

Characterization of cellular ceramics made by porcelain tile residues

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Abstract

This work deals with physical, structural and mechanical characterization of cellular ceramics obtained from porcelain tile polishing residues expanded with silicon carbide abrasive residues [A.M. Bernardin, et al., Proceedings of the VIII World Congress on Ceramic Tile Quality, vol. 3, 2004, pp. 195–199]. In a previous work it was studied the expansion process, that means, SiC decomposition simultaneous with the polishing residue melting, both at $\sim 1200^\circ\text{C}$. Starting at 1000°C SiC particles decomposes into SiO_2 and CO_2 , the last one used as an expanding agent, promoting the expansion of the melt (porcelain tile residue) formed at 1200°C . Now, the microstructure, expansion, density and mechanical properties (flexural tests) were determined to characterize the product. Cellular ceramics can substitute polymers (expanded polystyrene) and wood in internal partition walls and linings, and cellular concrete in the building industry.

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1. Introduction

Recycling was established with the Industrial Revolution, when the available metals were continuously processed. Nowadays recycling is an international industry. All kind of materials are collected, separated, processed and commercialized according rigid specifications, resulting in standard materials that can be traded around the world. The use of standard materials is, in theory, circular: primary substances extracted or collected are transformed in products that eventually are redundant and can be recycled in the manufacturing process [2,3].

Without recycling the process is not circular, becoming a sequence of events without a logical resolution. Potentially useful materials become a menace, not a resource [4]. The use of secondary, that is, recycled materials results in a great energy saving related to the primary production. Among the several raw materials that can be recycled are the industrial solid residues from the ceramic industry [5–8].

Currently there is no recycling of the solid residues from the tile ceramic industry in Santa Catarina State, South of Brazil. The region is the second tile ceramic producer in Brazil, with approx-

imately 30% of the overall tile production and the first exporter. The ceramic residues are discarded directly on landfills, without any kind of separation [9–11]. The polishing ceramic residues, locally named “mud”, are the remaining portions of the “grés porcellanato” polishing process containing abrasive particles detached from the abrasives used to polish the tiles, normally chlorine–magnesium cement impregnated with silicon carbide or diamond particles.

The residue is collected, stored and filtered in effluent treatment stations that remove the residual water, producing mud as a subproduct. The mud is stored on landfills, causing environmental impact [12,13]. An alternative to the discard of ceramic solid residues is their use to make new products [14,15]. Specifically in this study the porcellanato polishing residues were used to form low density ceramics. As presented by Bernardin et al. [1], low density ceramics can be used as building materials according their thermal and acoustic characteristics.

As previously stated, the polishing residues are the rest of porcelain tiles, mixed with water and organic substances. Porcelain tiles are characterized by a dense microstructure with low and close porosity. This product present a minor crystalline phase formed by mullite and quartz crystals immersed in a vitreous phase. The vitreous phase is formed by a siliceous glass containing alkaline and earth alkaline oxides, mainly potash, soda and magnesia, with a low melting

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Table 1
Chemical analysis (wt.) of the sample residues

Samples	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	CaO	MgO	K ₂ O	Na ₂ O	LOI
Porcelain residue	59.5	17.3	0.7	0.3	1.6	5.3	2.8	3.6	5.6
Abrasive residue	10.5	1.6	1.6	0.1	4.1	34.8	0.1	6.3	40.2

point as the porcelain tiles, or “grés porcellanato”, are fired at 1200 °C during 40–50 min. Combining a glassy material with a relative low melting point allows its recycling in new products.

In its turn, the silicon carbide that is present in the polishing abrasives can be decomposed above 1000 °C in presence of oxygen. Its decomposition 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 decomposition can result in a cellular ceramic material because of gas formation, in this case, carbon dioxide.

The amount of expansion is related to the number and particle size of SiC particles present in the vitreous material. After cooling, the structure formed is a vitreous material containing large and rounded pores, as obtained and discussed in a previous study [1].

In this study, the mechanical and physical properties of the cellular ceramic are related to the amount of abrasive added to the residue from an effluent treatment station. The results show good mechanical resistance with very low density and a microstructure formed by rounded and large pores.

2. Materials and methods

Samples from porcelain tile polishing residue and from SiC abrasives were 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 (Phillips PW2400, molten sample) and the phase analysis by X-ray diffraction (Phillips PW1830, Cu K α , 0–75°, analysis with X’Pert HighScore software). The particle size analysis was carried out by LASER diffraction (CILAS 1064, 60s ultrasound). Finally, the thermal analysis was determined by differential thermal analysis (BP Engenharia RB 3000, 20–1200 °C, 10 °C/min, air atmosphere).

After characterization, polishing residue samples were dried (110 °C, 24 h) and sifted (200 mesh) forming a residue powder. The abrasives used were the part not used of SiC abrasives; they were disaggregated in a hammer mill, dried (110 °C, 24 h) and sifted (200 mesh) forming an abrasive powder. In sequence, the abrasive powder was added to the residue powder in mass fractions of 0.5%, 1.0%, 1.5%, 3.0%, 6.0% and 12.0% forming six formulations. The formulations were mixed with 6% of water (mass fraction) and pressed (300 kgf/cm²) in cylindrical specimens (5 cm diameter, 1 cm height), five specimens for each formulation.

The compacts were sintered during 20 min at 1180 °C with 30 °C/min heating rate and cooled in the furnace (laboratory muffle oven). After heat treatment the expanded specimens of all

formulations were submitted to linear expansion, volume density and mechanical resistance determination. The density was determined by immersion in mercury and the mechanical resistance was determined by the flexural test (Ceramic Instruments MOR3E, 10 mm/min).

3. Results and discussion

Table 1 shows the chemical analysis of the samples (porcelain tile polishing residue and abrasive residue) used in this study; Fig. 1 shows the phase analysis. As observed the major part of the abrasive residue is composed of chlorine–magnesium cement used to form the abrasive. It was not possible to identify the silicon carbide (SiC) because of the procedure used to analyze the abrasive samples: the samples were calcined at 1000 °C during 3 h, causing total conversion of the SiC present in the samples.

Regarding the porcelain residue, it 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 (13.3 wt%) shows the good vitrification of this system.

The thermal analysis essay of the abrasive sample (Fig. 2) shows endothermic peaks between 260 and 500 °C, probably regarding the decompositions of hydroxides (mainly portlandite and clinocllore) present in the sample. The carbonates (calcite, magnesite and dolomite) were not identified, probably due its low content. Between 1020 and 1030 °C it can be seen endothermic and exothermic peaks, probably related to the dissociation of the silicon carbide into silica and carbon dioxide.

The thermal analysis of the porcelain tile residue (Fig. 3) shows endothermic peaks at 60 and 400 °C approximately, probably due thermal decompositions of the organic substances present in the residue obtained from the effluent treatment. At

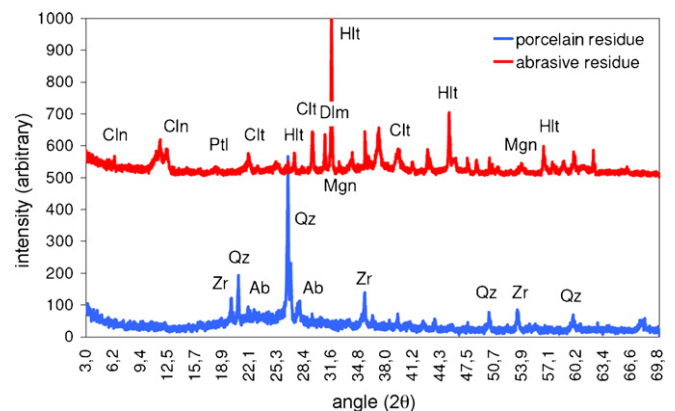


Fig. 1. Phase analysis of the porcelain and abrasive residues. Qz is quartz; Zr is zircon (SiO₂-ZrO₂); Ab is albite; Hlt is halite (NaCl); Dlm is dolomite; Clc is calcite; Mgn is magnesite; Cln is clinocllore; Plt is portlandite (Ca(OH)₂).

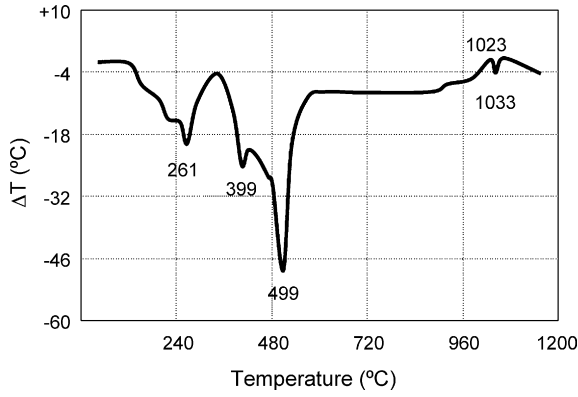


Fig. 2. Differential thermal analysis of the silicon carbide abrasive sample.

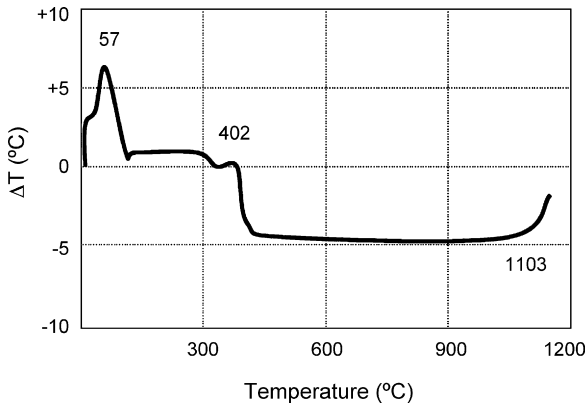


Fig. 3. Differential thermal analysis of the porcelain tile polishing residue sample.

1100 °C approximately there is an endothermic peak related to the beginning of the residue glass transition (T_g).

Fig. 4 shows the particle size distribution of the abrasive and the polishing residue. The polishing residue is 100% under 75 μm with a mean particle size of 10 μm . The abrasive residue is 100% under 90 μm with 15 μm mean particle size.

Regarding the apparent density of the sintered material an increase in the polishing residue addition causes a decrease in the density of the cellular ceramic, Fig. 5. Starting at 1000 °C the expansion promoted by the silicon carbide present in the

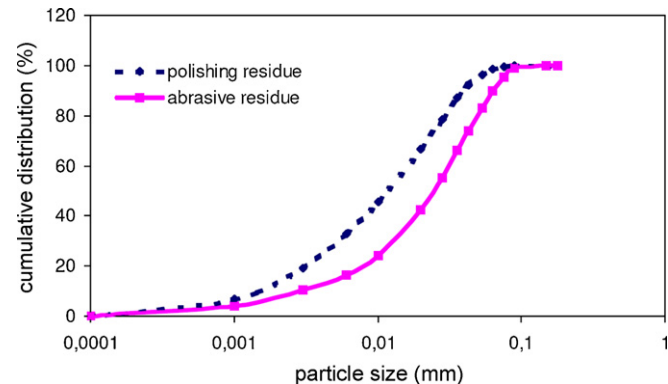


Fig. 4. Particle size distribution for the abrasive residue and the polishing residue.

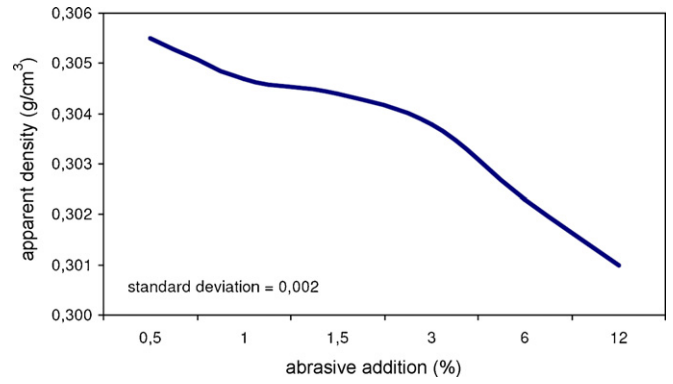


Fig. 5. Evolution of the apparent density of the cellular ceramic due the amount of abrasive addition.

abrasive (and in the polishing residue) forms large and rounded pores in the final product.

Even without abrasive addition the apparent density is very low because the residue contains many abrasive particles and volatile materials, probably organic substances incorporated in the residue during its polishing, transport and treatment processes. The very low mean density observed (0.301 g/cm³) allows the use of the cellular material as an acoustic or a thermal insulator.

An increase in the amount of abrasive addition on the polishing residue causes, as expected, a reduction in the mechanical resistance of the cellular ceramics obtained, Fig. 6. There is a great reduction in the flexural resistance of the samples starting at 3% of abrasive addition, probably due the quantity and size of the formed pores.

Besides the mechanical resistance reduction with the abrasive addition, the product presents an acceptable mechanical resistance adequate to several uses, mainly the building industry, as a substitute for cellular concrete. After sintering, all samples presented a great and gradual expansion with the addition of the abrasive residue. It is obvious the relation between abrasive addition and expansion, Fig. 7.

Again, even without the addition of the abrasive residue it was observed a great expansion of the sintered residue. 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

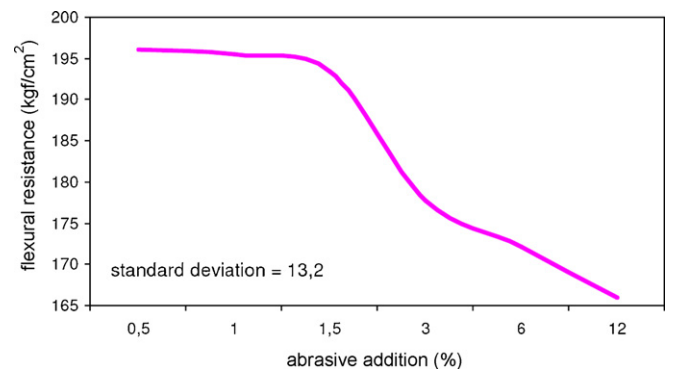


Fig. 6. Evolution of the flexural resistance of the cellular ceramic due the amount of abrasive addition.

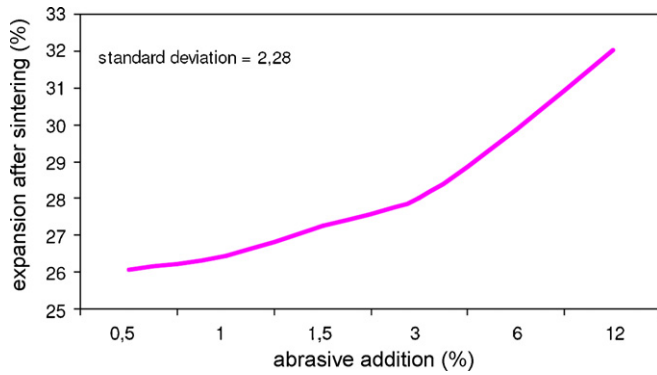


Fig. 7. Evolution of the linear expansion of the cellular ceramic due the amount of abrasive addition.



Fig. 8. Microstructure of the sample with 6% abrasive addition in the polishing residue after sintering (digital camera).

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 Fig. 8 it is observed the microstructure of one sample with 6% abrasive addition. The pores are big, closed and rounded, what can explain the good mechanical resistance besides the very low porosity observed for these products. It seems the product can present good thermal and acoustic insulation, but these properties were not analyzed in this study.

4. Conclusion

It is possible the use of 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. Even without addition of the residue containing silicon carbide it was observed a good expansion of the samples, already indicating the presence of SiC particles in the residue in function of the polishing process.

The product expansion (and consequent density reduction) occurs by the presence of closed pores in the microstructure of the samples. More the addition of silicon carbide (by abrasive addition) more the expansion of the samples, showing the effect of silicon carbide dissociation in pore formation, with density reduction and increasing expansion.

The mechanical resistance of the product reduces with the addition of abrasive residue because of the increasing of porosity. Besides the amount of pores present in the samples it was observed good flexural resistance (average of 180 kgf/cm²) showing that the product could be used as a building material. The mechanical resistance is due the rounded form of the pores present in the samples redistributing the tensions applied on the product, avoiding its concentration.

Finally, the expansion process occurs due the silicon carbide dissociation simultaneously to a viscous glass phase formed during sintering of the samples. The glass melting at the same time of SiC decomposition 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 added to (or present in) the porcelain residue result in the form, size and distribution of the pores in the final product.

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