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# Effect of water-extraction on characteristics of melting and solidification of fly ash from municipal solid waste incinerator

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# ABSTRACT

This study was conducted to investigate the effect of water-extraction process on the removal of major elements and heavy metals in the fly ash from Municipal Solid Waste Incinerator (MSWI), and their thermal stability in the following melting process. The water-extraction was first applied to extract soluble elements and heavy metals from the fly ash from MSWI at different liquid-to-solid rates (L/S) of 2, 5, and 10, respectively. The extracted fly ash and the raw fly ash were then melted at the temperatures of 1000-1350 °C in an electrically heated furnace. The results showed that the compounds of Ca, Na, K, and Cl achieved high removal rates of 30.7-72.8% at L/S = 10, respectively. The water-extractable heavy metal with removal rate of 12.3% among the several heavy metals tested. The water-extracted fly ash had better stability as compared to raw one, which was indicated by lower weight loss and better immobilization ability of heavy metals such as Zn, Cu, and Pb in the melting process. The results showed that combing water-extraction and melting process could provide one of the alternatives for treating MSWI fly ash in China for reutilization.

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# 1. Introduction

Incineration is considered to be one of the effective treatment approaches for municipal solid waste (MSW) for its simultaneous energy recovery and pollution minimization. Municipal solid waste incineration (MSWI) has attracted increasing attentions recently in China due to site unavailability for landfill as well as rapidly increasing land price. A few cities, especially large ones like Beijing and Shanghai, are considering or already have constructed several MSWI facilities. However, MSW incineration also produces considerable amount of solid residues such as bottom ash, fly ash, and slag, which account for 30-35% weight (wt) of total MSW incinerated [1]. MSWI fly ash is enriched in various harmful components such as heavy metals, and dioxins, which could pose serious threats to environment and human health. MSWI fly ash is classified as hazardous waste according to Chinese regulations and needs to be appropriately pretreated before final disposal and reutilization. Currently, 400,000 tons of MSWI fly ash is generated in China annually and the amount will keep going up due to increasing number of incineration facilities constructed across China. This makes it essential to recover or recycle

MSWI fly ash for other purposes such as construction materials [2].

Ash melting process is one of the most promising technologies to stabilize MSWI fly ash, which is characterized by its capability to destroy dioxins and immobilize heavy metals into a glass matrix [3-6]. And the glass matrix could be used in glass-ceramics, landscaping or building materials [7-9]. However, ash melting process still suffers from the reputation of producing toxic gaseous by-products and having poor heavy metal stabilization, due to the presence of high alkali chloride and soluble metal salt contents [10]. Typically, chlorine contents between 10 and 20 wt.% were found in MSWI fly ash. Whereas, ordinary construction material requires low chlorine content, for instance, the chlorine content of Portland cement needs to be less than 0.02 wt.% to avoid corrosion and heavy metal leaching [11]. This implies that the high chlorine content in MSWI fly ash has to be removed before its recovery and reuse. Extraction is one of the common processes used as a pretreatment to remove soluble elements and heavy metals. Various extraction technologies have been used by different researchers [12-16], such as extracting with acid and/or alkaline, water followed by moderate phosphoric acid addition, and ferrous sulphate solution followed by ferrous sulphate oxidation. These extraction processes were reported to be able to remove most of the chlorides in the form of alkali chlorides such as NaCl, KCl, and CaCl, leachable salts, and amphoteric heavy metals from MSWI fly ash. Such





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extracted ash may limit the formation of metallic chlorides during its subsequent thermal treatment, hence reducing and eliminating the generation of toxic gases in the melting process [17].

In most cases in China, MSW is collected in a complex way and contains much more complicated components than those of the developed countries, resulting in the significant difference in physical and chemical characteristics of the MSWI fly ash from developed countries. Although considerable research has been conducted on extraction and melting or sintering technologies [18–22], little information is available for the application of such technologies for Chinese MSWI fly ash. Therefore, specific research needs to be carried out for Chinese MSWI fly ash with specific characteristics. This study was conducted to investigate the effect of the waterextraction process on the removal of major elements and heavy metals as well as their thermal stability in the following melting process; the aim was to explore the feasibility of integrating waterextraction and melting process to provide one of the alternatives for reuse and final disposal of Chinese MSWI fly ash.

# 2. Materials and methods

# 2.1. Materials

The fly ash used in this study was obtained from a MSW incineration plant in Shanghai city of China. The daily treatment capacity of the incinerator is 1200 tons. The incinerator is equipped with a cyclone, an adsorption reactor, and a fabric filter to control air pollution. The fly ash is captured by a bag-filter and removed twice a day. To ensure the representative samples, enough amounts of fly ash samples were taken for 20 days; and 10 kg of fly ash samples were taken once a day, total 200 kg of fly ash samples were collected in the period of 20 days. The samples taken in different days were then mixed homogeneously and stored for later analytical and experimental uses. When the test began, the fly ash samples were placed into a pan and dried in an oven at 105 °C for 24 h. After cooled down to ambient temperature, the sample was sieved by an auto sieve (Retch contol-g-200, Germany). Several batches of dried and sieved fly ash were put together, mixed and homogenized. The prepared samples were then kept in glass bottles for later use. The fly ash tested had a moisture content of 0.50%, and a loss-on-ignition of 3.75%. The major compositions of the fly ash samples were shown in the Table 1 (given by the form of oxides percent). The alkaline fly ash had a pH of 12.45 in a distilled water solution (L/S = 10), and a basicity (CaO/SiO<sub>2</sub>) of 0.78. The definition of basicity was given by Park and Hyun [23].

# 2.2. Experimental designs and methods

The fly ash was first water-extracted. Distilled water was used as extractant for water-extraction experiment. Different L/S ratios were employed by other researchers. Three L/S ratios of 2, 5, and 10 were used in this study. The ratios were determined based on the results by Wang and Zhang, who used L/S ratios of 2–100 and 2–20, respectively [20,24]. Extraction time of 120 min was applied for water washing process, which was determined by one of the our previous experiments. 100 g of fly ash was first put into the distilled water for extraction at a given L/S ratio and stirred in an agitation apparatus at a speed of 30 rpm. The mixture was separated by a vacuum filter. After a filter cake has been formed, it still contains

Table 1	
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Гhe major	compositions	of the fly ashes
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CaO	SiO <sub>2</sub>	$Al_2O_3$	Na <sub>2</sub> O	K <sub>2</sub> O	Fe <sub>2</sub> O <sub>3</sub>	Cl	SO <sub>3</sub>
16.4	20.7	6.79	16.1	7.25	3.66	16.0	6.73

a certain amount of leaching liquid that is trapped in the pores. Displacement washing is used to remove this remaining leaching liquid by washing it twice with distilled water. The resulting filter cake was oven dried at 105 °C for 24 h, then ground and kept in glass bottles. The extracted liquor was kept in a refrigerator at the temperature of 4 °C. The samples of extracted fly ash and extracted liquor were taken when analyses on major elements, heavy metals, and crystalline species were performed.

The melting experiment was performed in an electric-heated furnace for both water-extracted and raw fly ashes under the designed operational parameters, including melting temperature and melting time. The temperature inside the furnace was monitored by a thermocouple controlled by a programmed temperature controller. The same amount of 100 g sample (dry matter) was taken for both water-extracted fly ash and raw fly ash for each test. The samples were put into alumina crucibles for melting test in a furnace. The melting temperatures of 1000–1350 °C were used. The temperature increased at a heating rate of 5 °Cmin<sup>-1</sup>. One of the crucibles was taken out from the furnace at an interval of 50 °C, starting from 1000 °C until 1350 °C. The samples were cooled down slowly in air to ambient temperature, then analyzed for their major elemental components, heavy metal contents, and TCLP leaching test.

Each water-extraction and melting experiment were performed in three parallel runs, each datum presented in this study was the mean of three measurements.

# 2.3. Analyzing methods

The major components of the raw fly ash, the extracted fly ash and the melted slag were analyzed by X-ray fluorescence spectroscopy (XRF), and the crystalline species were identified with X-ray diffractometer (XRD) using Cu K $\alpha$  radiation and 2 $\theta$  [24]. The leaching tests were determined by toxicity characteristic leaching procedure (TCLP) testing, and extraction fluid #2 and L/S ratios of 20 were used in the test [25]. The raw and the melted fly ashes were digested with HNO<sub>3</sub>/HCIO<sub>4</sub>/HF according to USEPA SW3050 [26]. The heavy metals were analyzed with Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES). All the targeted elements of the extracted liquor, except for chloride, were analyzed with ICP-AES too. Chloride was determined by a volumetric method using a 0.1N silver nitrate solution and K<sub>2</sub>CrO<sub>4</sub> as an indicator.

#### 3. Results and discussion

#### 3.1. Water-extraction

# 3.1.1. Extraction of major elements and heavy metals

Three L/S ratios of 2, 5, and 10 were used to investigate the effect of water-extraction on the removal of major elements and heavy metals in the raw fly ash. The raw fly ash, the water-extracted fly ash, and the extracting solution were analyzed for the contents of the major elements and heavy metals. The mass balances of major elements were checked. The removed mass of each element and heavy metal was calculated based on the extracting solution volume and its concentration in the solution. The removal rate was calculated in terms of the mass in the initial fly ash and the mass extracted into the extracting solution obtained after filtration, but excluding the minimum mass of the elements and heavy metals remaining in the washed fly ash with the absorbed water. The results are presented in Table 2 (given by the form of elements). It could be seen that the elements in the MSWI fly ash exhibited three distinct leaching behaviors. Ca, Na, K, and Cl, showed stronger leaching abilities in water and reached high removal rates of 30.7%, 68.2%, 62.7%, and

Major element	Mass initial raw ash (g) (S.D.)	L/S = 2		L/S=5		L/S = 10	
		Mass in EW (g) (S.D.)	Removal rate (%)	Mass in EW (g) (S.D.)	Removal rate (%)	Mass in EW (g) (S.D.)	Removal rate (%)
Ca	16.25 (0.94)	2.07 (0.14)	12.70	3.05(0.17)	18.7	4.98(0.34)	30.7
Si	10.57 (0.84)	0.17 (0.02)	1.60	0.22(0.01)	2.00	0.34(0.01)	3.20
Al	3.79 (0.25)	0.09 (0.01)	2.40	0.11(0.01)	2.90	0.15(0.02)	4.00
Na	11.57 (0.51)	3.16 (0.56)	27.3	5.46(0.86)	47.2	7.89(0.14)	68.2
K	8.010 (0.75)	1.91 (0.09)	23.8	3.23 (0.56)	40.3	5.03 (0.43)	62.7
Fe	3.790 (0.36)	0.03 (0.01)	0.80	0.03 (0.01)	0.90	ND	-
Cl	19.72 (1.92)	7.24 (0.84)	36.70	9.31 (0.92)	47.2	14.36 (1.85)	72.8
S	3.160 (0.31)	0.10 (0.01)	3.20	0.13 (0.02)	4.10	0.16 (0.02)	5.00
Pb (10 <sup>-3</sup> )	211.1 (20.6)	0.16 (0.02)	0.10	0.39 (0.05)	0.20	0.51 (0.05)	0.20
$Cr(10^{-3})$	23.02 (3.26)	0.41 (0.04)	1.80	1.43 (0.15)	6.20	2.82 (0.34)	12.3
Cu (10 <sup>-3</sup> )	52.03 (7.37)	0.03 (0.008)	0.10	0.03 (0.01)	0.10	0.05 (0.01)	0.10
Zn (10 <sup>-3</sup> )	498.3 (36.8)	0.02 (0.004)	-	0.03 (0.01)	-	ND	-
Cd (10 <sup>-3</sup> )	4.74 (0.32)	ND	-	ND	-	ND	-

Table 2	
Effects of water-extraction on removals of major elements and heavy metals (based on initial fly ash of 100 g dry matter	er)

Note: S.D., standard deviation; EW, extracted water; ND, not detected.

72.8% at L/S = 10, respectively, Si, Al, and S were relatively hard to be extracted by water, which were represented by the low removal rates of 3.2%, 4.0%, and 5.0% at L/S = 10, respectively. The reason is that Si, Al, and S compounds are regarded as the nucleation ones and could form center matrixes of fly ash particles, making them hard to be transferred into water. Fe seemed not to be extractable in the water. This phenomenon basically agrees with the report from other researchers [18]. 12.3% Cr was removed and found to be easily extracted one among the heavy metals tested in the MSWI fly ash used in our test, and the other heavy metals showed very poor leachability with removal rate less than 1% at all three L/S ratios. It was also found that L/S = 10 achieved the highest removal rates of elements and heavy metals among the three L/S ratios tested. Thus, L/S = 10 was selected and used for all the water-extraction experiments in this study.

# 3.1.2. Analyses of crystalline species

The raw fly ash and the water-extracted fly ash were identified by X-ray diffraction (XRD) in order to get insight into the transferring characteristics of major components in the water-extraction process.  $SiO_2$ ,  $CaSO_4$ , KCl, and NaCl were found to exist in the form of crystal in the raw fly ash as shown in Fig. 1, while  $SiO_2$ ,  $CaSO_4$ , and  $Ca_2Al(AlSi)O_7$  were identified in the water-extracted fly ash (Fig. 2), and the diffraction angles and interplanar distance (d-spacing) in the XRD pictures of the fly ashes were shown in Table 3. No peaks of NaCl and KCl appeared in the water-extracted fly ash, indicating that the alkali chlorides in the raw fly ash were removed by the waterextraction process. The result was in agreement with the previous report [27].  $Ca_2Al(AlSi)O_7$  in the water-extracted fly ash might have



Fig. 1. The XRD patterns of the raw fly ash components.

been formed by recrystallization in the water-extraction process or fly ash sample drying.  $SiO_2$  and  $CaSO_4$  were found in both fly ashes, showing that the two components were not effectively removed through the water-extraction process. This could be explained by the fact that both components are less soluble and not easily transferred into water.

# 3.2. Melting and solidification

# 3.2.1. Weight loss

The water-extracted fly ash was melted to further stabilize in order to meet the requirements for safe utilization as construction materials or other purposes. The fly ash was unstable at high temperature, and its weight would decrease in the melting process. The raw fly ash and the water-extracted fly ash were melted at different temperatures to compare the difference in melting behavior. The results are shown in Fig. 3. As temperature went up, the weight losses of the both fly ashes increased gradually in the early stage and started to increase quickly after 1150 °C until the stable state was finally reached. However, the weight loss of the raw fly ash was significantly higher than that of the water-extracted one. The weight loss reached 14.6%, 30.3%, 36.8%, and 38.0% at the temperatures of 1150, 1200, 1250, and 1350 °C, respectively, for the raw fly ash, while 8.1%, 15.3%, 16.9%, and 17.6%, respectively, for the water-extracted fly ash at the corresponding temperatures. It appeared that the water-extracted fly ash had lower weight loss than the raw one in the melting process, which was mainly attributed to the removal



Fig. 2. The XRD patterns of the water-extracted fly ash components.

The diffraction angles and interplanar distances	(d_spacing) in the YPD	nictures of the fly ashes
The unitaction angles and interplanar distances	(u-spacing) in the ARD	pictures of the fly asiles

Crystalline species	SiO <sub>2</sub>	CaSO <sub>4</sub>	KCl	NaCl	CaAl(AlSi)O7
	20.63	25.52	28.39	27.47	25.48
Diffraction angles	26.68 50.17	31.40 40.54	40.54 50.17	31.73 45.45	26.68 28.01
	-	45.45	66.36	66.36	50.11
	4.242	3.488	3.141	3.244	3.493
d Spacing	3.338	2.846	2.224	2.817	3.338
u-spacing	1.872	2.224	1.872	1.994	3.183
	-	1.994	1.407	1.407	1.819



Fig. 3. Weight losses of raw fly ash and extracted fly ash over the temperature of  $1000{-}1350\,{}^\circ\text{C}.$ 

of some heavy metals and major components in water-extraction process. The result indicated that a water-extraction is effective in decreasing the production of volatile gases, thus reducing the pollution from melting process.

#### 3.2.2. Changes of major components

The changes of major elements in the raw fly ash and the waterextracted fly ash in the melting process are shown in Figs. 4 and 5, respectively. For the raw fly ash, the contents of Cl, K, and Na in the melted fly ash started to decrease remarkably until reached zero as the temperature went up to 1350 °C. This mainly resulted from the volatilization of Cl, K, and Na. The components of Ca, Si, and Al had lower volatilization, and their contents kept increasing in the whole melting process due to the high weight losses of Cl, K, and Na. Similar profiles of changes of major elements were found for the water-extracted fly ash. However, the volatilization amounts of Cl, K, and Na were significantly different between both fly ashes.



Fig. 4. Changes of main elements in the raw fly ash in the melting process.

For instance, for 100 g of raw fly ash, the volatilization amounts of Cl, Na, K, and S at the temperature of 1350 °C were 19.7, 11.6, 8.0, and 3.2 g, respectively. Whereas, for 100 g of extracted fly ash, the corresponding volatilization amounts were 5.4, 3.7, 3.0, and 3.2 g, respectively, which were significantly lower than the former's. It can be seen that the volatilizations of the elements of Cl, Na, K, and S were the main contributing factors for the weight loss of both fly ashes, but the higher amount of Cl, Na, K, and S in the raw fly ash led to more volatilization in the form of chloride and sulphates in the melting process. It is evident that the water-extraction was capable of pre-removing Cl, Na, K, and S, thus significantly reduced the generation of gaseous by-products and the pollution in the melting process.

#### 3.2.3. Immobilization of heavy metals

Heavy metals could be either immobilized in the melting body or volatilized in the melting process. It is expected to immobilize as much heavy metals as possible in order to minimize volatilization amount and air pollution. The samples of the raw fly ash and the water-extracted fly ash were melted at different temperatures (1000–1350 °C), and the analyses were made to evaluate the stability of the residual heavy metals in the melting process. The stability of heavy metals was determined in terms of immobilization rate, which was defined as follows:

$$K = \frac{m_2 c_2}{m_1 c_1} \times 100\%$$

where *K*: heavy metal immobilization rate (%);  $m_1$ : mass of the initial fly ash;  $m_2$ : mass of the melted fly ash (kg);  $c_1$ : heavy metal content of the initial fly ash;  $c_2$ : heavy metal content of the melted fly ash.

For both raw fly ash and water-extracted fly ash, the immobilization rates of Zn, Cu, Pb, and Cr decreased with the increase of temperature (Fig. 6), indicating that high temperature led to more



Fig. 5. Changes of main elements in the extracted fly ash in the melting process.



Fig. 6. Immobilization rates of different heavy metals in the melting process.

volatilization and thus had adverse effect on the immobilization of heavy metals, and the temperature control was critical for reducing volatilization of heavy metals. The significant difference between both fly ashes was the K values of Zn, Cu, and Pb at the temperature of 1350 °C (Fig. 6), which were 70.6%, 73.9%, and 58.1% in the water-extracted fly ash, respectively, obviously higher than 50.6%, 58.5%, and 48.5% in the raw fly ash, respectively. However, no evi-

dent difference in K value was found for Cr and Cd. The high heavy metal immobilization rate implies the low volatilization amount of heavy metals. The amount of heavy metals volatilized into gaseous by-products would decrease with the increase of immobilization rate. The mass balance calculation shows (Fig. 7) that for the same amount of 100 g of fly ash melted at the temperature of 1350 °C, the volatilization amounts of Zn, Cu, and Pb in the extracted fly

## Table 4

Comparisons on leachability of heavy metals in different fly ashes

Heavy metal	Raw fly ash	Melting temperature						
		Melted raw fly ash			Melted water-extracted fly ash			
		1000 (°C)	1200 (°C)	1350 (°C)	1000 (°C)	1200 (°C)	1350 (°C)	
Zn (mg/l)	28.27	10.65	10.04	5.68	10.43	9.65	5.63	
Cr (mg/l)	3.310	0.730	1.220	ND	0.700	1.24	0.05	
Pb (mg/l)	12.42	0.580	0.370	0.04	0.280	0.04	ND	
Cu (mg/l)	10.18	1.430	1.080	0.25	0.970	0.52	ND	
Cd (mg/l)	1.920	ND	ND	ND	ND	ND	ND	



Fig. 7. Mass balance of different heavy metals at  $1350\,^\circ\text{C}$  (based on initial 100 g fly ash).

ash decreased 99.6. 7.6. and 33.1 mg than those in the raw fly ash, respectively. The enhanced thermal stability of some heavy metals was believed to be due to the previous removal of alkali metals. The alkali metals and alkaline metal oxides such as Na, K, and Mg, known as network modifiers, could work against the primary oxides of network formers and result in breaking of the glass-forming bond and lowering of the melt viscosity [28]. As mentioned above, water-extraction process was capable of removing more than 60% of alkaline metal oxides at L/S of 10. The removal of such network modifiers suppressed heavy metal volatilization and enhanced the immobilization. Furthermore, more than 72% chlorides were also removed by the water-extraction, which also inhibited the formation of volatile metal chlorides and reduced their volatilization in the melting process. The result showed that water-extraction process could increase immobilization rate and enhance the melting stability of some heavy metals.

#### 3.2.4. Leachability of heavy metals

The raw fly ash, the melted raw fly ash, and the melted water-extracted fly ash were analyzed using Toxicity Characteristic Leaching Procedure (TCLP) to determine and compare their leachabilities. The results are shown in Table 4. It shows that the leachabilities of Zn, Cr, Pb, Cu, and Cd in the raw fly ash were significantly higher than those in the melted raw fly ash and the melted water-extracted fly ash, indicating that leachabilities of heavy metals could be greatly improved through the melting process. The lower leachabilities of Zn, Cr, Pb, and Cu in both melted fly ashes were due to the heavy metal ions replacing other ions and holding in the framework of glass [29]. Compared to the melted raw fly ash, the leachabilities of Zn, Cr, Pb, and Cu in the melted water-extracted fly ash were slightly lower, implying that the lower leachability of heavy metals were mainly affected by the melting process. Generally speaking, the leaching contents of the heavy metals decreased as temperature increased for both melted raw fly ash and melted water-extracted fly ash. The leaching contents of Pb, Cu, and Cd were below the detection limits when the temperature reached 1350°C.

# 4. Conclusions

Water-extraction proved to be effective in removing some components and heavy metals in MSWI fly ash. Ca, Na, K and Cl were removed by 30.7-72.8% at L/S = 10, Si, Al, S, and Fe were found hard to be extracted by water. Cr was the most extractable heavy metal among five heavy metals tested. As compared to the raw fly ash, the water-extracted fly ash had lower weight loss of components and heavy metals due to the pre-removal in the water-extraction process, thus significantly reducing the pollution of by-products generated in the melting process. The water-extracted fly ash had more thermal stability as indicated by higher immobilization ability of heavy metals in the melting process. The results suggested that integration of water-extraction and melting process could be one promising method for reuse and final disposal of MSWI fly ash.

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