

# PONENCIA 41 bloque C1

## CERAMICS FOAMS MADE FROM PLAIN GLASS CULLETS

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**Abstract:** This work deals with physical, structural and mechanical characterization of ceramic foams obtained from plain glass cullets expanded via bubble formation during sintering. The expansion process was caused by air expansion simultaneous to glass melting at 850°C. The expansion is due the presence of air trapped with the glass particles during milling and pressing processes. As the temperature is raised during firing the glass becomes viscoplastic and impermeable to gas flow. During the fast cooling stage the bubbles are transformed into rigid pores, and the glass foam is formed. The microstructure, expansion, density and mechanical properties (flexural tests) were determined to characterize the glass foam. Cellular ceramics can substitute polymers (expanded polystyrene) and wood in internal partition walls and linings, or cellular concrete in the building industry. In this previous study the thermal properties were not determined. This approach is a non expensive way to obtain low density materials with good mechanical resistance, avoiding the discard of pollutant residues from ceramic processing.

**Keywords:** cellular ceramics; glass residues; recycling

### 1. Introduction

Ceramic foam is a porous material with porosity ranging from 70% to 90%, volume density from 0.3 to 0.6 g/cm<sup>3</sup>. It has 3D frameworks structure, and interconnected or isolated pores [1]. Because of its many advantages, such as low density, high porosity, low heat transfer rate, high temperature resistance, corrosion resistance and excellent acoustic property, the cellular materials are applied in a variety of industries such as filtration, heat insulation, sound insulation, catalyze, and extends to electron, optics and biochemistry over the past decade [1,2-5].

Closed-cell ceramic foam manufacturing techniques can be classified into three general categories: sponge-replication, adding foaming agent and organic filling. The sponge-replication was first developed in the early 1960's. It uses a natural sponge or polyurethane foam as a form, which is infiltrated with ceramic slurry. The ceramic slurry is then fired to form ceramic foam. Based on gas bubbles in preceramic melts, gas evolving constituents are added to the melt. The generated bubbles cause the foam. Foaming uniformity and cell geometry can be adjusted by careful selection of surfactants and foaming agents [1,6-10].

The organic filling technique is based on a space holder concept, i.e., polyurethane foam infiltrated with ceramic slurry is dried and indurated at room temperature. The ceramic foams developed in this experiment were made by the foaming agent technique [11,12]: the unusual aspect is that the foaming agent is only the air trapped among the particles of a milled glass. It is a well known feature in the ceramic tile industry the problem when a milled glaze is rapidly heated along its melting temperature: if the glaze is melted and rapidly cooled without an adequate soaking time at the melting temperature several bubbles are formed and maintained in the glaze layer until the glaze is hardened in the cooling stage. The bubbles not eliminated during the heat treatment are transformed in permanent pores, reducing the glaze quality. In this work residues of plain glass (soda-lime glass) are milled, granulated, pressed and fired in a industrial roller kiln in a rapid firing cycle that prevents the elimination of the air trapped at the surface particles and particle interspaces. As a result, the air expands forming bubbles during the firing cycle due the impermeability of the glass as it is melted, imprisoning the air. At the cooling cycle, the imprisoned air bubbles in the glass are transformed in large pores, forming the glass foam.

### 2. Materials and Methods

The plain glass residue was submitted to physical-chemical characterization to determine its chemical, phase and thermal analyses. The chemical analysis was carried out by X-ray fluorescence (wavelength dispersive

spectrometry) and the thermal analysis was determined by differential thermal analysis (DTA, 10°C/min, in air atmosphere). A 100kg batch of plain glass cullet was milled in a 200l industrial mill with 100l water. The milling time was 12h and a mixture of 0.25% w/w of acrylic emulsion and 0.25% w/w of polyvinyl alcohol was added. The particle size analysis was carried out by laser diffraction (10s reading time). After characterization the glass residue was dried (110°C, 24h), granulated with 7% water and pressed (350kgf/cm<sup>2</sup>) in 120mm×50mm specimens. The compacts were dried (100°C, 24h) and sintered in an industrial roller kiln at 800°, 850°C and 900°C maximum temperature in a 50min firing cycle (figure 1). After firing the expanded samples at each heat treatment temperature were analyzed regarding linear expansion, water absorption, apparent density and mechanical resistance. The density was determined by mercury immersion and the mechanical resistance was determined by three point loading test (10mm/min, ISO 10545). The microstructure was determined by scanning electron microscopy (5kV-10kV).



Figure 1. Samples fired at 850°C in a roller kiln

### 3. Results and Discussion

Table 1 shows the chemical analysis of the glass cullet residue used in this study. As observed, the glass residue is a typical soda-lime glass. The thermal analysis of the glass cullet (figure 2) shows an endothermic peak at 756°C related to the residue glass transition temperature (T<sub>g</sub>). At 1163°C there is another endothermic peak related to the beginning of the residue melting (T<sub>m</sub>).

Table 1. Chemical analysis of the porcelain tile residue

simple (%) w/w	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	K <sub>2</sub> O	Na <sub>2</sub> O	LOI
glass cullet	66.0	3.0	0.3	11.2	3.7	3.3	12.2	0.3

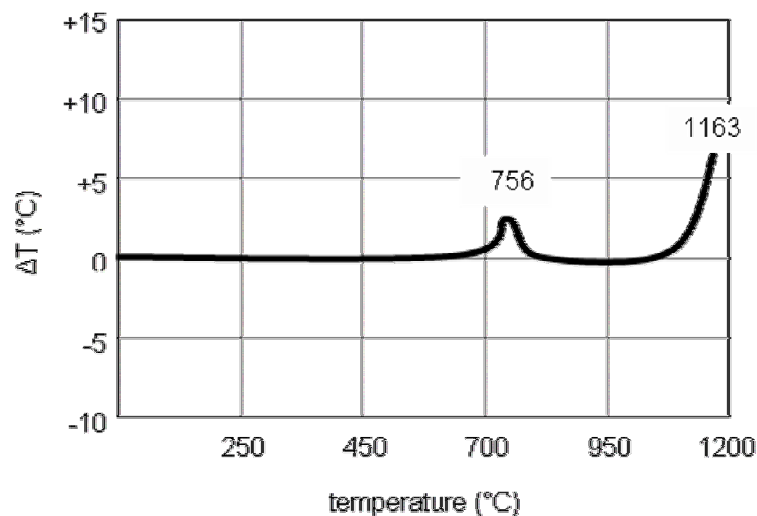


Figure 2. Differential thermal analysis of the glass cullet residue

Figure 3 shows the particle size distribution of the glass residue after milling. The residue is 100% under 77 $\mu$ m with a mean particle size of 30 $\mu$ m.

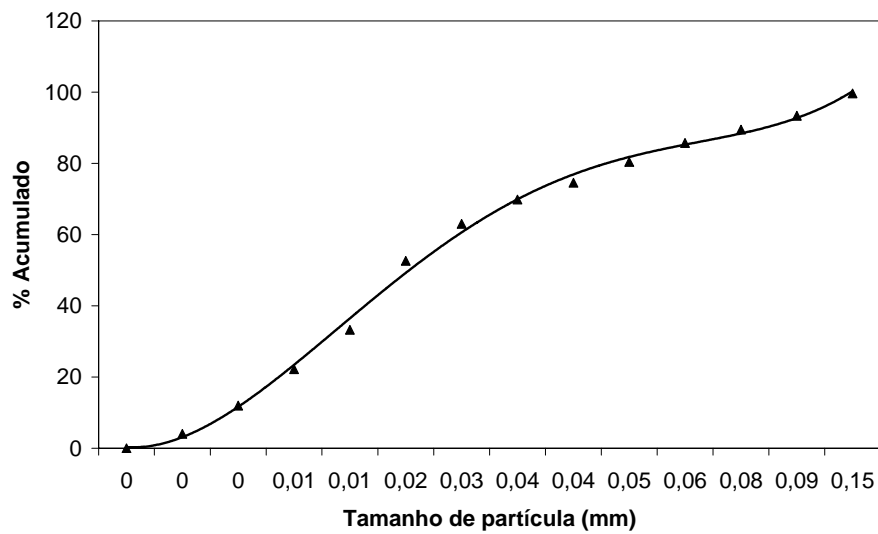


Figure 3. Particle size distribution for the glass residue after milling

Regarding the apparent density of the sintered material an increase in the firing temperature causes a decrease in the apparent density of the cellular ceramic, figure 4. Starting at 800 $^{\circ}$ C, the expansion promoted by the air trapped in the glass particles forms large and rounded pores in the final product. The very low average density observed (0,24g/cm $^3$ ~0,34g/cm $^3$ ) allows the use of the ceramic foam as an acoustic or a thermal insulator.

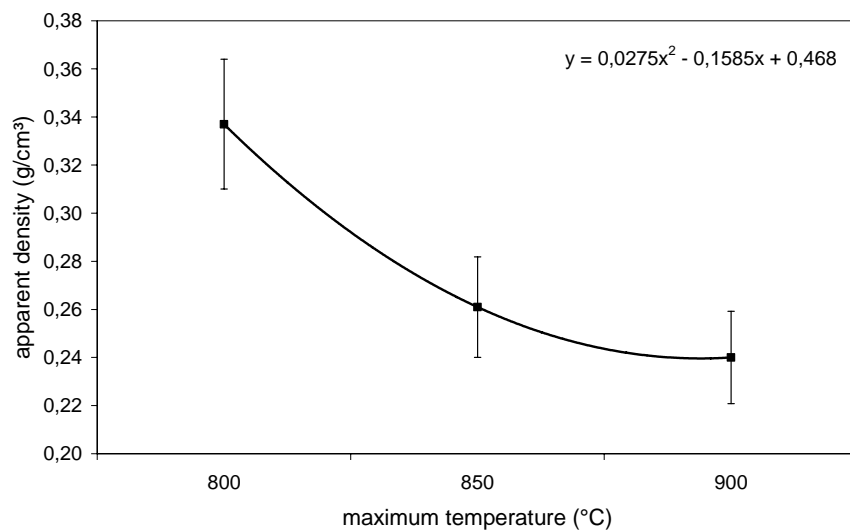


Figure 4. Evolution of the apparent density of the ceramic foam due the firing temperature

An increase in firing temperature causes a great variation in the mechanical resistance of the obtained ceramic foams, figure 5. Initially, there is a raise in the flexural resistance of the samples starting at 800 $^{\circ}$ C firing temperature due the quantity and size of the formed pores. At 850 $^{\circ}$ C the maximum resistance is obtained and an additional heat treatment at 900 $^{\circ}$ C results in a decrease of the mechanical resistance.

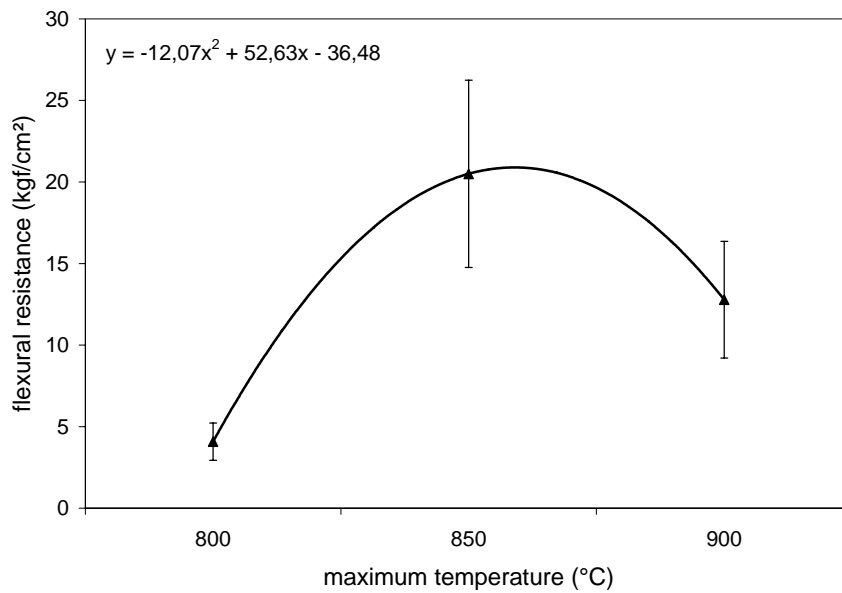


Figure 5. Evolution of the flexural resistance of the ceramic foam due the firing temperature

The foam presents an acceptable mechanical resistance, adequate to several uses, mainly the building industry, as a substitute for cellular concrete or expanded polystyrene. After sintering, all samples have presented a great and gradual expansion with the firing temperature. It is obvious the relation between firing temperature and linear expansion, figure 6. It must be observed the very high increment in linear expansion with temperature: at 800°C the foam expands 52%, at 850°C expands 64%, and at 900°C expands 68%, showing a stability region.

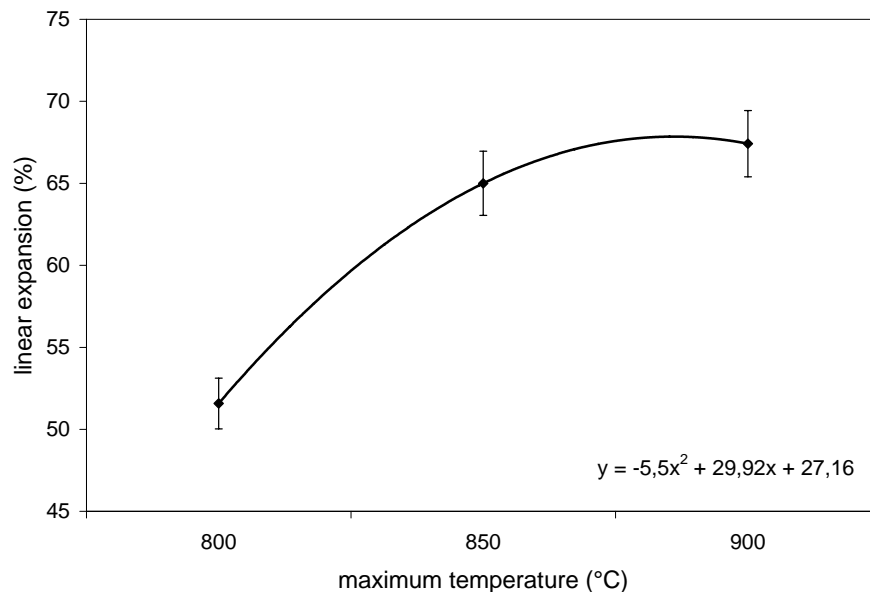


Figure 6. Evolution of the linear expansion of the ceramic foam due the firing temperature

The expansion is due the presence of air trapped with the glass particles during milling and pressing processes. As the temperature is raised during firing the glass becomes viscoplastic and impermeable to gas flow. If the glass becomes impermeable (by liquid viscous phase formation in temperatures above the glass transition) more rapidly than the air trapped with the particles is expelled from the compacts the air forms bubbles. During the fast cooling stage the bubbles are transformed into rigid pores, and the glass foam is formed.

Figure 7 shows the water absorption of the ceramic foams in function of the firing temperature. At 800°C the water absorption is very high (~31%) because the foam is not totally sintered. At 850°C the foam is completely

formed and the absorption is low (~3%). Higher firing temperatures increase the water absorption due the bigger pores formed.

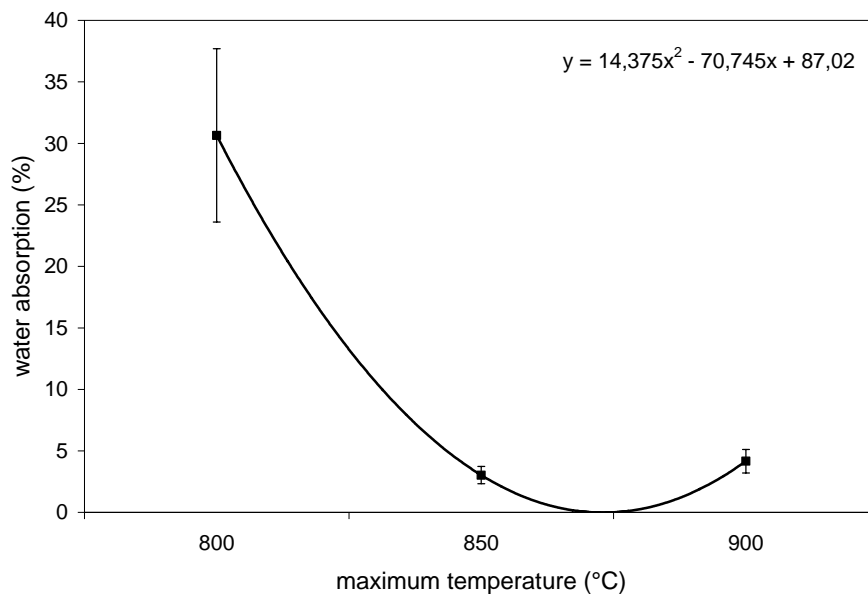


Figure 7. Evolution of the water absorption of the ceramic foam due the firing temperature

Finally, in figure 8 it is observed the microstructures of the samples treated at 850°C and 900°C firing temperatures. The pores are big, closed and rounded, what can explain the good mechanical resistance besides the very high porosity observed in these products. It seems the product can present good thermal and acoustic insulation, but these properties were not analyzed in this study.

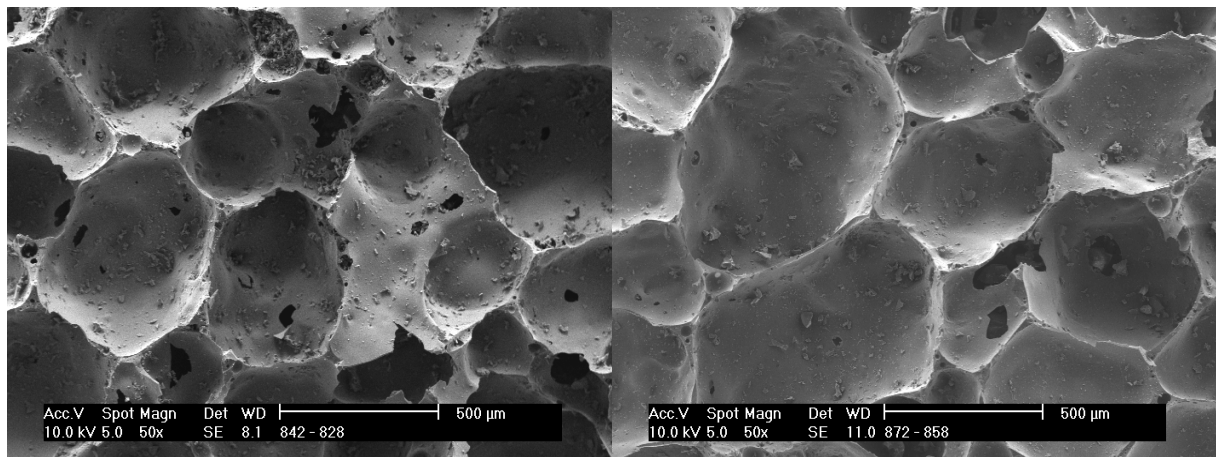


Figure 8. Microstructure after sintering for 850°C and 900°C firing temperatures

#### 4. Conclusion

Residues from package and plain glasses can be used to form ceramics foams with very low densities. The cellular ceramics can be used in the building industry as substitutes for cellular concrete due their low density, resulting in light weight structures with acceptable mechanical resistance. It seems the product has good acoustic and thermal insulation and could be used as substitutes for wood and polymers in internal walls and linings, but these properties have not been determined at this moment.

The reduction of the apparent density is related to the presence air trapped with the glass particles during the glass processing. The product expansion (and consequent density reduction) occurs by the presence of closed pores in the microstructure of the samples. The mechanical resistance of the product is reduced with high firing temperatures because the increased porosity. Besides the amount of pores present in the samples it was observed

good flexural resistance (20kgf/cm<sup>2</sup>) showing the product could be used as a building material. The mechanical resistance is due the rounded form of the pores present in the samples; these pores redistribute the tensions applied on the product, avoiding its concentration.

Finally, the expansion process occurs due air expansion simultaneously to a viscous glass formation during sintering. The firing above glass transition results in a product that permanently holds air, producing bubbles that during cooling form the observed pores in the microstructure of the material. The quantity, dispersion and size of the pores present in ceramic foams result in the final properties of this product.

## 5. References

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