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Characterization of Solid Waste Incineration Fly Ashes and their Heavy Metal Leaching Behavior

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ABSTRACT

Two fly ashes from municipal solid waste incineration were selected to study their heavy metal leaching behavior. The main purpose of this research is to investigate the characteristics of fly ashes and compare the leaching of heavy metals in different leaching environment. pH and acid neutralization capacity analysis showed that fly ashes were highly alkaline. Fly ashes also contained a variety of heavy metals including Pb, Cu, Cr, Zn, Cd and Ni etc. Leaching studies showed that the alkalinity of fly ashes raised the pH of leaching solution from acidic to basic. Ni, Cu and Zn were strongly bound to ashes and manifested low leaching. In contrast, Cr and Cd had high mobility but their leaching was inhibited by the low solubility of carbonate Cr and Cd. Pb was highly leachable in the alkaline environment with concentration in the leaching solution reached as high as 9.74 mg/L. In addition, the presence of EDTA in the environment also increased leaching. Pb concentration was raised to 16.63 mg/L. This could be attributed to the chelating capacity of EDTA which means that the presence of organics in natural environment should be taken into consideration.

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INTRODUCTION

Increasing amount of municipal solid waste (MSW) fly ash was generated each year as the amount of MSW being incinerated continued to grow. City authorities, faced with limited landfill capacity and more stringent regulation, had turned to incineration for MSW disposal due to its mass and volume reduction capability and energy recovery possibility (Atanes et al. 2019; Cao et al. 2021). Bottom ash and fly ash accounted for about 25-30% and 3-5% of the MSW mass respectively (Ma et al. 2019). Between them, fly ash was considered more environmentally hazardous. It contained high contents of chloride, sulfate salts and heavy metals as well as toxic substances such as dioxin and furan (Wang et al. 2018). Therefore, MSW fly ash was designated as hazardous waste in many countries (Loginova et al. 2019). This means that fly ash had to undergo proper treatment to reduce its impact on environment before it can be sent for landfill or reused. One of the requirements fly ash had to meet before further disposal was heavy metal leaching toxicity. Many studies on fly ash focused on the reducing of metal leaching potentials by washing, stabilization/solidification and thermal treatments etc. (Chen et al. 2017; Xue and Liu, 2021)

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Leaching toxicity tests before and after treatment were often conducted to evaluate the efficacy of these treatments. Many methods on leaching were developed by various authorities. Among them were European standard leaching tests (EN12457-4), the Toxicity Characteristics Leaching Procedure (TCLP, US EPA method 1311) and Synthetic Precipitation Leaching Procedure (SPLP, US EPA method 1312). China also published its methods including sulfuric acid/nitric acid method (HJ/T 299-2007) and acetic acid buffer solution method (HJ/T 300-2007). As a matter of fact, sulfuric acid/nitric acid and acetic acid buffer solution method were similar to the SPLP and TCLP methods respectively.

Among them, TCLP method simulates the leaching through a landfill and could provide a rating that can prove if the waste is dangerous to the environment or not. This rating can dictate the waste management methodology for disposal. Therefore, TCLP is a measure employed regularly for comprehensive toxicity testing (Al-Ghouti et al. 2021). TCLP method was employed in this research for detailed measurement of heavy metal leaching of fly ash.

Leaching of heavy metals from fly ash was a complicated process. Chemical compositions, heavy metal speciation, characteristics of extractant and leaching environment all had significant influences on the leaching behavior (Liu et al.2019a). For example, Jiao et al. (2016) argued that chloride or sulfate salts of metals were easily mobilized because of their high solubility. In contrast, silicate salts of metal were quite stable. On the other hand, Lu et al. (2019) believed that the high final pH in the leaching solution of fly ashes hindered the release of heavy metal which led to serious underestimation of toxicity. The high pH of fly ash resulted from its high alkalinity. Due to its small size and low density, fly ash exited incinerator with flue gas which was treated with lime to neutralize acid compounds such as HCl, HF and SO_2 (Zhang et al. 2021). Many studies have suggested that TCLP may not be an ideal test to reflect the mobility of organic and inorganic pollutants in fly ashes (Guo et al. 2017; Yakubu et al. 2018). For instance, Liu et al.(2019b) argued that, due to the presence of organic acid in the landfill environment, TCLP was not able to reflect realistically the extent of heavy metal leaching. Instead, an EDTA-modified TCLP may be more suitable.

This research aimed to characterize the physical and chemical properties of fly ashes and analyze their heavy metal contents, speciation and leaching toxicity via TCLP and EDTA-modified TCLP. The ultimate goal is to clarify the effects of fly ash on the leaching environment and compare the toxicity leaching of heavy metals with or without EDTA. Results from this research could help to advance the understandings on how to evaluate the potential hazards of fly ashes to the environment.

MATERIALS AND METHODS

Fly ashes

Two municipal solid waste incineration fly ashes from Zhejiang and Guangdong Provinces of China were used and named as FA1 and FA2 respectively.

Fly ash characterization

The moisture content of fly ash was determined according to the national standard method of China (GB/T 212-2008) while proximate analysis was conducted according to ASTM standard method E1131-08. pH and acid neutralization capacity (ANC) were analyzed according to methods by Liu et al. (2019b) and Yue et al. (2019). Specifically, 1.0 g of fly ash was added to 100 mL of deionized water and stirred for 15 min at 150-200 rpm. The pH of the mixture was then measured and designated as the pH of fly ash. For ANC, 1.0 g of fly ash was added to a 250 mL conical flask and mixed with 100 mL of deionized water. The mixture was titrated with 1 mol/L of acetic acid to pH 7.0 after 15 min of mixing. The amount of acetic acid consumed was ANC.

X-ray Fluorescence Spectrometer (XRF-1800, Shimadzu, Japan) and X-ray Diffraction (XRD, Rigaku UItima IV, Japan) were used to determine the chemical compositions and crystalline minerals.

Heavy metal contents and speciation

To analyze the heavy metal content, fly ash was digested with 1 mL hydrochloric acid, 4 mL nitric acid, 1 mL hydrofluoric acid and 1 mL hydrogen peroxide first. The digested liquid was brought to a volume of 50 mL with deionized water and analyzed for heavy metals (Pb, Cu, Cr, Zn, Cd, Ni etc.) via Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES, Optima8000, PerkinElmer, US). Content of each metal in g/Kg fly ash were calculated.

Tessier sequential chemical extraction method was adopted to investigate the speciation of heavy metals (Tessier et al. 1979). The extraction was divided into five steps. Extractants of increasing strength were employed sequentially. The extractants used step by step were 10 mol/L $MgCl_2$, 1 mol/L NaAc, 0.04 mol/L $NH_2OH \cdot HCl$ in 25% (v/v) HAc, 0.02 mol/L HNO_3 and 5 mL 30% H_2O_2 and finally 5 mL of 3.2 mol/L NH_4OHAc in 20% (v/v) HNO_3. At the end of each step, the fly ash was filtered out and supernatant was brought to 50 mL with deionized water and analyzed for heavy metals via ICP-OES. Metals detected from step 1 to step 5 were classified as exchangeable, carbonate-bound, Fe-Mn oxide-bound, organic matter-bound and residuals and represented by F1, F2, F3, F4 and F5 respectively. Sum of F1 and F2 (in percentage) was calculated as mobility factor (MF) to describe the potential mobility of metals (Rassaei et al. 2020).

Heavy metal leaching toxicity tests

TCLP was used to simulate the leaching process of fly ash in landfill. Fly ash was sieved to sizes of less than 9.5 mm. Acetic acid (pH=2.88 ± 0.1) was used as leachant. Liquid/solid ratio (L:Kg) was set at 20:1. The fly ash/acetic acid mixture was put on an end-over-end rotator for agitation for 18 hours and filtered. Filtrate analyzed for heavy metals by ICP-OES. For comparison, an EDTA-modified TCLP method was also employed. EDTA was added to acetic acid leachant to a concentration of 1 mmol/L. All other procedures were the same as TCLP.

RESULTS AND DISCUSSION

Fly ashes properties

Results from proximate analysis and other characterization of fly ashes were shown in Table 1. Both ashes were low in moisture and volatiles as it is the product of high temperature incineration.

The pH of fly ash is the natural pH of the material as it resulted from contact with water. pHs of the fly ashes were high at 12.6 and 12.3. Lime was sprayed to treat flue gas which was later retained by bag filter as part of the fly ash which resulted in the high pH of fly ash.

Acid neutralization capacity (ANC) is the material's ability to neutralize acetic acid. Although FA1 and FA2 showed similar pH values, ANC of FA1 was three time more than that of FA2. It seems that alkalinity in FA1 was more readily available by acetic acid. Ramanathan and Ting (2016) believed that the speciation of alkaline compounds varied with flue gas treatment. Metal oxides generated during incineration could react with moisture in flue gas to produce alkali such as $Ca(OH)_2$ and NaOH. Their contents varied with the amount of moisture that is available. ANC results showed that the alkaline compositions in these two fly ashes may be quite different.

Table 1. Proximate analysis and other characteristics

	Proximate analysis			Moisture (wt%)	pH	ANC (mL/g)
	Volatile (wt%)	Fixed carbon (wt%)	Ash content (wt%)			
FA1	6.70	9.20	84.10	0.83	12.6	7.8
FA2	3.60	7.75	88.65	0.34	12.3	2.4

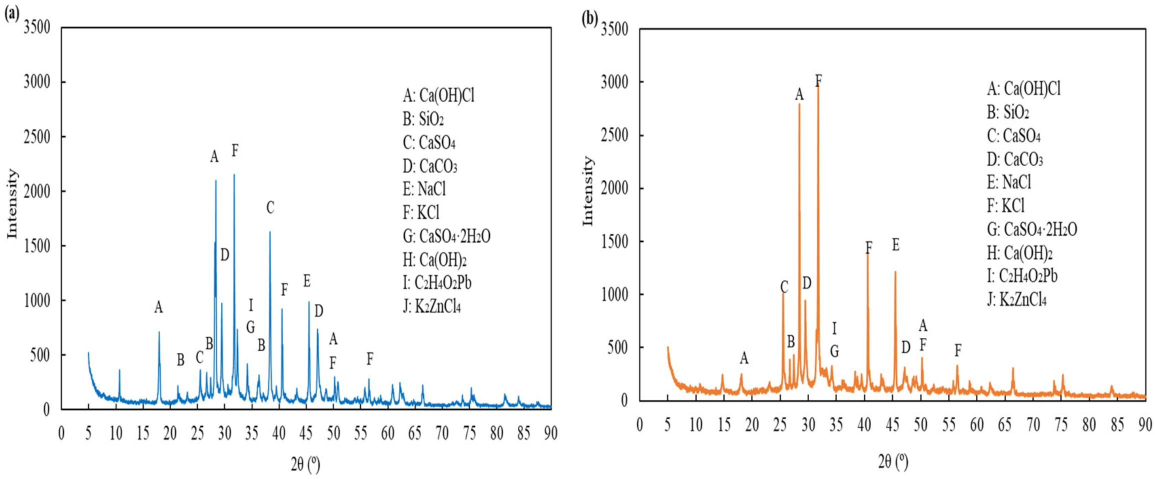

Fig. 1. XRD patterns: (a) FA1; (b) FA2

Crystalline structures by XRD

XRD analysis (Fig.1) was carried out to elucidate the crystalline composition. XRD patterns demonstrated that the two fly ashes had similar crystalline compositions. The crystalline chemicals identified included Ca(OH)Cl, $CaCO_3$ and $CaSO_4$, SiO_2, NaCl, KCl etc. and those are commonly found in ashes. It is a common practice in China for flue gas to be treated by $Ca(OH)_2$ or CaO to neutralize acidic compounds such as HCl and SO_2. The resultant Ca(OH)Cl and $CaSO_4$ are products of reactions such as those of (1)-(3) (Chen et al. 2012).

$$\mathrm{Ca(OH)_2 + HCl \rightarrow Ca(OH)Cl + H_2O} \tag{1}$$

$$\mathrm{Ca(OH)_2 + SO_2 \rightarrow CaSO_4 + H_2O} \tag{2}$$

$$\mathrm{CaO + HCl \rightarrow Ca(OH)Cl} \tag{3}$$

The presence of $C_2H_4O_2Pb$ and K_2ZnCl_4 indicating the existence of toxic heavy metals. It is believed that the complex mineralogy of fly ashes is the result of vaporization, melting, crystallization, vitrification, condensation and precipitation which occurred during flue gas generation and treatment that followed (Li et al. 2004).

Chemical compositions by XRF

XRF analysis was next conducted for the chemical compositions of fly ashes as shown in Table 2. The most abundant element is Ca (49.21 wt% for FA1 and 42.42 wt% for FA2) followed by Cl (18.58 wt% for FA1 and 14.48 wt% for FA2). Besides Ca and Cl, ashes contained high amount of K and Na together with Pb, Cu, Cr, Sr, Zn, Ni of varying contents. These could also trace their origins back to the components in municipal solid wastes.

In summary, characterizations showed that fly ashes were high in alkalinity with a large amount of Ca-containing alkaline products. In addition, they were rich in chemicals both soluble and insoluble. XRD and XRF analysis revealed the presence of toxic metals such as Pb, Ni, Cu, Cr etc. indicating potential hazards. The fly ashes in the research were from two incineration plants. They showed similarity and variation in characteristics. This variability added to the difficulty in treatment and disposal (Weibel et al. 2017).

Table 2. XRF analysis results

Element	Content (wt%)		Element	Content (wt%)	
	FA1	FA2		FA1	FA2
Ca	49.21	42.42	Pb	0.39	0.52
Cl	18.58	14.48	Br	2.02	0.78
K	5.68	8.20	P	0.086	0.32
Na	4.42	6.39	Cu	0.32	0.51
S	1.51	3.12	Ba	0.076	0.47
Si	1.37	2.01	Cr	0.037	0.33
Zn	1.77	3.62	Sr	0.076	0.17
Fe	0.90	3.04	Mn	12.42	0.14
Al	0.38	0.90	Sn	0.18	0.11
Ti	0.24	0.90	Zr	0.037	0.059
Mg	0.40	0.73	Ni	/	0.046

Table 3. Heavy metal contents in fly ashes

	Cr (g/Kg)	Ni (g/Kg)	Cu (g/Kg)	Zn (g/Kg)	Pb (g/Kg)	Cd (g/Kg)
FA1	0.28	0.14	2.59	2.08	2.69	0.16
FA2	2.38	0.38	4.10	3.12	3.45	0.30

Heavy metal contents

Landfill was one of the main methods of fly ash disposal. However, fly ash was designated as hazardous and direct landfill was no longer an option in China since 2008. Authorities in China urged innovation in reuse and recycling of fly ash to recover valuable minerals. Owing to the complexity of chemicals in the ashes, especially the existence of various heavy metals, fly ashes often had to be pretreated to reduce their adverse effects on environment and ecosystem. The total contents of heavy metals and their leaching toxicity were a major factor in the determination the degree of pretreatment (Shao et al. 2022). XRF analysis showed presences of a number of heavy metals in both fly ashes. It was reported that XRF may not be able to detect trace amount of metals. Therefore, fly ashes were digested and digested solutions analyzed for heavy metal via ICP-OES.

Table 3 lists the contents of 6 major toxic heavy metals in the two fly ashes via digestion+ICP-OES method. Other metals such as As, Ba, and Be, were relatively low and did not pose serious leaching and therefore were not of concerns.

As shown in Table 3, the contents of heavy metals varied with fly ashes. FA2 contained more heavy metals than FA1. Cr content in FA2 was more than 8 times that of FA1. These differences could be attributed to the difference in the compositions of municipal solid wastes or incineration conditions which resulted in fly ashes of difference chemical compositions or physical properties.

Heavy metal speciation

Besides contents of heavy metal, speciation study could give some ideas how these metals were bound to fly ashes. The strength of binding determined how mobile these metals are in the environment. Chaudhary and Banerjee (2007) argued that biological and environmental impacts of heavy metals was affected by their morphologies even when total amount of heavy metals was the same. Therefore, heavy metal speciation study could be used to evaluate the bioavailability and mobility of heavy metals and indirectly assess the risk to the environment. Tessier sequential chemical extraction method was one of most commonly used speciation method (Race et al. 2015). Results of Tessier extraction were shown in Fig. 2.

Among the five fractions from Tessier analysis, the fraction of exchangeable are considered to be most easily leached (Fu et al. 2019). Carbonate-bound ones were sensitive to solution pH and prone to leach out in acidic environments while remain stable in alkaline environment. Fe-Mn oxide-bound heavy metals were those bound with minerals and tended to present in fine particles (Zhou et al. 2015a). Zhou et al. (2015b) reported that Fe-Mn oxide-bound metals were susceptible to changes in pH and redox environment. Organic matter-bound heavy metals were those linked to organic matter formed in the process of waste incineration. Residuals were generally tightly encapsulated in the solids and difficult to leach out except in the extreme environment (Ye et al. 2016). Studies have shown that among the 5 fractions, exchangeable and carbonate-bound fractions were considered much more mobile in environment.

Residual Ni and Cr predominated in both ashes. Indicating that these metals may be stable. The next most abundant speciation for both Ni and Cr was Fe-Mn oxides bounds. More than half of Cu, Pb and Cd was categorized as Fe-Mn oxides bound with a relatively small percentage as residuals. By comparison, the speciation of Zn varied greatly with fly ash. Most of Zn (about 61%) in FA1 was in residual fraction while Zn in FA2 were mostly Fe-Mn oxides bound (80.45%).

Another feature from speciation is that very little of the heavy metals were in exchangeable and organic matter-bound fractions. The absence of exchangeable heavy metals could be attributed to the sintering effects of incineration. Heavy metals were mostly bound after going through high temperature treatment. On the other hand, the lack of organic matter-bound metals could be explained by the fact that organics were mineralized during incineration as shown by the low volatile and fixed carbon contents in Table 1.

Mobility factor (MF) values were calculated and shown in Table 4. MF was used to assess the mobility of heavy metals. MF less than 1% indicates no risk, while 1-10% means low risk, 10-

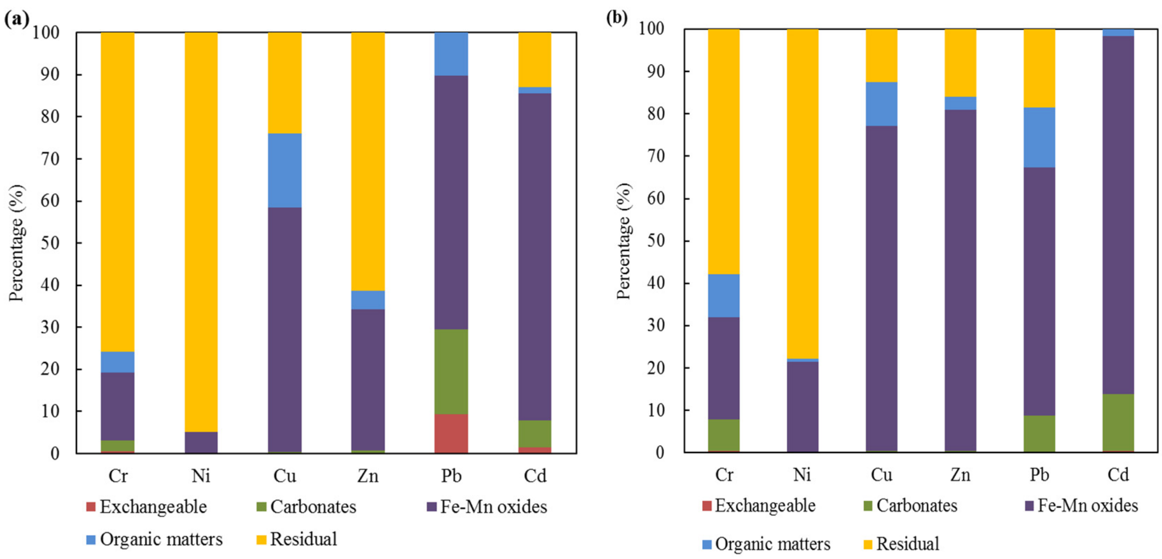

Fig. 2. Heavy metal speciation: (a) FA1; (b) FA2

Table 4. Mobility factor values (%) of heavy metals

	Cr	Ni	Cu	Zn	Pb	Cd
FA1	3.09	0.23	0.33	0.67	29.44	7.47
FA2	7.79	0.12	0.52	0.47	8.78	13.92

Table 5. Leaching concentrations of heavy metals and leaching standards

	Cr (mg/L)	Ni (mg/L)	Cu (mg/L)	Zn (mg/L)	Pb (mg/L)	Cd (mg/L)	pH
FA1-TCLP	0.082	0.11	9.48	3.99	**6.53**	0.08	9.8
FA2-TCLP	1.35	0.47	17.57	17.04	**9.74**	0.09	8.8
FA1-EDTA/TCLP	0.092	0.13	10.67	3.34	**9.11**	**3.49**	9.5
FA2-EDTA/TCLP	1.11	0.47	24.75	17.82	**16.63**	**7.72**	8.0
Leaching toxicity standard	4.5	0.5	40	100	0.25	0.15	-

30% medium risk, 30-50% high risk and 50-75% very high risk. As shown by MF values, Ni, Cu and Zn presented no risk while Cr, Pb and Cd were in low or medium risk. MF of metals in FA1 followed the order of Pb > Cd > Cr > Zn > Cu > Ni and Cd > Pb > Cr > Cu > Zn > Ni for FA2.

Heavy metal leaching

Leaching toxicity of heavy metals via TCLP and EDTA-modified TCLP were summarized in Table 5. The last row in Table 5 is the leaching toxicity standards for pollution control on the landfill site of municipal solid waste (GB 16889-2008) which was developed by the Ministry of Ecology and Environment of China.

The extent of heavy metal leaching appeared to be related to its mobility factor. Leaching of metals of no risk (Ni, Cu and Zn) was much lower than that required by the national standards. However, extent of heavy metal leaching can not all be explained by mobility factor. For instance, Cr and Cd were of low to medium risks. But leaching toxicity for both met the standards. As shown by the speciation in Fig.2, carbonate bound Cr and Cd contributed to the higher mobility factor values of these two metals. These carbonate compounds of Cr and Cd were stable in basic environment. The last column in Table 5 listed the pH of leaching solution at the end of leaching tests. pH was raised from the original 2.88 to basic because of the alkalinity of fly ashes. Acetic acid in the solution was consumed by the alkalinity from the ashes. Overall, it showed that the basicity of the leaching environment did contribute to the inhibition of leaching of heavy metals. Of the 6 major metals, Pb was the only one that had leaching beyond that required by the landfill standards. Pb in both fly ashes were of low to medium risks. Unlike Cr and Cd., $PbCO_3$ could dissolve to form soluble Pb hydroxide compounds such as $Pb(OH)_3^-$ at high pH thus increased leaching.

In addition, the addition of EDTA had significant effects on heavy metal leaching. Leaching toxicities were much high via EDTA-modified TCLP than those via indicating the presence of chelating agents could affect leaching. This is in accordance with studies by Jain et al (2022) and Xin et al. (2022). Leaching from both Pb and Cd were higher than stipulated by the standards. Therefore, it is possible that leaching may be underestimated by TCLP when organic acids were present in the environment. Lu et al. (2019) proposed that EDTA-modified TCLP could more closely simulate the real leaching environment where chelating organics may co-exist.

Overall, TCLP and EDTA-modified TCLP results proved that heavy metals leaching process was complicated. Many factors (heavy metal speciation, solubility of heavy metal compounds, leaching environment) could all play a role.

CONCLUSIONS

Fly ashes varied in features such as chemical composition, alkalinity, crystallinity, heavy metal content and speciation because of their origins. Ca-bearing compound such as $Ca(OH)_2$, $CaSO_4$ and $CaCO_3$ etc. stemmed mostly from flue gas treatment and were the main contributor to the alkalinity. A number of heavy metals were found in the fly ashes. These metals varied in their binding with fly ashes which resulted in their varying degrees of risks. These binding had significant effects on leaching toxicity. For examples, heavy metals of low mobility (no risk) were not leached thus relatively safe to the environment. At the same time, mobility factor of heavy metal was not the only factor that determined leaching. Leaching environmental also played a significant role. The alkalinity of the fly ashes turned the leaching solution from acidic to basic which in turned inhibited or facilitated heavy metals leaching depending on the solubility of metal compound in the environment. In addition, EDTA-modified TCLP results showed that the presence of organic acid was another factor. Overall, heavy metal leaching toxicity tests had to take into consideration of all these factors for an accurate evaluation of the hazards of fly ashes.

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CONFLICT OF INTEREST

The authors declare that there is not any conflict of interests regarding the publication of this manuscript. In addition, the ethical issues, including plagiarism, informed consent, misconduct, data fabrication and/ or falsification, double publication and/or submission, and redundancy has been completely observed by the authors.

LIFE SCIENCE REPORTING

No life science threat was practiced in this research.

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