



## Effects of wet ball milling on lead stabilization and particle size variation in municipal solid waste incinerator fly ash

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### ABSTRACT

Water-extracted municipal solid waste incinerator (MSWI) fly ash was treated by a process of wet ball milling, using desalinated water as the milling solution. We investigated the influence of the milling process on the partitioning and leaching characteristics of lead (Pb) and the particle size distribution. The results show that 93.11% of the Pb was partitioned into the milled ash, 2.60% to the milling balls, and 0.17% to the inner surface of the milling jar, while amounts lower than the detection limit remained in the milled solution. As tested by the toxicity characteristic leaching procedure (TCLP), the leaching of Pb was inhibited after short-term grinding (from 5.2 to 1.2 mg/L after 1 h of milling), and further reduced by about 96% after 96 h of ball milling. The mobility of the heavy metal was analyzed after a sequential extraction procedure. The results also show that Pb tended to become more stable after milling. The size distribution of particles was analyzed by a laser particle diameter analyzer and their morphology during grinding was observed using scanning electron microscopy. The median size of the fly ash decreased significantly from 36 to 5  $\mu\text{m}$  after 0.5 h of milling, but then only slightly, from 5 to 2  $\mu\text{m}$ , with further milling from 0.5 to 96 h, due to the concurrent actions of fragmentation and/or agglomeration. The reason for the stabilization of Pb by ball milling was probably that Pb was sealed in the milled fly ash during the fragmentation and agglomeration of particles.

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### 1. Introduction

The presence of contaminants such as heavy metals means that municipal solid waste incinerator (MSWI) fly ash has always been classified as a hazardous waste. The common treatment process in Taiwan has been to place this material in landfills following a cement-based solidification process. However, new landfill sites have often been opposed by the surrounding inhabitants. Efforts are therefore being focused on ways to render the MSWI fly ash innocuous and recyclable. However, MSWI fly ash also includes large amounts of chloride salt, which is very disruptive to heavy metal stabilization and affects the quality of recycled products produced therefrom. It is therefore preferable to remove these chloride salts before the recycling process. Water extraction is a simple and useful pre-treatment method [1–3] that can be used to remove most of the soluble salts and even some of the heavy metals from fly ash. Nevertheless, the presence of heavy metals in the extracted fly ash still often exceeds the leaching test regulatory limits, making it necessary for further stabilization after the water extraction process.

The sequential extraction procedure (SEP) was originally used to investigate the distribution of heavy metals in river sediment. The procedure can identify five fractions of heavy metals using different leaching liquids [4] according to increasing stability: the exchangeable, carbonated, Fe–Mn, organic matter, and residual fractions. The procedure has been modified and developed continuously, and can now be employed for the analysis of many other heavy metals that may be present in hazardous wastes including MSWI ash [5–7]. This method has been used in many studies to evaluate the leachability and stabilization effects of MSWI fly ash. The Fe–Mn fraction, organic matter fraction, and residual fraction have been classified as difficulty of leachability in adverse circumstances [7–9].

Heavy metal stabilization treatment is beneficial for reducing harm to the operator and environmental risks during the recycling process, and could help to expand the scope of recycling technology. Recently, a novel method has been introduced, the so-called mechanochemical (MC) treatment method, designed to inhibit the elution of heavy metals from fly ash [10]. In that study, fly ash was milled with CaO in a dry planetary ball mill. It was found that Pb elution from fly ash could be inhibited by 93% by MC treatment as compared to untreated fly ash. This effect may have been the result of the high surface energy of the ground particles, which caused the formation of insoluble compounds. Montinaro et al. [11] further demonstrated that it was possible to achieve immobilization

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**Table 1**  
SEP experimental conditions.

Stage	Fraction	Reagents/1 g sample	Shaking time and temperature
1	Exchangeable fraction	8 mL 1 M MgCl <sub>2</sub> (pH = 7.0)	1 h at room temperature
2	Bound to carbonates	8 mL 1 M NaOAc (pH = 5.0 adjusted by HOAc)	5 h at room temperature
3	Bound to Fe–Mn oxides	20 mL NH <sub>2</sub> OH·HCl in 25% HOAc	6 h at 96 ± 3 °C
4	Bound to organic matter	3 mL 0.02 M HNO <sub>3</sub> and 5 mL 30% H <sub>2</sub> O <sub>2</sub> (pH = 2.0 adjusted by HNO <sub>3</sub> ) 3 mL 30% H <sub>2</sub> O <sub>2</sub> (pH = 2.0 adjusted by HNO <sub>3</sub> )	2 h at 85 ± 2 °C 3 h at 85 ± 2 °C
5	Residual	5 mL 3.2 M NH <sub>4</sub> OAc in 25% (v/v) HNO <sub>3</sub> 0.52 mL HNO <sub>3</sub> , 1.5 mL HCl, 1 mL H <sub>2</sub> O <sub>2</sub> , 2 mL HF in a teflon tube	1 h at room temperature Microwave digestion, 20 min at 180 ± 5 °C

of Pb(II) in contaminated soils by dry ball milling. The study suggested this was probably induced by mechanical treatment, such as entrapment of the Pb(II) due to agglomeration, solid diffusion of Pb(II) into the crystalline reticulum of the soil particles, and so on. In both of these studies, it was shown that mechanical treatment methods have potential for the stabilization of heavy metals.

The ball milling process was originally applied to grind particles for mineral processing, and for the manufacturing of cement and ceramics. Apart from the reduction in particle size, there are changes in the crystalline structure during milling. Chemical reactions in ground materials, usually called mechanical activation, are increasingly being studied [12,13]. Palaniandy et al. [14] reported that the agglomeration of particles was a major factor when the treated particles were ground to less than 10 μm, especially with particles having a high surface energy. Agglomeration was found to be detrimental to the production of nano-particle materials because the particles could not be ground to a finer level. During the MC process, heavy deformation occurs to the particles. The deformation is confirmed by the presence of a variety of crystalline defects such as dislocations, vacancies, stacking faults, and an increased number of grain boundaries. The presence of this defect structure enhances the diffusivity of solute elements into the matrix [15]. However, these phenomena might help in the stabilization of heavy metals by sealing them within the ground matrix due to a transformation of the material from crystalline to amorphous phase during the milling process [11]. Therefore, in this study, we explored the influences of milling on particle size variation and the leaching characteristics of Pb. Our efforts were focused on providing a reasonable explanation for Pb immobility as Pb was the only heavy metal exceeding the toxicity characteristic leaching procedure (TCLP) limitations in fly ash in this study.

## 2. Materials and methods

### 2.1. Sampling and pre-treatment

Fly ash was collected from a large MSWI plant in northern Taiwan. This plant has a series of air pollution control devices, including cyclone dust collectors and semi-dry powder activated carbon spray towers combined with bag filters. Two types of fly ash were collected: boiler fly ash from the cyclone dust collector and reaction fly ash from the bag filter. The experimental material in this study consisted of a homogeneous mixture of both boiler and reaction fly ashes, at weight percentages of 37 and 63, respectively, mixed in accordance with the daily output of the incineration plant. The mixed fly ash samples were placed on different plates to be dried in an oven at 105 °C for 24 h. After this, water was used to leach out the soluble elements such as chloride, sodium, and potassium. The water extraction was a two-stage process with a water/solid ratio of 5; the extraction (washing) time was 5 min for each stage [16].

### 2.2. Wet ball milling experiments

Wet milling can be used to avoid the dispersion of hazardous dust into the working environment. Water was selected as the

general milling solution in the experiments. The conventional ball mill used included a milling jug consisting of a ceramic cylindrical chamber (6.8 L) that rotated around a horizontal axis. The chamber contained aluminum oxide balls of two diameters, 5 and 15 mm, combined in equal amounts by weight. Wet milling runs were performed with the operating conditions fixed according to those commonly used in industrial ceramic production. In this study we used the same traditional ball milling apparatus used in the ceramics industry for a short period of time. The critical rotation speed of the chamber was fixed at around 93 rpm [17]. The volume fractions of the balls and slurry filling were equal, each being 30%, with 40% of the space left vacant [16]. The liquid/solid ratio was 9, indicating 1 g of ash mixed with 9 g of water. The ratio was obtained from the results of a preliminary experiment, in which the effects of milling with liquid/solid ratios of 2, 3, 4, and 9 were tested for 24 h of milling time. After the milling process, the slurries with liquid/solid ratios of 2, 3 and 4 became too thick to mill, due to the reduction in the particle size of the ash, thus we proceeded with the liquid/solid ratio of 9. The milling time varied from 0.5 to 96 h.

Wet milling was used in this experiment. The Pb could possibly be dissolved in the milling solution or adhere to the surface of the milling balls or milling jar during the milling process. A mass balance method was applied in order to determine the distribution of Pb in the sample after 96 h of milling time. After the milling process, a small fixed amount of water was used to wash the milling balls and milling jar. The washing water and all of the milled ash were filtered through 0.3 μm filter paper (ADVANTEC, Japan) to separate the milled ash and milled solution. Then, the milling balls and milling jar were washed and soaked for 3 days in 15.7N of concentrated nitric acid. Finally, the extracted ash, milled ash, milled solution, and the concentrated nitric acid from the washed milling balls and jar were analyzed to calculate the distribution of Pb.

### 2.3. Analytical methods

The diameter of fly ash particles was analyzed using a laser particle diameter analyzer (Honeywell Microtrac X-100). The analysis method for the total amount of heavy metals was the same as in the final step of the sequential extraction procedure (SEP). The toxicity characteristic leaching of the heavy metal was determined by TCLP (USEPA method 1311). The SEP was employed to investigate the leaching behavior of the heavy metals. The different solvents and extraction liquids used during the SEP to extract species with similar chemical properties are shown in Table 1 [4,18]. This was followed by the determination of the heavy metal concentrations by flame atomic absorption spectroscopy (FAAS) (GBC Avanta series/932B). The elements in the fly ash were determined by inductively coupled plasma spectroscopy (JOBINYON JORIBA Ultima-2000). Scanning electron microscopy (SEM) (Leo 1530) was used to observe the morphology of the fly ash particles before and after grinding. The crystalline structure of the fly ash particles was identified by X-ray diffraction (XRD) (Bruker D8A) with Cu Kα radiation ( $\lambda = 0.1542$  nm).

**Table 2**  
The main cations and heavy metals in the fly ash.

Cations	Mixed ash	Water-extracted ash
Ca <sup>a</sup>	29	33
Si <sup>a</sup>	3.8	9.0
Al <sup>a</sup>	1.9	4.7
K <sup>a</sup>	5.8	3.2
Na <sup>a</sup>	5.2	3.0
Mg <sup>a</sup>	1.0	1.8
Fe <sup>a</sup>	0.7	1.8
Ti <sup>a</sup>	0.5	0.9
Zn <sup>b</sup>	11,820	17,560
Pb <sup>b</sup>	6,790	7,395
Cu <sup>b</sup>	2,195	2,270
Cr <sup>b</sup>	760	1,390
Cd <sup>b</sup>	440	460

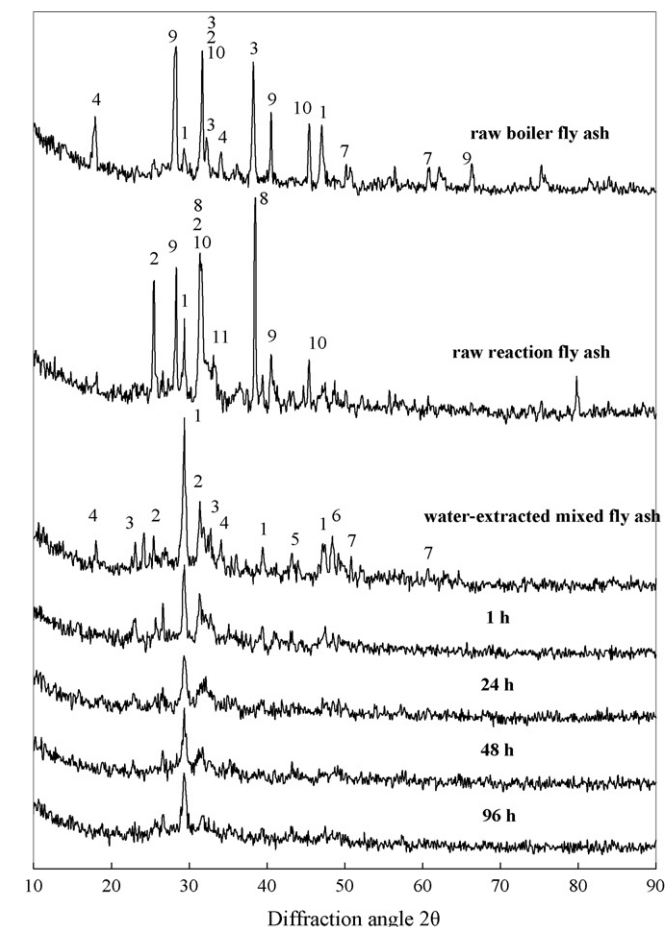
<sup>a</sup> Unit: % (w/w).

<sup>b</sup> Unit: mg/kg.

### 3. Results and discussion

#### 3.1. Characteristics of the mixed fly ash and water-extracted mixed fly ash

The mixed fly ash (comprised of the boiler and reaction fly ash) was strongly basic, with a pH higher than 11. The ignition loss of the mixed fly ash was lower than 7%. The main cation and the heavy metal contents of the mixed ash and the water-extracted mixed ash are shown in Table 2. Clearly, the main cation with the highest concentration was calcium. Of the five main heavy metals found,



**Fig. 1.** XRD results for the fly ash after various grinding periods. 1: CaCO<sub>3</sub>, 2: CaSO<sub>4</sub>, 3: CaSiO<sub>3</sub>, 4: Ca(OH)<sub>2</sub>, 5: Al<sub>2</sub>O<sub>3</sub>, 6: PbO, 7: SiO<sub>2</sub>, 8: NaOH, 9: KCl, 10: NaCl, 11: ZnO.

**Table 3**  
TCLP test results for fly ash (mg/L).

Elements	Mixed ash	Water-extracted mixed ash	Threshold
Pb	31.98	5.20	5
Zn	2.93	0.59	–
Cu	0.38	0.05	15
Cd	0.04	0.02	1
Cr	0.13	0.91	5

zinc had the highest concentration, at over 11,000 mg/kg, followed by lead. The concentrations of calcium, silicon, and heavy metals increased after water extraction, while the amount of soluble salts, such as sodium and potassium, decreased in correspondence to the increase in the concentration of heavy metals in the water-extracted mixed ash. The variation in crystalline compounds in the fly ash was analyzed by XRD. Fig. 1 shows that the highly soluble compounds originally found in the boiler/reaction fly ash (NaOH, NaCl and KCl) were removed by water extraction, as evidenced by the lack of observable peaks in the XRD patterns of the water-extracted mixed ash. This largely explains the disappearance of the soluble salts, especially the NaCl and KCl.

The TCLP results, both before and after water extraction, are shown in Table 3. The amount of Pb in the mixed ash was higher than with the USEPA regulatory limit of 5 mg/L. After water extraction, not only was the Pb concentration still higher than the regulatory limit, but the total amount of Pb was also high, as shown in Table 2. Consequently, the water-extracted mixed ash still required further treatment to stabilize the Pb in the ash and reduce its risk to human health during the recycling and reuse processes.

#### 3.2. Partitioning of Pb and leaching characteristics of the milled mixed ash

The quantities of Pb in the milled ash, milled solution, milling ball, and milling jar were analyzed to determine the partitioning of Pb after the milling process. The experimental results show that 93.11% of Pb was in the milled ash, 2.60% on the milling balls, 0.17% on the inner surface of the milling jar, and Pb remaining in the milled solution was lower than the detection limit (see Table 4). After the milling process, most of the Pb remained in the ash, with only small percentages transferred to the milling balls and milling jar. It was very interesting that the quantity of Pb in the milled solution was lower than the detection limit of the ICP. This phenomenon showed that Pb could be sealed into the milled ash during the milling process.

The TCLP Pb results for the milled mixed ash are shown in Table 5. The leaching concentrations were lower than the regulatory limit after 1 h of milling. The leaching was reduced by 96% after 96 h of milling. It is well-known that the leaching concentration of Pb is usually affected by pH, since Pb is an amphoteric metal that is liable to form ampholyte. Researchers have found that it is difficult to leach the Pb from the MSWI fly ash with the modified TCLP when the pH is between 9 and 11 [19]. In this study, according to the leaching test, the pH of the TCLP leached solution was

**Table 4**  
The partitioning of Pb after 96 h of milling time.

	Weight (mg)	Percentage
Water-extracted mixed ash	578.02	100
Milled ash	538.20	93.11
Milling ball	15.00	2.60
Milling jar	0.98	0.17
Milled solution	– <sup>a</sup>	– <sup>a</sup>
Cannot be measured	23.84	4.12

<sup>a</sup> The quantity was lower than the detection limit of the ICP.

**Table 5**  
TCLP results after milling.

	Time (h)				
	0 <sup>a</sup>	1	24	48	96
Concentration (mg/L)	5.20	1.05	0.89	0.77	0.20

<sup>a</sup> Water-extracted mixed ash, not milled.

between 9.6 and 10.2, before and after 96 h of milling, respectively. In other words, the milling process made a contribution toward the stability of the Pb, not to the change in the pH.

SEP was also used to analyze the leaching status after the different lengths of milling time. This was done to track the change in the level of Pb leaching. Although it was not possible to accurately determine the chemical bonding of the heavy metals from the SEP results, SEP was useful in helping us to comprehend the mobility of Pb in a natural environment [20]. The SEP results are shown in Fig. 2. Carbonate was the main fraction of the water-extracted mixed ash. After wet ball milling, the carbonate fraction decreased with milling time, while the other three fractions increased. These results indicate that the mobility of Pb was decreased by the milling process, making it more difficult for the Pb to leach out into the natural environment. The SEP results further reveal that mechanical loading induced by milling could alter the status of Pb in fly ash.

3.3. Variation in the particle size of the milled mixed ash

The evolution of the median particle size,  $d_{50}$ , as a function of the grinding time is shown in Fig. 3. The median size of water-extracted mixed ash was about 36  $\mu\text{m}$ ; the smallest size was 2  $\mu\text{m}$  after 96 h of milling. The reduction in size was very apparent during the initial period, e.g., after 0.5 h of milling, the size was reduced to 5  $\mu\text{m}$ . This shows that the fragmentation of particles was the dominant phenomenon in the early stage. However, after 0.5 h of milling, as shown in Fig. 3, the rate of particle size reduction tended to decline. This indicates that the alternate and/or concurrent actions of fragmentation and agglomeration were caused by milling.

An analysis of the size distribution during the milling process (Fig. 4a–c) shows several interesting features. The particle size distributions were poly-modal after sample milling. As seen in Fig. 4a, the distribution rapidly shifted to the left (towards smaller particle size) in the first 0.5 h of milling. After 1 h of milling, particles smaller than 0.45  $\mu\text{m}$  disappeared, forming another sub-peak with a median size of around 1  $\mu\text{m}$ . Whenever these sub-peaks appeared, the leftward spreading of the distribution diminished. These results reveal that smaller particles could agglomerate to form larger particles (agglomeration action). The sub-peak weakened after 2 h of milling (fragmentation action). While the sub-peaks repeatedly formed and weakened during the milling process, the main-peaks shifted slightly to the left (Fig. 4b and c). A comparison with Fig. 1 shows that although the reduction of the main particle sizes was limited, the majority of the milling energy was used to destroy the crystalline structure of the fly ash particles. This decrease in inten-

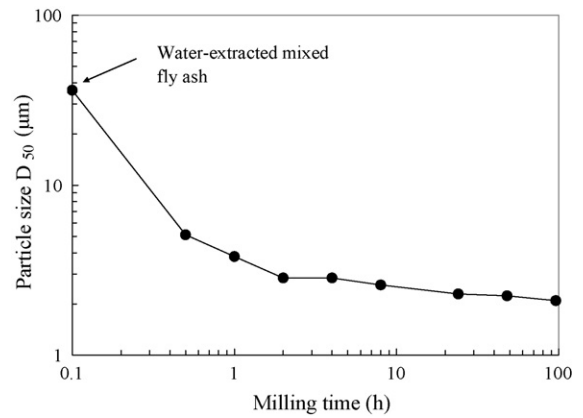


Fig. 3. Median particle size after the various grinding periods.

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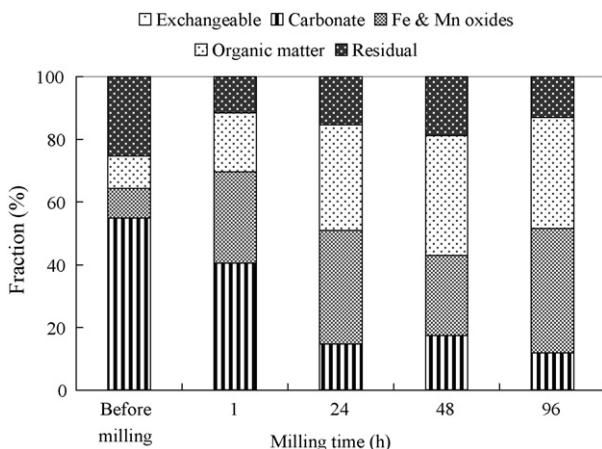


Fig. 2. SEP results for lead.

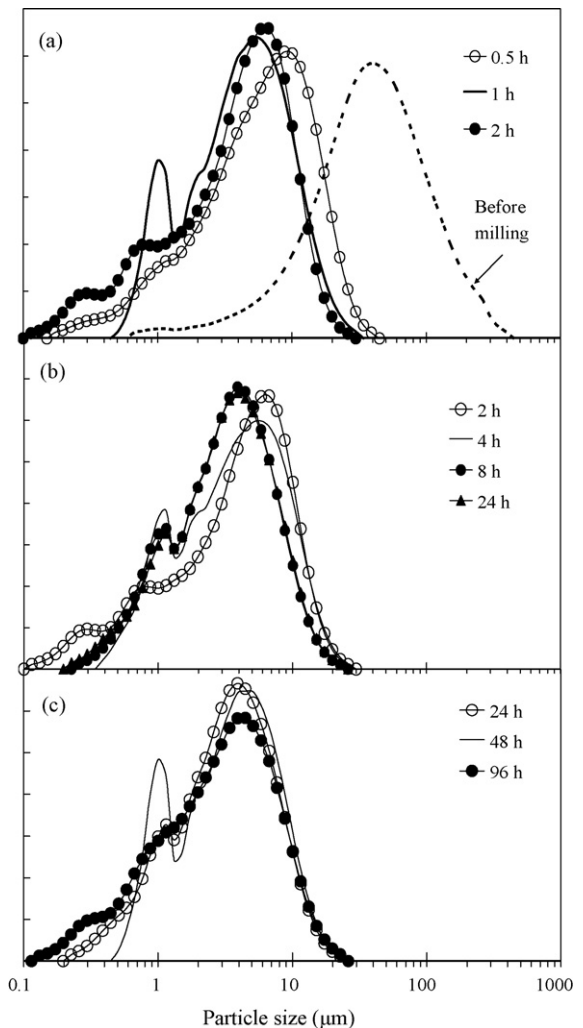
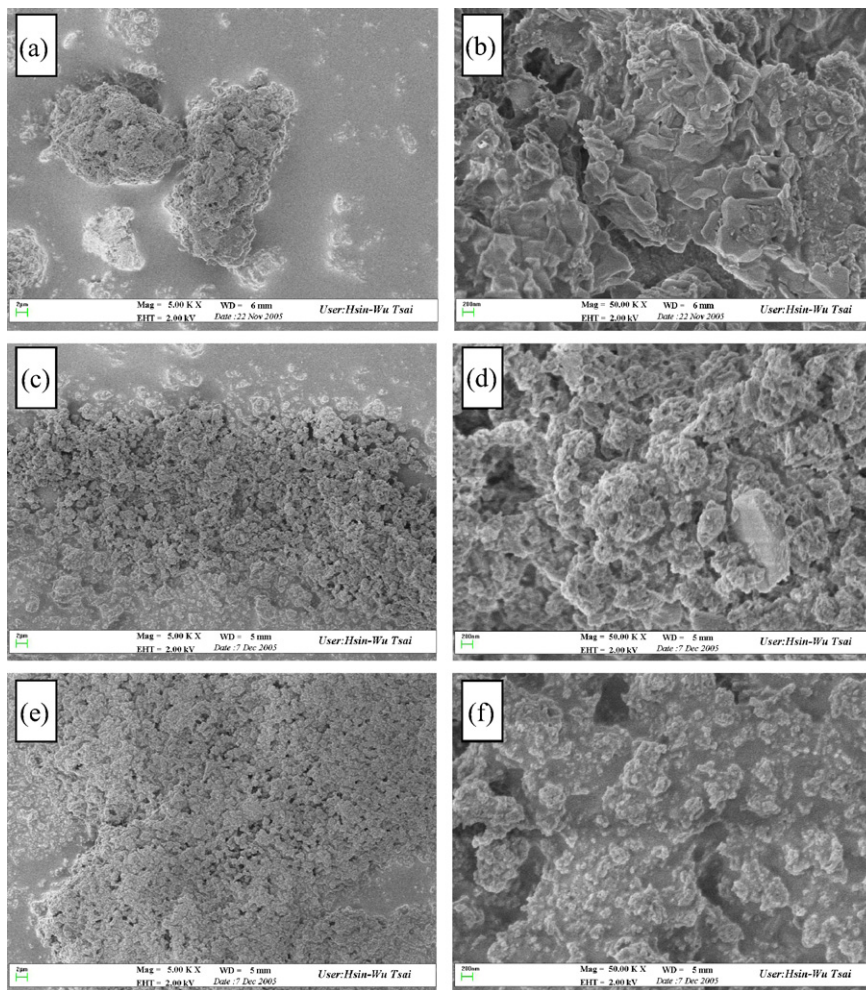


Fig. 4. Cumulative particle-size distribution after the various grinding periods: (a) 0–2 h; (b) 2–24 h; and (c) 24–96 h.



**Fig. 5.** The variation in particle size and the fragmentation and/or agglomeration phenomena were directly observed by SEM micrographs for: (a) water-extracted mixed fly ash at 5k $\times$ ; (b) water-extracted mixed fly ash at 50k $\times$ ; (c) 24 h at 5k $\times$ ; (d) 24 h at 50k $\times$ ; (e) 96 h at 5k $\times$  and (f) 96 h at 50k $\times$ .

sity is presumed to be due to the introduction of a certain degree of crystal deformation, causing changes in the crystal grain size and leading to the development of amorphization [12,21]. The amorphization of ground particles can be regarded as the result of the constantly increasing number of defects that are due to the deformation of the crystalline structure by milling. The presence of this defect structure enhances the diffusivity of solute elements into the matrix [15]. Montinaro et al. [11] also found that when the number of crystalline defects increased, Pb could diffuse within the solid matrix. The result would be the chemical entrapment of Pb during the process of amorphization. Therefore, the decrease in the XRD peaks during the milling treatment suggests that this might have led to the observed stabilization of Pb in our case.

The variation in particle size and the fragmentation and/or agglomeration phenomena were directly observed by SEM. Fig. 5a shows images of water-extracted mixed ash is composed of differently sized particles. After 24 h of milling, the particles were ground to smaller sizes, as shown in Fig. 5c. After 96 h of milling, the particles appeared to be only slightly smaller than they were after the previous milling time (see Fig. 5e).

The agglomeration could clearly be observed at higher magnifications (50k $\times$ ). The solitary particle of water-extracted mixed fly ash seen in Fig. 5b is actually composed of many bulky, irregularly shaped pieces of debris with relatively smooth surfaces. After 24 h of milling, the original particle configuration changed, forming agglomerations of particles consisting of many small bits (Fig. 5d). The agglomeration action finally led to the melted-looking surface

displayed on the particles after 96 h of milling (Fig. 5f). The SEM images clearly verify the agglomeration of particles.

Fragmentation and/or agglomeration happened repeatedly throughout the milling process, similarly to the mechanism observed in MC treatment. The MC process is conducted during high-energy milling, where the powdered particles are repeatedly flattened, cold-welded, fractured, and re-welded [15]. Owing to the fact that the variation in particle size found in this study was similar to that which occurs during the MC process, we suggest that the mechanism of MC might be useful for the stabilization of Pb by ball milling. Heavy metals could be dispersed onto the surface of the ash and strongly adsorbed into the newly fragmented surfaces of the particles during the milling process [11]. Furthermore, when fine particles aggregate to form larger ones, as during re-welding in the MC process, it is conjectured that heavy metals could become entrapped inside the new particles, making it difficult for them to leach out.

#### 4. Conclusions

In these experiments, we observed the occurrence of alternate and/or concurrent actions of fragmentation and agglomeration of water-extracted mixed ash particles during the milling process. Large particles were fragmented into small particles and when enough surface energy accumulated, the small particles agglomerated to form particles that were larger than the original small ones. The fragmentation and/or agglomeration acted to decrease

the size of the water-extracted mixed ash particles, although the  $d_{50}$  decrease was limited to around 2  $\mu\text{m}$ . We suggest that this phenomenon was the reason for the stabilization of Pb by milling. The wet ball milling process caused most of the Pb to remain in the milled ash and even helped to detoxify the water-extracted mixed ash after a short period. This technology has the potential to be applied as the first stage in the recycling of the MSWI ash and/or industrial waste ash. It also has the potential to improve the working environment of the operators during later parts of the waste-ash recycling process.

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