

CERAMICS INTERNATIONAL

Ceramics International 37 (2011) 2437–2444

www.elsevier.com/locate/ceramint

# Production of glass-ceramics from incinerator ash using lab-scale and pilot-scale thermal plasma systems

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 Received 1 October 2010; received in revised form 7 March 2011; accepted 25 March 2011
 Available online 26 May 2011

#### Abstract

This research employed two thermal plasma melting systems and vitrification technology to treat incinerator ash. The melted slag was used for the production of glass–ceramics via powder sintering and heat treatment. When using the pilot-scale plasma molten system, the melt was rapidly quenched in water to yield glassy slag. The properties of quenched slag glass–ceramics were superior to that glass–ceramics produced by slowly cooling the slag with air. The glass–ceramics with the best physical/mechanical properties and chemical resistance was produced by heat treatment at 1150 °C for 2 h. Diopside and gehlenite were formed as the major crystalline phases. Glass–ceramics produced from incinerator ash demonstrate great potential for reutilization as non-porous or water permeable materials.

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Keywords: Incinerator ash; Thermal plasma technology; Glass-ceramics

# 1. Introduction

Environmental regulations are becoming increasingly stringent, making the disposal of hazardous waste a major problem, both in developed and developing countries. In Taiwan, incineration has become an important method for the treatment of combustible solid waste, due to the increasing difficulty of finding suitable sites for traditional landfills. However, incineration also poses a number of problems, particularly for the treatment of ash. It is estimated that over 1.5 million tonnes of incinerated ashes is generated annually in Taiwan, and developing a safe and reliable immobilization technology where transforming the ash into a stable form is essential. Thermal plasma technology has been undergoing development for many decades, offering a wide range of benefits including: the generation of less gas than conventional incinerators; operational temperatures high enough to destroy dioxins and other toxic species; strong chemical reactivity to reduce or decompose toxic contaminants in gas phases; high energy densities providing high processing rates and high quench rates [1,2]. Prior studies on treating toxic waste, such as radioactive waste, medical waste, and incinerator ash using thermal plasma technology have shown satisfactory results [3–13]. A critical review of thermal plasma technology for the current status of waste treatment was provided in a report by Gomez et al. [14]. The treatment of waste using thermal plasma technology produces a stable, vitrified slag, and determining the means to reutilize the melted slag is a key point in the further development of this high temperature molten process. Glass—ceramics have been studied by many researchers, and appear to provide a sensible alternative to the reuse of slag [15–23].

Lab-scale and pilot-scale plasma systems developed at the Institute of Nuclear Energy Research (INER) exclusively for the treatment of hazardous waste, were employed for vitrification processes in the current research. This study examined the slag generated by thermal plasma technology, exploring the feasibility of using the plasma system to treat incinerator scrubber ash and fly ash. Large quantities of SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, and CaO in the ash could be recycled as raw materials for the production of glass—ceramics. The initial composition and heat treatment methods were carefully selected to produce various crystalline phases, thereby providing glass—ceramics with a variety of beneficial properties. The molten slag produced from lab-scale and pilot-scale plasma systems was

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characterized using the following: the toxicity characteristic leaching procedure (TCLP) for resistance to leaching; X-ray diffractometry (XRD) for the determination of crystal structure; scanning electron microscopy (SEM) for the observation of microstructural characteristics. Other properties including micro-hardness, porosity, water absorption, and physical/mechanical properties were also evaluated.

#### 2. Experimental procedures

The incinerated ash used in this investigation was obtained from a municipal solid waste incinerator in Taipei. Table 1 shows the chemical composition of the scrubber ash and fly ash from the incinerator. Two different mixtures of ash were tested in this study. Sample I mixed scrubber ash and fly ash at a ratio of 3:1 which was also the production ratio of scrubber ash to fly ash at this incinerator. However, the basicity of the CaO/SiO<sub>2</sub>, of sample I was high, reaching 5.65. To lower the pour point, it mixed sample II at a ratio of 1:3 of scrubber ash and fly ash, thereby yielding a basicity of 1.92. A lab-scale 100 kW nontransferred plasma torch was first used as the heat source. An Al<sub>2</sub>O<sub>3</sub> crucible containing approximately 300 g of sample material (sample I or II) was placed in the center of the chamber to provide uniform temperature distribution in the crucible. The heating rate was controlled at approximately 7 °C/min. The molten slag was maintained at 1500 °C for 1 h, before being aircooled to room temperature. For the pilot-scale process, it used a 1200 kW DC plasma furnace (capacity 250 kg/h). Before introducing ash into the plasma furnace, 67 kg of scrubber ash and 200 kg of fly ash were mixed (at a ratio of 1:3). The molten sample was maintained above 1500 °C for 1 h to ensure homogeneous melting. The melt was then rapidly quenched in water to yield quenched slag.

Each sample of slag was then ground to less than 74  $\mu$ m. The ground samples were pressed at  $150 \text{ kgf/cm}^2$  into a

Table 1 Chemical composition of scrubber ash and fly ash.

Chemical composition	Scrubber ash (wt.%)	Fly ash (wt.%)
SiO <sub>2</sub>	2.05	21.63
$Al_2O_3$	0.45	8.60
$Fe_2O_3$	0.27	2.45
CaO	42.57	28.04
MgO	0.98	2.87
Na <sub>2</sub> O	4.83	4.55
K <sub>2</sub> O	4.64	4.43
TiO <sub>2</sub>	0.09	2.00
CuO	0.13	0.10
MnO	0.02	0.19
$SO_3$	3.79	8.31
$Cr_2O_3$	0.01	0.13
ZnO	1.47	1.26
CdO	0.47	0.14
PbO	0.47	0.22
$ZrO_2$	< 0.01	< 0.01
$P_2O_5$	0.37	3.07
NiO	< 0.01	0.02
$SnO_2$	0.15	0.26
Cl <sup>-</sup>	31.5	9.8

 $4 \text{ cm} \times 1.5 \text{ cm} \times 0.7 \text{ cm}$  stainless mold without the use of any binder. The green bodies thus formed were subsequently sintered and heat treated using a conventional laboratory electric furnace at temperatures ranging between 850 and 1150 °C for 2 h, before cooling to room temperature. Glassceramics (I, II, and Q) were produced and then cut in addition to polished for analysis. The experimental procedure is shown in Fig. 1. For SEM examination, a Hitachi S-4700 scanning electron microscope was employed to examine the microstructure of the materials polished and etched using 2% HF for 1 min. XRD was performed with a Rigaku D/MAX-VB diffractometer operating with a Cu Ka radiation at scanning speeds of 4° min<sup>-1</sup> from 5–90°. Crystalline phases were identified by comparing the intensities and positions of Bragg peaks with those listed in the Joint Committee on Powder Diffraction Standards (JCPDS) data files. Thermal properties, such as glass transition, crystallization, and melting temperatures, were analyzed using differential thermal analysis (DTA). Physical properties such as density and water adsorption were evaluated using the Archimedes method. Four-point bending strength and compressive strength tests were carried out with a Testometric 220D bending test machine. To test for chemical resistance, 5 wt.% of acid/alkali solutions were prepared by mixing HCl, H<sub>2</sub>SO<sub>4</sub>, CH<sub>3</sub>COOH, or NaOH, respectively, with DI water. Bulk samples of 5 g were boiled in 50 ml of the above acid/alkali solutions for 1 h. Weight loss was then measured to determine chemical resistance. For each procedure, at least five samples were tested and the results were averaged.

## 3. Results and discussion

Table 2 lists the properties and volume/weight reduction of slag samples after the melting process. It was interesting to discover that the hardness, porosity, and water absorption characteristics of the quenched slag were better than those of slag-I and slag-II. Generally, feeding higher proportions of scrubber ash induces large reductions in volume following lab-scale plasma vitrification. The ratio of the reduction of volume for slag-I and slag-II was 7.3 and 4.2, respectively. This could be due to the fact that the scrubber ash had more combustible or organic constituents.

Results of the toxicity characteristic leaching procedure of the ashes and slags in this study are provided in Table 3. Samples showed insignificant leachability characteristics for Zn, Cd, Pb, and Cu following vitrification in the form of slag. The quantity of heavy metal extracted was below the limits established by the Environmental Protection Agency (EPA) of Taiwan. The leachability characteristics for heavy metals were low because the heavy metal ions replaced the Ca<sup>+2</sup> or Al<sup>+3</sup> ions held within a framework of glass. It should be noted that the total elemental mass-balance was not considered in this study because volatile metals, such as Cd, Zn, and Pb, may be released as gas during the melting stage. Therefore, a secondary air pollution control system should be established to detect metals that are easily volatilized at lower temperatures, when using thermal plasma molten technology to treat incinerator ash.

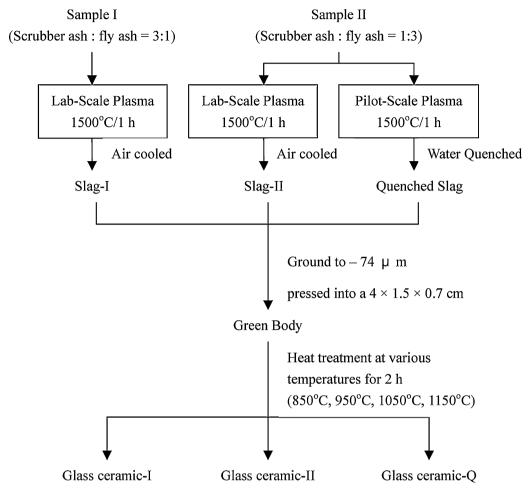


Fig. 1. Experimental procedures.

Thermal properties, such as glass transition, crystallization, and melting temperature, were analyzed using differential thermal analysis at a heating rate of 10 °C/min, and the results are shown in Fig. 2. No apparent glass transition or crystallization peaks were observed in the slag-I and slag-II samples; however, the melting point for slag-I and slag-II appeared at 1451 °C and 1435 °C, respectively. This is because mineral phases had already been formed in the slag at a slower cooling rate. For quenched slag, the onset of the glass transformation temperature was 720 °C, with the first shallow endothermic peak at approximately 750 °C, representing the dilatometric softening point of the low viscosity

Table 2 Slag properties and volume/weight reductions after melting process.

Properties	Slag-I	Slag-II	Quenched Slag					
	The ratio of scrubber ash:fly ash							
	3:1	1:3	1:3					
Volumetric density (g/cm <sup>3</sup> )	2.9	3.0	3.0					
Porosity (%)	1.3	4.7	1.1					
Water absorption (%)	0.5	1.6	0.4					
Knoop hardness (Hv)(GPa)	0.8	1.3	5.6					
Volume reduction ratio	7.3	4.2	_					
Weight reduction ratio	1.4	1.6	_					

separated phase. An exothermic crystallization peak occurred at 889  $^{\circ}$ C, and the endothermic reaction was observed at 1227  $^{\circ}$ C representing the formation of the liquid-phase. Normally, the nucleation temperature is 50–100  $^{\circ}$ C above the dilatometric softening point. Consequently, the temperature selected for heat treatment in this study was between 850 and 1150  $^{\circ}$ C.

X-ray diffractometry analysis of the glass—ceramic samples (2 h of heat treatment at various temperatures) showed various trends towards crystallization as a function of thermal treatment temperature. The results are shown in Figs. 3–5. As seen in the figure, mayenite ( $Ca_{12}Al_{14}O_{33}$ ) was formed in slag-I; while gehlenite ( $Ca_{2}Al_{2}SiO_{7}$ ) and calcium magnesium chloride silicate ( $Ca_{8}Mg(SiO_{4})_{4}Cl_{2}$ ) were formed in slag-II, due to the low cooling rate. Following heat treatment between 850 and

Table 3 TCLP results for ashes and slags.

Elements (mg/l)	Zn	Cd	Pb	Cu
Scrubber ash	3.7	< 0.1	84.3	0.2
Fly ash	0.1	0.2	0.8	< 0.1
Slag-I	< 0.1	< 0.1	0.4	< 0.1
Slag-II	< 0.1	< 0.1	0.3	< 0.1
Quenched slag	N.D.	N.D.	0.3	< 0.1
Taiwan-EPA regulated TCLP limit	25.0	1.0	5.0	15.0

ND indicates not detected.

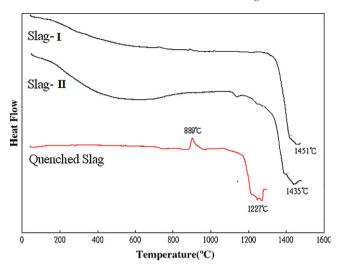


Fig. 2. DTA curves for slag-I, slag-II, and quenched slag.

1150 °C, the main mineral phases remained the same. Mayenite and calcium magnesium chloride silicate are rarely found in slag or glass–ceramics produced from incinerator ash. The reason for the formation of these two mineral phases requires further study. However, for the quenched slag, the main mineral phases changed from amorphous to diopside (Ca(Mg,Al)(-Si,Al)<sub>2</sub>O<sub>6</sub>) and gehlenite following heat treatment between 850 and 1150 °C. With an increase in temperature during heat treatment, the intensities decreased slightly as the rate of nucleation and crystal growth declined at higher treatment temperatures. Similar glass–ceramic results have been observed in various slag-based systems [24–26].

SEM microstructural examinations after heat treatments at different temperatures were performed and the results are shown in Fig. 6. As seen in the figure, the glass–ceramic material was not entirely homogeneous. Several cracks can be observed in glass ceramic-I at 850 °C/2 h, caused by treatment temperature producing low to fully sintered results. Following heat treatment at 1150 °C, the sample was completely sintered.

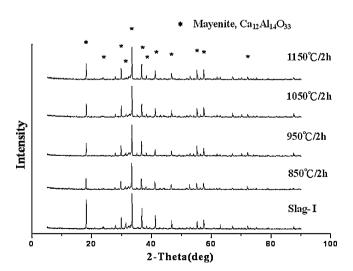


Fig. 3. XRD results of the Slag-I and Glass Ceramic-I after different heat treatment temperature for 2 h.

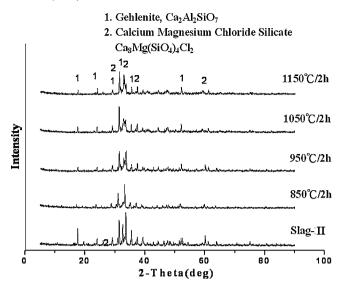


Fig. 4. XRD results of the slag-II Glass Ceramic-I -II after different heat treatment temperature for 2 h.

As for Glass Ceramic-II, the size of the crystals increased with an increase in heat treatment temperature to 1150 °C/2 h, reaching 0.5–1.0  $\mu m$ . Superior crystallinity was achieved using quenched slag as the treated sample, at Glass Ceramic-Q at temperatures between 850 and 1150 °C, and the grain size increased from 0.2  $\mu m$  to 1.0  $\mu m$ . For samples prepared from quenched slag, the quantity of nuclei increased sharply in the parent glass at lower temperatures, thereby forming material with a fine grain. Nevertheless, with an increase in heat treatment temperature, the rate of crystal growth increased and the number of nuclei decreased, resulting in the formation of grains of a larger size.

Various properties of glass-ceramics obtained from different slags and heat treatment temperatures are listed in Table 4. With the exception of glass-ceramics produced from quenched slag at 1150 °C, the density, porosity, and water absorption of most glass-ceramics were well correlated, increasing with an

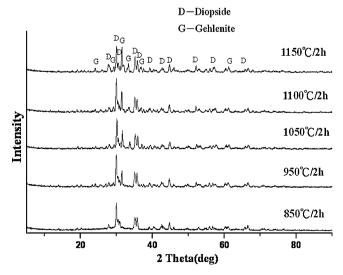


Fig. 5. XRD results of the Glass Ceramic-Q after different heat treatment temperature for  $2\,h.$ 

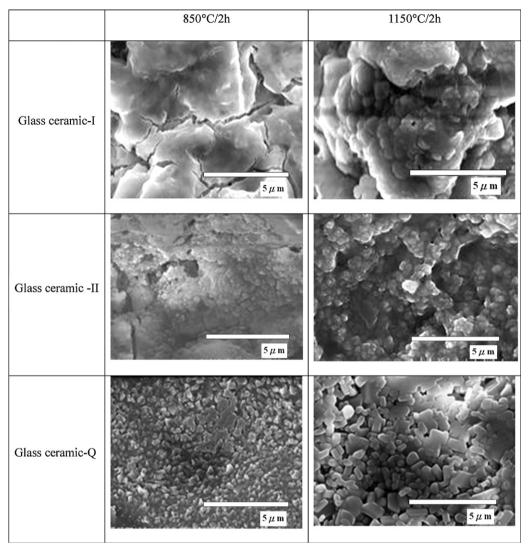


Fig. 6. SEM micrographs of microstructure material samples heat-treated at 850 °C/2 h and 1150 °C/2 h.

increase in temperature. This could be attributed to the coexisting evolution of gas resulting from the reduction iron or decomposition of alkaline metal salts releasing gas [27,28]. In the case of Glass Ceramic-Q formed at 1150 °C, both density and hardness increased, while porosity and water absorption decreased sharply. Following the release of gasses at temperatures around 1000–1100 °C, crystal began melting together at higher temperatures. As a result, the Glass Ceramic-Q sample condensed, providing improved characteristics.

For mechanical properties, the compressive strength and four-point bending strength of glass—ceramics were found to increase with an increase in temperature from 850 °C to 1150 °C. Generally, glass—ceramics produced by heat treatment at 1150 °C showed the best characteristics, particularly Glass Ceramic-Q that obtained from the quenched slag at a temperature of 1150 °C. During heat treatment of slags in this system, crystallization yielded a fine grain microstructure, due to the separation of the liquid-phase at temperatures

Table 4 Properties of the glass–ceramics.

Properties	Glass ceramic -I (prepared from slag-I)			Glass ceramic -II (prepared from slag-II)			Glass ceramic-Q (prepared from quenched slag)				Marble[30]	Granite[30]		
Temp. (°C)	850	950	1050	1150	850	950	1050	1150	850	950	1050	1150	_	_
Volumetric density (g/cm <sup>3</sup> )	2.4	2.3	2.4	2.3	2.2	2.2	2.2	2.1	2.3	2.3	2.4	2.9	2.6-2.8	2.6-2.8
Porosity (%)	7.2	9.3	9.3	11.0	29.7	29.2	31.0	33.1	26.9	27.2	26.9	7.2	_	_
Water absorption (%)	2.8	3.9	3.9	4.6	13.7	13.1	14.2	15.5	11.6	11.6	11.5	4.1	_	_
Knoop hardness (Hv) (GPa)	1.0	1.0	1.1	1.1	1.1	1.1	1.5	1.8	1.0	1.4	1.8	6.5	_	_
Compressive strength (MPa)	7.6	8.8	13.6	20.4	29.9	44.6	45.9	56.2	25.5	35.5	50.1	107.4	90-230	60-300
Binding strength (MPa)	5.7	7.6	7.7	13.4	30.8	30.2	35.5	40.7	9.1	14.2	17.5	29.3	14–17	12–15

Table 5 Chemical resistance for glass–ceramics (wt.% loss).

Properties	Glass co	eramic –I (p	prepared from	n slag-I)	Glass ceramic –II (prepared from slag-II)			m slag-II) Glass ceramic-Q (prepared from quenched slag)				om	Marble[30]	Granite[30]
Temp. (°C)	850	950	1050	1150	850	950	1050	1150	850	950	1050	1150	_	_
5% HCl	13.6	12.3	11.7	12.0	10.9	13.2	11.1	10.7	7.6	1.1	2.9	2.8	0.01N  HCl > 5	0.01N HCl 2.2-4.2
5% H <sub>2</sub> SO <sub>4</sub>	1.7	5.3	5.5	2.5	3.2	2.9	2.0	1.9	0.9	2.7	1.1	0.9	_	-
5% CH <sub>3</sub> COOH	10.5	9.6	9.6	9.2	5.4	5.6	5.3	5.2	1.6	0.9	0.6	0.5	_	_
5% NaOH	1.5	1.6	3.0	1.5	0.9	1.1	1.2	1.0	1.2	1.2	0.5	0.2	0.01N NaOH >5	0.01N NaOH 2.4-3.6

Table 6
Physical and mechanical properties of glass–ceramics made from incinerator ashes.

Reference	This study (glass-ceramic Q)	Park et al. [31]	Karamanov et al. [30]	Kim et al. [32]	Cheng et al. [33]	Bernardo et al. [9]	Roether et al. [34]
Main starting material Main crystalline phase	Incinerator fly ash Diopside, gehlenite	Incinerator fly ash Diopside	Incinerator fly ash Ferrobustamite	Incinerator fly ash Gehlenite, augite, calcium aluminium oxide	Incinerator fly ashes Gehlenite, wollastonite, anorthite, mayenite, augite	Incinerator fly ashes Gehlenite, wollastonite	Incinerator fly ashes Melilite, wollastonite, anorthite, kyanite
Hardness (GPa)	7.9	6.73	5.6-5.9	6.7	1.0–2.2	_	6.0
Density (g/cm <sup>3</sup> )	2.9	2.78	2.61-2.65	2.78	2.1-2.4	2.56-2.84	2.6
Compressive strength (MPa)	314.7	_	220-360	_	13.9-98.1	_	_
Bending strength (MPa)	110.0	127	48–69	127	5.1–24.7	57.5-110.7	81

between 850 and 1150 °C. Generally, an increase in the strength of glass-ceramic materials is a result of their fine grain and uniform microstructure. As the temperature of heat treatment increased, the number of nuclei began increasing, causing the formation of fine grains and an increase in the grain boundary area, thereby improving the physical and mechanical properties. Table 5 shows the results of testing glass-ceramics produced from various slags for chemical resistance. As seen in the table, the durability of glass-ceramics is not generally well correlated with heat treatment temperature. The chemical stability of glassceramic materials is directly associated with the composition and quantity of crystalline phase as well as morphology. The microstructure of glass-ceramics influences chemical resistance to acid and alkaline solutions. However, high weight losses were noted for the HCl durability test, due to gelatinization of gehlenite in an HCl solution [29]. The glass-ceramics produced from the quenched slag at 1150 °C still showed the greatest chemical resistance. Tables 5 and 6 compare the results from other glass-ceramics derived from incinerator fly ash waste with those of the current study. The resulting physical/mechanical and chemical resistance values in this study demonstrate that the properties of glass-ceramic produced from incinerator ash are on par with those of natural marble and granite [30].

#### 4. Conclusions

This study carried out the treatment and characterization of scrubber ash and fly ash from incinerators at a mixing ratio of 3:1 and 1:3, respectively, using lab-scale and pilot-scale thermal plasma systems. Both treatment processes produced glassceramic products with desirable properties on par with those of natural marble or granite, following vitrification of incinerated ash and a subsequent powder sintering process. The major phases in glass-ceramics produced from air-cooled slags were mayenite, gehlenite, and calcium magnesium chloride silicate. In addition, gehlenite and diopside were formed in quenched slag at a heat treatment temperature of 850-1150 °C. Comparing the physical/mechanical properties and chemical resistances of the glass-ceramics fabricated in this study indicates that the quenched slag from the pilot-scale plasma system possesses the best characteristics, implying that the quenching process could offer an improved method for the treatment of incinerator ash. The slag-based glass-ceramic shows great potential for use as floor tiles and other building applications.

## Acknowledgements

The National Science Council of ROC and Institute of Nuclear Energy Research, ROC, supported this study under contract NSC95-2221-E027-057-MY2, which was gratefully acknowledged

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