PHYSICAL, STRUCTURAL AND MECHANICAL CHARACTERIZATION OF EXPANDED CERAMICS OBTAINED FROM POLISHING RESIDUES

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ABSTRACT

This work deals with physical, structural and mechanical characterization of expanded ceramics obtained from porcelain tile polishing residues expanded with silicon carbide abrasive residues. In the first work it was studied the expansion process, that means, SiC decomposition simultaneous with the polishing residue melting, both at ~1,200°C. Starting at 1,000°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 1,200°C. Now, the microstructure, expansion, density and mechanical properties (flexural tests) were determined to characterize the product. Expanded ceramics can substitute polymers (expanded polystyrene) and wood in internal partition walls and linings, and cellular concrete in the building industry.

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 [3,1].

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 [5]. 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 [8,10,11,14].

Currently there is no recycling of the solid residues from the tile ceramic industry in the State of Santa Catarina, in the south of Brazil. The region is the second tile ceramic producer in Brazil, with approximately 30% of the overall tile production and the first exporter. The ceramic residues are discarded directly on landfills, without any kind of separation [2,7,13]. The polishing ceramic residues, locally named "mud", are the remaining portions of the "gres porcellanato" polishing process containing abrasive particles detached from the abrasives used to polish the tiles, normally a 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 by-product. The mud is stored on landfills, causing environmental impact [12,9]. An alternative to the discard of ceramic solid residues is their use to make new products [4,6]. Specifically in this study the porcellanato polishing residues were used to form low-density ceramics. As presented in Qualicer 2004, 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 closed porosity. This product presents 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 oxides, mainly potash, soda and magnesia, with a low melting point as the porcelain tiles, or "gres porcellanato", are fired at 1,200°C during 40 to 50 minutes. Combining a glassy material with a relatively 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 1,000°C in presence of oxygen. Its decomposition results in silica and carbon dioxide according: $SiC+O_2 \rightarrow SiO_2+CO_2$. Therefore, the mixture of a vitreous material that melts at the same temperature of SiC decomposition can result in an expanded 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.

In this study the mechanical and physical properties of an expanded ceramic are related to the amount of abrasive added to a 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 subjected 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, CuKα, 0° to 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°C to 1,200°C, 10°C/min, air atmosphere).

After characterization, polishing residue samples were dried (110°C, 24h) 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, 24h) 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 (300kgf/cm^2) in cylindrical specimens (5cm diameter, 1cm height), five specimens for each formulation.

The compacts were sintered during 20min at 1,180°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 subjected 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, 10mm/min).

3. RESULTS AND DISCUSSION

Table 1 shows the chemical and phase analysis of the samples (porcelain tile polishing residue and abrasive residue) used in this study. 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 the procedure used to analyze the abrasive samples: the samples were calcined at 1,000°C during 3h, causing total conversion of the SiC present in the samples.

Sample	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO	MgO	K_2O	Na_2O	LOI.	Phases
Porcelain tile residue	59,5	17,3	0,7	0,3	1,6	5,3	2,8	3,6	5,6	Q, Z, Al
Abrasive residue	10,5	1,6	1,6	0,1	4,1	34,8	0,1	6,3	40,2	H, D, C, Mg, Cl, Q, P
Where: Q is quartz; Z is zircon ($SiO_2.ZrO_2$); Al is albite; H is halite (NaCl); D is dolomite; C is calcite; Mg is magnesite; Cl is clinochlore; P is Portlandite ($Ca(OH)_2$)										

Table 1. Chemical and phase analysis of the sample residues

Regarding the porcelain residue, it is formed by quartz, albite and zircon, the major phases of a porcelain tile paste. The amount of alkaline oxides present in the residue (13.3% in weight) shows the good vitrification of this system.

The thermal analysis test of the abrasive sample (figure 1) shows endothermic peaks between 260°C and 500°C, probably regarding the decompositions of carbonate (calcite, magnesite and dolomite) and hydroxide (Portlandite) present in the sample. Between 1,020°C and 1,030°C it can be seen endothermic and exothermic peaks, probably related to the dissociation of the silicon carbide into silica and carbon dioxide.

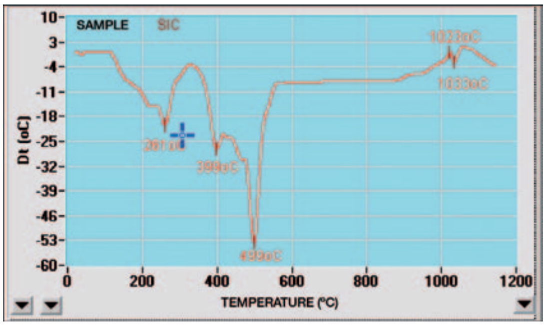

Figure 1. Differential thermal analysis of the silicon carbide abrasive sample

The thermal analysis of the porcelain tile polishing residue (figure 2) shows endothermic peaks between 20°C and 150°C, probably due to the thermal decomposition of organic substances that came from the effluent treatment, and another endothermic peak at 400°C, probably another organic decomposition. At 1,100°C approximately, related to the melting of the residue and beginning of glass formation.

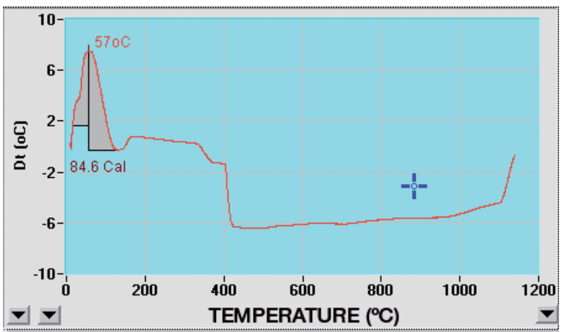

Figure 2. Differential thermal analysis of the porcelain tile polishing residue sample

Figure 3 shows the particle size distribution for the abrasive and the polishing residue. The polishing residue is 100% under 75μm with a mean particle size of 10μm. The abrasive residue is 100% under 90μm with 15μm mean particle size.

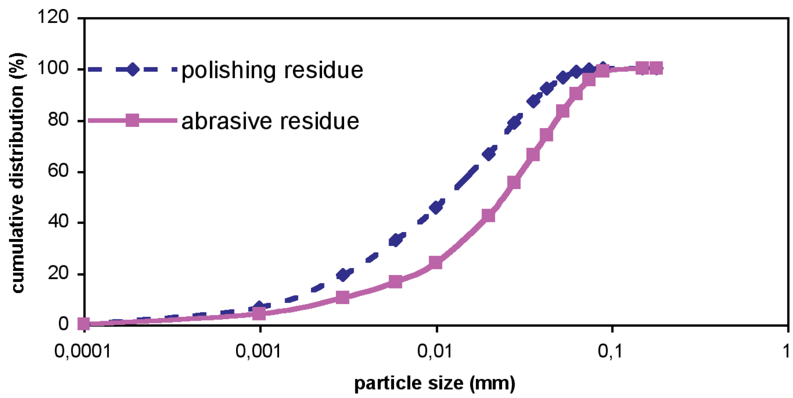

Figure 3. Particle size distribution for the abrasive residue and the polishing residue

Regarding the apparent density of the sintered material, an increase in the amount of abrasive addition on the polishing residue causes a decrease in the density of the expanded ceramic, figure 4. Starting at 1,000°C the expansion promoted by the silicon carbide present in the abrasive (and in the polishing residue) forms large and rounded pores in the final product.

Figure 4. Evolution of the apparent density of the expanded ceramic due the amount of abrasive addition

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 (0,3g/cm^3) allows the use of the expanded material as acoustic or 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 expanded ceramics obtained, figure 5. 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 pores formed.

Figure 5. Evolution of the flexural resistance of the expanded ceramic due the amount of abrasive addition

Besides the mechanical resistance reduction with the abrasive addition, the product presents an acceptable mechanical resistance 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, figure 6.

Figure 6. Evolution of the linear expansion of the expanded ceramic due the amount of abrasive addition

Again, even without the addition of the abrasive residue it was observed a great expansion of the sintered residue. The expansion, probably because of the presence of volatile materials added during the polishing residue processing, does not allows the use of this kind of residue in ceramic formulations for very dense tiles, as the porcelain tiles. Many attempts to add residues from treatment stations in the composition of dense tiles failed due the great porosity of the products after firing.

Finally, in figure 7 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 a 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.

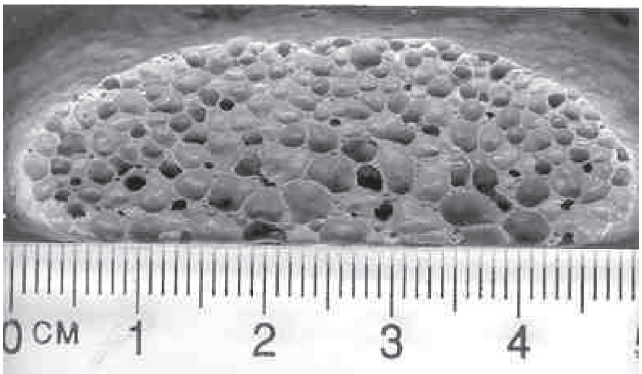

Figure 7. Microstructure of the sample with 6% abrasive addition in the polishing residue after sintering (digital camera)

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 expanded ceramics with low density. The expanded ceramics can be used in the building industry as substitutes for cellular concrete because their low density, resulting in low weight of the structures, and acceptable mechanical resistance.

It seems the product has good acoustic and thermal insulation and could be used as a substitute for wood and polymers in internal walls and linings, though these properties have not been determined at this moment.

The reduction of the apparent density is related to the presence of volatile materials. Even without addition of the residue containing silicon carbide it was observed a good expansion of the samples, probably indicating the presence of organic materials in the polishing residue added during its processing and treatment.

The product expansion (and consequent density reduction) occurs by the presence of closed pores in the microstructure of the samples. The larger the addition of silicon carbide (by abrasive addition), the greater 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, a mean of 180kgf/cm², 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, what redistribute the tensions applied on the product, avoiding tension concentration.

Finally, the expansion process occurs because the silicon carbide dissociation and organic material decomposition develop simultaneously with a viscous glass phase formation during sintering of the samples. The glass formation related to degasification 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 the porcelain residue result in the form, size and distribution of the pores in the final product.

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