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## Production of pyroxene ceramics from the fine fraction of incinerator bottom ash

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### ABSTRACT

Incinerator bottom ash (IBA) is normally processed to extract metals and the coarse mineral fraction is used as secondary aggregate. This leaves significant quantities of fine material, typically less than 4 mm, that is problematic as reuse options are limited. This work demonstrates that fine IBA can be mixed with glass and transformed by milling, calcining, pressing and sintering into high density ceramics. The addition of glass aids liquid phase sintering, milling increases crystalline reactivity and calcining reduces volatile loss during firing. Calcining also changes the crystalline phases present from quartz (SiO<sub>2</sub>), calcite (CaCO<sub>3</sub>), gehlenite (Ca<sub>2</sub>Al<sub>2</sub>SiO<sub>7</sub>) and hematite (Fe<sub>2</sub>O<sub>3</sub>) to diopside (CaMgSi<sub>2</sub>O<sub>6</sub>), clinoenstatite (MgSiO<sub>3</sub>) and andradite (Ca<sub>3</sub>Fe<sub>2</sub>Si<sub>3</sub>O<sub>12</sub>). Calcined powders fired at 1080 °C have high green density, low shrinkage (<7%) and produce dense (2.78 g/cm<sup>3</sup>) ceramics that have negligible water absorption. The transformation of the problematic fraction of IBA into a raw material suitable for the manufacture of ceramic tiles for use in urban paving and other applications is demonstrated.

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### 1. Introduction

There are currently 25 energy from waste (EfW) plants operating in the UK with more than 450 plants in Europe, the majority of which combust residual waste, the material that remains after commercially viable recyclable materials have been extracted from municipal solid waste (MSW) (Nixon et al., 2013). The combustion process produces incinerator bottom ash (IBA) and this is normally quenched on exiting the combustion chamber. After ageing and weathering, IBA forms a loosely agglomerated granular material with 250–320 kg typically produced per tonne of input residual waste to an EfW plant. This heterogeneous mix of concrete, ceramics, glass, brick, clinker and particles of fused materials contains significant ferrous metals (~10 wt.%) and non-ferrous metals (~1–3 wt.%), mainly copper and aluminium, which are extracted for recycling. Additional IBA processing typically uses standard drum or flat screen decks with mesh sizes to sort different size fractions with further metal recovery using magnetic and eddy current separators. The metals extracted have significant value and the mineral fraction greater than 4 mm is extensively used as secondary recycled aggregate. In excess of 5 million tonnes of IBA aggregate have been used in the UK, predominantly as a sub-

base and capping material in numerous civil engineering applications. IBA also contains approximately 45 wt.% of fine material, most of which is significantly less than 4 mm in diameter, which is not ideal for use as aggregate (Chimenos et al., 1999; Crillesen and Skaarup, 2006; Sabbas et al., 2003). There are currently limited alternative beneficial reuse options for this material which is typically either blended back into IBA aggregate or disposed of to landfill, neither of which is ideal.

Previous research has used IBA for the production of ceramics and glass–ceramics. Ceramic manufacturing typically involves sintering compacted powder samples, while in the glass–ceramic process raw materials are melted to form a glass at relatively high temperatures which is then held at lower temperatures to promote crystallisation. An advantage of these processes is that they encapsulate heavy metals in the ceramic or glass–ceramic formed, so that metal leaching is normally extremely low (Rawlings et al., 2006).

A significant amount of research has involved melting IBA to form a glass which is then ground, compacted and sintered. For example, IBA was mixed with glass and melted at 1500 °C for 5 h and the glass formed was quenched in water and dry milled to give a homogeneous powder with an average particle size of <38 μm (Barbieri et al., 2002). This powder was then pressed and sintered at 1000 °C for 20 min to form glass–ceramics with densities between 1.8 and 2.3 g/cm<sup>3</sup>. In similar research IBA was vitrified

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at 1400 °C for 1 h and the glass formed ground to less than 65 µm. This powder was then mixed with fine corundum-based waste from an aluminium foundry, uniaxially pressed and sintered at temperatures between 760 and 830 °C for 1 h, to produce glass–ceramic tiles with densities between 2.64 and 2.72 g/cm<sup>3</sup> (Appendino et al., 2004; Ferraris et al., 2001). IBA has been melted at 1400 °C for 1 h to produce a glass frit that was ground to less than 75 µm and mixed with different amounts of fine alumina waste. The powder formed was uniaxially pressed and sintered at 1250 °C to produce glass–ceramics with densities between 2.3 and 2.4 g/cm<sup>3</sup> (Aloisi et al., 2006). Vitriified IBA has also been processed into a powder for the production of porcelain stoneware (Rambaldi et al., 2010). The IBA was melted at 1400 °C for 1 h and the glass formed was ground and sieved to <40 µm. It was then substituted for between 5 and 10 wt.% of the feldspar used to manufacture porcelain stoneware and this resulted in a 20 °C reduction in the sintering temperature. These processes clearly produce high quality glass–ceramics but involve melting at high temperatures and therefore high energy. They have also used all the mineral fraction of IBA remaining after metal extraction, whereas industrial application already exists for much of this material as secondary aggregate.

Sintered ceramics have been produced by mixing IBA with clay (Schabbach et al., 2012). The mixes were ground to <75 µm, uniaxially pressed and sintered at 1200 °C for 1 h to produce ceramic tiles with maximum densities of 2.78 g/cm<sup>3</sup>. IBA has been blended with marine dredging spoil (Baruzzo et al., 2006) and the powders formed pressed and sintered at 1130 °C to form ceramics with densities of 2.70 g/cm<sup>3</sup>. IBA has also been mixed with water treatment sludge cake and sintered at 1100 °C to give ceramic bricks with densities of 2.5 g/cm<sup>3</sup> and water absorption of 2.8 wt.% (Lin et al., 2006).

Sintered ceramics have also been formed using the <8 mm mineral fraction of IBA (Bethanis et al., 2002; Cheeseman et al., 2003). The processing involved ball milling, uniaxially pressing and sintering at 1100 °C and this produced diopside-containing ceramics with densities of ~2.6 g/cm<sup>3</sup>. These samples exhibited high (~20%) linear shrinkage during sintering which is problematic because high shrinkage causes warping and deformation and this increases scrap production. The linear shrinkage of commercial clay ceramics is typically in the range between 7% and 8% (Acchar et al., 2006).

The research reported in this paper has investigated transforming the fine (<4 mm) fraction of IBA into a raw material suitable for the production of ceramic tiles. The research has focussed specifically on the problematic fine fraction of IBA for which alternative reuse applications are not available. The work demonstrates that this material can be transformed into a viable raw material for manufacturing sintered ceramics with high density and hardness that exhibit low shrinkage during firing.

## 2. Experimental procedure

### 2.1. Characterisation of fine IBA

A representative 50 kg batch of <4 mm processed IBA was obtained from a major supplier of secondary IBA aggregate in SE England (Day Group, Brentford facility). This had previously been aged in the environment for approximately 2 months and the ferrous and non-ferrous metals extracted, with the remaining mineral components size sorted for use as secondary aggregate. The problematic less than 4 mm fraction of IBA represents about 45 wt.% of the total IBA received at the processing plant. Representative samples were dried at 105 °C and disc milled (Gy–Ro, Glen Creston Ltd., UK) to form powders with a mean particle size of 35 µm and

pressed into discs for analysis by X-ray fluorescence (XRF, Spectro 2000 Analyser). The crystalline phases in the milled powder were characterised by X-ray diffraction (Philips PW 1830 diffractometer with PW1820 goniometer using Cu K $\alpha$  radiation with an accelerating voltage of 40 kV). The effect of temperature on mass loss was investigated using thermogravimetric analysis (TG/DTA, Stanton Redcroft, STA-1500 Series) on 25 µg samples of milled powder using a ramp rate of 10 °C min<sup>-1</sup>.

### 2.2. Ceramic processing

Initial trials demonstrated that the addition of 20 wt.% of recycled soda lime silica glass significantly improved liquid phase sintering of fine IBA. 500 g sample batches consisting of 400 g of IBA and 100 g glass were therefore prepared using 1 wt.% of polyethylene glycol (PEG-8000) added as a binder. This was wet milled for 24 h in a porcelain ball mill using high-density alumina milling media, a water to charge ratio of 2 and a milling media to charge ratio of 5. The particle size distribution of the milled slurry was determined by laser diffraction (Beckman Coulter, LS-100 Series).

Milled slurries were dried overnight at 105 °C and passed through a 500 µm sieve to form powder suitable for pressing. ‘Green’ tile samples (110 mm × 55 mm × 20 mm) and ‘green’ disc samples (40 mm diameter) were formed by uniaxial pressing at 48 MPa (Nannetti S hydraulic press) in a steel die. Pressed samples were then sintered in an electric furnace (Lenton Thermal Design Ltd., ECF 12/45) at a heating rate of 6 °C min<sup>-1</sup> to temperatures between 1020 and 1100 °C with a dwell time of 1 h at peak temperature.

### 2.3. Production of calcined milled powders

After wet ball milling, drying and sieving, the IBA: glass powder was heated in an alumina crucible to temperatures between 600 and 1100 °C to investigate the effect of calcining temperature. The powders were then lightly ground using a mortar and pestle to produce calcined powders suitable for processing into ceramics using the same procedures as for un-calcined powders.

### 2.4. Physical property characterisation of products

The linear shrinkage LS occurring during sintering was calculated from disc samples by comparing the diameter before and after firing using:

$$LS = (D_o - D_s) * 100/D_o$$

where  $D_o$  is the diameter of the green disc and  $D_s$  the diameter of the sintered disc.

The density of the sintered samples was determined using Archimedes method, taking into account the effect of temperature on the specific gravity of water. The water absorption of sintered samples was determined from the increase in weight of dried samples after immersion in water for 24 h under vacuum to remove the air entrapped within open porosity.

The Vickers micro-hardness of sintered samples was measured with a Leitz Wetzlar 8423 microhardness tester using a 25 g load. Prior to hardness testing the samples were diamond-polished to a 1 µm finish. Average hardness values were calculated from five measurements on five different samples with the Vickers number (HV) calculated from:

$$HV = 1.854(F/D^2)$$

where  $F$  is the applied load (kg) and  $D$  is the length of the diagonal of the indentation measured in mm.

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