Effects and mechanism of the conditions of sintering on heavy metal leaching characteristic in municipal solid waste incineration fly ash

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Abstract
Municipal solid waste incineration (MSWI) fly ash treated with toxicity metals holds enormous potential for construction use to economize on resources and protect environment. To reach the goal, this study investigated the effects of sintering conditions on leaching characteristic of heavy metals for MSWI fly ash, especially Cr, Cr\(^{6+}\), Ag, and Ba, with the orthogonal and Box-Behnken design experiment, which considered grain size (\(D_{50} = 30, 45, \) and \(60 \, \mu m\)), fluxing agent (\(CaO = 0, 2.5, \) and \(5\%\)), setting temperature (1000, 1050, and 1100 °C), and setting time (120, 180, and 240 min). The mechanism of immobilization for heavy metals was also discussed through the analyses of morphological characterizations, mineral phases, chemical composition, and leaching values of metals. The results indicated that changing grain size and adding fluxing agent of CaO have positive influence on reducing the leaching of heavy metals compared with direct sintering. The leaching values of As, Pb, Cd, Cu, Ni, Mn, Hg, Be, Se, and fluoride are not detected after sintering. Ideal sintering condition with desirability of 1.00 was predicted and optimized by the Box-Behnken response method in grain size of \(D_{50} = 30 \, \mu m\), fluxing agent of \(CaO = 5\%\), setting temperature = 1050 °C, and setting time = 180 min, which immobilized Cr, Cr\(^{6+}\), Ag, and Ba lower than the limitation of standards. Actual experiment was consistent with numerical optimization. Furthermore, the model of leaching characteristic for heavy metals in MSWI fly ash was established with the discussion on species distribution of heavy metals to better explain the mechanism during sintering.

Keywords MSWI fly ash · Heavy metal · Leaching · Sintering · Grain size · Fluxing agent · Setting temperature · Speciation fraction

Introduction
Incineration power generation plays a crucial role in municipal solid waste disposal structure. In China, the throughput of municipal solid waste incineration (MSWI) for harmlessness was 897,700 t per day, and the removal efficiency was 99.32% up to the end of 2020 as shown in China Ecological and Environment Bulletin, 2020. In the technological process of MSWI power plant as seen in Fig. 1, waste is burned in incinerator, meanwhile chemical energy is converted into electric energy, which contains a large quantity of MSWI fly ash from the flue gas purification and heat recovery system, accounting for 3–5% of the waste (Ji et al. 2022). Though bottom ash as the other by-product accounts for nearly 80% of all MSWI ash (Li et al. 2012), only the concentration of few heavy metals from MSWI bottom ash exceed the standard value; thus, MSWI bottom ash can be reused or disposed to landfills directly (Xiang et al. 2022; Sirico et al. 2022). Particularly, MSWI bottom ash can be sorted out to recycle into minerals, glass, ferrous metals, and non-ferrous metals, considered a lower greenhouse emissions than primary metals production (Gökelma et al. 2021), and becomes a secondary source of ferrous metals and non-ferrous metals (Šyc et al. 2020), while the management of MSWI fly ash is facing a great deal of challenges. The main components of MSWI fly ash include CaO, CI, Na\(_2\)O, K\(_2\)O, SO\(_3\), SiO\(_2\), and Al\(_2\)O\(_3\) (Luan et al. 2016), in the present, considered as a substitute for a part of the cement
binder. According to the research data, MSWI fly ash without treatment is used in mortar constitutes 5% of cement showing a greater compressive strength (Kirkelund et al. 2016). The proportioning of special concrete with MSWI fly ash is up to 20% in compressive strength of 43.73 MPa (Turullo and Mallisa 2018). Besides, dosage of MSWI fly ash in 30% as cement mixed in concrete was found that cementitious materials are generated leading to increasing of compressive strength (Zeng et al. 2020). However, MSWI fly ash is also an acknowledged hazardous waste formulated in the Directory of National Hazardous Wasted (Version 2021), causing harm to environment, human body, and the ecosystem due to the enriched in soluble chloride (Čarnogurská et al. 2015; Xu et al. 2022), the leaching of some heavy metals, such as Cd, Pb, Cr, and Zn (Alorro et al. 2009; Kirk et al. 2002; Mangialardi 2001; Mangialardi et al. 1999), and persistent toxic organic compounds, such as PAH, and PCDD/Fs (Min et al. 2018).

Consequently, to make the industrial waste more acceptable and keep resource conservation, treatment measure must be taken for MSWI fly ash in construction use. Different methods have been developed, including washing (De Casa et al. 2007; Qiu et al. 2019; Wang et al. 2016), chemical stabilization (Ma et al. 2019; Wang et al. 2015), and thermal treatment (Fujii et al. 2019; Ma et al. 2017; Peng et al. 2020). Several studies about the treatment have been performed in construction resource utilization and environmental coordination. Some sources indicated that MSWI fly ash under washing used in mortar helps to increase compressive strength compared with traditional mortar (Bie et al. 2016; Keppert et al. 2015). Melting is utilized in MSWI fly ash for producing clinker of Alinite cement (Wu et al. 2012a, b) and Calcium Sulfoaluminate cement (Guo et al. 2014).

To date, high temperature sintering as a way of thermal treatment has been regarded as one of the most effective measures for stabilizing harmful metals of MSWI fly ash (Soltanian et al. 2022; Shen et al. 2022). Besides, chloride salt has high thermal stability (Wang et al. 2022a, b), and transition into stable chemical forms during sintering (Wang et al. 2021). However, the effect about stabilization of sintering usually suffers from many factors, such as temperature, time, grain size, and fluxing agent. The solidification conditions are variant in heavy metals. Moreover, fewer efforts were made to illuminate the sintering mechanism and realistic prediction which is critical to get deeper insight into contaminant transport.

In this work, the migration and transition behavior of heavy metals for MSWI fly ash during the high temperature sintering process were investigated, and a series of experiments were carried out: (a) to work over the effects of grain size, fluxing agent, setting temperature, and setting time on the chloride’s distribution, and leaching of heavy metals; (b) to explore the optimum sintering conditions for the leaching of heavy metals lower than the limitation of standard during the sintering process; (c) to probe into the sintering and immobilization mechanism of heavy metals by characterizing crystalline phases, leaching concentration, and phase transformation. This study was expected to further understand the heavy metals existence form and transition behavior during the multiple factors sintering treatment process.

The rest of this paper is organized as follows: The materials, methods, and theory were introduced in Section 2. Morphological characterizations, chemical composition, mineral phase, and leaching of heavy metals analyses with the orthogonal and Box-Benhnken experiment for raw and sintered MSWI fly ash were discussed in Section 3. The model for sintering mechanism was established and discussed in Section 4. The conclusion was drawn in Section 5.

**Material and method**

**Materials**

The raw samples used for study were MSWI fly ash collected from Baiyun Waste Incineration Plant in
Guangzhou, Guangdong. The raw sample of MSWI fly ash was dried in electric drying oven at 105 °C for 8 h. The dried powder was used for dry ball milling without grinding aid in Planetary Ball Milling (JC-QM, Guangxi University) in 400 r/min to the target of grain size, and then mixed with CaO in alumina crucible for sintering. The process of sintering was in Muffle Furnace (SX2-10-12A, Guangxi University) with a heating rate of 10 °C/min. Besides, the cooling mode of sintered MSWI fly ash was natural cooling in Muffle Furnace.

The grain size distribution curves of MSWI fly ash in different ball milling time are shown in Fig. 2. MSWI fly ash can be classified as ML (silt with low liquidity) according to Standard for Engineering Classification of Soil, and is made up of spherical and smooth particles with $D_{50}$ of 60 μm and small pores. It also shows that ball milling makes no difference on grain size of MSWI fly ash after 45 s. The achievable minimum grain size of $D_{50}$ by dry grinding is 30 μm. As shown in Table 1, the primary chemical constituents of MSWI fly ash are noteworthy that, CaO, SiO$_2$, Na$_2$O, and K$_2$O take up about 60% jointly, yet SO$_3$ and Cl contents are over 30%. The chemical of CaO as fluxing agent in form of pellets with its analytical grade was purchased from a supplier of chemicals and reagents in Jiaxing, Zhejiang.

**Selection of variables**

The solid-state sintering is a method of powder densification under high temperatures without melting by proving surface energy and grain-boundary energy, including physical and chemical transformation. The production of crystal phase is the key to sintering (Wang et al. 2022a, b). The theory of enhancing driving force and forcing atomic motion of powders in sintering for crystallization was used when designing experiment. The chlorine and heavy metals can be surrounded firmly by other atoms after sintering theoretically (Bordia and Olevsky 2009; Kang 2005).

In this work, solid-state sintering of MSWI fly ash was affected by setting temperature, setting time, grain size of MSWI fly ash, and dosage of fluxing agent. Changing grain size of MSWI fly ash could be a pre-treatment to alter granular surface regularity and surface energy by ball milling. The other hand, ball milling is conducive to lattice defect by crushing and extrusion on MSWI fly ash with energy storage (Annenkov et al. 2017; Pourghahramani et al. 2008). The impact and induction of tensile and compressive from particles lead to the defects of the material structure, such as changes of the surface, lattice distortion, interfacial forces, and conversion of the range order (Guzzo et al. 2015; Pourghahramani and Forssberg 2006). The setting time and

![Grain size distribution analysis in different ball milling time](image)

**Table 1** Chemical composition of raw MSWI fly ash for experiment (mass, %)

|        | SiO$_2$ | Fe$_2$O$_3$ | Al$_2$O$_3$ | CaO | MgO | Na$_2$O | K$_2$O | SO$_3$ | Cl | P$_2$O$_5$ | TiO$_2$ | Other |
|--------|---------|-------------|-------------|------|-----|---------|-------|-------|----|---------|---------|-------|
| FA1    | 4.477   | 1.467       | 1.581       | 42.319 | 1.699 | 9.497   | 5.721 | 7.499 | 21.525 | 1.378   | 0.953 | 1.884 |
| FA2    | 4.808   | 1.816       | 1.028       | 41.547 | 0.086 | 7.013   | 6.79 | 6.99  | 26.503 | 0.266   | 1.337 | 1.816 |
| FA3    | 2.168   | 1.56        | 1.102       | 45.58 | 1.132 | 7.359   | 6.562 | 5.772 | 25.431 | 0.559   | 0.897 | 1.878 |

FA in different numbers indicates that MSWI fly ash is collected in different dates.
setting temperature were related to the diffusion process of atoms in MSWI fly ash, influencing the effect of crystallization. Fluxing agent was used to change temperature and efficiency of sintering, and to diversify type and structure of crystalline phase.

**Orthogonal and Box-Benhnken experiment methods**

In this study, all the experiments for MSWI fly ash were performed according to the orthogonal and Box-Benhnken design. Four factors were to be considered in this work, which were grain size of MSWI fly ash, dosage of fluxing agent, setting temperature, and setting time. To optimize the test, orthogonal test was applied to preliminary find out the effects of the four factors on content of chloride and the leaching of heavy metals, to provide a series of detection guiding for sintering mechanism and process, and to identify some heavy metals whose leaching value tending to zero or being far below standards (Germanta and Seberry 1979). The Box-Benhnken response surface design was applied to analyze the influence of the four factors on leaching characteristic for subsequent analysis of residual heavy metals and chlorine, and to predict the optimal value and the corresponding conditions for the test.

According to the orthogonal tables, four factors were to be considered in three level. L₉(3⁴) orthogonal table was applied in this paper shown in Table 2. The sintering temperature is 0.8–0.9 times that of melting temperature of materials (Marfunin 1979). In addition, the sintering setting temperature was considered: 1000, 1050, or 1100 °C. The raw MSWI fly ash was heated with setting time (120, 180, or 240 min) at the target temperature. Grain size was concentrated in D₅₀ of 30, 45, or 60 μm by the method of dry ball-milling. The pellets of CaO were chosen as fluxing agent with the ratio of 0, 2.5, or 5%, by considering the effect of chemical composition of MSWI fly ash.

| No | Factor      | Grain size of D₅₀ (μm) | Dosage of fluxing agent (%) | Setting temperature (°C) | Setting time (min) |
|----|-------------|------------------------|----------------------------|--------------------------|-------------------|
| S1 |             | 60                     | 0                          | 1000                     | 120               |
| S2 |             | 45                     | 0                          | 1050                     | 180               |
| S3 |             | 30                     | 0                          | 1100                     | 240               |
| S4 |             | 60                     | 2.5                        | 1050                     | 240               |
| S5 |             | 45                     | 2.5                        | 1100                     | 120               |
| S6 |             | 30                     | 2.5                        | 1000                     | 180               |
| S7 |             | 60                     | 5                          | 1100                     | 180               |
| S8 |             | 45                     | 5                          | 1000                     | 240               |
| S9 |             | 30                     | 5                          | 1050                     | 120               |

When it comes to the Box-Benhnken response surface table, a total of 27 experiments with four variables at three different levels were used, which were chosen as the same as orthogonal design. It can be seen from the Table 3 that three center points per block was adopted in the test.

**Testing methods**

**Particle size test**

To explore a target field of grain size of MSWI fly ash in different time under dry ball milling, laser particle size analyzer (MAZ 3000, Malvern, UK) was carried out for the samples with various ball milling time (0, 15, 25, 30, 45, or 60 s). The samples were measured by the theory of Michelson and Franhoff. The bulk density of MSWI fly ash was tested by powder heap densimeter (FT-100E, Rooko, China) according to Methods of Dust Character Test (GB/T 16,913–2008).

**Micro-structure test**

The mineral structure, crystallinity, and phase transformation of raw MSWI fly ash and sintered MSWI fly ash were identified X-ray diffraction (XRD) (Ultima IV, Rigaku, Japan). They were analyzed over a range of 10–80° (2θ) at the scanning speed of 4°/min. The measured results were identified by comparing with the standard powder diffraction database of the International Centre for Diffraction Data (ICDD PDF-2 Release 2008). The microstructure and elements distribution of MSWI fly ash were observed by scanning electron microscopy (SEM) (S-3400 N, Hitachi, Japan) and energy-dispersive spectrometer (EDS) (E8-SPR, Heleex, China). The chemical composition of MSWI fly ash was measured by X-ray fluorescence (XRF) (S8 TIGER, Bruker, German). The concentration of water-soluble chlorine of MSWI fly ash was tested by ion chromatograph (IC) (ICS-5000, Dionex, USA), according to Solid Wastes-Determination of Fluoride, Bromate, Chloride, Nitrite, Cyanate, Bromide, Nitrate, Phosphate and Sulfate-Ion Chromatography from Identification Standards for Hazardous Wastes-Identification for Extraction Toxicity (GB 5085.3–2007). Electron binding energy and chemical shift were measured by X-ray photoelectron spectroscopy (XPS) (ESCALAB 250XI, Thermo Fisher, USA).

| No | Factor      | Grain size of D₅₀ (μm) | Dosage of fluxing agent (%) | Setting temperature (°C) | Setting time (min) |
|----|-------------|------------------------|----------------------------|--------------------------|-------------------|
|    | A           | Grain size of D₅₀ (μm) | 30                         | 60                       |
|    | B           | Dosage of fluxing agent (%) | 0                         | 5                        |
|    | C           | Setting temperature (°C) | 1000                      | 1100                     |
|    | D           | Setting time (min) | 120                       | 240                      |

Table 2 Orthogonal experiment design of L₉(3⁴)

Table 3 The Box-Benhnken experiment design
Heavy metals leaching characteristics test

The leaching characteristics of fourteen heavy metals were analyzed conforming to Solid Waste-Extraction Procedure for Leaching Toxicity-Sulphuric Acid & Nitric Acid Method HJ/T 299, CN. Extraction liquid was used in pH 3.2 as a leaching solution. The samples were soaked for 18 h with liquid/solid ratio of 10 mL/g. The leaching concentrations of heavy metals were measured by inductively coupled plasma-mass spectrometry (ICP-MS) (NexION 2000, Perkin Elmer, USA). The species distributions of heavy metals in MSWI fly ash were explored on the basis of BCR sequential extraction method dividing into four fractions as acid soluble, reducible, oxidable, and residual fraction (Xie et al. 2020; Usero et al. 1998). The extractions of speciation for heavy metals were tested by inductively coupled plasma-optical emission spectrometry (ICP-OES) (Optima 8000, Perkin Elmer, USA). The same sample was tested for three times, and the average value of the three results was used to evaluate the leaching characteristic of heavy metals. Moreover, the blind test for all samples was taken to ensure the reliability of the results.

Results and discussions

Morphological characterizations analysis

The samples in Fig. 3 of different grain sizes show that MSWI fly ash under ball milling increased with bulk density in two grain size of D_{50} (60 and 30 μm). Particles change into powders from granules. In addition, SEM results in Fig. 4 of raw MSWI fly ash in two grain size of D_{50} (60 and 30 μm) supported the hypothesis of original experiment design that ball milling only changes grain size and distribution by crushing and extrusion on MSWI fly ash without impacting on micromorphology. The results showed that the surface of raw MSWI fly ash particles is surrounded by floculent and globosity with high porosity.

According to bulk density test, the average bulk density of raw MSWI fly ash is 0.8 ± 0.05 g/cm³, while the range of bulk density of sintered MSWI fly ash is from 2.17 to 2.69 g/cm³. The samples of sintered MSWI fly ash in S1, S4, and S7 conditions shown in Fig. 5 are in agreement with the results of bulk density test. Comparing with raw sample, it was observable that sintered MSWI fly ash bulk density grows
up and the gap of particles is dropped. The color has noticeable change from gray to malachite green, which suggests to the formation of new minerals. The external morphology of sintered MSWI fly ash in S2, S6, and S9 conditions was studied by SEM, as shown in Fig. 6. The results revealed that treatment significantly affects the microstructure of the MSWI fly ash. Sintering causes particles to become lower porosity and to weld together forming a dense structure. In previous studies, sintered coal fly ash can be seen many small granular hydroxy-sodalite aggregates on the surface with SEM (Luo et al. 2018). Red mud-fly ash is formed a mass of bar-structure from spherical particles under sintering (Samal et al. 2015). After sintering, the spherical particles of coal fly ash disappeared (Wu et al. 2014). In addition, sintering leads to dramatic particles aggregation with bar or rod-shape in microstructure, as a result of formation and growth of crystal nucleus. The raised surfaces indicated that directional crystals grow at fracture. The upturn of crystallinity helps to improve bulk density and mechanical properties. The migration and motion rate of atomics build up by enhancing driving force during sintering.

### Chemical composition and XRD analysis

The chemical compositions tested with XRF of raw MSWI fly ash and sintered MSWI fly ash under different condition in orthogonal design are given in Fig. 7. It can be seen that CaO, SiO₂, Al₂O₃, SO₃, and Cl are the main compositions for sintered MSWI fly ash, which is notable differences with raw MSWI fly ash. The chemical compositions of sintered MSWI fly ash are under different group remain level. The content of CaO, SiO₂, and Al₂O₃ all increased to 50.16%, 11.96%, and 5.25% from 41.55%, 4.81%, and 1.03%, respectively. Sintering contributes to optimize compositions of MSWI fly ash for construction use as cement, because of more similar components. CaO is beneficial to the growth of crystal with high value, while, Na₂O, K₂O, and Cl for sintered MSWI fly ash were all lower than raw MSWI fly ash. Particularly, the percentage of Cl nearly fell from 26.50 to 12.74%, which provides the evidence that sintering helps to reduce the content of Cl being harmful to cement (Chen et al. 2016). In addition, the content of chlorine in MSWI fly ash can be determined by water-soluble chlorine and

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**Fig. 5** Images for the sintered MSWI fly ash in S1, S4, and S7 conditions

**Fig. 6** SEM images analysis for sintered MSWI fly ash in S2, S6, and S9 conditions
non-water-soluble chlorine (Hwang et al. 2006), and water-soluble chlorine (Gao et al. 2022). Thus, the content of water-soluble chlorine in MSWI fly ash should be limited under 2% when using for concrete, which is in light of the requirements stipulated in the Technical Specification for Pollution Control of Fly-Ash from Municipal Solid Waste Incineration (HJ 1134–2020). The results of the content of water-soluble chlorine in MSWI fly ash under sintering are shown in Fig. 8. The contents of water-soluble chlorine in sintered MSWI fly ash were lower than 2% except for S1, while, whose value of raw MSWI fly ash was much higher than sintered MSWI fly ash, which suggests the transformation of chlorine from water-soluble salts to non-water-soluble chlorine salts or stable chlorine-containing minerals during sintering.

Figure 9 demonstrates the XRD patterns of raw and sintered MSWI fly ash are under various orthogonal conditions. It was discerned that the main differences in the raw and sintered samples were the variation in mineral peak intensity. By comparison with raw MSWI fly ash, the relative contents of CaCO₃ and CaClOH declined and even disappeared as the results that CaCO₃ and CaClOH decomposing at high temperature during sintering. It can be well explained as the change of chemical composition on CaO and Cl, whereas the peak intensity of CaTiO₃, MgO, and Ca₁₂Al₁₄O₃₃ increased or occurred. This phenomenon supports the analysis of SEM that crystallinity and intensity of the crystal phases increased in degree by sintering. Ca₁₂Al₁₄O₃₃ is created by the solid-state reaction with CaO and Al₂O₃ (Rudradawong et al. 2020), which belongs to orthorhombic crystal with cyan-gray Ng and blue-green Np surface, which is in good explication with sintered MSWI fly ash images that the noticeable change of color from gray to malachite green.

The content of soluble chloride salts as NaCl and KCl in sintered MSWI fly ash was reduced by 50% nearly comparing raw samples in Fig. 9. However, Ca₅(PO₄)₃Cl raised practically four times under sintering. The results showed that a part of Cl is immobilized as a stable mineral reducing the possibility of escaping, which are in accord with the previous analysis in Figs. 7 and 8. Moreover, the other Cl is attached on the sintered MSWI fly ash particles as water-soluble chloride salts. Thus, sintering is limited to remove
the whole water-soluble chlorine in this study, especially in a sharper limit to reinforced concrete, which may be taken off by washing for those soluble chloride salts.

Figure 10 shows the change for crystallinity of raw and sintered MSWI fly ash. The value with a mean of 89.78% under sintering, while raw MSWI fly ash only is 65.93%. This result provided specific evidence that the treatment in this study helps MSWI fly ash to improve the growing of crystals and reduce the leaching of heavy metals. It is worth notice that the value of group S8 is 99.99% which means a greatly tight structure in particles.

**Leaching of toxicity metals analysis**

The leaching performances of sintered MSWI fly ash were evaluated using toxicity threshold included in the standard of Identification Standards for Hazardous Wastes-Identification for Extraction Toxicity (GB 5085.3–2007), Integrated wastewater Discharge Standard (GB 8978–1996), and Technical Specification for Coprocessing of Solid Waste in Cement Kiln (GB 30,760–2014). All the results were summarized in Table 4. It is shown that leaching characteristics of toxicity elements in the most orthogonal samples are much lower than the standard limits. Specifically, the leaching values of As, Pb, Cu, Mn, Hg, Be, Se, and Fluoride were even not detected, indicating that proposed sintering methods have immobilization contribution on the most toxicity elements. In spite that, the effects of sintering design have some limitations in reducing the leaching of Cr, Cr$^{6+}$ Ag, and Ba. In group S2 and S3, the immobilization of Cr is only 25.74% and 19.68%, respectively. Even the leaching of Cr$^{6+}$ increased from 5.54 to 6.5 mg/L. The result showed that As, Pb, Cd, Cu, Ni, Zn, Mn, Hg, Be, Se, and fluoride barely indicate leaching characteristic in orthogonal test.

**Table 4 Orthogonal test table for sintering experiment**

| No | Leaching of toxicity metal elements (mg/L) |
|----|------------------------------------------|
|    | As  | Pb  | Cd  | Cr  | Cu  | Ni  | Zn  | Mn  | Hg  | Cr$^{6+}$ | Be  | Ag  | Ba  | Se  | Fluoride |
|----|-----|-----|-----|-----|-----|-----|-----|-----|-----|----------|-----|-----|-----|-----|----------|
| FA | 0.41| 0.64| 0.04| 8.74| 0.54| 0.29| 2.4 | 1.5 | 0.07| 5.54     | 0.008| 0.94| 115 | 1.65| 0.65     |
| S1 | ND  | ND  | 0.01| 2.93| ND  | ND  | ND  | ND  | ND  | 1.85     | ND  | 0.62| 13.45| ND  | ND       |
| S2 | ND  | ND  | 0.03| 2.49| ND  | ND  | 0.3 | ND  | 0.02| 2.5      | ND  | 0.59| 5.29 | ND  | ND       |
| S3 | ND  | ND  | 0.02| 3.15| ND  | ND  | 0.02| ND  | ND  | 1.45     | ND  | 0.41| 4.22 | ND  | ND       |
| S4 | ND  | ND  | 0.29| 0.38| ND  | ND  | 0.12| 0.01| ND  | 1.612    | ND  | 0.28| 34.4 | ND  | ND       |
| S5 | ND  | ND  | 0.02| 0.68| ND  | ND  | 0.01| ND  | ND  | 0.323    | ND  | 0.18| 10.72| ND  | ND       |
| S6 | ND  | ND  | 0.89| ND  | ND  | ND  | 0.19| ND  | ND  | 0.78     | ND  | 0.23| 26.8 | ND  | ND       |
| S7 | ND  | ND  | 0.03| 0.26| ND  | ND  | 0.23| ND  | ND  | 0.289    | ND  | 0.36| 38.3 | ND  | ND       |
| S8 | ND  | ND  | 0.03| 0.65| ND  | ND  | 0.18| ND  | ND  | 0.759    | ND  | 0.36| 38.3 | ND  | ND       |
| S9 | ND  | ND  | 0.04| 0.28| ND  | ND  | 0.22| ND  | ND  | 0.254    | ND  | 0.16| 39.7 | ND  | ND       |
| ST | 0.1 | 0.3 | 0.03| 0.2 | 0.5 | 0.2 | 1   | 1   | 0.05| 0.5      | 0.005| 0.5 | 100 | 1   | 100      |

ND indicates not detected, ST indicates standard thresholds
Response surface of the Box-Benhnken analysis

The leaching of Cr, Cr$^{6+}$, Ag, and Ba was chosen to investigate with the Box-Benhnken response surface method for further revealing the influence of four factors on above toxicity elements, and calculating optimal value and experiment design with sintered MSWI fly ash. It was considered that residual toxicity elements can be greatly restricted in crystal phase according to the results of orthogonal experiment. The findings as shown in Table 5 provide substantial evidence for four variables having a major impact on the immobilized toxicity metals.

Effect of grain size

The experiment results were analyzed with response surface plots and ANOVA of grain size for model, which indicates grain size has influence on the leaching of Cr, Ba, and Cr$^{6+}$ whose $P$-values less than 0.05 are significant, yet $P$-value of Ag is greater than 0.1 being not significant. Figure 11 is represented the regression model demonstrating the impact of grain size on toxicity metals when fluxing agent, setting temperature, and setting time is 2.5%, 1050 °C, and 180 min, respectively. It can be seen that Ag leaching remains level with grain size. However, Ba leaching showed significant changes. The leaching of Ba, and Cr$^{6+}$ increased as grain size up while Cr decreased steadily.

Effect of fluxing agent

The analysis depicts that fluxing agent of CaO makes marked impression on immobilization of all toxicity metals in the experiment. As can be seen from Fig. 12, great changes have taken place in leaching of Ba, Cr, and Cr$^{6+}$ with grain size, setting temperature, and setting time is $D_{50} = 45$ μm, 1050 °C, and 180 min, respectively. Surprisingly, Ba leaching sharply went up with content of CaO rising between 0 and 1%. Then, the rate of increase slows down after 1%. The situation nearly reached a peak of 4%. The adding of CaO

| No | Factor | Grain size of $D_{50}$ (μm) | Dosage of fluxing agent (%) | Setting temperature (°C) | Setting time (min) | Leaching characteristics of heavy metals (mg/L) |
|----|--------|-----------------------------|-----------------------------|--------------------------|-------------------|-----------------------------------------------|
|    |        |                             |                             |                          |                   | Cr    | Ag    | Ba    | Cr$^{6+}$ |
| 1  | 45     | 5                           | 1050                        | 240                      |                   | 0.322 | 0.1   | 26.5  | 0.803     |
| 2  | 30     | 2.5                         | 1100                        | 180                      |                   | 1.08  | 0.091 | 19.8  | 0.834     |
| 3  | 45     | 0                           | 1100                        | 180                      |                   | 2.83  | 0.47  | 5.4   | 3.21      |
| 4  | 60     | 5                           | 1050                        | 180                      |                   | 0.165 | 0.16  | 39.2  | 0.345     |
| 5  | 60     | 2.5                         | 1000                        | 180                      |                   | 0.25  | 0.58  | 32.1  | 0.732     |
| 6  | 45     | 2.5                         | 1100                        | 120                      |                   | 0.56  | 0.22  | 25.6  | 1.32      |
| 7  | 60     | 2.5                         | 1050                        | 240                      |                   | 0.34  | 0.493 | 30.4  | 1.03      |
| 8  | 45     | 2.5                         | 1050                        | 180                      |                   | 0.446 | 0.312 | 30.4  | 0.476     |
| 9  | 45     | 5                           | 1050                        | 120                      |                   | 0.22  | 0.23  | 32.9  | 0.345     |
| 10 | 45     | 2.5                         | 1050                        | 180                      |                   | 0.435 | 0.451 | 29.4  | 0.443     |
| 11 | 60     | 2.5                         | 1100                        | 180                      |                   | 0.287 | 0.21  | 32.6  | 1.98      |
| 12 | 30     | 2.5                         | 1000                        | 180                      |                   | 0.82  | 0.57  | 27.5  | 0.367     |
| 13 | 45     | 5                           | 1100                        | 180                      |                   | 0.73  | 0.05  | 27.3  | 0.892     |
| 14 | 45     | 0                           | 1000                        | 180                      |                   | 2.1   | 0.72  | 6.87  | 2.26      |
| 15 | 45     | 2.5                         | 1050                        | 180                      |                   | 0.58  | 0.45  | 26.9  | 0.623     |
| 16 | 45     | 0                           | 1050                        | 240                      |                   | 2.49  | 0.66  | 3.6   | 2.9       |
| 17 | 60     | 0                           | 1050                        | 180                      |                   | 0.9   | 0.59  | 11.2  | 2.25      |
| 18 | 30     | 2.5                         | 1050                        | 240                      |                   | 0.764 | 0.222 | 24.1  | 0.786     |
| 19 | 60     | 2.5                         | 1050                        | 120                      |                   | 0.241 | 0.47  | 34.2  | 0.98      |
| 20 | 45     | 2.5                         | 1000                        | 120                      |                   | 0.598 | 0.56  | 27.9  | 0.562     |
| 21 | 45     | 5                           | 1000                        | 180                      |                   | 0.65  | 0.283 | 34.7  | 0.432     |
| 22 | 30     | 0                           | 1050                        | 180                      |                   | 3.123 | 0.621 | 7.9   | 1.65      |
| 23 | 45     | 0                           | 1050                        | 120                      |                   | 1.42  | 0.692 | 4.12  | 1.72      |
| 24 | 45     | 2.5                         | 1000                        | 240                      |                   | 0.61  | 0.554 | 28.3  | 0.98      |
| 25 | 30     | 2.5                         | 1050                        | 120                      |                   | 0.732 | 0.481 | 21.3  | 0.34      |
| 26 | 30     | 5                           | 1050                        | 180                      |                   | 0.2   | 0.148 | 33.2  | 0.423     |
| 27 | 45     | 2.5                         | 1100                        | 240                      |                   | 0.59  | 0.23  | 20.6  | 1.45      |
as fluxing agent helps to immobilize Ag, Cr, and Cr\(^{6+}\), but it is limited to Cr and Cr\(^{6+}\) in the number of 4.5% for CaO.

**Effect of setting temperature**

As is illustrated in the response surface plots, setting temperature has influence on Ag, Ba, and Cr\(^{6+}\) leaching when grain size, fluxing agent, and setting time is \(D_{50} = 45\ \mu\text{m}, 2.5\%\), and 180 min, respectively. The tendency of heavy metals leaching is proved in Fig. 13. Ag and Ba leaching were gradual decline in higher temperature which are in contrast to Cr\(^{6+}\). Ba is the most sensitive to temperature.

Figure 14 is used to explore interaction between fluxing agent and setting temperature on Cr\(^{6+}\), Cr, and Ba by 3D surface mode, which grain size of \(D_{50}\) is 45 \(\mu\text{m}\), and setting time is 180 min. The leaching of Cr\(^{6+}\) and Cr showed an extreme influence on interaction rather than one factor. Therefore, it was decreased when adding dosage of CaO and receding the temperature at the same time. However, Ba leaching values were control with interaction approaching one factor for fluxing agent of CaO. The trends of change for three metals are similar.

**Effect of setting time**

Figure 15 clearly shows that setting time only has significant influence on immobilization of Cr\(^{6+}\) with grain size, fluxing agent, and setting temperature being \(D_{50} = 45\ \mu\text{m}, 2.5\%\), and 1050 °C, respectively, and proves that high temperature has negative impact on immobilization of Cr\(^{6+}\). Especially, Cr\(^{6+}\) leaching showed a steep growing after 1035 °C.

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**Fig. 11** Response surface plots of one factor in grain size of \(D_{50}\) for Cr, Cr\(^{6+}\), Ag, and Ba leaching when fluxing agent, setting temperature, and setting time is 2.5%, 1050 °C, and 180 min, respectively

**Fig. 12** Response surface plots of one factor in fluxing agent of CaO for Cr, Cr\(^{6+}\), Ag, and Ba leaching when grain size, setting temperature, and setting time is \(D_{50} = 45\ \mu\text{m}\), 1050 °C, and 180 min, respectively

**Fig. 13** Response surface plots of one factor in setting temperature for Cr, Cr\(^{6+}\), Ag, and Ba leaching when grain size, fluxing agent, and setting time is \(D_{50} = 45\ \mu\text{m}, 2.5\%\), and 180 min, respectively
Figure 16 shows the interaction of fluxing agent of CaO and setting time on Cr$^{6+}$, Cr, and Ba in 3D model when grain size of $D_{50}$ and setting temperature are set up in 45 μm, and 1050 °C, respectively. The tendency is like the results of the interaction between fluxing agent and setting temperature. However, the trend for change of Cr and Cr$^{6+}$ was slow down.

Optimal prediction analysis

The model condition was predicted response by desirability function in the Box-Behnken response surface. The leaching of heavy metals as functions was set for the goal under the standard limitation for numerical optimization, which was used to calculate the highest desirability and to design the subsequent experiment. In this work, the ideal selection focused on a desirability of 1.00, and the reduction of energy consumption. Thus, the optimal condition is grain size of $D_{50}$ = 30 μm, fluxing agent of CaO = 5%, setting temperature = 1050 °C, and setting time = 180 min, resulting in the leaching of Cr, Ag, Ba, and Cr$^{6+}$ is 0.121, 0.131, 30.25, and 0.266, respectively. The theoretical variables were compared with experiment in three tests with average results.

**Fig. 14** Response surface plots of the interactions between fluxing agent of CaO and setting temperature for Cr, Cr$^{6+}$, and Ba leaching

**Fig. 15** Response surface plots of one factor in setting time for Cr, Cr$^{6+}$, Ag, and Ba leaching when grain size, fluxing agent, and setting temperature being $D_{50}$ = 45 μm, 2.5%, and 1050 °C, respectively

**Fig. 16** Response surface plots of the interactions between fluxing agent of CaO and setting time for Cr, Cr$^{6+}$, and Ba leaching
being shown in Table 6. The experimental results supported the theoretical optimization that the average is observed to be 0.129, 0.16, 31.51, and 0.23, respectively, which implies that the Box-Behnken response surface contributes to optimize the sintering process.

**Sintering mechanism of MSWI fly ash**

In light of the above analysis, the sintering mechanism of MSWI fly ash in this study was drawn by the analyses of EDS, XPS, and ICP-OES, which was explored on the discussion of sintering mechanism on Cr, Ag, and Ba. The raw MSWI fly ash are compared with sintered MSWI fly ash in the optimal condition ($D_{30}=30\ \mu m$, $CaO=5\%$, setting temperature $=1050\ °C$, and setting time $=180\ min$).

The analysis of EDS helps to measure the heavy metal distributions of the particles and the changes of atomic ratio of heavy metals in MSWI fly ash. The results of SEM–EDS map scanning are shown in Fig. 17. The heavy metals of Cr, Ag, and Ba were gathered and uniformly dispersed in the particles of sintered MSWI fly ash. Besides, the atomic ratio of Cr, Ag, and Ba from surface area of sintered MSWI fly ash increased, especially Ag of $57.09\%$ and Ba of $44.36\%$, compared to raw MSWI fly ash, which declared that the heavy metals are solidified in the particles during sintering.

However, the mechanism of sintering immobilization can be a chemical or physical process (Zhao et al. 2022); thus, XPS was analyzed to explore the mechanism of sintering immobilization in MSWI fly ash. The survey XPS spectrum (Fig. 18) shows the distribution of elements and the changes of the intensity of the spectra in counts per second in the same peaks. The major peaks noted in the survey spectrum were equivalent to O 1 s, Ca 2p, C 1 s, and Cl 2p. Though the most peaks of heavy metals were weak, and some even are undetected. It is clear that the same peak of the majority heavy metals in different samples appeared the increase or decrease of the intensity of the spectra in counts per second, which confirmed that the mechanism of sintering immobilization should be caused by the electronic transition in elements and mainly a chemical process. The MSWI fly ash was treated with direct heating for sintering accumulating limited internal energy. Therefore, the results of crystallinity and solidification for toxicity metals were shown a low performance. Crystal was formed in geometry as rules spontaneously when temperature up in a period by breaking bonds. Rising temperature provides thermal energy for internal motif to gain kinetic energy (Pan et al. 2020). The particles were formed from other aggregation structures to crystalline state, which storage the least internal energy and possess the most stable shape. On the other hand, the physical process played an assistant role with the overlap of peaks at a low change for the mechanism of sintering immobilization, which also can be proved in the above analysis of SEM images that the particles of sintered MSWI fly ash became lower porosity and to weld together forming a dense structure. At the same time, arrangement space was sharply reduced for whole atoms as the result of density decreasing as characterization. In addition, solid materials were transferred and interchanged due to the dual effects of physics and chemistry, exposing the shortage with an immediate heating.

It is hard to measure the heavy metals in MSWI fly ash, because of the low contents, and to confirm the chemical compositions and process with heavy metals during sintering, while, which is the key to understand the mechanism of sintering immobilization. Therefore, the measurement of the chemical phases and speciation in heavy metals helped to analyze the mechanism of sintering in MSWI fly ash, according to the determination of percentages of each species with ICP-OES analysis using the BCR sequential extraction method (Xie et al. 2020; Usero et al. 1998), as shown in Fig. 19. Four types of fractions and distribution of heavy metals are acid soluble (carbonate bound), reducible (Fe–Mn oxide bound), oxidizable, and residual fraction. Heavy metals in acid soluble fraction are easy to leach in acid environment as carbonate minerals (Gao et al. 2014). In reducible fraction, heavy metals can be combined with Fe–Mn oxide bound types and limited about its leaching under oxidizing environment (Taylor 1997). Oxidizable heavy metals species are more steady but damaged under dramatic oxidative conditions. Specially, residual species is the most stable and hardly affected by environment, whose heavy metals exist in silicate, primary minerals, and secondary minerals (Gao et al. 2010).

The results showed that acid soluble and residual fraction of Cr in raw MSWI fly ash accounted for 41.6% and 35.5%, respectively. Thus, the leaching characteristic of raw MSWI fly ash is active, while the Cr in sintered MSWI fly ash was mainly concentrated in residual, reducible, and oxidizable fraction accounted for 44.1%, 22.3%, and 20.4%, respectively. Sintering shaken down the leaching characteristic of MSWI fly ash through decreasing acid soluble fraction and especially increasing residual fraction. Cr is in the form of $Cr^{3+}$ and $Cr^{6+}$ in MSWI fly ash, and $Cr^{6+}$ can be reduced as $Cr^{3+}$ with a low boiling point by $CO_2$ from decomposition of $CaCO_3$ at high temperature during sintering, which lead to

| Toxicity metals | Optimal value | Experimental value in average |
|-----------------|--------------|------------------------------|
| Cr              | 0.121        | 0.129                        |
| Ag              | 0.131        | 0.16                         |
| Ba              | 30.25        | 31.51                        |
| $Cr^{6+}$       | 0.266        | 0.23                         |
the increase of residual fraction. Besides, CaO can promote the reaction with Cr and the formation of stable chemical composition as residual fraction, such as CaCr$_2$O$_4$ (Chen et al. 2021), which was in agreement with Fig. 12 that the agent of CaO has positive on the sintering immobilization of Cr in MSWI fly ash. When it comes to grain size of $D_{50}$, the leaching characteristic of Cr is different to Cr$^{6+}$, Ag, and Ba, and increased as grain size of $D_{50}$, decreasing as shown in Fig. 11, which was reported that the grain size of $D_{50}$ has a negative influence on residual fraction of Cr caused the adsorption of fine particles (Abanades et al. 2002). Besides, as setting time and setting temperature growing up, the environment became reductive, where a lot Cr in reducible fraction was released. Consequently, setting time and setting temperature have negative influence on immobilization of Cr and Cr$^{6+}$ (Wang et al. 2019) as shown in Figs. 13 and 14.

The heavy metal Ag of raw MSWI fly ash was mainly in oxidable accounted for 67.3%, and the leaching value exceeded the limitation because the environment of leaching test simulating the acid environment has oxidizing property. Sintering helped to enhance the percentage of residual fraction from 20.2 to 61.2%, and reduce oxidable fraction to 20.4%. The formation of residual fraction is the important reason to explain sintering mechanism of Ag. Ba of raw MSWI fly ash in acid soluble and residual fraction accounted for 44.2% and 46.3%, respectively, and it was simple for leaching due to the half acid soluble fraction. While, residual species of Ba in sintered MSWI fly ash increased to 64.7% and became the major species. The mechanism of sintering on Ag was similar with Ba according to raising the crystallization for residual fraction such as primary minerals. It was helpful to reach the sintering residual fraction on Ag and reduce the energy for supporting the growth of crystal in MSWI fly ash, and fluxing agent, setting temperature, and setting time were used to shorten sintering time and reduce consumption. However, the addition to fluxing agent of CaO showed negative effect (Fig. 12) on Ba leaching which was different to Ag. The reduction of Ba can react with most of oxide types and increase the oxidable fraction, at the same time, Ba leaching increased.

The treatment of sintering was improved through adding two additional factors as the method to promote crystallization leading to residual fraction in MSWI fly ash and immobilization of heavy metals. Changing grain size with ball milling was utilized to provide energy under the mechanical work for atoms and Irregular surface. Besides, it

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Fig. 17 SEM–EDS analysis of MSWI fly ash on three heavy metals of Cr, Ag, and Ba: (a) raw MSWI fly ash and (b) sintered MSWI fly ash

Fig. 18 XPS survey spectrum of raw and sintered MSWI fly ash
is useful to reduce the distance of particles (Luo et al. 2017). MSWI fly ash has saved a large energy before sintering. CaO contributes to decrease melting point in SiO₂-CaO or SiO₂-Al₂O₃-CaO system as high-melting oxide (Zha et al. 2021). The results of sintering were considerable with previous treatment. Particles are push to a drastic motion as temperature growing. Mineral phases, such as CaTiO₃, and Ca₁₂Al₁₄O₃₃ are continuously generated at setting temperature and time, occupying a large space in particle. Toxicity metals are locked and surrounded by other crystals when particles compacted. Gigantic binding force becomes the limitation on leaching of metals.

The specific mechanism is shown in Fig. 20 giving a rational explanation for inherent change during treatment. Atoms keep moving with certain speed and distance in particles for MSWI fly ash. It is simple for toxicity metals in MSWI fly ash to depart from particles when leaching, for acid soluble and oxidable fraction are the main species of metals, which were easy to leach in acid and oxidized environment during leaching test. The conditions of sintering have different influence on species distribution according to the characteristics of heavy metals to affect the leaching values. The sintering mechanism is mainly a chemical process. Moreover, the main species distribution of heavy metals become stable residual fraction, and the leaching values reduce after sintering as the key to explain the sintering mechanism in MSWI fly ash.

Sintering of MSWI fly ash as potential materials for concrete consumes 1200 MJ/t according to the Muffle Furnace with a full load in this study, which is far well below the cement production of 3–4 GJ/t (Mirzakhani et al. 2017; Yin et al. 2016). The consumption of MSWI fly ash sintering will be sharped down when sintering becomes the intensive industrial process. Thus, sintering for MSWI fly ash is a reliable environmental solution for reutilization and stabilization, while the findings of this study were restricted to

![Species distribution of Cr, Ag, and Ba in raw (R) and sintered (S) MSWI fly ash samples](image1)

![Main mechanism diagram with leaching characteristic of heavy metals for MSWI fly ash during sintering](image2)
other factors that may contribute to sintering, for example, sintering atmosphere, and pressure. The leaching of As, Pb, Cd, Cu, Ni, Zn, Mn, Hg, Be, Se, and fluoride hardly was detected as the values being over the lowering. Thus, they were left out to consider the relation with setting temperature, setting time, grain size of MSWI fly ash, and dosage of fluxing agent. The mechanism of sintering on heavy metals except Cr, Ag, and Ba has not been explored. Besides, the findings did not imply the leaching characteristic for MSWI fly ash in the environment permanently.

### Conclusion

In this study, the treatments of ball milling and sintering for MSWI fly ash were conducted to investigate the effect of grain size ($D_{50}=30$, 45, and 60 μm), fluxing agent (CaO with 0, 2.5, and 5%), setting temperature (1000, 1050, and 1100 °C), and setting time (120, 180, and 240 min) on the leaching characteristic of toxicity metals, and to explore the mechanism of sintering on heavy metals. The main findings are represented as follows:

a) The growth of crystal in particles is connected and welded under sintering to reduce internal space and restrict the leaching characteristic of heavy metals in MSWI fly ash. The leaching values of As, Pb, Cd, Cu, Ni, Zn, Mn, Hg, Be, Se, and fluoride are not detected, in addition that the most leaching of Cr, Ag, Ba, and Cr$_{6}^{3+}$ are below the limitation after sintering and considerable with standards for construction use as hazardous waste.

b) Decreasing grain size with ball milling has a significant influence on restraining the leaching characteristic of Cr$_{6}^{3+}$, Ag, and Ba. CaO also is seen as the main elements for crystallization during sintering, which is significant to reduce the leaching values of Cr$_{6}^{3+}$, Ag, and Cr as its rising. Setting time is shown less influence on leaching characteristic than setting temperature. Increasing temperature can be seen a positive influence on Ag and Cr, and negative influence on Cr$_{6}^{3+}$ and Ba.

c) Based on the Box-Behnken experiment, the ideal condition is that grain size of $D_{50}=30$ μm, fluxing agent of CaO = 5%, setting temperature = 1050 °C, and setting time = 180 min. Besides, the mechanism of sintering can be seen as the results on the change of speciation fraction from acid soluble fraction and oxidable fraction to reducible fraction and residual fraction.

### Author contribution

Sheng He: methodology, investigation, formal analysis, original draft writing, validation, building model, writing-review and editing; Yitong Zhou: data analysis, methodology, formal analysis, validation, writing-review and editing; Peng Yu: supervision, resources, funding acquisition, visualization. Xin Xia: building model, formal analysis, writing-review and editing; Hongtao Yang: building model, formal analysis, writing-review and editing.

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### Data availability

Not applicable.

### Declarations

### Ethics approval

Not applicable.

### Consent to participate

Not applicable.

### Consent for publication

Not applicable.

### Conflict of interest

The authors declare no competing interests.

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