Production and Characterisation of Glass Foams for Thermal Insulation

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In this work, glass compositions from glass bottles with different colours and graphite (2 - 12 vol%) as the pore-forming agent were prepared for the production of materials with controlled porosity for applications where thermal and acoustic insulations are main technical requirements. The formulated compositions were, in a later stage, uniaxially pressed (40 MPa) and fired at different temperatures (800 - 950 °C) and cycles with holding times between 15 and 120 minutes. The results showed that it is possible to produce glass foams for thermal insulation from optimized compositions containing 90 vol% glass and 10 vol% graphite, fired between 800 and 950 °C for 30 minutes, which showed porosities between 53 and 76 %, with thermal conductivities between 0.16 and 0.08 W/mK and compressive strength between 20 and 4.6 MPa. The obtained glass foams are strong candidates for applications requiring thermal insulation with suitable combination of thermal conductivity, porosity and mechanical strength.

1. Introduction

Ceramic materials are typically employed as insulators when the working temperatures are greater than the room temperature. The application of these materials depends on the thermal conductivity and refractoriness which may vary depending on the chemical composition and pore structure. The best thermal insulation is vacuum. However, due to the difficulties to get it and keep it, its application is limited. A practical solution is the utilization of air, which also exhibits low thermal conductivity (0.026 W/mK) (Holman, 1983). Thus, a technical solution is the use of porous or cellular materials with closed pores such that air or other gases from the decomposition of foaming agents or pore formers remain enclosed within the formed pores. Porous ceramics, including the vitreous ones, are relatively fragile materials which exhibit high porosity with closed, open or interconnected pores (Zeschky et al., 2003).

The growing interest in porous ceramics have been mainly associated to their specific properties such as high surface area, high permeability, low density and thermal conductivity which are still related to characteristics of ceramic materials such as high refractoriness and resistance to chemical attacks (Ortega et al., 2003). Among the materials used for the manufacture of porous materials, could be cited alumina, mullite, silicon carbide, zirconia, hydroxyapatite and some composite systems (Salvini et al., 2000).

Furthermore, there is the possibility of obtaining porous materials from vitreous materials. In these cases, the use of glass from recycling is explored and can be enhanced in applications involving temperatures lower than 500 °C as in the case of isolated grills and fireplaces and in particular in building systems.

Waste glasses from the glass industry and glass containers used in daily life are discarded with household garbage (Alves et al., 2001).

The recycling glass not only reduces power consumption but also the extraction, processing and transportation of minerals used as raw materials. According to the Brazilian Technical Association of Automatic Glass Industries (ABIVIDROS, 2014) it is possible an energy gain of 4 % and a 5 % reduction in CO_2 emissions when 10 % of glass scraps are used in the manufacture of glass. In 1991 only 15 % of glass

containers were recycled but in 2004 it was 45 % (Rosa et al., 2007). Moreover, to recycle and to use glasses can contribute to the national energy matrix through the large amount of energy savings, since to produce 1 kg of new glass 4,500 kJ are needed, whereas to produce 1 kg of recycled glass there is need of 500 kJ (Alves et al., 2001).

Thus, the use of waste is a great opportunity to elongate the life cycles of elements in the anthroposphere, reducing the need for extraction of the environment (Galembeck, 2013).

In this context, this article presents results of a research work aimed to the production of glass foams from recycled glass and graphite electrodes from melting furnaces for steel, for applications where thermal and acoustic insulations are the main technical requirements.

2. Materials and methods

In this work, glass bottles (VG) of different colours (clear, green and brown) and graphite as pore forming agent (a residue from electrodes of electric melting furnaces for steel) were used as raw materials. Chemical compositions of transparent glass bottles (VGT), green glass bottles (VGV) and brown glass bottles (VGM) were obtained by X-ray Fluorescence (Philips, model PW 2400) as shown in Table 1.

Table 1: Chemical composition of the glasses used as raw materials

Materials/Oxides (wt%)	SiO_2	Al_2O_3	Fe_2O_3	CaO	K_2O	MgO	Na_2O	P_2O_5	TiO_2
VGT	71.2	2.17	0.10	9.64	0.02	-	16.82	0.02	0.04
VGV	68.3	2.07	0.41	8.94	0.44	1.80	17.95	0.01	0.06
VGM	67.1	1.97	0.83	9.55	0.30	0.65	19.58	0.01	0.03

The glass bottles (VG) had sodium-calcium composition, such that the major differences in composition may be related to the chromophore oxides, particularly iron oxide (Fe_2O_3), which, as expected, increases when the coloration of the glasses becomes transparent/clear to green and brown.

The glass bottle (each colour) was milled on a crusher (SERVITECH, CT-058), and the resulting powder was dry milled for 90 minutes in a fast mill (SERVITECH, CT-242) by using a porcelain jar containing alumina balls to yield particle sizes (D_{50}) of 3.5 µm (using a laser scattering particle size analyser, Malvern, ZEN-3600).

The graphite was milled into a crusher (SERVITECH, CT-058) resulting in powders with particle sizes smaller than 106 µm. Subsequently, compositions containing glass bottles (mixture of powders in equal proportions of the three glass bottles) and graphite varying from 2 to 12 vol% were prepared. The formulated and prepared compositions were humidified with 5 % water and homogenized in a fast mill (SERVITECH, CT-242) with a porcelain jar containing alumina balls for 10 min.

The prepared compositions were, in a later step, uniaxially pressed (40 MPa) in a steel die using a hydraulic press (Bovenau P10 ST). The obtained disc samples (30 x 10 mm) were dried in a laboratory dryer (SP LABOR®) at 110 °C for 2 h.

The thermal behaviour during firing of raw materials and prepared compositions were studied by means of an optical dilatometer (Expert System Solution Misura ODHT) and by a thermal analysis equipment (TA Instruments, SDT Q600 - Simultaneous TGA-DSC) at a heating rate of 10 °C/min with a flow of synthetic air of 10 cm^3/min.

Based on the thermal analyses green compacts were fired at different temperatures (800, 850, 900 and 950 °C) for different holding times (15, 30, 60 and 120 minutes) and subjected to several measurements and analyses.

The geometric density (ρ_{geo}) was calculated from the dimensions and mass of the samples. The true density of the glass foams (ρ_t) was determined by gas (He) pycnometry (Multi-Pycnometer, MVP-4DC). The void fraction (porosity (ε)) was determined by considering the relation between the densities, that is, [1- (ρ_{geo}/ρ_t)]. The microstructure of pores could be seen from images of fracture surfaces of fired samples, obtained in a scanning electron microscope, SEM (JEOL JSM-6390LV).

To determine the mechanical strength of the samples, compression tests (EMIC DL 2000 model) were performed on 5 disc samples (30 x 10 mm) with loading speed of 1 mm/min.

The thermal conductivity of the obtained materials was determined by a TCi Thermal Conductivity C-THERM TECHNOLOGIES on disk-shaped samples of 30 mm diameter and 10 mm thick.

3. Results and discussion

Figure 1 shows a curve of linear shrinkage of the glass bottle (VG) and thermograms (DSC/TG) of graphite (G). Figure 2 shows curves of linear shrinkage of the glass bottle (VG) and compositions containing between 2 and 12 vol% graphite.
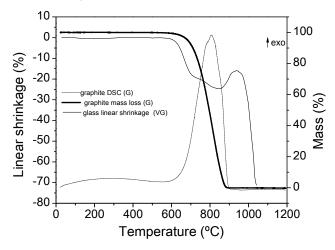

Figure 1: Curves of linear shrinkage and thermograms (DSC/TG) referring to the glass bottle (VG) and graphite (G)

It is observed in the linear shrinkage curve (Figure 1) of the glass bottle that densification (in terms of shrinkage) occurs at a greater rate between 550 and 600 °C and continues to shrink at a slower rate until 900 °C. From 900 °C initiates the glass expansion (glass softening region) before the beginning of its melting (950 °C). In the DSC curve of the graphite (Figure 1) it can be seen only an exothermic event between 650 and 900 °C regarding the graphite oxidation to CO and CO_2. Such decomposition event can be confirmed by the mass loss (TG) curve (loss of 70 % of the original mass).
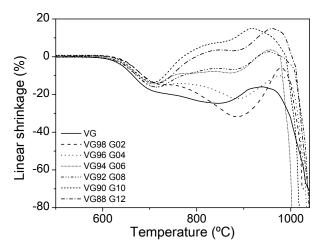

Figure 2: Curves of linear shrinkage referring to the glass bottle (VG) and formulated compositions

It is noteworthy that the oxidation of graphite occurs in the temperature range in which the glass is already in an advanced state of densification. This behaviour indicates that the gas generated by the oxidation of graphite remained trapped in the glass matrix may generate pores. In general, it can be seen in Figure 2 that the linear shrinkage (densification) of the compositions begins between 550 and 600 °C. Between 725 and 875 °C it can be seen the expansion of the compositions (glass + graphite) generated by the oxidation of the graphite. From 900 °C occurs the characteristic expansion of the glass bottle. It is observed that the expansion is increasing as the amount of graphite added increased from 2 to 10 %. For larger additions of graphite (12 %) the expansion decreases. This behaviour is probably related to an increase in volume of the graphite gases which results in increased internal pressure and thus the rupture of the walls of the pores allowing the

escape of the generated gases. Thus, the composition with 90 % glass bottle and 10 % graphite was chosen because it provides the best conditions in terms of the relative amounts of the used raw materials. Figure 3 shows curves relating the porosity as a function of the firing temperature and holding time of compacts containing 90 % glass and 10 % graphite.
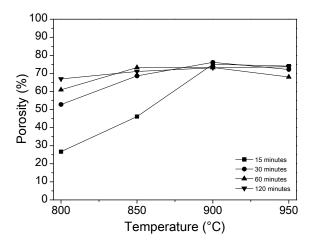

Figure 3: Curves relating the porosity as a function of the firing temperature and holding time of compacts containing 90 % glass and 10 % graphite

As can be seen (Figure 3), the porosity increases (~25-75 %) as the firing temperature increases from 800 °C to 900 °C (temperature of maximum porosity) remaining practically constant between 900 °C and 950 °C, independently of the holding time in a specific temperature. The maximum porosity at 900 °C is in agreement to Figure 2 since the composition containing 90 % glass bottle and 10% graphite shows greater expansion. The images relating to the samples of porous compacts fired at 900 °C for 30 min (up to 90 % glass bottle and 10 % graphite) are shown in Figure 4, in which one can view the porosity progress as a function of temperature. It is also observed a homogeneous distribution of pores. Furthermore, there is an increase in the size and number of pores as the temperature increases to 900 °C, which is in agreement with the porosity data shown in Figure 3. At 950 °C (Figure 4 (d)) there is a decrease in pore size due to the high firing temperature that favours the increase of internal pressure leading to rupture of the pore walls promoting thus the reduction of the pore sizes and the escape of gas.
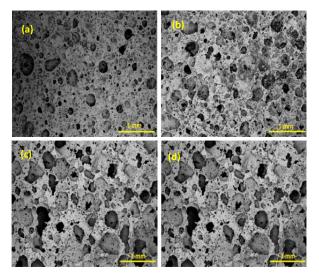

Figure 4: Micrographs (SEM) of samples containing 90 % glass and 10 % graphite fired at different temperatures: (a) 800 °C; (b) 850 °C; (c) 900 °C (d) 950 °C for 30 min

The thermal conductivity and mechanical strength as a function of the firing temperature of porous compacts (composition containing 90 vol% glass bottle and 10 vol% graphite) are shown in Figures 5 and 6.
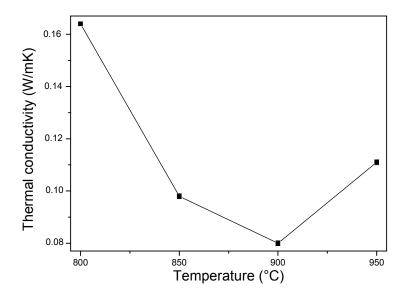

Figure 5: Thermal conductivity as function of the firing temperature for 30 min for samples containing 90 % glass and 10 % graphite

As can be seen from Figure 5, the thermal conductivity decreases (~ 0.16 to 0.08 W/mK) as the porosity increases to 900 °C. However, between 900 °C and 950°C the thermal conductivity increases (up to approximately 0.11 W/mK) due to a decrease in pore size as seen in Figure 4.
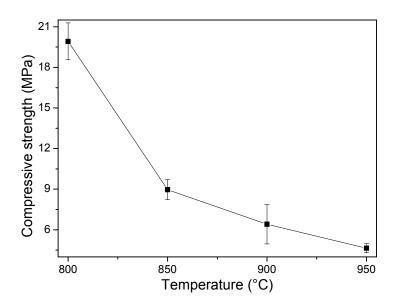

Figure 6: Mechanical strength as function of the firing temperature for 30 min for samples containing 90 % glass and 10 % graphite

The mechanical strength (Figure 6) decreases (from 20 to 4.6 MPa) as the firing temperature increases. This result is in good agreement with the observed porosity results, i.e., as it increases the compressive strength decreases. The mechanical strength values achieved are typical of ceramic foams.

4. Conclusions

The results showed that it is possible to produce thermal insulators from optimized compositions containing 90% glass bottle and 10 % graphite, fired at temperatures between 800 and 950 °C for 30 min with porosities between 53 and 76 %, thermal conductivities between 0.16 and 0.08 W/mK and compressive strengths between 4.6 and 20 MPa. The obtained materials are therefore potential candidates for applications which require appropriate combination of thermal conductivity, porosity and mechanical strength.

Acknowledgements

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