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Controlling reactions during heavy metal leaching from municipal solid waste incineration fly ash

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Abstract: This research aimed to characterize the physical and chemical properties of municipal solid waste (MSW) incineration fly ash, as well as to investigate the leaching of heavy metals during toxicity characteristics leaching procedure (TCLP) process. To understand the leaching behaviour, concentrations of heavy metals, chloride, sulphate and calcium at various leaching time was monitored together with pH of the leaching solution. Results showed that the chemical compositions of fly ashes led to differences in leaching environment. For the two fly ashes under study, one resulted in a leaching environment with pH of 10–12 while the other had pH about 6–7. Based on pH, anions and cations concentrations, Minteq software was employed to investigate the speciation of heavy metals. Results showed that the shift in precipitation/dissolution balance of carbonate and hydroxides of heavy metals could explain the fluctuation in metal concentrations during the leaching process, which indicates that leaching was probably controlled by these reactions. On the other hand, addition of EDTA changed the controlling reactions. Chelating reactions between heavy metals and EDTA led to much higher leaching toxicity due to the presence of heavy metals, showing that the presence of chelating organics in natural environment may facilitate heavy metal leaching.

Keywords: characterization; toxicity characteristics leaching procedure; Minteq; precipitation/dissolution.

INTRODUCTION

Industrialization and urbanization in recent decades had led to alarming rises in the generation of municipal solid waste. It was reported that, in 2020, China produced 242 million tons of MSW which were mainly disposed of by landfill and incineration.¹ The disposal by landfill was intensive and the leaching of heavy metals into groundwater was problematic.² Incineration, on the other hand,

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was effective in reducing both volume (up to 90 %) and mass (up to 70 %) while generating electricity.^{3,4} Therefore, it was considered a promising method and had witnessed rapid developments.

However, air pollution and generation of residues are some of the concerns related to incineration. Solid residues from MSW incineration include bottom ash and fly ash.⁵ Between them, fly ash is considered hazardous as it contains large amount of heavy metals and toxic organics.⁶ Besides residues from MSW incineration, fly ash also contained reagents and products from flue gas neutralization. Therefore, the main elements in fly ash include O, Cl, S, Ca, Si, Al, Fe, Mg, Na and K, as well as toxic elements such as Pb, Cd, Cr, Ni, Zn, Cu, *etc.*⁷

MSW fly ash accounted for about 3–15 % of the MSW depending on the incineration technology.⁸ This means that, in China, millions of tons of fly ash need to be processed annually. Due to the continuous growth in fly ash generation, many countries have called for reuse and recycling. Fan *et al.* believed that MSW fly ash is basically an aluminosilicate material.⁹ Therefore, it could be reused as cementitious materials, soil amendment or light-weight aggregate in construction and road pavement materials.¹⁰

However, it is important to remove harmful elements before fly ash can be reused. Luo *et al.* classified the fly ash treatment into three categories, that is, chemical and physical separation, stabilization/solidification and thermal treatment.¹¹ The purposes of these treatments are to re-configure ashes (*e.g.*, vitrification or sintering) to remove pollutants (*e.g.*, washing), or to immobilize pollutants (*e.g.*, solidification, geo-polymerization).^{12–15}

Toxicity leaching tests were often conducted to compare the effects of heavy metal removal or immobilization. The most commonly used method was toxicity characteristics leaching procedure (TCLP) recommended by US EPA. Leaching of heavy metal was a complicated process. Many reactions, involving precipitation/dissolution, diffusion, sorption and surface coating, *etc.*, could occur during leaching.¹⁶ That is why physical and chemical factors could all influence the leaching of heavy metals. The former includes particle size, shape and porosity, temperature, time, *etc.*, while the later includes pH, redox, complexation or sorption conditions and leaching kinetics, *etc.*^{17,18} Therefore, the conditions of leaching tests have to be specified so that the results are comparable. Sample preparation, composition of leachant, mixing method, liquid to solid ratio, time, temperature and filtration method were all elucidated in the TCLP method. Still, researchers have argued that TCLP may not be able to truly reflect the leaching behaviour of ashes of high alkalinity. Lu *et al.* compared the leaching potentials of raw and cement-solidified bottom ashes *via* TCLP, multi-pH TCLP and EDTA-mediated TCLP and found that TCLP underestimated the leaching of heavy metal.¹⁹ They recommended that EDTA-mediated TCLP may be more

suitable for the evaluation the heavy metal leaching by alkaline materials, especially in the environment where organic acid may be present.

This research intended to study the properties of two fly ashes and investigate the heavy metal leaching characteristics during TCLP tests. pH, heavy metal, chloride, sulphate, carbonate and calcium concentrations in leaching solutions were monitored. Software Minteq 3.1 was used to calculate the metal speciation and solubility under the leaching conditions. At the same time, the leaching of heavy metal with addition of EDTA was also studied for comparison. Our ultimate goal was to study the feature of heavy metal leaching in fly ashes and advance the understanding of interaction between fly ash and leachant in order to pave the way for a better prediction of heavy metal impact on the environment.

EXPERIMENTAL

Fly ashes

Fly ashes employed in this research were from municipal solid waste incineration plants in Zhejiang and Guangdong Provinces of China. The samples were designated as FA1 and FA2, respectively.

Fly ash characterization

The moisture content was determined according to the national standard method of China (GB/T212-2008). The proximate analysis was conducted according to ASTM standard method E1131-08. The chemical compositions of the samples were determined by X-ray fluorescence spectroscopy (XRF-1800, Shimadzu, Japan) and the crystalline phases were characterized by X-ray diffraction (XRD, Rigaku Ultima IV, Japan) with a CuK α radiation at 40 kV and 30 mA, as 2θ ranging from 5 to 90°, while FTIR analysis was carried out *via* Nicolet iS10 (Thermo Scientific, Germany).

pH and the acid neutralization capacity (ANC) of fly ash were measured according to the methods proposed by Liu *et al.*²⁰ Specifically, 1 g of fly ash and 100 mL of deionized water were put into a 300 mL conical flask and stirred at 200 r/min for 15 min and pH was measured. Half of the mixture was next titrated with 1 mol/L acetic acid to pH 7.0. The amount of acetic acid consumed was the ANC.

Analysis of heavy metal contents

The contents of heavy metals in fly ash were measured based on a standard method of China (HJ803-2016) which was designed specifically for the analysis of metal in soil/sediments, *via* digestion with nitric acid/chloric acid. Heavy metals in the digested solution were determined by inductively coupled plasma optical emission spectrometry (ICP-OES, Optima8000, PerkinElmer, US) after pretreatment.

Heavy metal leaching toxicity tests

Fly ash was dried and sieved with a 200-mesh sieve before use. TCLP (USEPA method 1311) and an EDTA-modified TCLP were adopted to analyse the leaching toxicity potentials of fly ashes for comparison. With EDTA-modified TCLP, the concentration of EDTA in the leachant was set at 1 mmol/L. All other procedures were conducted as stipulated by the TCLP method. To study the leaching behaviour of heavy metal during TCLP tests, instead of the 18 h recommended by the standard method, leaching time was set at 2, 4, 6, 8, 10, 12, 14, 16 and 18 h, respectively. After mixing for the pre-determined amount of time, the mixture was fil-

tered and the filtrate analyzed for pH, chloride, sulfate, calcium, carbonate and heavy metal concentrations.

RESULTS AND DISCUSSION

Fly ash characterization

The proximate analysis was carried out on both fly ashes to understand their basic properties as shown in Table I. 78 and 89 % of FA1 and FA2 were ash. This is reasonable as fly ashes were the results of high temperature incineration and most of the volatiles or organics were burned off. The high ash contents could be attributed to Ca, Si compounds and other residual metals.²¹ According to the proximate analysis, about 22 and 11 % of the fly ash were volatile and fixed carbon indicated the residual organics.

TABLE I. Physicochemical properties of FA1 and FA2

Sample	Proximate content ^a , wt. %			Moisture content, %	pH	ANC / mL g ⁻¹
	Volatiles	Fixed carbon	Ash			
FA1	9	13	78	0.34	12.7	7.3
FA2	3	8	89	0.34	12.4	3.8

^aContent of fixed carbon = 100 – Content of ash – Content of volatiles

Besides the proximate analysis, Table I also listed the moisture content, pH and ANC of these ashes. Both ashes had low moisture contents (only 0.34 %) but high in pH and acid neutralization capacity (ANC). The pH measured here was the natural pH in contact with water. Fly ashes in this research originated from bag filters which were used to treat flue gas. Lime was sprayed to neutralize acidic gases. Therefore, the high pH was most likely caused by the neutralization products or CaO and Ca(OH)₂ that remained. ANC was measured to evaluate the presence of alkaline compounds and their availability and reactivity.²² Although both fly ashes showed similar pHs as shown in Table I, their ANC values differed. FA1 had a much higher ANC indicating that the alkalinity in FA1 may be more readily neutralized by acetic acid.

XRF and XRD analysis were next performed to further clarify the chemical compositions and crystalline structures of fly ashes. The XRF results are presented in Table II.

XRD results in Fig. 1 proved the presence of chloride salts, *i.e.*, NaCl and KCl. Other crystals observed include portlandite (Ca(OH)₂), anhydrite (CaSO₄), calcium hydrochloride (CaCl(OH)), calcite (CaCO₃) and silica (SiO₂). XRF and XRD results were consistent with the high pH and ANC values of both fly ashes in Table II. Due to the presence of these calcium-containing compounds, raw fly ashes manifested strong alkalinity.²¹

FTIR spectrograms were obtained to investigate the surface functional groups as shown in Fig. 2. The wideband at 3445 cm⁻¹ corresponded to the stretching

vibration of -OH in hydration water, while the band at 1634 cm^{-1} was its bending vibrations.²⁴ Absorbances centred at 2510 , 2360 , 1795 , 1451 and 875 cm^{-1} were caused by vibrations of CO_3^{2-} (carbonates).²⁵ Peaks at 1120 and 660 cm^{-1} indicated vibration stretching of S-O bonds and SO_3^{2-} , respectively.²⁶ Besides, Si-O bonds were observed at 560 cm^{-1} .²⁷ It appears that that SiO_2 , SO_4^{2-} and CO_3^{2-} salts were all found to be present in these fly ashes.

TABLE II. Element compositions of FA1 and FA2 *via* XRF (wt. %)

Item	Ash	
	FA1	FA2
Ca	55.74	50.97
Cl	14.27	11.44
O	11.41	12.70
K	7.01	5.68
Na	4.22	4.32
Mg	1.79	0.64
S	1.32	2.38
Si	0.68	1.70
Fe	1.31	2.65
Al	0.36	0.78
Ti	0.33	0.76
Br	0.18	1.02
P	0.13	0.20

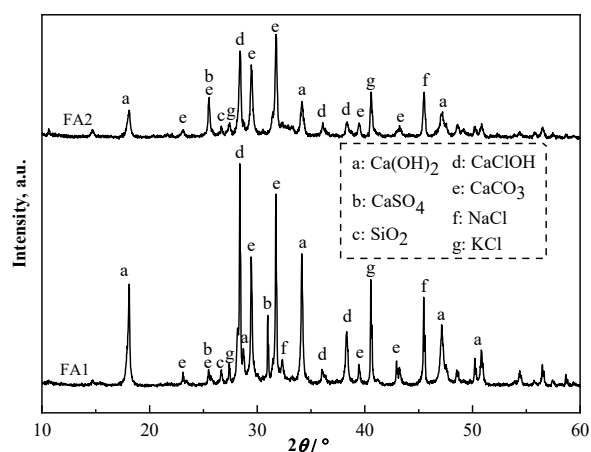


Fig. 1. XRD pattern of FA1 and FA2.

Overall, the characterizations showed that both ashes were high in alkalinity with an abundance of chemicals such as Ca-containing compounds, salts of chloride, sulphate and carbonate, and oxides (*e.g.*, SiO_2). However, the exact amount of these elements differed as their origins varied. This variability could be added to the difficulty in fly ash treatment.

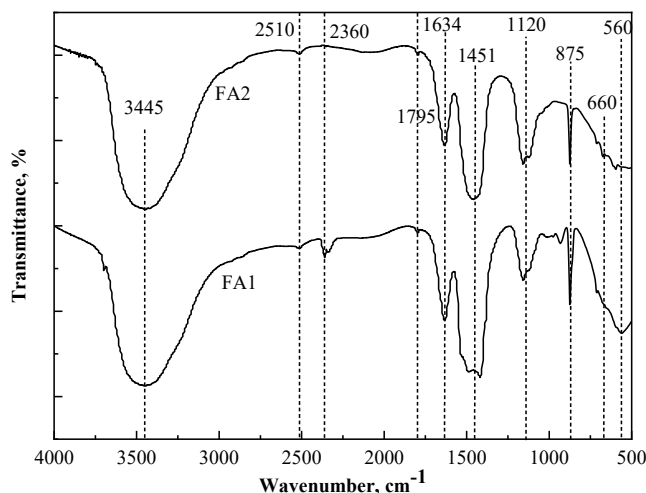


Fig. 2. FTIR spectra of FA1 and FA2.

Heavy metal contents

Heavy metals were of the most concern for their toxicity. Digestion+ICP-OES analysis was employed to analyse the heavy metal contents in fly ashes. This method was able to detect even trace amount of heavy metals in solids.

Table III lists the contents of 6 major heavy metals in fly ashes. Contents of other metals such as As and Sb, were also detected, but their contents were extremely low and were not of concern here. Studies have shown that many heavy metals were transported in the form of volatile chloride and condensed into fly ash particles.^{28,29} FA1 had 1.08 g/kg of Pb and 7.90 g/kg of Zn. By comparison, the contents of Cu, Cr and Cd were much smaller. However, the heavy metals contents also varied with fly ash. FA2 showed a much higher metal contents than FA1, probably due to the difference in solid wastes compositions in different city. The municipality where FA1 came from implemented a more stringent garbage sorting practice.

TABLE III. Contents of major heavy metals in FA1 and FA2

Sample	Heavy metal content, g kg ⁻¹					
	Cr	Ni	Cu	Zn	Pb	Cd
FA1	0.25	0.06	0.53	7.90	1.08	0.18
FA2	2.22	0.69	1.98	26.74	2.46	0.25

Heavy metal leaching toxicity

The main purpose of this research was to investigate the leaching behaviour of fly ashes by studying the leaching of heavy metals at different time point and exploring the important factors affecting the leaching behaviour.

TCLP leaching. Fig.3a shows the changes of pH and heavy metals concentrations at different leaching time. As shown in Fig. 3a, pH of the FA1 leaching solution rose quickly from the original 2.88 to about 10 after just 2 h. It continued to climb gradually, from 10 at 2 h to about 12 at 10 h. The changes in pH were caused by the consumption of acetic acid by the alkalinity in fly ash. For FA1, Cr, Ni, Cu, Zn and Cd concentrations remained low and stable during the whole leaching process. On the contrary, there is an obvious increase in Pb leaching as time was extended.

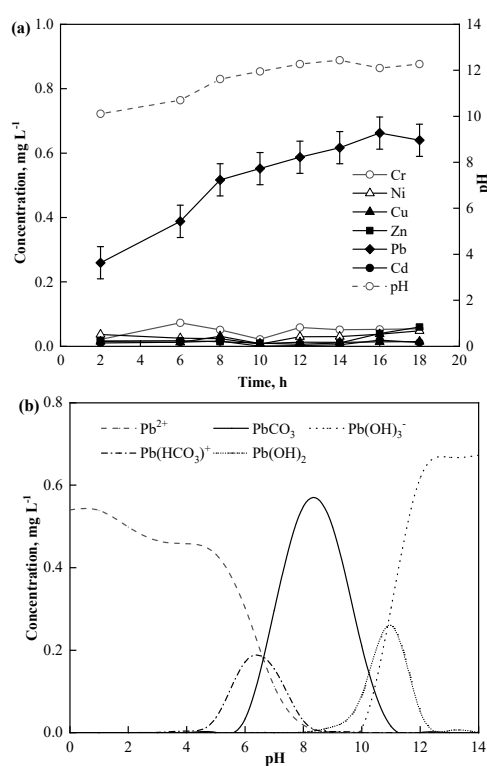


Fig. 3. Leaching of heavy metals FA1 during TCLP test (a); speciation of metals in leaching environment *via* Minteq (b).

To understand the different leaching phenomena of metals, besides heavy metals, other anion and cation concentrations in leaching solution were also monitored. As shown by XRD, XRF and FTIR analysis, fly ashes contained a variety of carbonate, sulphate and chloride salts and metal oxides. The release of these compounds into leaching solution could change the leaching environment thus affecting the release of heavy metals. Results showed that high concentrations of potassium, sodium, chloride, sulphate, carbonate and calcium were detected in leaching liquid. For instance, the leaching solution of FA1 contained a

total inorganic carbon (TIC) of about around 123.3 mg/L with a sulphate concentration about 120 mg/L. The release of carbonate would not only change the acidity of leaching solution but also reacting with heavy metals to form different compounds thus affecting the solubility of metals.

Software Minteq 3.1 was used to elaborate the speciation of metals in the leaching environment. Concentrations of heavy metals, calcium, chloride, sulphate and carbonate and pH were entered into the software. Minteq results showed that Cr, Ni, Cu, Zn and Cd mainly existed as carbonate or hydroxide precipitates at the pHs of the leaching solution (10–12). For example, for Cd, CdCO_3 and Cd(OH)_2 precipitates dominated at high pH. This is in accordance with the low leaching of these metals shown in Fig. 3a. That is, the high alkalinity of FA1 resulted in a very alkaline leaching solution. Metals Cr, Ni, Cu, Zn and Cd were bound up in carbonate or hydroxides precipitates.

The only exception is Pb, which has an obvious trend of increasing leaching. This can also be explained by the reactions occurring during leaching process. Fig. 3b represents the speciation of Pb-containing compounds at different pHs at the presence of carbonate, calcium, sulphate and other heavy metals. Only major species were presented here. Concentrations of other species such as PbOH^+ , $\text{Pb(HCO}_3)^+$ and PbSO_4 , were negligible. Unlike Cr, Ni, Cu, Zn and Cd, Pb existed mainly as PbCO_3 at pH 6–10, while Pb(OH)_3^- became dominant as pH rose to higher than 10. It seems that as the pH of leaching solution increased, Pb was dissolved gradually to form species the like of Pb(OH)_3^- .

Fig. 4a shows the leaching concentrations of heavy metals and pH changes during leaching of FA2. pH of the leaching solution reached 5.2 after 2 h, then rose to 6.0 at 6 h and fluctuated between 6.5 and 7.2 after that. FA2 had a much lower alkalinity and acid neutralization capacity. Thus, the leaching solution remained acidic to near neutral.

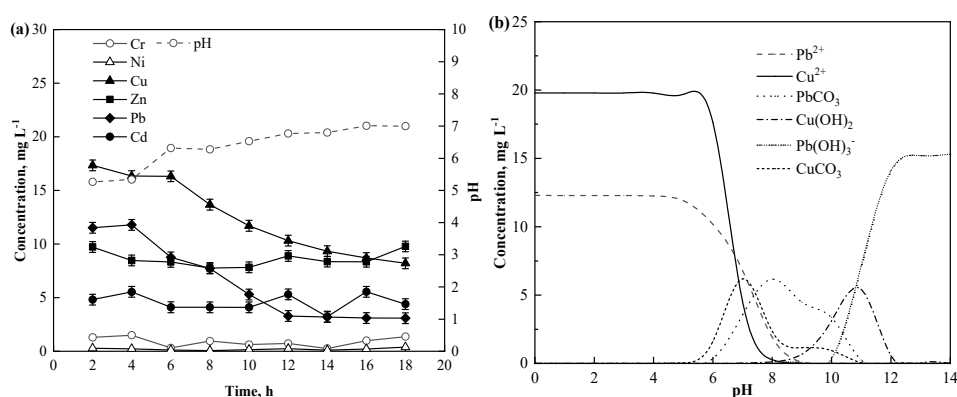


Fig. 4. Leaching concentrations of heavy metal against leaching time (a) and speciation Pb and Cu at different pH *via* Minteq (b).

The characterization of FA2 (Table III) showed that FA2 contained much higher amount of heavy metals. Therefore, higher leaching was observed for all metals. For instance, the leaching of Zn at 18 h from FA1 was 0.022 mg/L, while that from FA2 reached as high as 8.21 mg/L.

However, the behaviour of metal leaching also was different from that of FA1. Cr, Ni, Zn and Cd leaching stabilized after 4 h. As with FA1, Minteq software was employed to calculate the predominant species. Results showed that Cr^{3+} , Ni^{2+} , Zn^{2+} and Cd^{2+} were the dominant species in the whole range of pH during the FA2 leaching. The formation of carbonate precipitate was very low at the pH range. Cr, Ni, Zn and Cd compounds that could be dissolved by acetic acid in TCLP were leached out at the first 4 h, after which time the leaching stabilized.

However, for Cu and Pb, at the first stage of leaching (2–10 h), pH fluctuated between 5 and 6. According to the calculation *via* Minteq, the predominant species at this stage were Cu^{2+} and Pb^{2+} , respectively (Fig. 4b). As leaching progressed, pH increased to about 7, and PbCO_3 and CuCO_3 precipitates became predominant. This is consistent with the decline in Pb and Cu leaching in Fig. 4a after 10 h.

Overall, it appears that the heavy metals leaching was a complicated process. Besides heavy metals, other chemicals from fly ash were also released, which changed the leaching environment. Of the two fly ashes under investigation, one was much more alkaline than the other, which resulted in two different leaching environments. However, the analysis *via* Minteq showed that regardless of the differences, the precipitation/dissolution of metal carbonate and hydroxides seemed to be the controlling reactions of heavy metal leaching, as the changes in metals containing compounds affected the extent of leaching.

EDTA-modified TCLP. As shown by TCLP leaching, the fly ash compositions could greatly affect leaching behaviour by changing the leaching environment where chemical reactions (mainly precipitation/dissolution) occurred. For the two fly ashes under study, their original difference in chemical composition, and other characteristics such as alkalinity, led to difference in conditions of leaching solutions. Thus, heavy metals behaved differently. In their study of the merit and demerit of TCLP for the leaching toxicity evaluation, Lzquierdo and Querol believed high alkalinity in fly ash led to a rise in pH of the leaching solution.³⁰ The leaching of metal was often repressed. Fly ash in natural landfill environment came into contact with an environment with a much lower pH and presence of organics. Thus, TCLP test may underestimate the real leaching potentials. Studies above already showed that the precipitation/dissolution reactions were related to the pH of leaching environment.

To investigate the leaching behaviour when organics were present, EDTA was used to represent chelating organics and its concentration was set at 1 mmol/L.

Fig. 5 shows the heavy metal leaching by FA1 and FA2 *via* EDTA-modified TCLP.

The addition of EDTA did not change pH of the leaching solution but increased the leaching of all metals. Cu and Cd leaching at 18 h for FA1 almost doubled. Other metals also observed various degree of increase. This proved that the chelating capability of EDTA facilitated the release of metals. Another proof of EDTA's chelating effects is the leaching of Pb and Cu from FA2. Without EDTA, the leaching of Pb and Cu declined as leaching progress (Fig. 4a). With the presence of EDTA, Pb leaching continued to increase from 2–8 h and stabilized after 10 h. These results showed that the chelating effects from EDTA changed the behaviour of metals. When acetic acid alone was used as leachant, the leaching process was controlled by the precipitation/dissolution of carbonate and hydroxides of metals. With the addition of merely 1 mmol/L of EDTA, chelating reactions also played a significant role. This shows that the presence of chelating organics in natural condition may have significant effects on the leaching of metals and had to be taken into consideration.

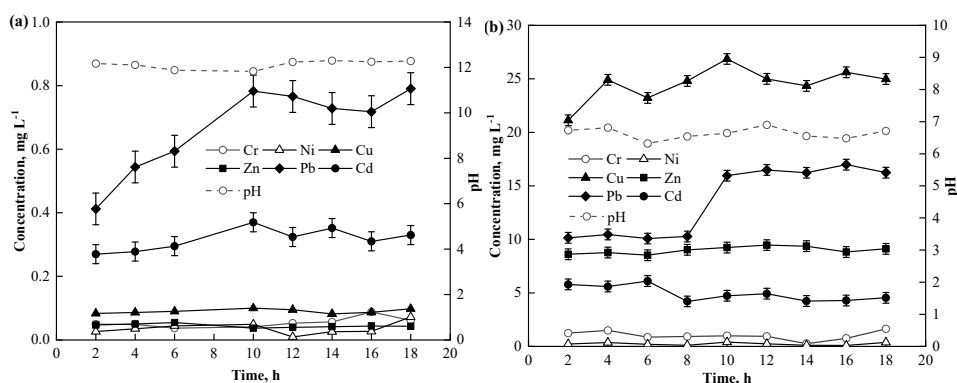


Fig. 5. Heavy metal leaching *via* EDTA-modified TCLP: a) FA1; b) FA2.

CONCLUSIONS

Fly ashes had high alkalinity because of the presence of various Ca-bearing compounds, though the strength of alkalinity and their acid neutralization capacity varied. Other major chemical composition in ashes include NaCl, KCl, SiO₂ and heavy metals such as Cr, Ni, Cu, Zn, Pb and Cd, *etc.* The exact contents of these chemicals could differ based on the compositions of municipal solid waste, incineration conditions and flue gas treatment method.

Study of heavy metals (Cr, Ni, Cu, Zn, Pb and Cd) leaching behaviour *via* TCLP showed that the process was complicated. The precipitation/dissolution reactions had significant effects on leaching. Because of the alkalinity in fly ash, pH of the leaching solution was raised. Calculations based on Minteq software

showed that the shifts in the precipitation/dissolution equilibrium of carbonate and hydroxides compound of heavy metals could explain the leaching of metals over time. The differences in characteristics of fly ash (alkalinity and heavy metal content, *etc.*) resulted in the variation of leaching environment and the concentrations of heavy metals. But it seems that the precipitation/dissolution reactions controlled the release of heavy metals from fly ash in TCLP test.

On the other hand, the addition of EDTA introduced chelating effects. Chelating of Pb with EDTA resulted in continuous leaching of Pb for 10 h. The amount of leaching was greatly increased, indicating that TCLP may lead to underestimation of the metal concentrations in natural environment.

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ИЗВОД

КОНТРОЛИСАЊЕ РЕАКЦИЈА ТОКОМ ЛУЖЕЊА ТЕШКИХ МЕТАЛА ИЗ ЛЕТЕЋЕГ ПЕПЕЛА ОД СПАЉИВАЊА КОМУНАЛНОГ ЧВРСТОГ ОТПАДА

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Ово истраживање је имало за циљ да окарактерише физичка и хемијска својства летећег пепела од спаљивања комуналног чврстог отпада и истражи лужење тешких метала током процеса TCLP (енгл. *Toxicity Characteristics Leaching Procedure* – поступак лужења са карактеристикама токсичности). Да би се разумело понашање током лужења, праћене су концентрације тешких метала, хлорида, сулфата и калцијума у различитим временима лужења, и рН вредности раствора за испирање. Резултати су показали да је хемијски састав летећег пепела довео до разлика у окружењу за лужење. Од два летећа пепела која су проучавана, један је резултирао рН вредношћу од 10–12 у окружењу за лужење, док је други имао рН од око 6–7. Софтвер Minteq је коришћен за истраживање специјације тешких метала на основу рН вредности и концентрације анјона и катјона. Резултати су показали да промена у равнотежи таложења/растварања карбоната и хидроксида тешких метала може објаснити флукуацију концентрација метала током процеса лужења, што указује на то да је лужење вероватно контролисано овим реакцијама. С друге стране, додавање EDTA је променило контролне реакције. Реакције хелирања између тешких метала и EDTA довеле су до много веће токсичности лужења тешких метала, показујући да присуство хелирајућих органских материја у природном окружењу може олакшати лужење тешких метала.

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