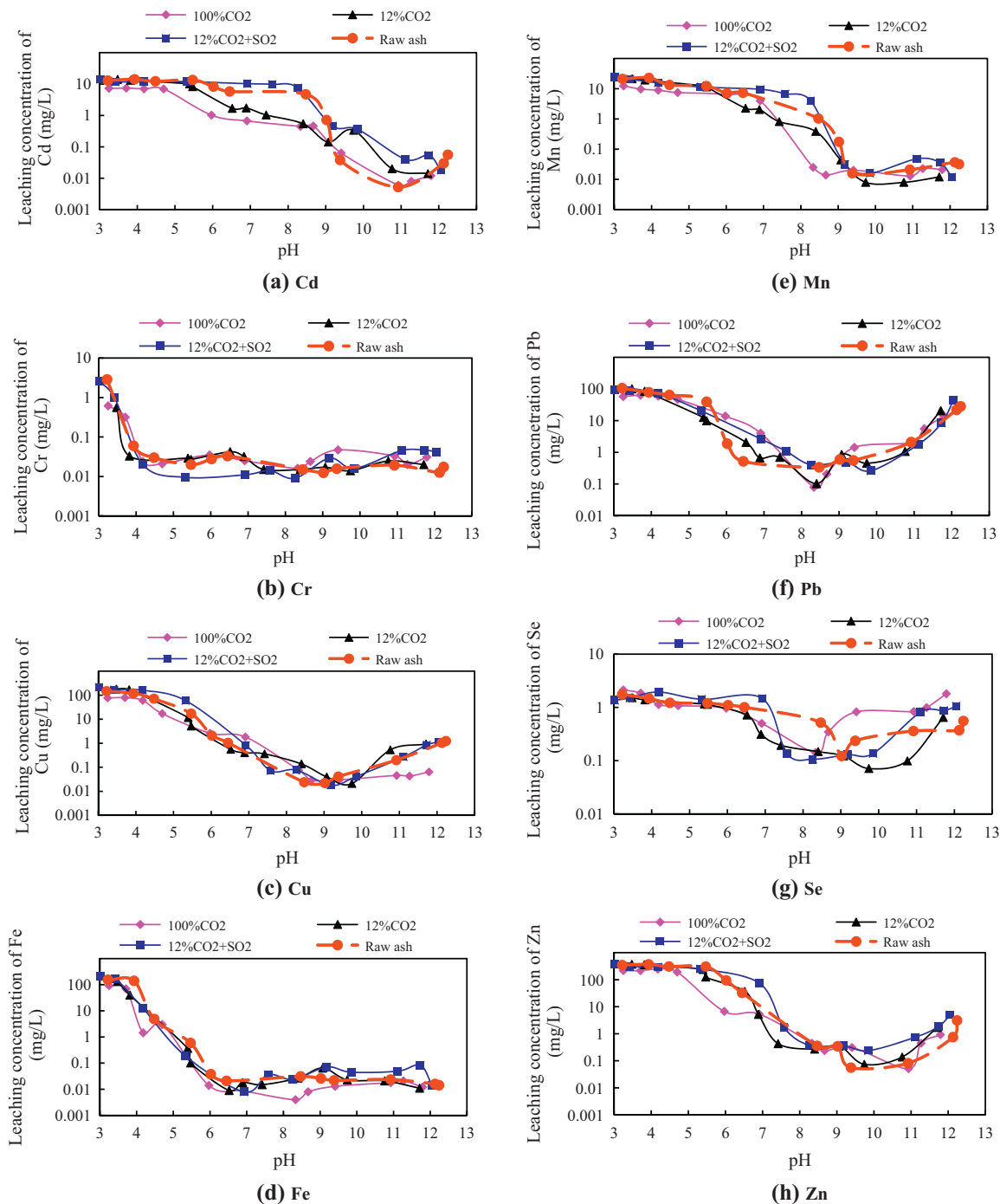


Fig. 2. Comparison of pH correlation-leaching characteristics of raw and carbonated fly ash.

content in flue gas during the carbonation stabilization reaction was an important factor influencing the leaching characteristics of Cd. The leaching concentrations for the same metal in the raw and carbonated ashes generally ranged up to three or four orders of magnitude with changes in pH, except for Se, whose leaching concentration ranged within only one order of magnitude. When the leaching solutions for raw and carbonated ash had similar pH, their leaching concentrations for each heavy metal were also relatively close, suggesting that the pH of the leaching solution was the key factor influencing the leaching concentrations of heavy metals [18,21]. The increase in buffer capacity with the pH change in carbonated ash caused an increase in heavy metal stability after the carbonation reaction. The leaching curves for Pb, Se, Cu, Zn, Cd, Cr,

Mn, and Fe as a function of pH from #3 carbonated ash are very useful for predicting the leaching behavior of heavy metals in the practical application of carbonation between fly ash and flue gas.

### 3.3. Long-term stability of heavy metals in raw and carbonated fly ash

An experiment examining the long-term stability of heavy metals in leaching toxicity was conducted using raw and carbonated fly ash (Fig. 3). According to the results in Section 3.1, the leaching concentrations of Cd, Cr, Fe, and Mn from raw ash were all below 0.05 mg/L, which is lower than the assured precision of the TCLP method. The large uncertainty in these results could probably lead



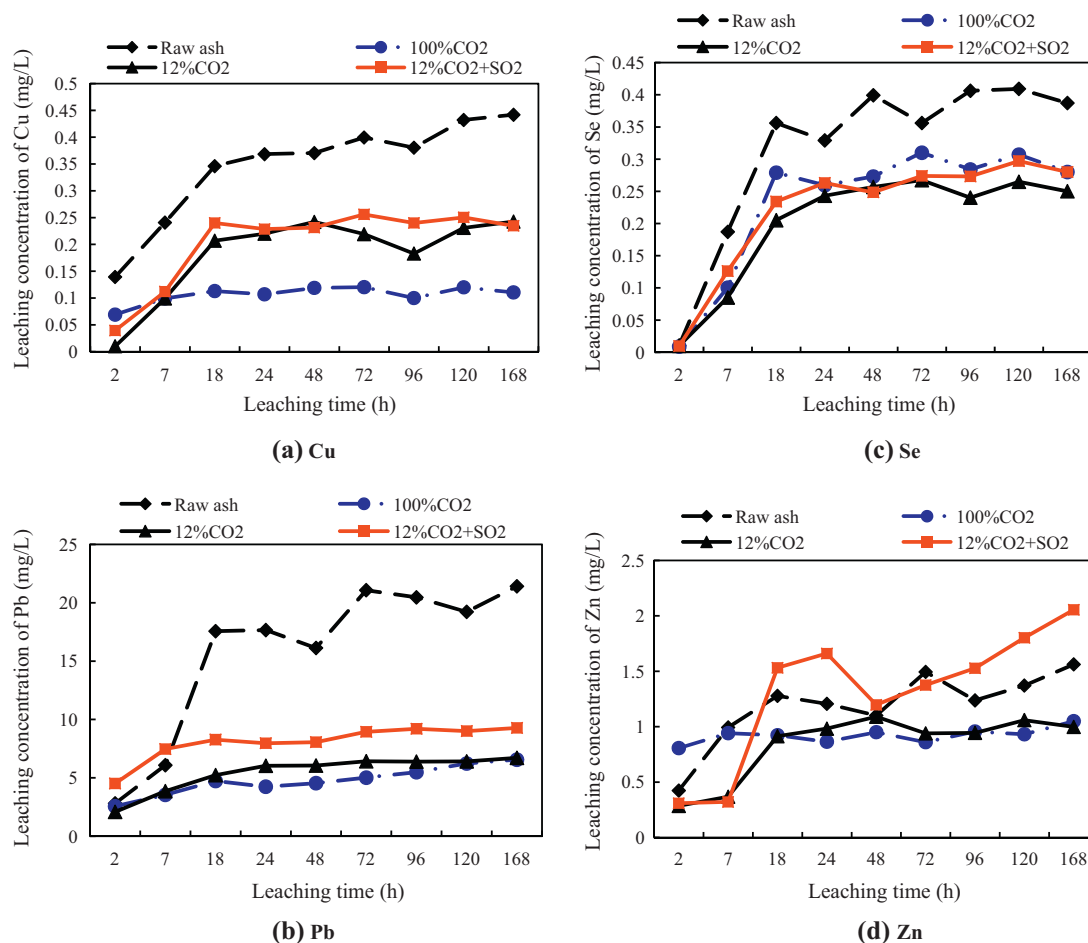


Fig. 3. Comparison of long-term leaching characteristics for raw and carbonated fly ash.

to large errors in subsequent analyses. Therefore, Cd, Cr, Fe, and Mn are not considered further in this section.

In Fig. 3, the long-term leaching concentrations of Cu, Pb, and Se from the three types of carbonated ash (#1, #2, and #3) were all lower than those from raw ash, and the long-term leaching concentrations of Zn from #1 and #2 carbonated ash were lower than those from raw ash, indicating that accelerated carbonation stabilization of MSWI fly ash could reduce its long-term leaching concentrations (toxicity) of Cu, Pb, Se, and Zn. The long-term leaching concentrations of Cu, Pb, Se, and Zn from gas #3 carbonated ash (12% CO<sub>2</sub> + SO<sub>2</sub>) were all higher than those from gas #2 ash (12% CO<sub>2</sub>); thus, the presence of SO<sub>2</sub> in the incineration flue gas resulted in a slight increase in the long-term leaching concentrations (toxicity) of Cu, Pb, Se, and Zn from the fly ash after the carbonation reaction. Based on the leaching concentrations of heavy metals at 168 h, the leaching concentrations at 24 h were converted to percentages in Table 3. Table 3 shows that the leaching concentration percentages of Cu, Pb, Se, and Zn from all three types of carbonated fly ash (#1, #2, and #3) were significantly higher than those from raw ash, except for the leaching concentration percentage of Pb from gas #1 carbonated ash. The leaching concentration percentages for heavy metals from gas #2 carbonated ash were almost all over 90%. Compared with raw ash, the leaching concentrations of heavy metals from carbonated ash showed better long-term stability. The leaching concentration percentages of Pb, Se, and Zn from gas #3 ash were lower than from gas #2 ash. Therefore, it can be inferred that the presence of SO<sub>2</sub> in the incineration flue gas reduced the long-term stability of heavy metals in the fly ash after the carbonation reaction.

#### 3.4. Distribution of chemical species of heavy metals in raw and carbonated fly ash

The modified Tessier sequential extraction procedure was used to investigate the chemical species of heavy metals in raw and carbonated fly ash (Fig. 4). According to order of activity from large to small, the chemical species of heavy metals in solid samples can be divided into five states: exchangeable, bound to carbonate, bound to Mn oxides, bound to organic matter, and residual. Metals in the first four states can gradually leach out in generally natural environments, whereas those in the residual state require a relatively long-term process to be released [23].

Fig. 4(a) shows the characteristics of the chemical species of different heavy metals in the raw fly ash; 83.5% of Cr, 81.2% of Fe, and 68.2% of Se existed in the residual state, suggesting that the extractive proportions of these metals were low. The corresponding values for Cu and Mn were 36.9% and 33.3%, respectively, while only 14.4% of Cd, 19.8% of Pb, and 17.5% of Zn existed in the residual state, suggesting that these three metals in the raw ash had a higher risk of being leached out than the other metals. Compared with raw ash, the proportions of residual state Cu, Fe, Mn, Pb, and Se in the carbonated ash were increased to different extents, which reduced the proportion of extractive state components, while the proportions of residual state Zn in the carbonated ash were reduced slightly. The proportions of the other metals, besides Cd, in the carbonated ash in the exchangeable state were significantly reduced, and the proportions of exchangeable state species of most heavy metals in gas #3 carbonated ash were relatively higher than those in gas #1 and gas #2 ash. Therefore, it can be inferred that the

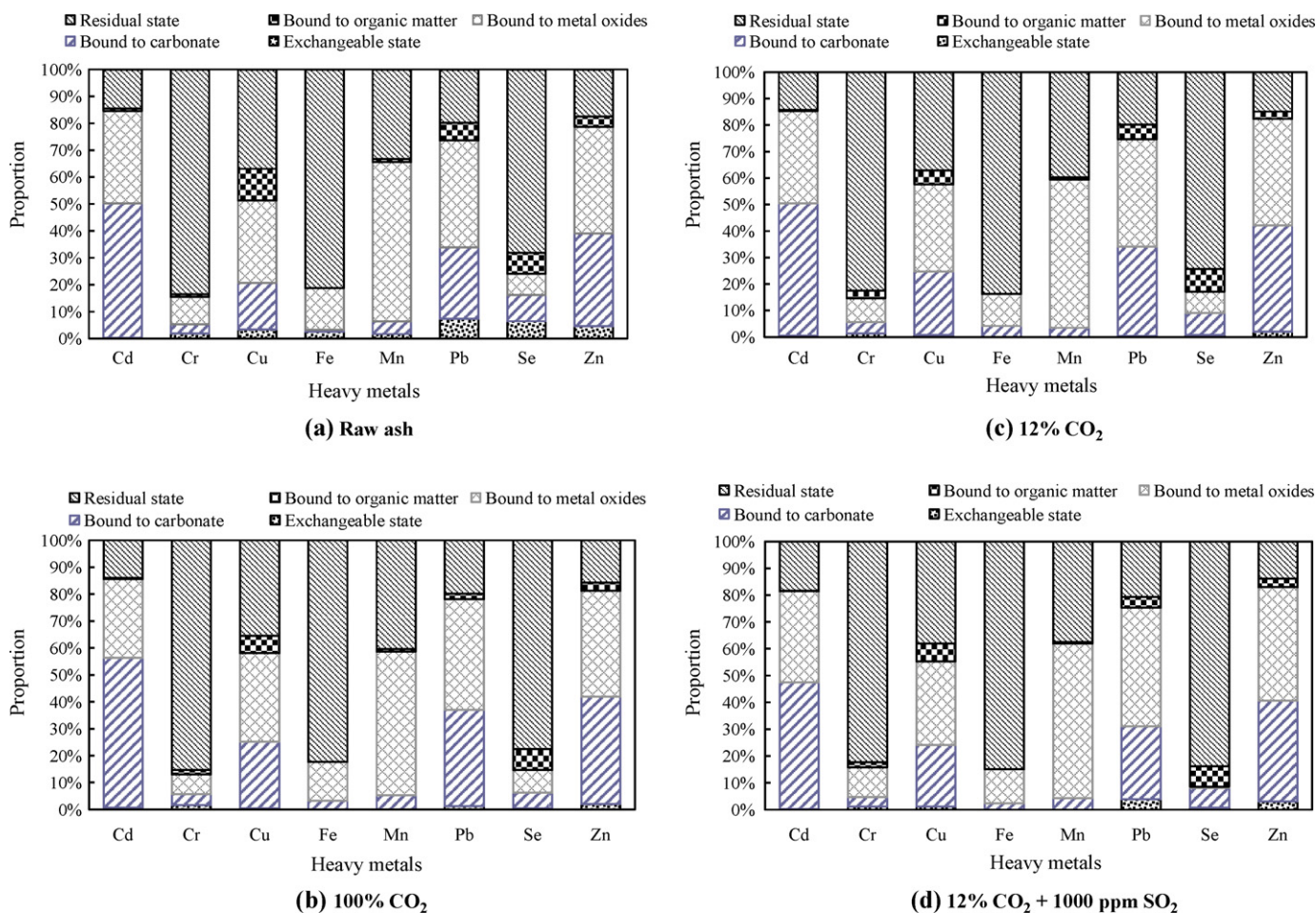


Fig. 4. Comparison of the chemical species of heavy metals in raw and carbonated fly ash.

Table 3  
Comparison of long-term leaching stability from raw and carbonated fly ash (%).

Ash sample	Cu	Pb	Se	Zn
Raw	83.34	82.48	85.01	77.21
#1 (100% CO <sub>2</sub> )	96.83	64.65	92.61	82.36
#2 (12% CO <sub>2</sub> )	90.65	89.91	97.20	98.40
#3 (12% CO <sub>2</sub> + SO <sub>2</sub> )	97.40	85.88	93.93	80.78

presence of SO<sub>2</sub> in the incineration flue gas increased the proportion of exchangeable state species of heavy metals in the fly ash after the carbonation reaction. The proportion of atoms bound in the carbonate state for almost all metals in the fly ash increased after the carbonation reaction and the metals bound in the carbonate state were quite sensitive to the range of pH.

#### 4. Conclusions

Compared with raw ash, the pH of the leaching solution was reduced for carbonated ashes (by almost 1 unit), and the leaching concentrations of heavy metals were generally lower, with Pb decreasing from 19.45 mg/L (raw ash) to 4.08 mg/L (1# carbonated ash). The presence of SO<sub>2</sub> in the incineration flue gas increased the leaching concentrations of heavy metals from the fly ash to different extents after the carbonation stabilization reaction. The pH correlation-leaching characteristic curves for carbonated fly ash were similar to those for raw ash. The leaching concentrations for the same metal (Cd, Cr, Cu, Fe, Mn, Pb, or Zn) in the raw and carbonated ashes generally ranged up to three or four orders of magnitude

with changes in pH, except for Se, whose leaching concentration ranged within only one order of magnitude. The pH of the leaching solution was the main factor influencing the leaching concentration of heavy metals, and the increase in buffer capacity with pH of carbonated ash caused an increase in heavy metal stability after the carbonation reaction. Accelerated carbonation stabilization of MSWI fly ash could reduce its long-term leaching concentrations (toxicity) of Cu, Pb, Se, and Zn, and the leaching concentrations of heavy metals from carbonated ash showed better long-term stability than those from raw ash. Comparing conditions when the CO<sub>2</sub> content was 12%, in the presence of SO<sub>2</sub>, the pH of the leaching solution for carbonated ash was closer to neutrality and the leaching concentration of Pb was lower, but the long-term leaching toxicities of Cu, Pb, Se, and Zn were slightly increased and their long-term stabilities were slightly reduced. Compared with raw ash, the proportions of residual state Cu, Fe, Mn, Pb, and Se were increased to different extents in the carbonated ash, the exchangeable state proportions of all other metals besides Cd were significantly reduced, and the proportions of metals bound in the carbonate state were increased. The presence of SO<sub>2</sub> in the incineration flue gas can increase the proportion of exchangeable state species for heavy metals in fly ash after the carbonation reaction.

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