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Production of glass–ceramic bodies from the bottom ashes of municipal solid waste incinerators

P. Appendino, M. Ferraris*, I. Matekovits, M. Salvo

Materials Science and Chemical Engineering Department, Politecnico di Torino, C.so Duca degli Abruzzi 24, I-10129, Torino, Italy

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Abstract

The bottom ashes coming from two different municipal solid waste incinerators were vitrified at 1400 °C. The obtained glass, mixed with other wastes coming from metallurgical and mineral industrial wastes, was used as raw material for the production of glass–ceramic tiles. Two different mixtures were used for the tile production: (a) glass from bottom ashes plus corundum-based waste from an aluminum foundry and (b) glass from bottom ashes plus kaolin-based waste from the kaolin ore extraction process. The tiles were prepared in air by a low-cost, low-temperature pressureless sintering process and were morphologically and mechanically characterized. The sintered materials were found to be good candidates for building applications (tiles, bricks, etc.), i.e. they showed bending strengths up to about 60 MPa and Young modulus up to about 53 GPa.

Keywords: Glass ceramics; Mechanical properties; Sintering; Tiles; Waste materials

1. Introduction

Significant amounts of residues, bottom and fly ashes, are produced by municipal solid waste incinerators (MSWIs): the incineration of one ton of municipal waste gives about 300 kg of bottom ash and 30 kg of fly ash. About 750,000 t/year of bottom ash and 130,000 t/year of fly ash are produced in Italy alone.[1] The MSWI ash vitrification has been generally accepted as a very promising alternative to their controlled landfilling. The melting technology reduces the volume of these residues, making the melted glassy slag stable and not toxic.[2,3] After the vitrification treatment, the incineration residues could potentially be reused, for example, as road-fill, concrete aggregate, building materials, etc.. In Japan, the melting process has been adopted in about 24 MSWIs and the effective reuse of the melted slag is under discussion.[2,4] In Europe, there are at least two main examples of vitrification and reuse of ashes: in Bordeaux, France, a MSWI has been integrated with a vitrification facility by plasma torch that can treat the total fly ash production of the site;[5] in Karlsruhe, Germany, a MSWI equipped with a melting furnace produces 7.5 t/h of glass, which is used as aggregate for concrete.[6]

Recent papers have reported on the use of bottom and fly ashes for the production of glass and glass–ceramics for potential architectural and decorative applications,[7–12] for the production of glass fibers[13] and glass matrix composites for potential technical and building applications[14,15] and as raw materials for synthetic aggregates and cement production.[16,17]

Another side of the waste-management problem is that many industries (i.e. metallurgy, ore extraction, etc...) produce wastes not suitable for vitrification: in most cases, these potential raw materials are sent to landfills.

The aim of this work is to utilize wastes coming from MSWIs and from metallurgical and mineral industries to produce glass–ceramic materials.

2. Experimental

Dry bottom ashes from MSWIs of Bergamo (BAS S.p.A, Italy) and of Vercelli (Italy) were vitrified. The

* Corresponding author. Tel.: +39-011-5644-687; fax: +39-011-5644-699.
E-mail address: monica.ferraris@polito.it (M. Ferraris).

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bottom ashes were analyzed by atomic absorption spectroscopy (AAS-Perkin-Elmer 1100). The vitrification of the bottom ashes was performed without any additives at 1400 °C for 1 h, in air, in a chamber furnace (Supertherm HT/17) and using alumina crucibles. The resulting glasses were dark brown. The glass derived from the Bergamo incineration bottom ashes was referred to as BA, while the one derived from the Vercelli incineration bottom ashes was referred to as VC. The experimental details and further information regarding the BA glass are reported in Ref. 15.

The glasses were subsequently ground for AAS and hot-stage optical microscopy (Leitz GmbH AII).

A corundum-based waste material, referred to as CW, from an aluminum alloy manufacturer (Electrolux Zanussi Metallurgica SpA, Italy) was used as additional raw material for the preparation of the sintered tiles. Similarly, a ceramic powder waste derived from the extraction of kaolin ore (ECC, Cornwall, England), referred to as KW, was also used as raw material in addition to the glass slag.

The chemical and mineralogical composition of the two glasses, CW and KW wastes were determined by X-ray diffraction analysis (XRD, Philips PW1710) and energy dispersion spectroscopy (EDS, Philips 525M). The densities were measured by a pycnometer. Thermal analyses were performed by a differential thermal analysis (DTA, Netzsch 4045).

Glass–ceramics were prepared by powder technology by sintering green bodies made of BA + KW and VC + CW powders. The particle size of the glass powders was chosen between 63 and 106 μm; the KW and CW particle size was lower than 45 μm. The glass and the other waste material powders were mixed at a volume ratio of 3:1.

Green bars (about $50\times10\times6.5$ mm^3) were uniaxially pressed using a laboratory hydraulic press (pressure = 165 MPa) at room temperature with the addition of a minimum amount of ethyl alcohol. In order to determine a temperature range for sintering, the green bodies were evaluated for linear shrinkage as a function of temperature in hot-stage optical microscopy, with a heating and cooling rate of 10 °C/min. The sintering temperature was varied from 800 to 1100 °C, according to specimen type (Table 1). Initial experiments saw the soaking time varied from 30 to 120 min; the optimized soaking time for samples of this size was found to be one hour, and the data to be presented here are for 1 h soaks.

Table 1
Sintering temperatures for VC + CW specimens and BA + KW specimens

Bodies	Sintering temperatures (°C)						
VC glass + CW	800	850	880	950	1000		1100
BA glass + KW	–	–	880	950	1000	1050	–

The soaking time is 1 h for each specimen.

The morphological and mineralogical characterization of the sintered samples was conducted by scanning electron microscopy (SEM, Philips 525M) and XRD; the mechanical strength was determined by the three point bending test with a 40 mm outer span and a crosshead speed of 0.1 mm/min (SINTEC D/10). The Young modulus of the sintered glass–ceramics and of the glasses was determined by means of the non-destructive resonance frequency technique (Grindosonic). The Vickers hardness (HV) was determined for the glasses and sintered samples by a macro Vickers tester (Volpert). The bulk density of the sintered materials was calculated by weighing and measurement of specimen dimensions. The total porosity of the samples was measured by a mercury porosimeter (Macropore Unit 120 e Porosimeter 2000, Fisons Instruments). The thermal expansion coefficient of the sintered bodies was measured by a dilatometer (Netzsch 4045).

3. Results

3.1. Characterization of the raw materials

The chemical analysis of the Bergamo and Vercelli MSWI bottom ashes is shown in Table 2: these ashes have similar composition with relatively high contents of SiO_2, Al_2O_3 and CaO.

The XRD analysis of the BA and VC glasses obtained by the bottom ash vitrification indicates that they were completely amorphous, as no crystalline peaks are observable. The glass compositions, listed in Table 2, are very similar: the vitreous systems are mainly constituted

Table 2
Chemical analysis (oxide contents in wt.%) of the two utilized bottom ashes (Bergamo and Vercelli MSWIs) and of the BA and VC glasses

Oxide (wt.%)	Bergamo bottom ash	Vercelli bottom ash	BA glass	VC glass
SiO_2	42.5	40.85	44.2	41.1
Al_2O_3	17.6	13.6	17.8	14.0
Fe_2O_3	5.7	5.7	3.5	4.1
CuO	0.2	0.2	0.1	0.4
MnO	0.1	0.1	0.1	0.2
PbO	0.2	0.3	0.1	4×10^{-2}
Cr_2O_3	4×10^{-2}	7×10^{-2}	0.1	0.1
NiO	2×10^{-2}	6×10^{-2}	2×10^{-2}	1×10^{-2}
ZnO	1.1	0.4	0.8	0.3
CaO	16.4	12.2	16.3	13.9
MgO	2.3	3.1	2.0	4.6
Na_2O	12.9	10.9	11.6	10.3
K_2O	1.8	1.8	1.4	3.2
TiO_2	2.5	1.2	2.0	2.9
Others	Balance	Balance	Balance	Balance

Table 3
Thermal and mechanical properties of BA and VC glasses

Properties	BA glass	VC glass
Glass transition (°C)	630–660	630–650
Crystallization temperature (peak, °C)	920–925	930–950
Melting temperature (onset °C)	1080–1100	1080–1100
Young modulus (GPa)	96±1	80±1
Vickers hardness (GPa)	6±0.1	6±0.1
Indentation toughness (MPa $m^{1/2}$)	0.5±0.2	0.5±0.2
Thermal expansion coefficient (10^{-6} K^{-1})	10.7 (100–500 °C)	9.6 (100–500 °C)
Density (gcm^{-1}	2.60±0.05	2.70±0.05

by silicon, calcium, aluminum and sodium oxides, with minor quantities of iron oxide. The thermal and mechanical properties of the two glasses, reported in Table 3, are comparable. Furthermore, their thermal and mechanical properties are similar to those of another glass obtained by direct melting of MSWI bottom ash.[15]

The CW, as revealed by XRD analysis, is principally constituted by corundum, with small amount of silicon and aluminum. The EDS analysis confirmed this result and also revealed traces of iron and zinc.

The XRD analysis of KW showed that its mineralogical composition is a mixture of kaolinite [$Al_2Si_2O_5(OH)_4$] and zinnwaldite [$KLiFeAl(AlSi_3)O_{10}(F, OH)_2$]. The zinnwaldite is a mica group mineral, typical of the region of origin of KW (St. Austell, Cornwall, UK).[18] The EDS analysis revealed silicon, aluminum, potassium and iron.

The differential thermal analysis of KW shows at about 100 °C an endothermic peak corresponding to the moisture removal and another large endothermic peak in the region 500–600 °C (with peak temperature 570 °C) as a result of dehydroxylation of the silicate lattice. As the temperature was increased, a smaller exothermic peak was formed at about 1020 °C, which is attributed to the formation of mullite.[19]

3.2. Glass–ceramic tiles sintering: process and characterization

The maximum shrinkage versus temperature (measured by heating microscopy) was recorded at about 800 °C for the VC + CW specimens and at about 830 °C for the BA + KW specimens. In both cases the total linear shrinkage is limited: about 5% for VC + CW and less than 3% for BA + KW (Fig. 1).

The SEM observations of the VC + CW specimens sintered at 800, 880, 950 and 1000 °C are shown in Fig. 2. At 800 °C, the samples are highly porous, not homogeneous and have a flaky appearance. Increasing the sintering temperature up to 880 °C, one sees that the specimen densifies and the pores become spherical and smaller. Even more densified are the samples fired at 950 °C (Fig. 2c): the porosity is reduced and pores are well distributed. For the specimens fired at 950 °C, the total porosity, measured by Hg porosimetry, was about 13%. As will be discussed later, this modification of the porosity increases the mechanical properties of the sintered samples.

A further increase in sintering temperature (Fig. 2d) resulted in an enlarged porosity. An analogous microstructural evolution was observed in the fired BA + KW specimens. Fig. 3 shows the SEM observations of BA + KW specimens sintered at 880, 950, 1000 and 1050 °C. For a first sintering temperature range (880–950 °C) the pore size decreases with an increase in temperature and, as a consequence, the sintered specimen is more uniform and compact. For the specimens fired at 950 °C, the total porosity, measured by Hg porosimetry, was about 11%. At temperatures above 950 °C, the

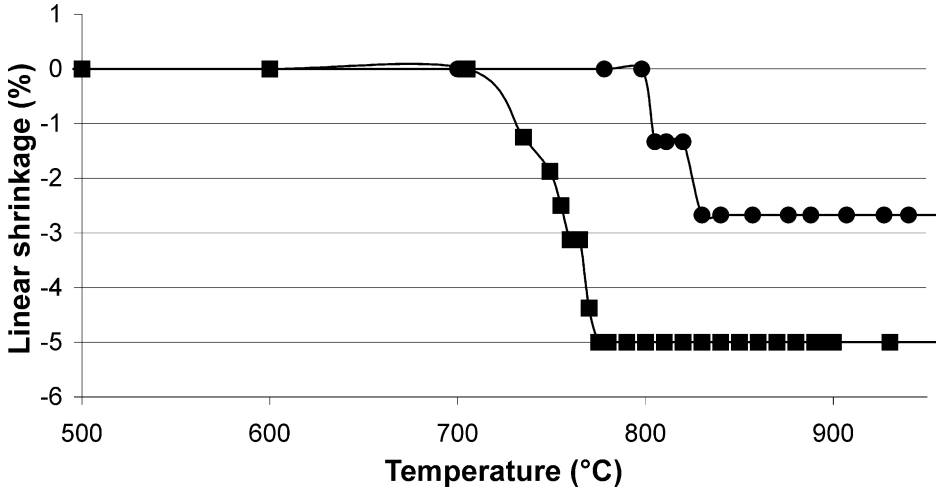

Fig. 1. Shrinkage vs temperature: BA glass plus KW (3:1 volume) green (•) and VC glass plus CW (3:1 volume) green (■), measured by heating microscopy.

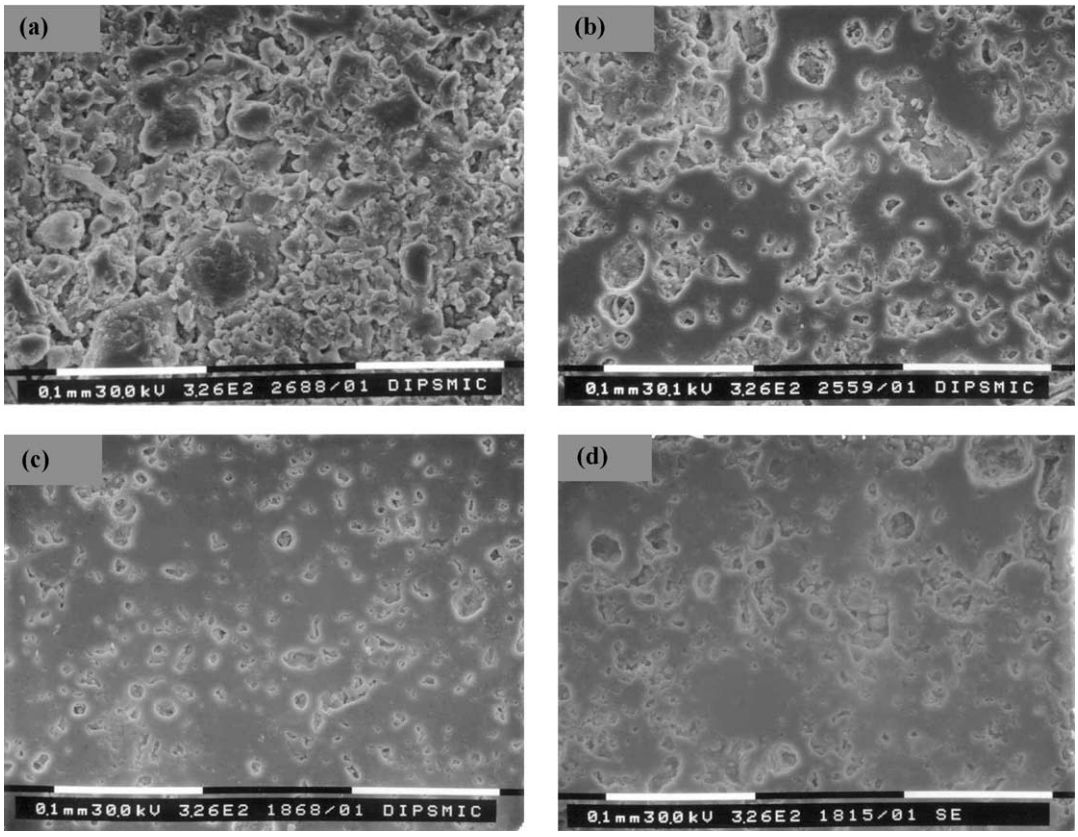

Fig. 2. SEM micrographs of VC + CW specimens sintered at different temperatures (1 h soaking time): (a) 800 °C, (b) 880 °C, (c) 950 °C, (d) 1000 °C.

bloating occurs: with a sintering temperature of 1050 °C (Fig. 3d), some pore diameters reach 150 μm.

The XRD analysis of samples heated to 950 °C for 1 h was performed. For the VC + CW the reflections were assigned to crystalline phases of corundum, feldspars (anorthite, $CaAl_2Si_2O_8$ and albite, $NaAlSi_3O_8$), pyroxenes [diopside, Ca(Mg, Al)(Si, Al)$_2O_6$, augite, Ca(Fe, Mg) Si_2O_6] and traces of metallic aluminum.

In the BA + KW sample sintered at 950 °C, the identified crystalline phases are feldspars (anorthite, $CaAl_2Si_2O_8$ and albite, $NaAlSi_3O_8$) and pyroxenes [diopside, Ca(Mg, Al)(Si, Al)$_2O_6$ and augite, Ca(Fe, Mg) Si_2O_6]. No mullite is present at 950 °C, in agreement with results of other researchers.[19]

The effects of sintering temperature on the density and on the mechanical characteristics (bending strength, Young's modulus and Vicker hardness) are presented in Figs. 4–7. The improvement of the mechanical properties and of the density of the fired bodies with increasing sintering temperature up to 950 °C is a common behavior for all the samples. However, above 950 °C an overall reduction in bending strength, Young's modulus, Vickers' hardness and density was observed.

The average bending strengths (Fig. 5) and Young's modulus (Fig. 6) for the samples fired at 950 °C reach $\sigma = 52$ MPa and $E = 57$ GPa for the VC + CW specimens and $\sigma = 61$ MPa and $E = 53$ GPa for the BA + KW specimens. The ratio of Young's modulus to flexural strength was of the order of 10^3 in agreement with that of most ceramic materials.[19]

The Vickers' hardness (Fig. 7) of the samples fired at 950 °C is 142HV for the VC glass plus CW and 200HV for the BA glass plus KW. These Vickers' hardness correspond to values between 3 and 4 in the Mohs scale.[20]

The thermal expansion coefficient for these specimens resulted 7.4×10^{-6} K^{-1} for VC glass plus CW and 6.4×10^{-6} K^{-1} for BA glass plus KW, in the temperature range 80–280 °C. Some preliminary tests of water absorption showed that the sample with BA glass plus KW has a water absorption of 2.61%.[21]

4. Discussion

4.1. Characterization of raw materials

The chemical composition of the MSWI bottom ashes evidenced the possibility of their direct vitrification by heating. Furthermore, the very similar composition of the bottom ashes coming from these two different MSWI indicates that the high temperature combustion

of municipal solid wastes produces residues of reasonably uniform composition. This result is in agreement with the conclusions obtained by Eusden et al.[22] As expected from this result, the glass obtained by direct vitrification of bottom ashes from different MSWIs showed almost the same thermal and mechanical behavior. The similarity of the compositions and of the thermal and mechanical properties verified for glasses derived from different ashes is an important aspect in their hypothetical use as raw materials in the tile production.

Otherwise, due to their compositions, the CW and KW industrial wastes are not suitable for a direct

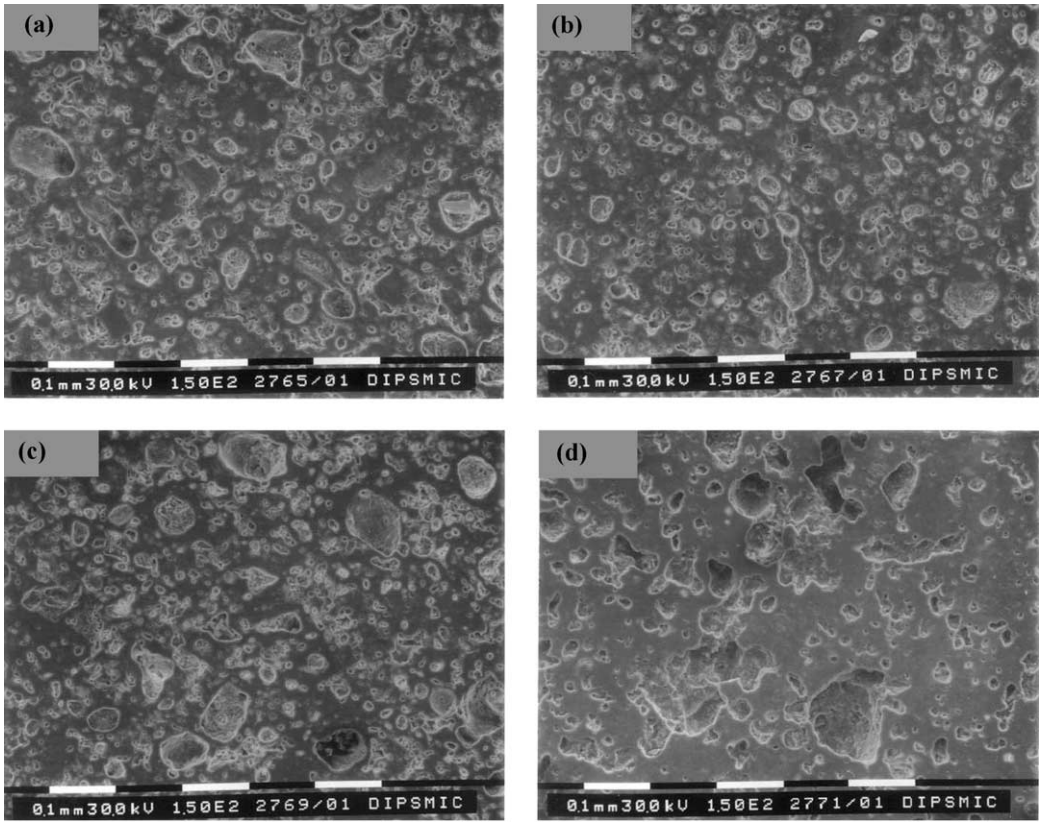

Fig. 3. SEM micrographs of BA + KW specimens sintered at different temperatures (1 h soaking time): (a) 880 °C, (b) 950 °C, (c) 1000 °C, (d) 1050 °C.

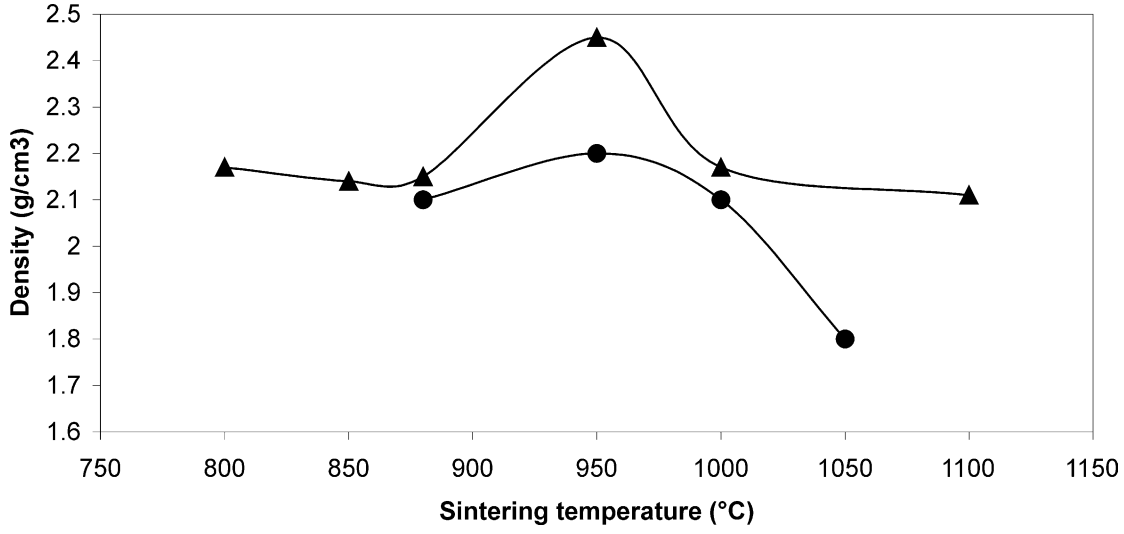

Fig. 4. Variation of density with sintering temperature for the sintered samples (1 h soaking time): ▲ = VC + CW specimens; ● = BA + KW specimens.

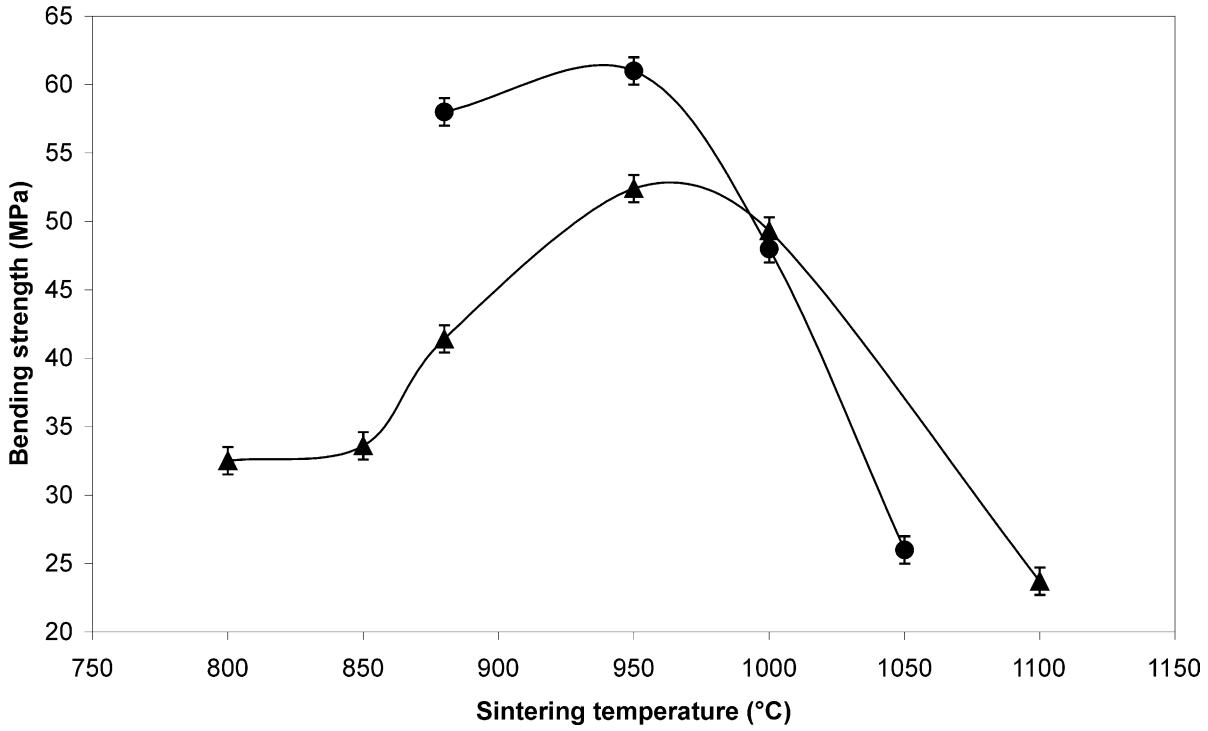

Fig. 5. Variation of bending strength with sintering temperature for the sintered samples (heating rate 10 °C/min; 1 h soaking time): ▲ = VC + CW specimens; ● = BA + KW specimens.

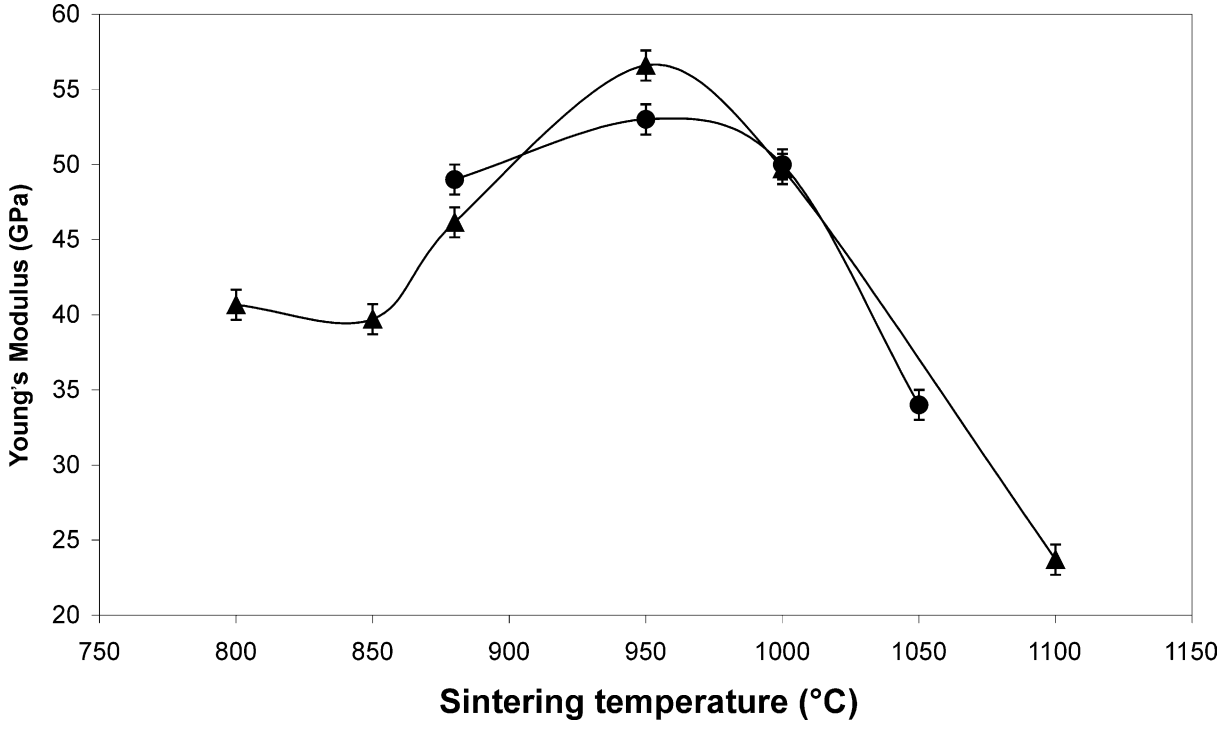

Fig. 6. Variation of Young's modulus with sintering temperature in the sintered samples (heating rate 10 °C/min; 1 h soaking time): ▲ = VC + CW specimens; ● = BA + KW specimens.

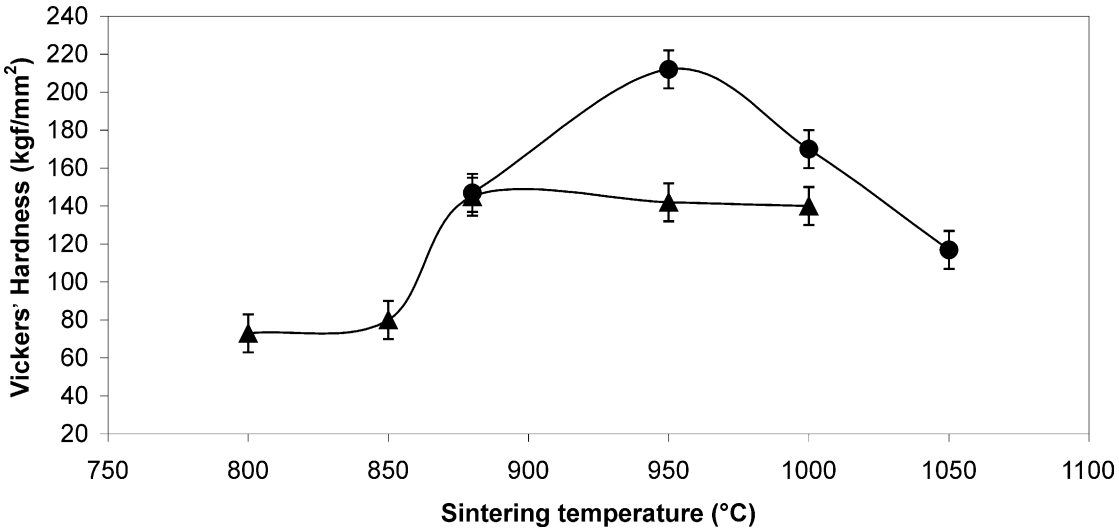

Fig. 7. Variation of Vickers' hardness with sintering temperature in the sintered samples (heating rate 10 °C/min; 1 h soaking time): ▲ = VC + CW specimens; ● = BA + KW specimens.

vitrification, but they can be successfully recycled as raw materials, in addition to BA or VC glass, for the production of ceramic materials.

4.2. Glass–ceramic tiles sintering: process and characterization

The sintering processes are performed at temperatures above the softening point[15] of the two glasses (BA and VC) and it is demonstrated by the presence of nearly spherical pores in the sintered samples (Figs. 2 and 3). Furthermore, the increase of the sintered material densities by increasing the sintering temperature up to 950 °C (Fig. 4) is in accordance with the viscous sintering theory, which correlates the temperature dependence of the sintering process to the temperature dependence of the glass phase viscosity.[23]

The bloating phenomenon, in the form of a high concentration of coarse pores, that starts at about 1000 °C for both the tile compositions (Figs. 2d and 3c and d), takes place by retention of gas within the mass when a considerable quantity of glass of adequate viscosity is present. Since the bloating is a process commonly used for the production of lightweight ceramic materials, e.g., aggregates for concrete, it has been studied by several researchers.[24–26] The mechanism of bloating by iron (III) oxide reduction is well known; Sandrolini et al.[24] gave a detailed description of the fundamental role of iron oxides in the bloating of vitrified ceramic materials with an iron (III) oxide content between 1 and 6 wt.%. At elevated temperature, Fe_2O_3 is partially reduced with the production of oxygen as the bloating gaseous phase, generating large pores within the fired body and determining a density decrease.

In this work, the sintered glass–ceramic bodies were prepared starting from the 70 vol.% of BA or VC glasses that have an iron oxide content between 3.5 and 4.1 wt.% and melting temperatures at about 1080–1100 °C: for these tile compositions, the presence of iron (III) oxide and of a large amount of glassy phase clearly determined the evidence of a relevant bloating when the green bodies are overfired above 950 °C. Anyway, the significant bloating could also be exploited for the preparation of low-cost lightweight ceramic materials (aggregates, insulators, ...) by heating just above 950 °C a mixture of the described wastes.

The overall decrease of the mechanical properties of the samples sintered above 950 °C (Figs. 5–7), can be attributed to a density reduction connected to the beginning of the bloating process, as observed during the SEM microstructural examination. The presence of the large pores, produced by bloating, was detrimental for the mechanical properties of the sintered samples: both the flexural strength and the Young's modulus are reduced to about the half after the bloating started. For the earlier discussed reasons, the sintering at 950 °C was chosen as optimal temperature for these samples.

The bending strengths and the Young's modulus of the materials sintered at 950 °C resulted very good and sufficiently high to satisfy the requests for ceramic wall tiles (i.e. bending strength $\geqslant 12$ MPa[27]) and are higher than those reported for four commercial ceramic tiles in Ref. 23. Their Vickers' hardness (Fig. 7) is sufficient for an application as wall tiles but not as floor tiles[27] which require, in general, a Mohs' hardness of 5.

Furthermore, the thermal expansion coefficient of the sintered materials is sufficiently low to satisfy the request for commercial tiles (i.e. $<9\times10^{-6}$ K^{-1}, Ref. 27).

The water absorption of the sample with BA glass plus KW sintered at 950 °C corresponds to tiles of BIb type in UNI EN 87 ceramic tile classification and it is in agreement with the bending strength versus water absorption behavior of ceramic pressed tiles.[28]

5. Conclusion

The present work was conducted to verify the feasibility of the MSWI bottom ash vitrification and the reuse of this glass as raw material for glass–ceramic tile production. We have produced glass–ceramic tiles starting only from waste raw materials of different sources: MSWIs bottom ash, metallurgical and mineral industrial wastes.

These metallurgical and mineral industrial wastes could not be vitrified, due to their composition; their use for the production of glass–ceramic tiles, mixed with vitrified bottom ashes, can be a way to reuse them.

The glass–ceramic bodies were produced by a low-cost viscous sintering process at moderate temperatures and without any pressure. The properties of the obtained samples are compatible with those requested for ceramic tiles.

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