



# Development of a sintering process for recycling oil shale fly ash and municipal solid waste incineration bottom ash into glass ceramic composite



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## ABSTRACT

Oil shale fly ash and municipal solid waste incineration bottom ash are industrial and municipal by-products that require further treatment before disposal to avoid polluting the environment. In the study, they were mixed and vitrified into the slag by the melt-quench process. The obtained vitrified slag was then mixed with various percentages of oil shale fly ash and converted into glass ceramic composites by the subsequent sintering process. Differential thermal analysis was used to study the thermal characteristics and determine the sintering temperatures. X-ray diffraction analysis was used to analyze the crystalline phase compositions. Sintering shrinkage, weight loss on ignition, density and compressive strength were tested to determine the optimum preparation condition and study the co-sintering mechanism of vitrified amorphous slag and oil shale fly ash. The results showed the product performances increased with the increase of sintering temperatures and the proportion of vitrified slag to oil shale fly ash. Glass ceramic composite (vitrified slag content of 80%, oil shale fly ash content of 20%, sintering temperature of 1000 °C and sintering time of 2 h) showed the properties of density of  $1.92 \pm 0.05 \text{ g/cm}^3$ , weight loss on ignition of  $6.14 \pm 0.18\%$ , sintering shrinkage of  $22.06 \pm 0.6\%$  and compressive strength of  $67 \pm 14 \text{ MPa}$ . The results indicated that it was a comparable waste-based material compared to previous researches. In particular, the energy consumption in the production process was reduced compared to conventional vitrification and sintering method. Chemical resistance and heavy metals leaching results of glass ceramic composites further confirmed the possibility of its engineering applications.

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## 1. Introduction

As one of the most important crude oil substitute resources, oil shale has been widely used to produce liquid shale oil due to the increase in the price of crude oil (Guo et al., 2014a; Jiang et al., 2007; Reinik et al., 2011). It is reported that shale oil (calculated based on the in situ oil shale) accounted for about 400 billion tons of oil that is higher than worldwide total for traditional crude oil (about 300 billion tons) (Han et al., 2014; Qian et al., 2008). However, the resulting oil shale fly ash (OSFA) is considered environmentally hazardous due to high alkalinity and heavy metals concentrations (e.g. Cr, Cd, Zn, Pb, Cu) (Blinova et al., 2012; Reinik et al., 2011). In order to reduce the hazards of oil shale fly

ash and achieve its resource utilization, the production of glass ceramics was studied in our previous study (Luan et al., 2010) and the results showed that it would be suitable raw material to synthesize glass ceramics by adding analytic reagent calcium oxides. Due to unbalance components in some solid wastes, in practice, the supplementing of some oxides or natural resources was necessary measurement to obtain desired crystals and good performances (Kang and Kang, 2012; Wang et al., 2010; Wu et al., 2013). From the viewpoint of natural resources conservation and maximal (100%) waste utilization, a cost-effective method involving the cheaper raw materials and economical process will be more popular for the recycling of large scale wastes. Therefore, the production of glass ceramic composites by utilizing various wastes with complementary components seems to be an attractive strategy. On the other hand, increasing incineration plants in China exhaust large quantities of wastes in the form of bottom ash (BA), leading to disposal, economical, and environmental problems. The generated volume of bottom ash usually ranged between 10% and

*Abbreviations:* MSWI, municipal solid waste incineration; OSFA, oil shale fly ash; BA, bottom ash; VS, vitrified slag; RAM, raw ash mixture of 80 wt.% oil shale fly ash and 20 wt.% bottom ash; SCS, slow cooling slag.

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12% of the initial volume of the wastes and weight between 20% and 35% of the initial wastes (Andreola et al., 2008; Barbieri et al., 2002; Monteiro et al., 2008). It is also identified as a hazardous waste due to the release of hazardous heavy metals and chlorides into the environment. Municipal solid waste incineration bottom ash generally contains high level of calcium content because the calcium hydroxide is injected into the waste gas stream to neutralize acid gas. Based on the analysis above, the comprehensive utilization of oil shale fly ash with high silica content and bottom ash with high calcium oxide content might be an attractive strategy to synthesize glass ceramics in which silica and calcium oxide are glass network former and modifier.

Glass ceramics as a kind of promising material have attracted much attention due to a variety of unique properties. Recent years, the conversion of wastes into glass ceramics has gradually become an important method to improve the recycling of hazardous inorganic wastes into value-added materials while simultaneously reducing the leaching concentrations of heavy metals. Various hazardous industrial (Erol et al., 2008; Kavouras et al., 2007; Zhao et al., 2012) and municipal wastes (Andreola et al., 2008; Cheng et al., 2011; Cyr et al., 2012; Yang et al., 2008) have been reported to synthesize glass ceramic materials by devitrifying a glass by single or two-stage heat treatment. Among these methods, the high-temperature melting as one of the most widely used methods could greatly reduce the volume of wastes and stabilize the heavy metals into the glass matrix, but the process (1400–1600 °C) is general energy-intensive and therefore expensive, which does not meet the economic strategy in terms of the recycling of wastes. Another common option for glass ceramics production is a direct heat treatment process. Although the raw materials are not melted, it need to be shaped firstly by compacting the mixed powder and then sinter the compact at targeted heat treatment temperatures (Aloisi et al., 2006; Appendino et al., 2004; Barbieri et al., 2002; Bernardo et al., 2009; Tang et al., 2013). It is also a cost in producing the powder and there are some limitations on the size and shape of compositions that may be compacted. Therefore, it is important to develop a new processing method in order to implement widespread availability of these hazardous industrial and municipal wastes.

Vitrification of hazardous wastes has been proved to be an attractive method for a safe immobilization of heavy metals in the glass matrix and the obtained vitrified slag (VS) can convert into glass ceramic materials through a two-stage heat treatment (Cheng, 2004; Lin et al., 2006; Luan et al., 2010; Kavouras et al., 2007). From the viewpoint of energy consumption reduction, if a certain amount of raw materials can directly reuse without melting, the energy consumption in the process can be reduced compared to common vitrification and sintering method. Based on this consideration, a more economical and simple method of co-sintering of vitrified amorphous slag and oil shale fly ash is proposed for the recycling of these two kinds of fly ash in the study. Oil shale fly ash and bottom ash were vitrified into the slag and then converted into the glass ceramic composite with the addition of various percentages of oil shale fly ash by the subsequent sintering process. The objective of our research is to explore the possibility of producing waste-based glass ceramic composites by the proposed method. To validate this hypothesis, the present study has been conducted to (i) produce glass ceramic composites using vitrified slag with various percentages of oil shale fly ash; (ii) characterize some important indexes of the products (density, weight loss on ignition, sintering shrinkage, compressive strength, chemical resistance and heavy metal leaching); (iii) study the phase transformation process and co-sintering mechanism of vitrified amorphous slag and oil shale fly ash by thermal analysis and XRD analysis. The results of the study will provide fundamental knowledge for the development of

waste-based glass ceramic composites through a simple and low energy consumption process.

## 2. Materials and methods

### 2.1. Raw material

OSFA used in this study was obtained from the thermal power plant, Jilin, China; and MSWI BA was obtained from municipal solid waste incinerator, Dalian, China. OSFA and MSWI BA that removed any coarse impurities were dried at 105 °C for 24 h in an electric dry oven, and then were graded to pass sieve NO. 150 (the diameter of mesh is 106 µm) for subsequent experiments. As a result of preliminary experiments, the raw ash mixture of 80% OSFA and 20% BA (marked as RAM) by weight were used in this study.

### 2.2. The preparation of glass ceramic composites

A total amount of 80 g RAM were separately put in a corundum crucible and melted at 1500 °C for 1 h to ensure complete melting. The melt was rapidly poured into water to obtain the vitrified slag. Meanwhile, the slow cooling slag (SCS) was obtained at a cooling rate of 4 °C/min from the melting temperature to room temperature. Then they were dried, ground and sieved to powders below 106 µm for subsequent experiments.

The vitrified slag (VS) with the addition of 10, 20 and 30 wt.% OSFA, denoting to VS10, VS20 and VS30, were homogenized in ball-mill for 1 h. The mixed powder was filled in the corundum crucible and a single sintering scheme with 2 h dwelling time was used for the targeted temperatures ranged from 850 to 1000 °C. The sintered samples were then cooled inside the furnace to room temperature to obtain the glass ceramic composites. Meanwhile, SCS and RAM samples with the addition of 10, 20 and 30 wt.% OSFA, labelled SCS10, SCS20, SCS30, RAM10, RAM20 and RAM30, were sintered at 1000 °C for 2 h, respectively.

### 2.3. Analysis and methods

#### 2.3.1. Chemical analysis

The chemical compositions of OSFA and BA were determined by X-ray fluorescence (XRF) (PDA-5500II, Shimadzu, Japan). The heavy metal concentrations of OSFA and BA were determined by inductively coupled plasma–optical emission spectrometry (ICP–OES).

#### 2.3.2. Thermal analysis

Thermal behavior of OSFA, BA and VS were tested by thermogravimetric (TG) and differential thermal analysis (DTA) (TG/DTA 6300, NSK, Japan) to determine the sintering temperatures. About 20 mg of samples was heated to 1100 °C at the rate of 10 °C/min in nitrogen atmosphere.

#### 2.3.3. X-ray diffraction (XRD) analysis

Crystalline phase components of raw materials and glass ceramic composites were analyzed by XRD (D/MAX-2400, Rigaku, Japan). They were analyzed over a range of  $2\theta$  angles from 10° to 60° with Cu K $\alpha$  radiation at 4° min<sup>-1</sup> scanning speed. The measured results were studied by the standard powder diffraction database of International Centre for Diffraction Data (ICDD PDF-2 Release 2004).

#### 2.3.4. Leaching toxicity

Toxicity assessment of selected glass ceramic composites were tested in a leaching experiment modified from the U.S. EPA SW-846 Method 1311-TCLP, using a pH 4.93 extraction fluid as

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