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Sintering of MSWI fly ash by microwave energy

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ABSTRACT

This study presents the sintering of municipal solid waste incineration (MSWI) fly ash assisted by microwave energy. The composition of fly ash was investigated by chemical sequential extraction and modified microwave digestion method. Effects of process time, container materials, aging time and salt contents were also discussed. The major elements of fly ash are Ca, Cl, Na, Si, K, Al, Mg, and Zn, and the metal species, Zn, Cr, Pb, Cu, and Cu, are mainly in the oxide phase. Under microwave processing, the fly ash was sintered into a glass-ceramics and the leaching concentrations of heavy metals were restrained. The stabilization efficiency increased with an increase in processing time in most of the cases. Better stabilization efficiency of fly ash was discovered by using the SiO₂ or Al₂O₃ container than by using the graphite plate/SiC plate. The presence of salt in the fly ash could enhance the sintering and stabilization of fly ash. During the aging time of 0–30 days, negligible Pb in the sintered fly ash was leached out, and the leaching concentration was lower than the criterion.

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1. Introduction

Municipal solid waste incineration (MSWI) fly ash is a residue found in waste after incineration. Fly ash in Taiwan amounts to about 160,000 tons/year. MSWI fly ash is regarded as hazardous waste. The components of MSWI fly ash are complex and unfixed [1]. Major contents are soluble salts [2], CaO, SiO₂, Al₂O₃, Fe₂O₃, MgO, Na₂O, K₂O, P₂O₅, TiO₂, MnO, CuO, ZnO, PbO, Cr₂O₃, and SO₃. Minor contents are Zn, Pb, Cr, Cd, Cu, Sn, Ba, Sb, Zr, As, Co, Mo, Rb, Bi, V, Ce, Ga, La, Nd, Nb, and Hg [3]. TCLP leaching of the heavy metals from MSWI fly ash still results in leachate concentrations above the legal limit for land disposal. Hence, it is important to dispose of this troublesome substance thoroughly before it is sent to landfills. The most commonly used treatment method for MSWI fly ash is cement solidification. However, this method increases the volume of ash, which reduces the useful lifetime of landfill sites. This study aims to find a new method to dispose of fly ash and avoid the disadvantage of cement solidification.

Although microwave heating was introduced over 50 years ago, its use in the treatment of MSWI fly ash is relatively new. Microwave energy is a nonionizing electromagnetic radiation with frequencies in the range of 300 MHz to 300 GHz. Compared with other conventional thermal treatment technologies, the microwave technique with its characteristics of polar oscillation and effect of dielectric losses offers the advantage of selective, uniform, and rapid heating.

The interaction of dielectric materials with electromagnetic radiation in the microwave range results in energy absorbance as heat [4].

The application of microwave radiation is widely used in many fields, including sintering and joining of ceramics [5–7], synthesis of composites [7], contaminated soil remediation [8], waste treatment [9,10], mineral processing [11], assisted digestion procedures [12], drying or dewatering of materials, polymer curing, and regeneration of activated carbon [10]. Moreover, a growing interest in the stabilization and immobilization of metal ions in soil and sludge through microwave radiation has also been reported. Results indicated that microwave radiation inhibits the leaching of metal ions from soil or sludge and that it makes these solid wastes acceptable for disposal or recycling [13–15].

This project aims to investigate the sintering of MSWI fly ash by microwave radiation. The objective of this study is to: (1) determine the proper procedure for stabilization of MSWI fly ash by microwave energy, (2) discuss the effects of processing time, (3) compare the effects of different crucibles/containers on microwave radiation of MSWI fly ash, and (4) discuss the effect of soluble salts and caustic substances on microwave radiation.

2. Materials and methods

Two specimens of MSWI fly ash from two different incineration plants were tested in this study. The MSWI fly ash was pretreated by drying in an oven at 105 °C (about 24 h), until the mass maintained a constant value within ±1%.

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The composition of the MSWI fly ash was determined by modified microwave digestion method reported in previous studies [15]. Samples of the MSWI fly ash (0.2 g) were mixed with a $\text{HNO}_3\text{--HCl--HF}$ solution (3 mL). These acidic slurries were processed using the following two-stage microwave program: (1) 650 W for 10 min (final temperature 160 °C) and (2) 800 W for 15 min (final temperature 200 °C). After the microwave-assisted digestion process, the mixtures were cooled to room temperature and filtrated, and the filtrate was adjusted to 100 mL before analysis.

Modified chemical sequential extraction [16] was also conducted to determine the phase of heavy metals in the MSWI fly ash. Portions of fly ash of 2 g were put in PVC tubes and mixed with extracting agents. The extraction procedure was as follows:

- (i) *Exchangeable*. The fly ash was extracted at 25 °C with 8 mL of magnesium chloride solution (1 M MgCl_2 , pH 7.0) and shaken for 1 h (125 rpm).
- (ii) *Bound to carbonates*. The residue from (i) was leached at 25 °C with 8 mL of 1 M NaOAc adjusted to pH 5.0 with acetic acid (HOAc). It was continuously shaken for 5 h (125 rpm).
- (iii) *Bound to Fe–Mn oxides*. The residue from (ii) was extracted with 20 mL of 0.04 M $\text{NH}_2\text{OH--HCl}$ in 25% (v/v) HOAc. This was performed at 96 ± 3 °C with 6 h shaking (125 rpm).
- (iv) *Bound to organic matter*. The residue from (iii) was added to 3 mL of 0.02 M HNO_3 and 5 mL of 30% H_2O_2 adjusted to pH 2 with HNO_3 , and the mixture was heated to 85 ± 2 °C for 2 h with shaking (125 rpm). A second 3-mL of 30% H_2O_2 (pH 2 with HNO_3) was then added and the sample was heated again to 85 ± 2 °C for 3 h with intermittent shaking (125 rpm). After cooling, 5 mL of 3.2 M NH_4OAc in 20% (v/v) HNO_3 was added and the sample was diluted to 20 mL and shaken continuously for 30 min.
- (v) *Residue*. The residue from (iv) was digested with a 10-mL of 10% HF and 10 mL HClO_4 mixture at 25 °C with 1 h shaking (125 rpm).

Between each successive extraction, separation was effected by centrifuging at 10,000 rpm for 30 min. The resulting solution was filtered through a membrane filter (0.45 μm) and analyzed by inductively coupled plasma-atomic emission spectrometry (ICP-AES), whereas the residue was washed with 8 mL of deionized water; after centrifugation for 30 min, this second supernatant was discarded.

In the sintering experiments, 20 g samples of fly ash were put in an SiO_2 ceramic crucible, Al_2O_3 ceramic crucible (11 cm diameter), and SiC plate with graphite plate, and the moisture contents were adjusted to 75 wt.% with deionized water. The experiments were conducted with microwave frequency of 2.45 GHz at 600 W. Respective process times were 10, 20, 30, 40, and 50 min. The effects of process time, container type, and aging time were discussed. Moreover, the effect of salt in the fly ash under microwave radiation was also studied. The fly ash was pre-washed by deionized water with liquid to solid ratio (L/S) of 5, 10, 15, and 20 mL/g and followed by the microwave process.

The raw fly ash and treated samples were tested by TCLP and the leaching concentrations were analyzed by ICP-AES. The crystal structure of fly ash samples before and after microwave processes were also determined by X-ray diffraction (XRD) to determine the species variation.

3. Results and discussion

3.1. Chemical composition of MSWI fly ash

Microwave-assisted digestion was performed for analysis of the chemical composition of MSWI fly ash. Table 1 lists the extractable

Table 1
Chemical composition of MSWI fly ash

Elements (mg/kg)	Plant A	Plant B
Zn	8,718	10,214
Cr	365	614
Pb	3,270	3,720
Ni	252	1,311
Cd	2,269	614
Ba	1,890	2,094
Co	183	1,137
Mn	671	N.D.
Fe	414	1,889
Mg	8,915	5,975
Ca	232,222	310,204
Cu	1,422	1,173
Ag	469	1,458
Al	16,407	15,617
Ga	111	506
Na	38,633	27,168
K	21,837	19,071
Si	33,236	24,745
Cl	118,365	165,816

D.L. = 0.1 mg/L and N.D. means the concentration of metal ion is below detection limit.

elemental composition measured in samples of the MSWI fly ash from plant A and B. The instrument detection limit (IDL) of the liquid extract was 0.1 mg/L, which translates into a detection limit of 0.05 mg/g for the MSWI fly ash. All the values of metal concentration, mg/kg, are listed in Table 1. The major elements in the MSWI fly ash from plant A, in decreasing sequence, were Ca, Cl, Na, Si, K, Al, Mg, and Zn, while those from plant B (decreasing sequence) were Ca, Cl, Na, Si, K, Al, Zn, and Mg. The amounts of Na, Si, K, and Al in the MSWI fly ash from plant A was higher than those from plant B. However, the amounts of Ca, Cl, Zn, and Pb from plant B was higher than those from plant A. The higher amount of Cl was generated from the composition of refuse in the incineration processes. An amount of HCl was also generated. The higher amount of Ca was generated without HCl/SO_x by the air pollution control device; the device adds excessive limestone to remove acidic gases. Several papers have reported a presence of CaCl_2 , CaSO_4 , NaCl, KCl, Al_2O_3 , SiO_2 , etc. in MSWI fly ash [1,2] determine, which cause the fly ash to be alkaline (pH value >12) because of the residual limestone.

Fig. 1(a) shows the XRD result of MSWI fly ash from plant A. There are amounts of NaCl, KCl, CaClOH , Ca(OH)_2 , Pb, AlO, SiO_2 , PbO_2 , and KNO_3 in the MSWI fly ash, which supports the finding that the alkali in MSWI fly ash is caused by CaClOH and Ca(OH)_2 . In addition, there is a special phenomenon regarding the MSWI fly ash. The present study discovered that the reaction product of Ca and Cl is CaClOH and not CaCl_2 . The CaClOH seems to deposit the excess limestone in the air pollution control process in plant A. At the same time, the Al species are AlO and not Al_2O_3 . The results in Table 1 and Fig. 1(a) support the finding that the major contents of the MSWI fly ash from plant A are NaCl, KCl, CaClOH , Ca(OH)_2 , AlO, and SiO_2 .

Table 2 shows the chemical composition of MSWI fly ash from plant A and B after chemical sequential extraction. The fly ash from the two plants showed similar tendencies, and the chemical phase of compositions (Zn, Cr, Pb, Ca, and Cu) for both plants was mainly oxide bound.

A result of TCLP leaching of MSWI fly ash (liquid to solid ratio L/S = 20) from both plant A and B, showed that the TCLP lead leaching concentrations for both raw and washed fly ash were much higher than the criterion in Taiwan (5.0 mg/L). The concentrations of Zn, Cr, Pb, Ca, and Cu ions from plant A were 14.29, 0.65, 49.10, 0.14, and 0.45 mg/L, respectively, while those from plant B were 6.11, 0.63, 74.58, 0.14, and 0.45 mg/L, respectively. The concentrations of

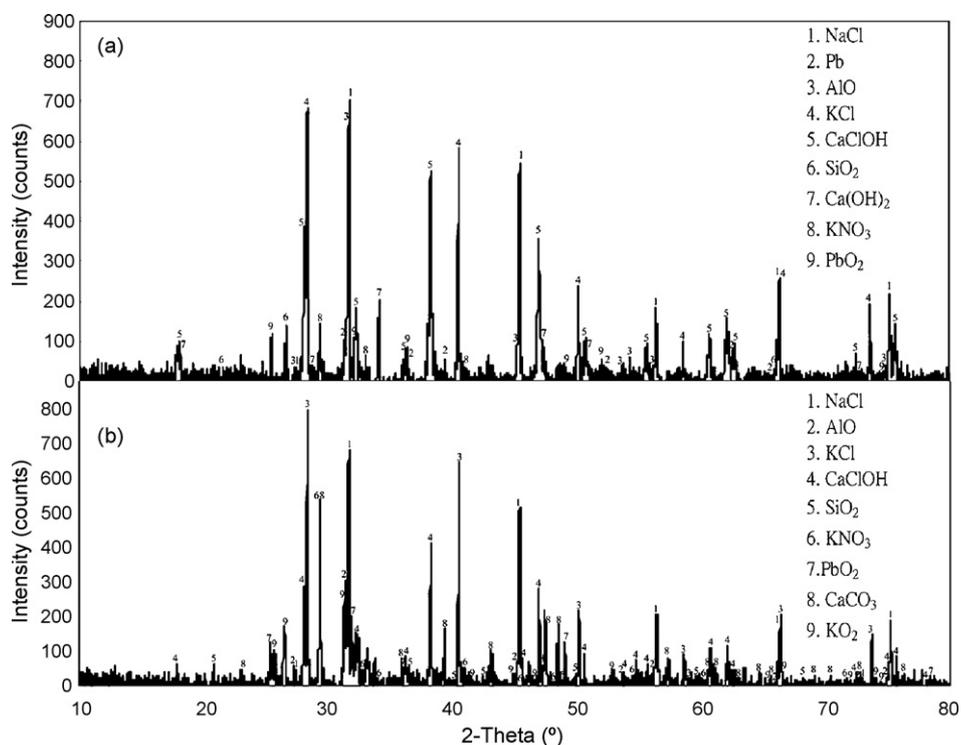


Fig. 1. XRD results of MSWI fly ash: (a) MSWI fly ash and (b) the sintered MSWI fly ash.

Table 2
Chemical composition of MSWI fly ash by chemical sequential extraction

Phase	Elements (mg/kg)									
	Zn		Cr		Pb		Cd		Cu	
	Plant A	Plant B	Plant A	Plant B	Plant A	Plant B	Plant A	Plant B	Plant A	Plant B
Exchangeable	26.2	4.89	12.5	11.6	80.2	38.6	4.06	2.42	2.75	3.44
Carbonate bound	5.96	5.13	10	15.6	29.2	24	2.22	2.57	2.55	2.13
Oxides bound	6627	6048	145	101	2487	2623	160	164	465	112
Organic bound	190	142	29.9	21.9	96.4	87.5	13.6	13.3	179	118
Residual	266	231	26.3	23.3	48.2	45.9	12.2	12.2	37.8	41.8

Zn, Cr, Pb, Ca, and Cu ions in the washed MSWI fly ash from plant A were 1.77, 0.71, 10.3, 0.09, and 0.18 mg/L, respectively; the concentration of Pb ions was 10 times higher than the criterion in Taiwan (5.0 mg/L). Similar results had been reported before. In the light of these results, further treatment of MSWI fly ash is required before landfill disposal.

3.2. TCLP concentration of heavy metals at different processing time

Fig. 2 illustrates the effect of different processing time and TCLP concentration as follows: (1) the TCLP lead concentrations dropped dramatically with an increase in processing time after 20 min when the MSWI fly was sintered. (2) There was an obvious tendency in the TCLP zinc concentrations under microwave radiation. After 10 or 20 min under microwave radiation, the MSWI fly ash had not started to sinter yet; however, half of the MSWI fly ash had sintered after 30 min, like Fig. 3(b). Most fly ash had sintered with minor part melting of the fly ash after 40 min, like Fig. 3(c). All of the fly ash had sintered and stabilized after 50 min, like Fig. 3(d) and the crucibles cracked at these cases (40 and 50 min). The TCLP leaching concentrations of lead were 107, 121, 69.1, 2.43, and 2.65 mg/L at 10, 20, 30, 40, and 50 min, respectively.

The results illustrate that the TCLP lead, zinc, and copper concentrations dropped dramatically with an increase in processing time after the MSWI fly ash had started to sinter. Several researches have reported that the sintering of MSWI fly ash can generate a glass-ceramic material by conventional sinter treatment and decrease the TCLP leaching concentration of heavy metals [17,18].

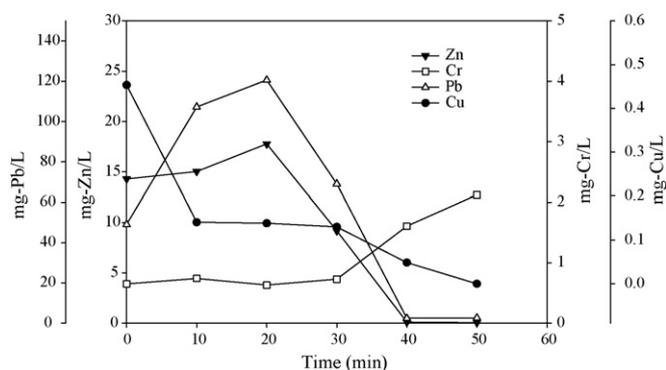


Fig. 2. The TCLP concentration of ions from the sintered MSWI fly ash (plant A) on the SiO₂ ceramic crucible by microwave process. (▼) Zn, (□) Cr, (△) Pb, and (●) Cu.

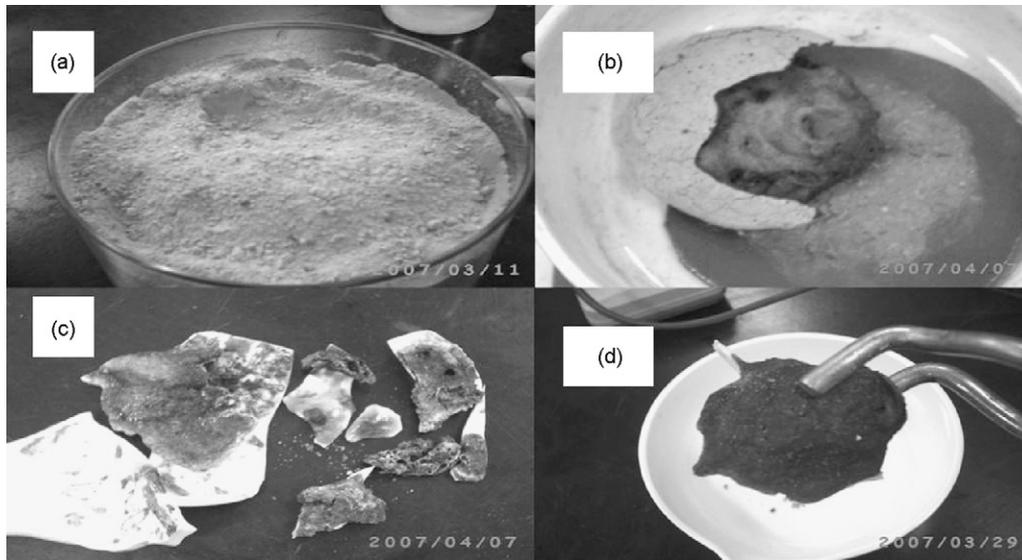


Fig. 3. The photos of fly ash before and after microwave process. (a) MSWI fly ash (raw ash from plant A); (b) the sintered MSWI fly ash from plant A by microwave radiation (at 30 min, 600 W); (c) the sintered MSWI fly ash from plant A by microwave radiation (at 40 min, 600 W); (d) the sintered MSWI fly ash from plant A by microwave radiation (at 50 min, 600 W).

MSWI fly ash can also pass the criteria for heavy metals after it has been sintered. The results in Fig. 2 show that the sintering of MSWI fly ash decreased the TCLP leaching concentration of heavy metals. However, when the MSWI fly ash did not sinter, the TCLP leaching concentration of heavy metals increased.

No literature reports the sintering treatment of MSWI fly ash by microwave process was found; however, several papers report the use of a conventional sinter or vitrification for the treatment of MSWI fly ash. Also, many articles mention that these processes can generate a glass–ceramic or glassy material [18]. The sinter causes particles to bind and immobilize heavy metals [17]. This generates a glass–ceramic material and causes the immobilization of heavy metals. Under microwave radiation, the higher leaching resistance of the sintered glass–ceramics may be due to: (1) a well-dispersed discontinuous phase containing those heavy metals; (2) layered microstructure comprising a core highly loaded with heavy metals surrounded by a shell free from those hazardous metals; (3) gradient concentration of the inclusion phase containing those heavy metals.

Haque [4] reports that Al_2O_3 , Fe_3O_4 , CuO , Fe_2O_3 , and C can penetrate the dielectric region. Hirota et al. [7] refer to the thermal runaway phenomenon, which indicates that the absorption ability of materials is different at different temperatures; however, it rises dramatically with an increase in temperature. This is helpful in the sintering of MSWI fly ash. We consider that elements like water, Al_2O_3 , Fe_3O_4 , CuO , Fe_2O_3 , and C absorb microwave energy first, then the absorption ability of other materials is changed so they too absorb microwave energy. This causes the MSWI fly ash to sinter. The thermal runaway phenomenon causes hot spots and a dramatic temperature gradient; this in turn causes the crucible to crack, as the SiO_2 ceramic crucible has poor heat conduction. Hirota et al. [7] found that the thermal runaway phenomenon caused samples to crack.

3.3. Effect of different crucibles (containers)

Graphite plate conducts heat well. In the present study, hot spots and the cracking of the crucible were at first avoided. Fig. 4 illustrates the effect of different containers and the following obser-

vations were made under microwave radiation: (1) the TCLP lead concentrations dropped dramatically with a graphite plate/ SiO_2 and Al_2O_3 ceramic crucible. (2) There was no obvious tendency with a graphite plate/ SiC plate. MSWI fly ash on a graphite plate/ SiC plate had not started to sinter after 10–50 min under microwave processing (600 W). The TCLP leaching concentrations of lead in fly ash from plant A were 76.3, 71.9, 68.0, 69.1, and 74.7 mg/L at 10, 20, 30, 40, and 50 min, respectively. Its leaching concentrations were similar to that of the raw MSWI fly ash and 12 times higher than the criterion. However, MSWI fly ash sintered on a graphite plate/ SiO_2 and an Al_2O_3 ceramic crucible had similar decreasing leaching concentrations. The TCLP lead leaching concentrations of MSWI fly ash from plant A and B would decrease with an increase of processing time under microwave radiation.

SiC has strong absorption ability for microwave radiation [4]. Therefore, SiC and MSWI fly ash would compete in absorbing

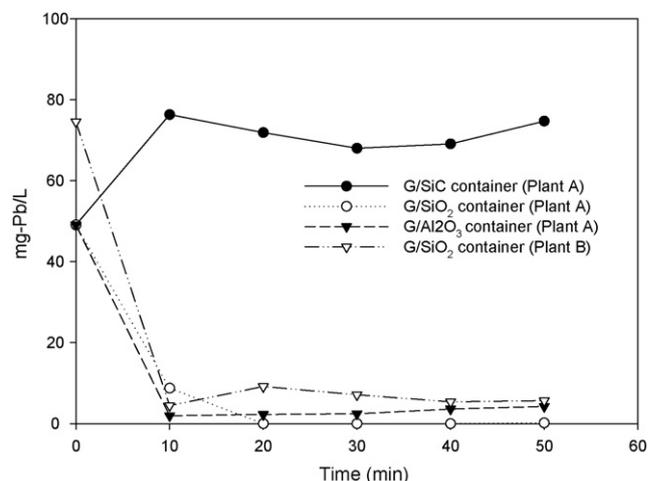


Fig. 4. The effect of different containers on the sintered MSWI fly ash by microwave process. (●) The MSWI fly ash from plant A on graphite plate/ SiC plate, (○) the MSWI fly ash from plant A onto graphite plate/ SiO_2 ceramic crucible, (▼) the MSWI fly ash from plant A onto graphite plate/ Al_2O_3 ceramic crucible, and (▽) the MSWI fly ash from plant B onto SiO_2 ceramic crucible with graphite plate.

Table 3
Pre-wash of MSWI fly ash with deionized water

	Liquid/solid ratio			
	5	10	15	20
Fly ash weight (g)	20.0	10.0	6.67	5.01
Residual ash after pre-wash (g)	13.2	6.05	3.89	2.68
Weight decrease (%)	33.9	39.6	41.7	46.5
pH	12.9	12.7	12.4	12.3

Table 4
The concentration (mg/L) of leaching ions in pre-wash water

Elements	Liquid/solid ratio			
	5	10	15	20
Pb	257	127	84	63
Ca	14,390	8,495	6,219	4,892
Na	4,235	2,733	1,807	1,278
K	4,766	3,216	2,159	1,532
Cl	34,228	17,002	14,806	11,484

the microwave energy when the SiC plate is used as container, and the MSWI fly ash would not sinter by microwave radiation. However, MSWI fly ash microwaved on a graphite plate/SiO₂ and an Al₂O₃ ceramic crucible did sinter. This is evidence that the absorption ability of SiC is better than that of MSWI fly ash.

3.4. Effect of soluble salts and causticity for microwave sintering

The results in Table 1 and Fig. 1(a) show that the major contents of the MSWI fly ash from plant A are NaCl, KCl, CaClOH, and Ca(OH)₂. This plays a role in sintering by microwave radiation. Deionized water (liquid to solid ratio L/S=5, 10, 15, and 20) was used to wash MSWI fly ash in advance. Table 3 shows the change in weight of MSWI fly ash due to the washing process. It appears that the leachate contained salts and causticity of 33.9%, 39.6%, 41.7%, and 46.5% (w/w), for the respective L/S ratios, while the pH value is over 12. Table 4 illustrates that there are abundant Ca, Na, K, and Cl salts and caustic Ca and K in the leachate. In addition, the concentration of Pb ion was higher than the criterion in Taiwan (5.0 mg/L).

During the process of microwave treatment, the soluble salts and causticity played an important role in the sintering of MSWI fly ash of plant A. Fig. 5 shows the effect of soluble salts and causticity at the different L/S ratios (5, 10, 15, and 20) in sintering. The MSWI fly ash in a graphite plate/SiO₂ ceramic crucible did not sinter under microwave radiation at 600 W. This supports the postulation that soluble salts and causticity affect the sintering of MSWI fly ash by microwave radiation. Most of the TCLP lead leaching concentrations were higher than the criterion because the MSWI fly ash did not sinter. The sintering temperature in conventional thermal treatment can be decreased to 700–800 °C when NaCl or KCl is present. This adds Ca(OH)₂, Na₂O, or K₂O which assist conventional sintering or vitrification. Kingman and Rowson [10] reported that molten caustic substances (NaOH and KOH) were shown to be effective absorbers of microwave radiation and that they led to the accelerated differential heating of coal/pyrite phases.

3.5. Aging

Fig. 6 shows the aging of sintered MSWI fly ash from plant A during 0–30 days indoors with open system. The results showed

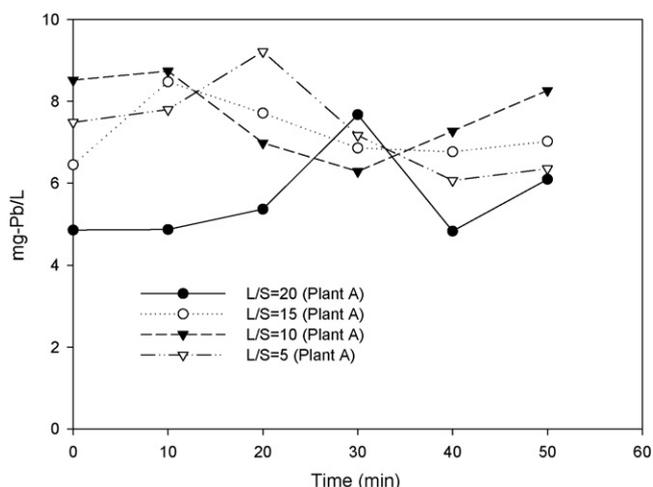


Fig. 5. The effect of soluble salt on microwave sintering. The MSWI fly ash from plant A: (●) L/S=20, (○) L/S=15, (▼) L/S=10, (▽) L/S=5.

no obvious increase in TCLP lead leaching concentrations with an increase in time, and most of these concentrations were lower than the criterion. Fig. 6 illustrates that the lead ions can become immobile after microwave sintering, which can decrease the TCLP lead leaching concentrations. It was also found that the weight of sintered MSWI fly ash increased with an increase in aging time. It appears that the sintering of MSWI fly ash can bind particles to create a ceramic-like material which can restrain the leaching of heavy metals in MSWI fly ash [17].

There are amounts of NaCl, KCl, CaClOH, Ca(OH)₂, Pb, AlO, SiO₂, PbO₂, and KNO₃ in the MSWI fly ash from plant A. Fig. 1(b) shows the XRD diagram of sintered MSWI fly ash from plant A. It appears that there are NaCl, KCl, AlO, CaClOH, SiO₂, KNO₃, PbO₂, CaCO₃, and KO₂ in the sintered MSWI fly ash; the Pb in the original fly ash was transformed into PbO₂, the Ca(OH)₂ into CaCO₃, and the part of KNO₃ into KO₂.

NaCl, KCl, CaClOH, CaCO₃, and KO₂ have a strong ability to absorb water vapor. This explains the weight increase of sintered MSWI fly ash with an increased aging time. In addition, the Ca(OH)₂ in the original fly ash was transformed into CaCO₃; for this there are two possible paths: (1) The carbon of MSWI fly ash forms into CO₂ at first, then the CO₂ and Ca(OH)₂ react into CaCO₃, (2) The Ca(OH)₂ in the sintered MSWI fly ash absorbs H₂O from the atmosphere, then

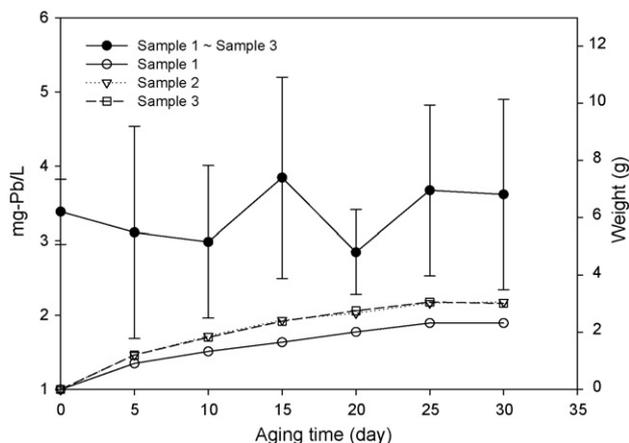


Fig. 6. The TCLP leaching concentration of Pb (MSWI fly ash from plant A) at different aging times. Leaching concentration (●) average of samples 1–3, and weight variation: (○) sample 1, (▽) sample 2, and (□) sample 3.

the CO₂ dissolves in the water. Finally, the carbonate and Ca(OH)₂ react into CaCO₃.

4. Conclusion

This study of the sintering of MSWI fly ash by microwave radiation led to the following conclusions:

1. The Zn, Cr, Pb, Ca, and Cu in the fly ash were mainly in oxide phase and only the leaching concentration of Pb was higher than the criterion of TCLP.
2. The MSWI fly ash could be sintered under microwave processing which could immobilize heavy metals and form a glass–ceramic material. The sintering efficiency increased as the microwave time was longer.
3. MSWI fly ash was effectively sintered by using graphite plate/SiO₂ container under microwave radiation when compared with the case of graphite plate/SiC plate. The SiC plate would compete with the MSWI fly ash in absorbing the microwave energy.
4. The pre-wash process before microwave process would leach out the soluble salt and causticity in the fly ash leading to the decreasing of sintering efficiency.
5. Aging of sintered MSWI fly ash during 0–30 days showed no obvious increase in the TCLP Pb leaching concentrations, and most of these concentrations were lower than the criterion (5 mg/L). This illustrates that the lead ions can be immobilized by microwave sintering. However, the sintered MSWI fly ash may adsorb the water content in the air leading to the weight increase of 40–60%.

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