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CELLULAR CERAMICS MADE FROM PORCELAIN TILE POLISHING RESIDUES: EFFECT OF SOAKING TIME

Aline da Silva Pereira¹; Diana de Souza Felisberto¹; Maisa Topanotti Daros¹; Glauco Luckmann¹; Adriano Michael Bernardin^{1,2}

¹Engenharia de Materiais, Universidade do Extremo Sul Catarinense, Criciúma, Santa Catarina, Brazil

²Tecnologia em Cerâmica, Serviço Nacional de Aprendizagem Industrial, Tijucas, Santa Catarina, Brazil
adriano@unesoc.net

Abstract: This work deals with physical, structural and mechanical characterization of cellular ceramics obtained from porcelain tile polishing residues expanded via bubble formation during sintering. The expansion process is caused by SiC particle oxidation simultaneous with the polishing residue melting, both at ~1200°C. Starting at 1000°C SiC particles coming from the abrasive used to polish the porcelain tiles oxidizes forming SiO₂ and CO₂, the last one used as an expanding agent, promoting the expansion of the melt (porcelain tile residue) formed at 1200°C. The microstructure, expansion, density and mechanical properties (compression 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/cm³. 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., a 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: the silicon carbide present in the polishing residues oxidizes above 1000°C in presence of oxygen. Its oxidation results in silica and carbon dioxide according: $\text{SiC} + 2\text{O}_2 \rightarrow \text{SiO}_2 + \text{CO}_2$. Therefore, the mixture of a vitreous material that melts at the same temperature of SiC oxidation can result in a cellular ceramic material because of gas formation, in this case, carbon dioxide [11,12].

2. Materials and Methods

The porcelain tile polishing residue was submitted to physical-chemical characterization to determine their chemical, phase and particle size distribution analyses. In addition, a thermal analysis was carried out. The chemical analysis was carried out by X-ray fluorescence (Philips PW2400, molten sample) and the phase analysis by X-ray diffraction (Philips PW1830, CuK α , 0° to 75°, 0.1°C). The particle size analysis was carried out by LASER diffraction (CILAS 1064, 10s reading time). Finally, the thermal analysis was determined by

differential thermal analysis (Netzsch 409, 20°C to 1200°C, 10°C/min, air atmosphere). After characterization the polishing residue was dried (110°C, 24h), granulated with 6% water and pressed (300kgf/cm²) in cylindrical specimens (5cm diameter, 1cm height). The compacts were sintered during 5min, 10min, 15min and 20min soaking times at 1200°C with 30°C/min heating rate and cooled in the furnace (laboratory muffle oven). After heat treatment the expanded samples at each soaking time were analyzed regarding linear expansion, volume density and mechanical resistance. The density was determined by mercury immersion and the mechanical resistance was determined by compressive test (EMIC DL 10000, 10mm/min, ASTM C496). The microstructure was determined by scanning electron microscopy (Philips XL 30, 5kV-10kV).

3. Results and Discussion

Table 1 shows the chemical analysis of the porcelain tile polishing residue used in this study; figure 1 shows the phase analysis. As observed, the polishing residue is formed by quartz, albite and zircon, the major phases of a porcelain tile paste. The amount of alkaline and earth alkaline oxides present in the porcelain residue (11.7 w/w%) shows a good vitrification tendency of this system. It was not possible to identify the silicon carbide (SiC) because the procedure used to analyze the residue samples: the samples were calcined at 1000°C during 3h, causing total conversion of the SiC present in the samples.

Table 1. Chemical analysis of the porcelain tile residue

sample (w/w%)	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	CaO	MgO	K ₂ O	Zr	Na ₂ O	LOI
porcelain residue	63.1	16.7	0.6	0.3	1.3	6.1	2.3	1.7	2.0	5.7

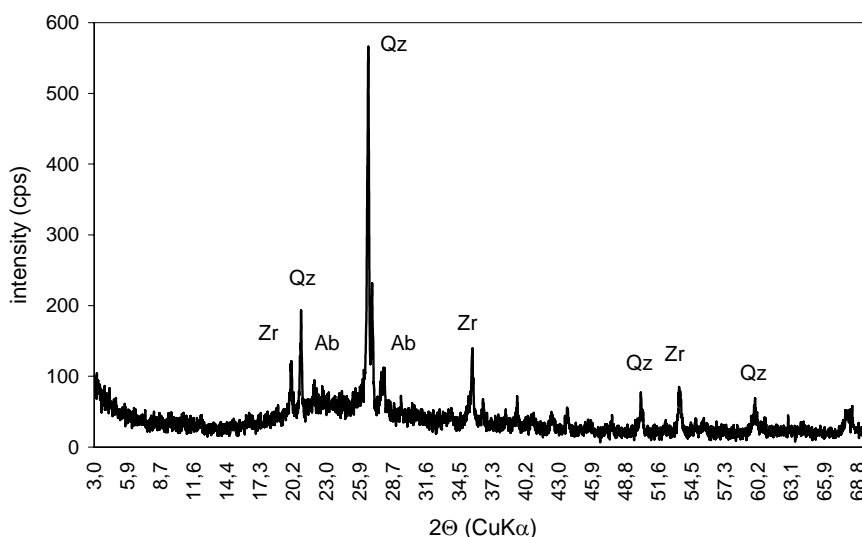


Figure 1. XRD pattern of the porcelain residue (Qz=quartz; Zr=zircon; Ab=albite)

The thermal analysis of the porcelain tile residue (figure 2) shows an endothermic peak at approximately 400°C, probably due thermal decompositions of the organic substances present in the residue obtained from the effluent treatment station. At approximately 1100°C there is another endothermic peak related to the beginning of the residue glass transition (T_g).

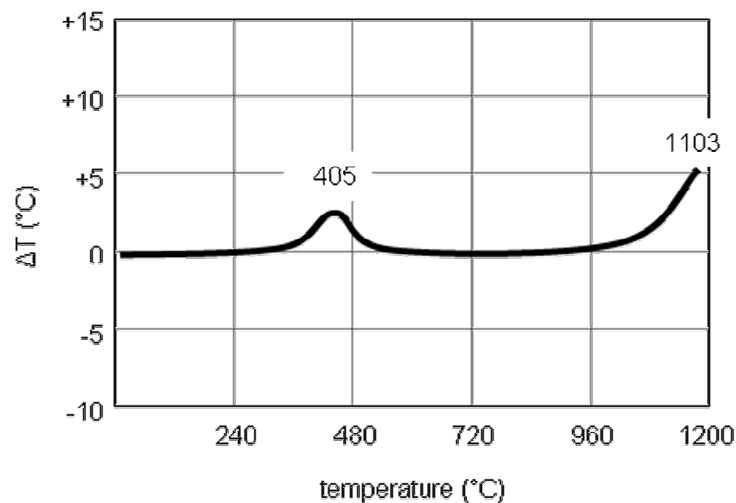


Figure 2. Differential thermal analysis of the porcelain tile residue

Figure 3 shows the particle size distribution of the polishing residue. The polishing residue is 100% under 75μm with a mean particle size of 10μm.

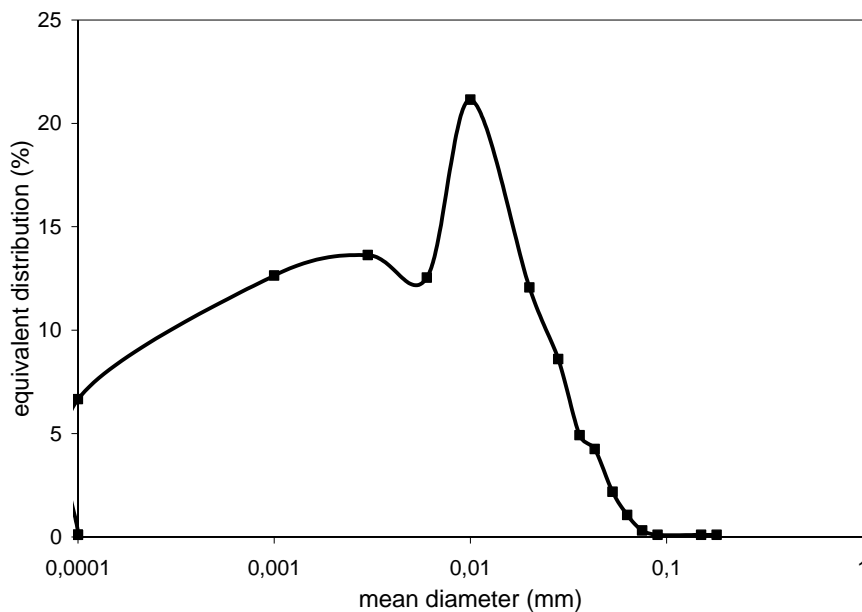


Figure 3. Particle size distribution for the polishing residue

Regarding the apparent density of the sintered material an increase in the soaking time causes a decrease in the density of the cellular ceramic, figure 4. Starting at 5min soaking time, the expansion promoted by oxidation of the silicon carbide present in the polishing residue forms large and rounded pores in the final product. The very low mean density observed (0,55g/cm³-0,75g/cm³) allows the use of the cellular material as an acoustic or a thermal insulator.

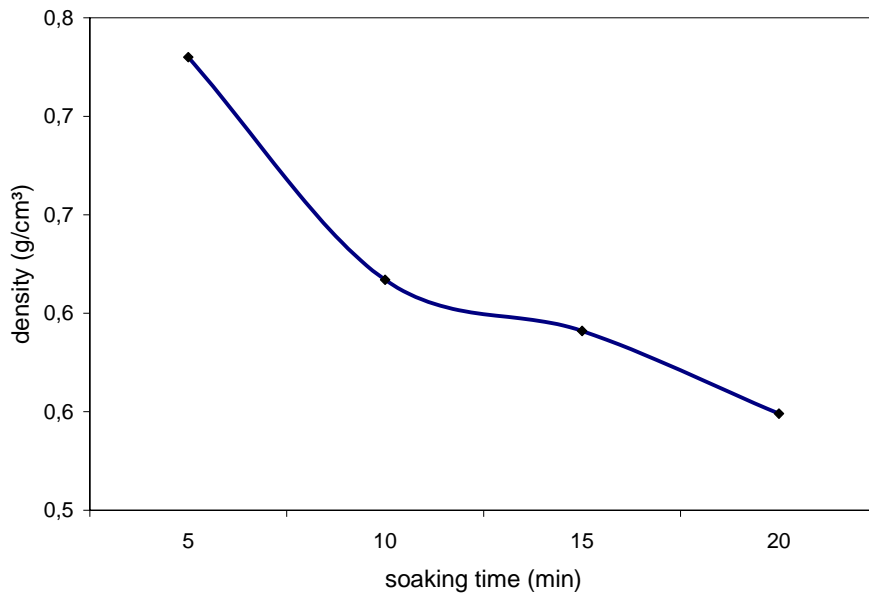


Figure 4. Evolution of the apparent density of the cellular ceramic due the soaking time

An increase in the soaking time causes, as expected, a reduction in the mechanical resistance of the cellular ceramics obtained, figure 5. There is a great reduction in the compressive resistance of the samples starting at 5min soaking time due the quantity and size of the formed pores.

Besides the mechanical resistance reduction with the soaking time, the product 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 soaking time. It is obvious the relation between soaking time and expansion, figure 6.

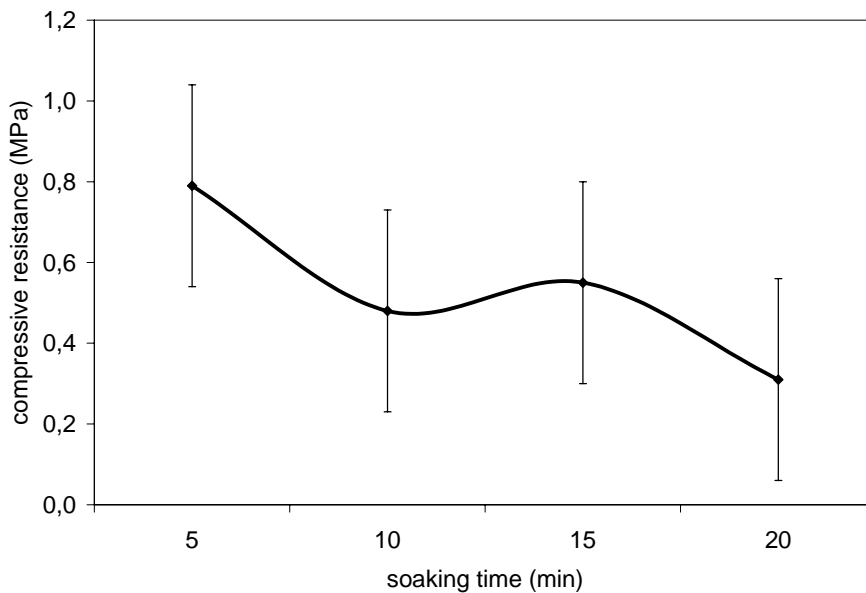


Figure 5. Evolution of the compressive resistance of the cellular ceramic due the soaking time

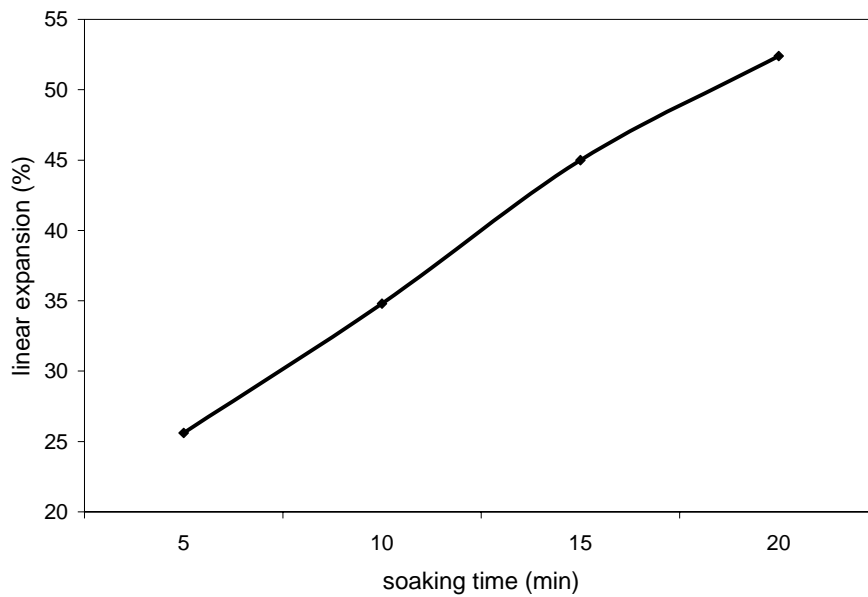


Figure 6. Evolution of the linear expansion of the cellular ceramic due the soaking time

The expansion is due the presence of SiC particles incorporated to the residue during the polishing process and this feature does not allow the use of this kind of residue in ceramic formulations for very dense tiles, as the porcelain tiles. Many attempts to add polishing residues from effluent treatment stations in the paste of dense tiles failed due the great porosity of the products after firing.

Finally, in figure 7 it is observed the microstructures of the samples treated at 1200°C during 5min and 20min soaking times. 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.

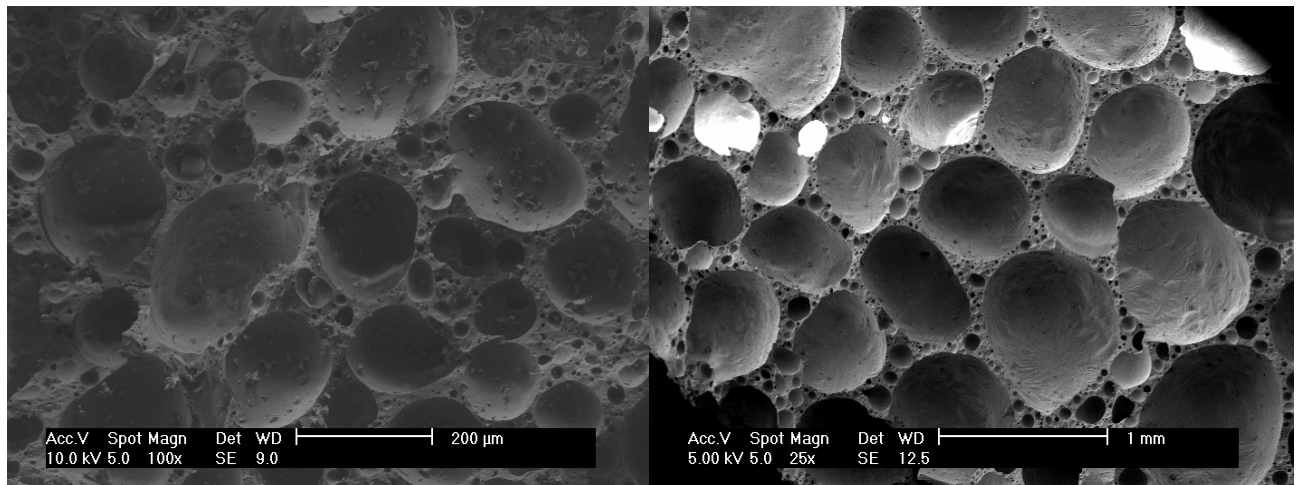


Figure 7. Microstructure after sintering for 5min and 20min soaking times

4. Conclusion

It is possible to use residues in ceramic processing. Residues from the polishing process and rests of silicon carbide abrasives can be used together to form cellular ceramics with low density. 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 of SiC particles. 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 the soaking time because the increased porosity. Besides the amount of pores present in the samples it was observed good compressive resistance (0.7MPa) 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 the silicon carbide oxidation simultaneously to glass formation during sintering. The glass melting at the same time of SiC oxidation results in a product that permanently holds gas, producing bubbles that during cooling form the observed pores in the microstructure of the material. The quantity, dispersion and size of the abrasive residue particles present in the porcelain residue result in the form, size and distribution of the pores in the final product.

5. References

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