



Research paper

Technological properties of ceramic produced from steatite (soapstone) residues–kaolinite clay ceramic composites



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ABSTRACT

Ceramic bodies (7.0 cm × 2.0 cm × 1.0 cm) of kaolinite clay and soapstone residuals collected from workshops in Ouro Preto and Mariana, Minas Gerais, Brazil, containing from 2.5 to 97.5 wt% steatite (soapstone) were prepared and firing at 500, 1000 and 1200 °C, for 2 h, in air. The linear shrinkage, compressive strength, water absorption and mass loss by heating were determined on the samples after heat treatment. The fired samples at 1000 and 1200 °C, with steatite percentages of 85, 90 and 95%, presented the best results for technological applications in ceramic industry. For these samples, the values of the compressive strength were higher than 10 MPa and those of water absorption varied between 8 and 22%, which means that the values of these properties are superior and inferior, respectively, to the reference values established by Brazilian Standards. The linear shrinkage was lower than 6%, which is the maximum value established by the Pólo Cerâmico de Santa Gertrudes, in São Paulo State. These samples were chemically, mineralogically, and morphologically analyzed using ICP/OES, X-ray diffraction, Mössbauer spectroscopy, SEM and BET.

Talc and kaolinite were the dominant minerals, followed by quartz, chlorite, magnetite and magnesite. When firing at 1200 °C, the talc changes to enstatite and the appearance of mullite, periclase, hematite, clinoenstatite and protoenstatite occurs. The partial fusion of the talc promoted an increase in the liquid phase diminishing porosity and, consequently, water absorption. This process and the combination with mullite and periclase, increased the strength, reaching the values of 78 MPa, which is much greater than the minimum value of 10 MPa defined by the Brazilian Standard 15270-1 (ABNT 2005) for application on structural ceramic blocks.

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1. Introduction

The southeastern region of Minas Gerais, Brazil, especially the Barroque cities of Ouro Preto and Mariana, is well-known for its soapstone art works that include traditional handicraft objects and kettles. This historical exploration of soapstone created handicraft shops, processing industries, separation and mass artifact production. These manipulation and production processes generate a great amount of fines that are normally discarded in inadequate places, being deposited in the soil and in waterways, causing silting and contamination. In addition, in this region, commonly occur kaolin deposits not yet industrially explored in spite of the great potential for the production of ceramics, paints, rubber, paper, pozzolanic material and mullite (Murray, 2007). The technological characterization of the kaolinite clay from this region has been the focus of various studies (Morales-Carrera et al., 2010;

Peralta-Sánchez et al., 2011a,b) which showed the need for additional researches to enhance the kaolinite clay application in the ceramic industry. Talc, which is the principal mineral present in the steatite, has a trioctahedron structure of 2:1 layers with no charge on the layers. The layers are bonded by van der Waal forces (Sánchez-Soto et al., 1997; Wiewióra et al., 1997). This mineral has important properties for the manufacturing of many industrial products, such as cosmetics, pharmaceuticals, filler in paper, pesticides, polymers, paints, rubber and also in ceramics because of its low thermal and electrical conductivity and capacity to improve the mechanical characteristics and dimensional stability of ceramics (Sánchez-Soto et al., 1997; Dellisanti et al., 2009). During the heating of the mineral, water is liberated and amorphous phase and enstatite appear. The thermal treatments even give rise to other polymorphic forms such as protoenstatite and clinoenstatite (Wesolowski, 1984; Sánchez-Soto et al., 1997).

Therefore, the use of steatite residuals as additives to the ceramic mass can be an alternative to improve the quality of the kaolinite clay products besides contributing to the reduction of environmental pollution.

The principal objective of this study was to evaluate the properties of the ceramics produced from mixing kaolinite clay with the steatite (soapstone) residuals from the region of Ouro Preto and Mariana.

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Table 1
Percentage (wt.%) of steatite (S) in the mixture steatite (S) + kaolinite clay (R5).

Sample	Mixture proportions	Sample	Mixture proportions	Sample	Mixture proportions
PR	R5	P5	R5 + S (20%)	P10	R5 + S (85%)
P1	R5 + S (2.5%)	P6	R5 + S (25%)	P11	R5 + S (90%)
P2	R5 + S (5%)	P7	R5 + S (50%)	P12	R5 + S (95%)
P3	R5 + S (10%)	P8	R5 + S (75%)	P13	R5 + S (97.5%)
P4	R5 + S (15%)	P9	R5 + S (80%)	PS	S

2. Materials and methods

Samples of steatite residuals (E) collected from handicraft shops in Cachoeira do Brumado, district of Mariana, and of kaolinite clay (R5) from a slope on highway BR-383, at 40 km from Ouro Preto, were dried at a temperature of 65 °C, for 72 h, in air, crushed and ground to yield a powder with a particle size suitable to pass through a #35 mesh (425 µm) sieve.

The humidity of steatite and kaolinite clay was measured in an electrical resistance ID200-Marte scale with 3 g of material for 3 min, followed by an adjustment of 10%; a value considered more adequate for the fabrication of the samples under pressure (Gaspar Júnior, 2003; Morales-Carrera et al., 2010). Fifteen samples were prepared containing different proportions of kaolinite clay and steatite (Table 1).

Bricks with a mass of 40 g and approximate dimensions of 7.0 cm × 2.0 cm × 1.0 cm, were molded at a velocity of 1 kN/s until reaching 50 kN, corresponding to a final pressure of 35 MPa, utilizing a uniaxial hydraulic press C1-SOLOCAP model LM-02, Digital Dynamometry Apnea. Then, the bricks were dried in an oven at 65 °C, in air. Their dimensions were measured every 2 h until present dimensional stability. After this time, the bricks were kept in the oven for 24 h, totalizing 72 h. After drying, the bricks were fired at 500, 1000 and 1200 °C, for 2 h (plateau at the maximum temperature of treatment), in air, using the electric furnace Giron 1200. The heating rate was 5°/min. Through macroscopic analysis, without using equipment, samples were chosen that were free of systematic defects, such as cracks, chips, and surface irregularities. They also needed to be homogeneous in color and free of other or imperfections (ABNT, 1983). Other tests, such as linear firing shrinkage (LS), compressive strength (CS), bulk density (BD), water absorption (WA) and mass loss by heating (ML) at 1200 °C for 2 h were performed. For each sample, a total of three bricks per firing temperature were tested, and the average was recorded. Linear firing shrinkage (LS) of bricks was obtained from:

$$LS (\%) = (l1 - l2) 100 / l1.$$

where: l1 = length of bricks before firing and l2 = after firing.

Bulk density (BD) was calculated by the ratio of the mass (*m*) to volume (*v*):

$$BD = m/v$$

For WA test, the mass of bricks before firing was measured (M1). The bricks were soaked in boiling water for 2 h and cooled with running water. Then, the bricks were reweighed (M2). Water absorption capacity was determined as

$$WA = (M2 - M1) \times M1 \times 100$$

To determine ML, the mass of bricks before firing (M1) and after firing was weighed. Mass loss by heating was calculated as

$$ML = (M1 - M2) \times M1 \times 100$$

For these tests a Vernier caliper Mitutoyo and a precision balance Kern KB were used.

Mechanical tests NBR 15270-3 (ABNT, 2005) were executed using a uniaxial hydraulic press at a velocity of 1 kN/s, C1-SOLOCAP model LM-02, Digital Dynamometry Apnea. Three samples, P10, P11 and P12, with the best results from the physical and technical tests were submitted to chemical, mineralogical and morphological analyses.

The chemical composition was determined by inductively coupled plasma/optical emission spectrometry (ICP/OES) with radial vision, Ciro CCD model, Spectro. The crystalline phases, before and after firing, were determined by X-ray diffraction (XRD) using a Panalytical EMPYREAN diffractometer with CuKα radiation, range of 2–70° 2θ, step size of 0.02 and counting time of 10 s. Morphological features were observed by scanning electron microscopy with X-ray microanalysis (SEM/EDS-Secondary Electrons), Vega 3 Tescam/Oxford, with gold coating. Volume fraction of porosity (pore volume) was measured by the nitrogen absorption using the Quantachrome BET (Brunauer-Emmett-Teller) model Nova 1200e. The N₂BET technique (Brunauer et al., 1938) is explained by the tendency of all solid surfaces of attracting molecules of surrounding gas, resulting in a process called gas sorption. The grain size distribution of the steatite and kaolinite clay was measured on a laser particle-measurement instrument, Cilas 1064, using the optical model Fraunhofer.

The Fe content and the type of Fe³⁺ and Fe²⁺ occupation in the raw and sintered phases were determined by Mössbauer spectroscopy (MS). Mössbauer spectra (MS) were collected at room temperature with a spectrometer using a constant-acceleration drive with triangular reference signal, 1024 channels (unfolded), and in the velocity range of −11 to +11 mm/s (increment of ~0.09 mm/s). The velocity was calibrated from the MS of a standard α-Fe foil at room temperature. The spectra were computer-fitted either with discrete Lorentzian sextets and/or doublets or with distributions of magnetic hyperfine fields.

3. Results

After drying at a temperature of 65 °C, for 72 h, the samples with the greatest quantity of R5 (Table 1), up to P7 (R5 + E(50%)), presented a reddish color (Fig. 1). This color turned to gray as the amounts of steatite content increased from P8 to PE. Laser particle size analysis for the kaolinite clay showed a mean particle size of 12.68 µm and a

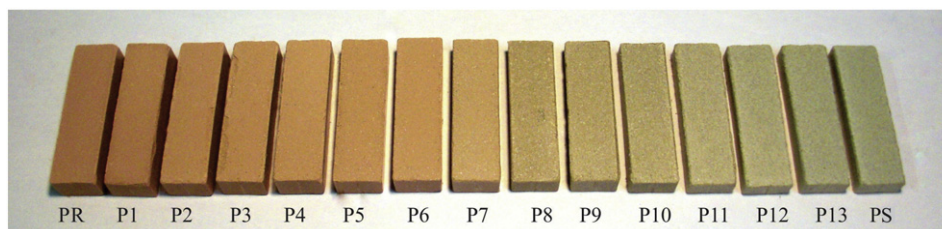


Fig. 1. Color evolution of the samples, evaluated macroscopically without instruments, after drying at a temperature of 65 °C, for 72 h, in air.

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