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Incorporation of SCBA in Red Ceramics and Sintering in Microwave Oven

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ABSTRACT

The present study investigated the incorporation of sugarcane bagasse ash (SCBA) in red ceramics, sintered in conventional oven and microwave oven, aiming to provide an alternative product, and a sintering process with higher energy efficiency in the production of red ceramics. The raw materials were characterized by XRF, XRD, thermogravimetry, particle size distribution and specific mass analyses. The specimens were shaped by extrusion in two different compositions, red clay and red clay with addition of 20 % SCBA and sintered at temperatures from 700 to 1100 °C. The conventional sintering occurred for 60 min with heating rate of 10 °C/min. In the microwave oven the sintering occurred in a hybrid way, with heating rate of 50 °C/min for 5, 10 and 15 mins. After sintering the tests of linear shrinkage, compressive strength, water absorption, apparent porosity and apparent specific mass were performed. The addition of SCBA causes an increase in the values of water absorption and decreases the compressive strength and specific mass of the red ceramic. This occurs due to the creation of pores inside the material from the volatilization of organic matter present in the ashes. The sintering in microwave oven, when compared to conventional sintering, promotes an increase in the values of compressive strength and specific mass and reduction of water absorption values of ceramics, probably due to the refinement of the microstructure and the higher densification. Thus the incorporation of ashes can be partially compensated by a more efficient sintering. The use of SCBA and the sintering in microwave oven, showed to be viable alternatives in the development of a more sustainable and light material, promoting the management of waste, reduction in the consumption of raw materials and energy saving.

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1. Introduction

Red ceramics is one of the main materials used in civil construction, accounting for 4.8 % of the sector. It is used mainly for the production of bricks, tiles and pipes^[1,2]. Although the data is outdated, it is estimated that in Brazil the total number of red ceramic factories is 6,903, contemplating industries from small to large^[2].

The composition of red clay, the raw material used in the manufacture of red ceramic materials, can vary according to the place of extraction, so the quality of the final product is mainly related to the composition and sintering (stage in which the physical and chemical reactions occur) of the material^[3]. Currently, the sintering of red ceramics occurs in a conventional way, which can reach 60 h, which entails a high energy demand^[4].

The reuse of waste has become essential to reduce the negative impacts caused to the environment. Because the chemical composition of red clay is heterogeneous, solid waste can be successfully incorporated into this raw material, giving them a destination and reducing the consumption of natural raw materials that are becoming scarce^[5].

Brazil is the world's largest producer of sugarcane, having produced around 657 million tonnes of sugarcane in the 2020/2021 harvest. The state of São Paulo is responsible for 54 % of national production^[6]. Each ton of sugarcane used in the production of sugar and ethanol generates on average 450 kg of waste (bagasse and straw). These residues are burnt in boilers for co-generation of electric energy, reducing the industry's costs with energy and generating ash^[7,8].

A study carried out in the laboratory indicated that for each kg of bagasse and sugarcane straw, approximately 30 g of ash are generated, so in the 2020/2021 harvest, if all the bagasse were burned, 8,869 million tons of SCBA could have been generated.

One of the alternatives adopted by the sugar and ethanol industries is the use of SCBA as fertilizers in agriculture due to their macro and micronutrients, but some properties can be negatively affected, which can be harmful to the soil^[9-12]. There are already several works that have studied the incorporation of SCBA in red ceramics, mainly to replace the silica found in clay, seeking to reduce the consumption of natural raw materials and reduce the negative impacts that waste can cause to the environment^[13-16].

The ceramics industry mostly consumes energy from non-renewable sources, such as natural gas which is a fossil fuel. In the production of ceramics, the stage that presents the highest energy consumption is sintering,

corresponding to more than 50 %, which occurs at high temperatures and over long periods. Thus it is necessary to seek alternative types of sintering with greater energy efficiency and use renewable sources^[17].

In conventional sintering energy is supplied to the material by convection, conduction, advection and radiation heat, thus initially heating the surface to the centre^[18,19].

In the microwave oven sintering happens in a volumetric way, because there is an interaction of the molecules with the electromagnetic field, thus the microwaves are absorbed by the material and transformed into heat in a uniform way, providing a fast heating with a greater energy saving, uniform porosity in the initial stages and greater densification, compared to the conventional method^[18,20-22].

Although microwave sintering has numerous advantages such as lower grain growth, higher densification, better mechanical properties, reduction in temperature, selective heating, rapid heating and energy saving^[23,24], some problems must be solved so that the heating occurs in an adequate way, such as the thermal instabilities, the low absorption of microwaves by transparent materials (low dielectric loss at low temperature) such as red ceramic, which allows the passage of microwaves through the material^[19,25,26].

Currently there are studies about the hybrid sintering of ceramic materials using a susceptor. The susceptor provides the heating of the transparent materials at low temperatures by the method of convection, conduction and radiation of heat until they begin to absorb microwaves (critical temperature) being then heated in two ways, by the susceptor and by the microwaves, this allows the reduction of thermal instabilities in the material^[27-31].

There are few studies on the sintering of red ceramics with the addition of residues. Taurino et al (2017)^[32], studied ceramic bricks with the incorporation of municipal solid waste sintered by microwave. The use of a waste and a more efficient sintering contributed to the generation of a more sustainable material, with lower consumption of energy and natural materials.

Lyra et al (2019)^[33] evaluated light aggregates of red ceramics with the incorporation of SCBA sintered in microwave oven. The incorporation of ash promoted a decrease in the specific mass of the aggregates, whereas the microwave sintering collaborated with an increase in the compressive strength and a reduction in water absorption.

Thus the objective of this study is to investigate the incorporation of sugar cane bagasse ash in red ceramics, sintered in conventional oven and microwave oven,

aiming to provide an alternative raw material, and a sintering process with greater energy efficiency, for the manufacture of materials for civil construction, as lightweight aggregates.

2. Experimental Procedure

The raw materials used in this work were, red clay (iron oxide above 4 % of its composition), provided by an industry manufacturer of ceramic tiles Top Telha of the city of Leme-SP, and sugar cane bagasse ash (SCBA) provided by a company producing sugar and ethanol Baldin Bioenergy, in the city of Pirassununga-SP. The raw materials were dried in oven at 100 °C for 48 h, where mass stabilization is performed.

The chemical compositions of the raw materials were determined by an X-ray fluorescence spectrometer (PANalytical model MiniPal4). The loss on ignition was performed at 1100 °C for 1 h, with a heating rate of 10 °C/min, according to ASTM C114:18^[34]. Mineralogical analysis of the raw materials was performed by X-ray diffraction (Rigaku Rotaflex model Miniflex 600) with graphite monochromator in the secondary beam, operating at 40 V/15mA, using scan angle of $5^\circ \leq 2\theta \leq 90^\circ$ and step 5 °/m. Particle size analysis was performed on a laser particle size analyser (Horiba model LA-950V2), the particles were dispersed in isopropyl alcohol. Differential thermal analysis was performed in a thermogravimetric analyzer (NETZSCH model STA449 F3 Jupiter) between the temperature of 25 to 1200 °C, using a heating rate of 10 °C/min under an inert atmosphere of nitrogen at a flow rate of 20 mL min⁻¹.

Two different mixtures were prepared for making the specimens, the first (reference) with 100 % clay, and the second with 80 % clay and 20 % SCBA. The amount of water used in each ceramic body was defined according to ABNT NBR 7180:2016^[35]. The raw materials were added and homogenized for 5 min in an intensive mixer (Eirich Industrial Ltda model RV02/E). Then the ceramic masses were extruded in a laboratory extruder, in the form of a cylindrical bar with 1.5 cm in diameter and cut to 3 cm in length. After forming, they were dried naturally for 24 hours and then dried in an oven at 100 °C for 48 hours. The specimens were characterized for green density (density of material before sintering).

The specimens were pre-sintered in an electric furnace (Jung Furnaces, model 10010, number 4718) at 600 °C for 60 min, at a heating rate of 10 °C/min. For sintering purpose an electric oven (Jung Ovens, model 10013, power 7 kW) and microwave oven (Panasonic, model NN-ST674SRUN, frequency 2450 MHz and output power 900 W) were used. The sintering in electric oven (conventional)

was performed with heating rate of 10 °C / min, and the specimens were exposed to maximum temperatures of 700 °C to 1100 °C for 60 min. In the microwave oven the temperatures used were the same, but the heating rate was 50 °C/min. The material was submitted to the maximum temperatures for 5, 10 and 15 mins. For microwave sintering, a silicon carbide susceptor was used so that at the initial temperatures it absorbed the microwave energy and transferred heated to the specimens.

Table 1. The proportions of the mixtures for the formulations (% by weight) and types of sintering.

Formulation	Clay	SCBA	Sintering
100A - 60CS	100	0	Conventional (60 min)
100A - 5MWS	100	0	Microwave (5 min)
100A - 10MWS	100	0	Microwave (10 min)
100A - 15MWS	100	0	Microwave (15 min)
60A - 20SCBA - 60CS	80	20	Conventional (60 min)
60A - 20SCBA - 5MWS	80	20	Microwave (5 min)
60A - 20SCBA - 10MWS	80	20	Microwave (10 min)
60A - 20SCBA - 15MWS	80	20	Microwave (15 min)

After sintering, the specimens were characterized by linear shrinkage, water absorption, apparent porosity, apparent specific mass, mechanical strength, X-ray diffraction (XRD) and scanning electron microscopy (SEM).

The linear shrinkage values were evaluated from the variation of the length of the specimens before and after sintering. The water absorption values were determined according to ASTM C373-88:2006^[36] (immersion in boiling water for 5 h). Apparent porosity and apparent specific mass were determined by Archimedes water immersion method according to ASTM C373-88:2006^[36]. The compressive strength test of sintered specimens was performed in a Universal Testing Machine EMIC DL 30000, based on ABNT NBR 5739:2007^[37].

The parameters used in the mineralogical analyses (XRD) of the already sintered specimens were the same used in the characterization of the raw materials.

The SEM characterization was performed only on the specimens sintered at 1100 °C, in a conventional oven and microwave at 10 min. This temperature was chosen because its better, and the time of 10 min for being intermediate between the times studied for the microwave. For microstructural characterization (SEM), a Philips XL 30 FEG scanning electron microscope was used. The surfaces of the specimens were sanded and then polished using diamond solution in the sequence of 9, 6, 3 and 1 µm.

3. Results and Discussion

Table 2 shows the chemical composition of the raw materials used. The clay analysed presented more than half of its composition formed by silicon dioxide (SiO₂), representing 66.11 %. The content of SiO₂ + Al₂O₃ (total 79.44 %) present in the raw materials influence the compressive strength of the sintered materials due to the formation of the vitreous phase. On the other hand, the presence of Fe₂O₃ above 4 % in the raw material, provides the reddish coloration of the pieces after sintering.

The SCBA sample presented a high percentage of loss on ignition, 58 % of its composition, which can represent the presence of organic matter and hydrocarbons, this is because the burning in boilers does not occur efficiently, thus not completely consuming the organic materials. The SiO₂ + Al₂O₃ compose approximately 24 % of the ash. As the clay presents higher silica content, the addition of SCBA in the same decreases the amount of SiO₂ of the total mass and increases the amount of organic matter, which when sintered in the material, volatilize forming voids. Thus, the materials with the addition of SCBA tend to present higher water absorption and lower mechanical strength.

Figure 1a and 1b show the results of the mineralogical composition of the crystalline solids of the raw materials, clay and SCBA, respectively. Peaks of the agrominerals muscovite and illite of the mica group, are found in the clay. The illite may appear as a result of the degradation of the muscovite.

As shown by XRF analysis, the diffractogram of the clay shows several peaks of quartz, indicating that it is the main mineral present. It is possible to identify an accentuated peak in the range of 2θ = 27 °, which can be an indication of the presence of quartz, being the same

one of the main formers of the glassy phase of the ceramic products. The magnetite peak identified, indicates the presence of iron, responsible for the red colour of the ceramics obtained after sintering, as mentioned.

Table 2. Raw materials chemical compositions obtained through XRF analysis (wt. %).

Oxides	Clay	SCBA
SiO ₂	66.11	20.62
Al ₂ O ₃	13.33	3.58
K ₂ O	4.87	4.51
Fe ₂ O ₃	6.82	2.82
TiO ₂	0.95	0.88
CaO	0.00	4.51
MnO	0.00	0.11
ClO ₂	0.00	0.93
P ₂ O ₅	0.00	2.39
SO ₃	0.00	1.65
LOI	7.92	58.00

The mineralogical composition of the SCBA is formed by an amorphous phase due to the organic matter present in the raw material, and quartz, originating from the contamination by sand during the harvest, and the silicon from the structural part of the sugar cane. The amorphous phase can be identified by the XRD baseline, which does not have well-defined peaks.

Table 3 presents the equivalent diameter values of clay and SCBA particles. Figure 2a and 2b present the curves of discrete and cumulative particle size distributions of clay and SCBA respectively. The clay showed 90 % of its particles smaller than 113 μm. According to the classification by soil particle diameter, the clay used has, 73,15 % silt (between 2 and 50 m), 20,24 % fine sand (between 50 and 200 m) and 6,60 % coarse sand (between 200 and 2000 m). SCBA has 90 % of the particles with

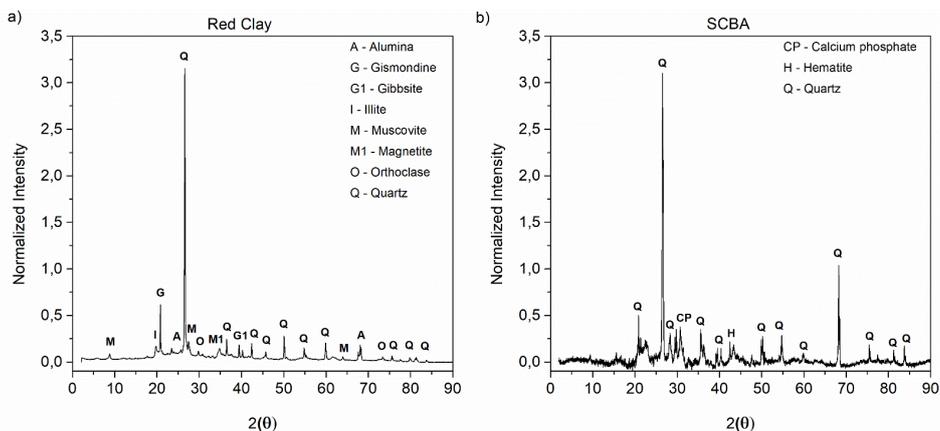


Figure 1. X-ray diffraction pattern of a) red clay and, b) SCBA. (The intensity was normalized by dividing the axis by 10⁴)

sizes less than 114 μm . Being 50,35 % silt, 47,72 % fine sand and 1,91 % coarse sand ^[38].

Table 3. Equivalent particle diameters.

Raw-material	D10(μm)	D50(μm)	D90(μm)	Average diameter (μm)
Clay	5.55	16.66	113.04	51.83
SCBA	13.02	44.54	114.90	56.51

The TG and DTA curves of the clay (a), show a mass loss throughout the thermal process of 11.85 %. The first peak occurs at approximately 44 $^{\circ}\text{C}$, and is related to the loss of surface water, adsorbed by the clay particles. Near 464 $^{\circ}\text{C}$ there is a broad peak, which indicates the decomposition of the organic matter. Around 572 $^{\circ}\text{C}$

occurs the decomposition of organic matter and the transformation of α to β -quartz ^[39]. From 900 $^{\circ}\text{C}$ on, there is a chemical reaction of the silica and alumina with the melting elements. Due to this reaction there is the formation of complex silicoaluminates, which give the ceramic material hardness, stability and resistance to some chemical substances.

The TG and DTA curves of the SBA (Figure 3b), present a total loss of mass of 70.51 %, possible to identify an exothermic peak around 56 $^{\circ}\text{C}$, which is related to the loss of water retained by the particles of SCBA. At approximately 319 $^{\circ}\text{C}$ there is an exothermic peak, indicating the dehydration of calcium phosphate and the volatilization of substances. At around 519 $^{\circ}\text{C}$ occurs the greatest loss of mass of the material, and an

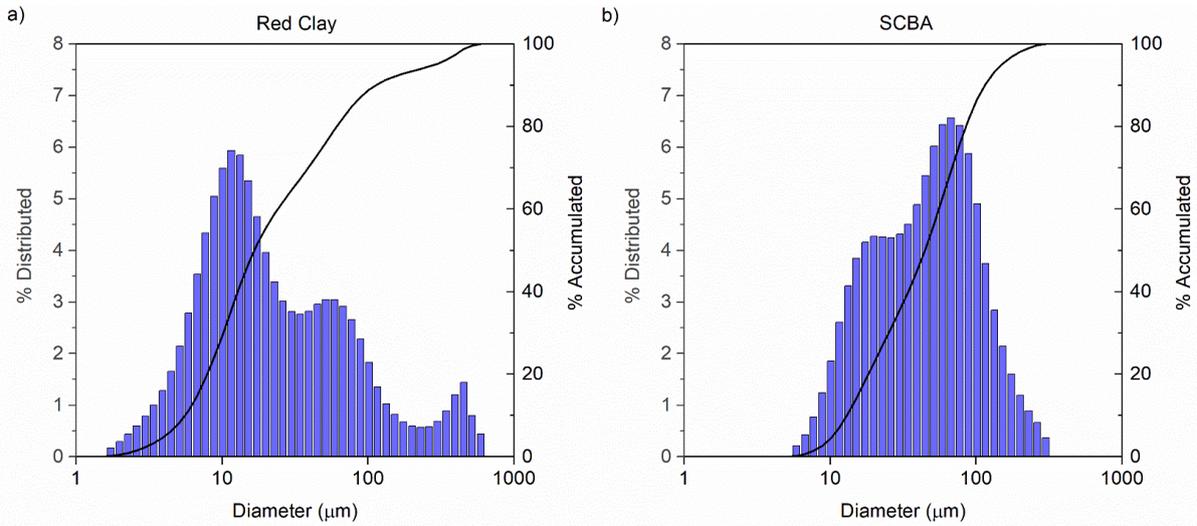


Figure 2. Discrete and cumulative particle-size distribution curves of a) red clay and, b) SCBA.

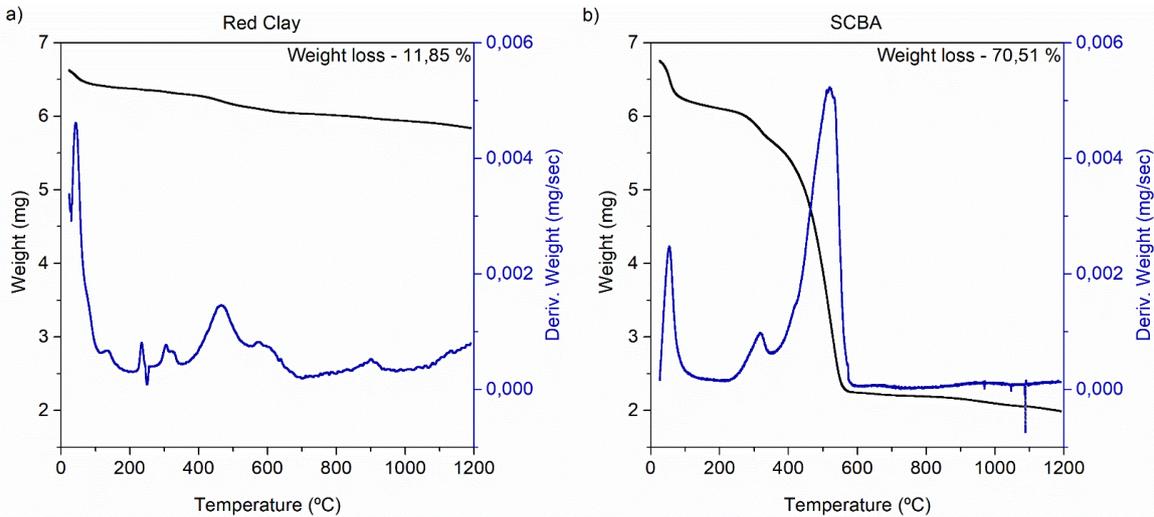


Figure 3. TG e DTA curves for a) clay and, b) SCBA.

intense exothermic peak can be observed. This indicates the decomposition of organic matter that was not completely volatilized during the burning of sugarcane bagasse in the industry. At approximately 575 °C occurs the transformation from α to β -quartz [39], this event may have been superimposed by the decomposition of organic matter.

The influence of the incorporation of 20 % SCBA and the sintering in microwave oven was determined through green density, linear shrinkage, water absorption, apparent porosity, apparent density mass, compressive strength, XRD and scanning electron microscopy. Being the specimens composed of 100 % considered the reference.

The green densities obtained for the different compositions of specimens before sintering are presented in Table 4. As expected, the density obtained for the specimens conformed only with clay is higher than those with added ash, since the real specific mass of the same is also higher. Being the real specific gravity of the clay 2,64 g/cm³ and of the SCBA 1,31 g/cm³.

Table 4. Green density

Compositions	Green density (g/cm ³)
Clay	1.76
Clay + 20 % SCBA	1.40

Figure 4a and 4b show the linear shrinkage values of red ceramic and red ceramic specimens with added SCBA, sintered in conventional oven (CS) and microwave oven (MSW).

Regardless of the composition of the specimens, as the sintering temperature increases, the linear shrinkage also increases, presenting significant values for the temperature

of 1100 °C.

The red ceramic specimens without addition were those that presented the lowest linear shrinkage, thus possessing greater dimensional stability. This occurred due to the greater presence of organic matter and hydrocarbons in the ceramics with added SCBA, this material volatilizes during sintering causing greater shrinkage.

In most cases the microwave oven provided greater shrinkage to the specimens when compared to the conventional oven. This occurs because this sintering method provides greater densification to the ceramic materials, thus occurring greater shrinkage. The temperature of 1100 °C was the one that presented the greatest statistical variability between the sinterings. For red ceramics the greatest shrinkage was obtained in the microwave oven in 10 mins being 62 % greater than in the conventional oven. For red ceramics with addition of 20 %, the highest value was obtained in a microwave oven in 15 mins, 41 % higher than in a conventional oven.

The water absorption is related to the portion of open pore volumes in the microstructure of the ceramic matrix. Analysing a and 5b, it is observed that the increase in temperature decreases the water absorption, due to the higher densification of the ceramic material, being the most expressive values verified for the temperature of 1100 °C.

As expected, the addition of SCBA provided an increase in water absorption due to the formation of pores in the material after sintering. For red ceramics, the specimens sintered in microwave oven for 15 min at 1100 °C showed a lower value of water absorption, being 58 % lower in relation to conventional sintering. The

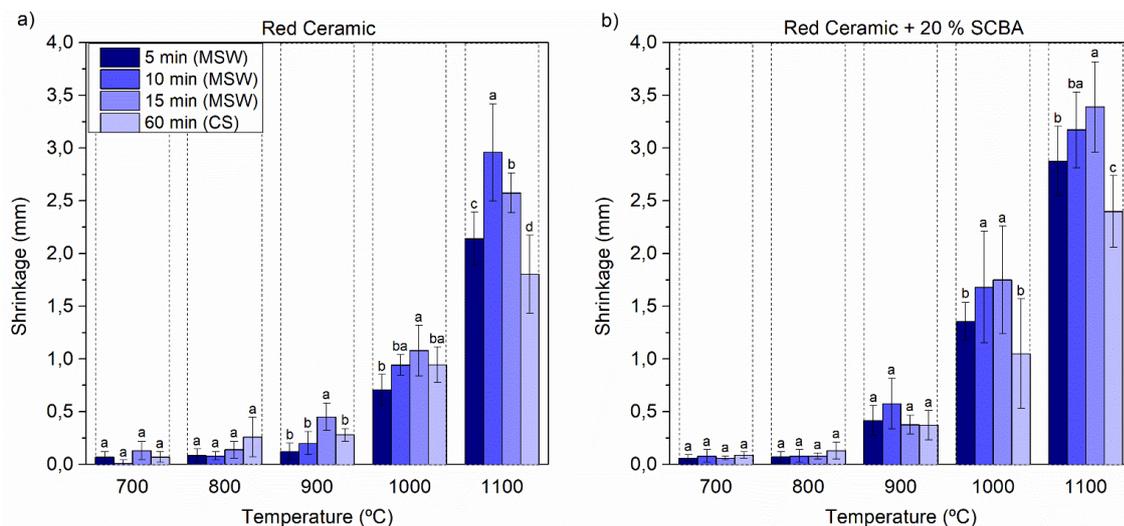


Figure 4. Shrinkage trend curves (%) of a) red clay ceramics and, b) SCBA-added red clay ceramics.

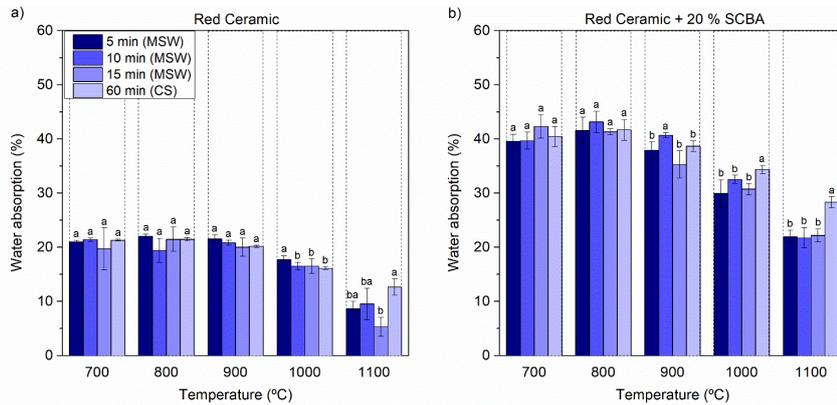


Figure 5. Water-absorption trend curves (%) of a) red clay ceramics and, b) SCBA-added red clay ceramics.

microwave. The red ceramic specimens with an addition of 20 % SCBA sintered in a microwave oven for 10 min, showed a decrease of 23 % of water absorption compared to conventional sintering.

Due to the volumetric heating provided by microwaves the materials have greater densification and a more uniform microstructure, which contributes to the decrease in water absorption.

The values of apparent porosity seen in Figure 6a and 6b are consistent with those found for water absorption. Thus, the red ceramic specimens presented the lowest results. The red ceramic specimens sintered in a microwave oven for 10 minutes showed a reduction in apparent porosity of 86 % compared to conventional sintering. For red ceramics with added SCBA, the specimens sintered in a microwave oven for 15 min at 1100 °C showed a lower value of apparent porosity, being 45 % lower compared to conventional sintering.

The specific mass is directly related to the values of water absorption and apparent porosity, so the higher these values, due to the number of pores, the lower the specific mass will be. For both compositions, the specimens sintered at 1100 °C presented higher specific mass due to greater sinterability which provides greater densification, as observed by the results of linear shrinkage, water absorption and apparent porosity.

The red ceramic specimens sintered in microwave oven for 10 mins, showed an increase of 10 % in specific mass compared to those sintered in conventional oven. The red ceramic specimens with addition of SCBA sintered in microwave oven for 10 mins, showed an increase of 8 % in specific mass when compared to those sintered in conventional oven.

Figure 8a and 8b show the compressive strength values of red ceramic and red ceramic specimens with added

SCBA, sintered in conventional oven and microwave oven. The mechanical strength of materials is one of the most affected properties when there are pores inside the material.

The increase in temperature, due to the decrease of pores provides consequently an increase in the values of compressive strength for the two compositions.

The results obtained showed that the addition of SCBA tends to decrease the mechanical strength of the specimens, so the higher the addition of SCBA, the lower the strength of the material, which was expected due to the organic matter present before sintering, which causes voids in the material.

The averages of compressive strength obtained for red ceramics are close to each other until the temperature of 900 °C, when occurs the completion of the phase transition (α -quartz) to hexagonal (β -tridimite). After this stage at the higher temperatures, the 10 min sintering in microwave oven presented superior performance for the ceramics as verified by the Tukey Test, resulting in compressive strength values about 57 % higher than the conventionally sintered ones at the temperature of 1100 °C. The ceramics with an addition of 20 % SCBA showed an increase in compressive strength of 58 % for the specimens sintered in a microwave oven for 15 mins at 1100 °C when compared to conventional ceramics. This fact probably occurred due to the beginning of the transition to the cristobalite phase^[39].

In most cases, the compressive strength results obtained for the specimens sintered in a microwave oven were higher than those sintered in a conventional oven. This effect can be attributed to the sintering process in microwave oven, which occurs more uniformly in the volume of the material.

The compressive strength results of the red ceramic

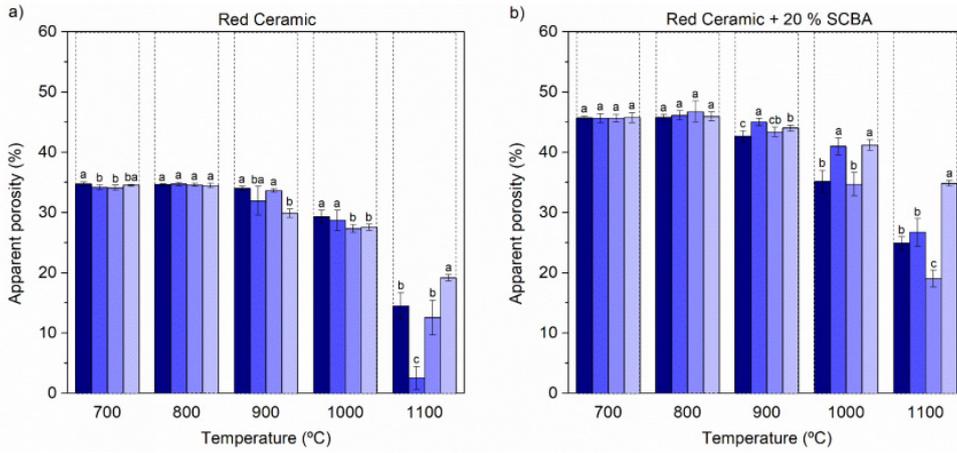


Figure 6. Apparent-porosity trend curves (%) of a) red clay ceramics and, b) SCBA-added red clay ceramics.

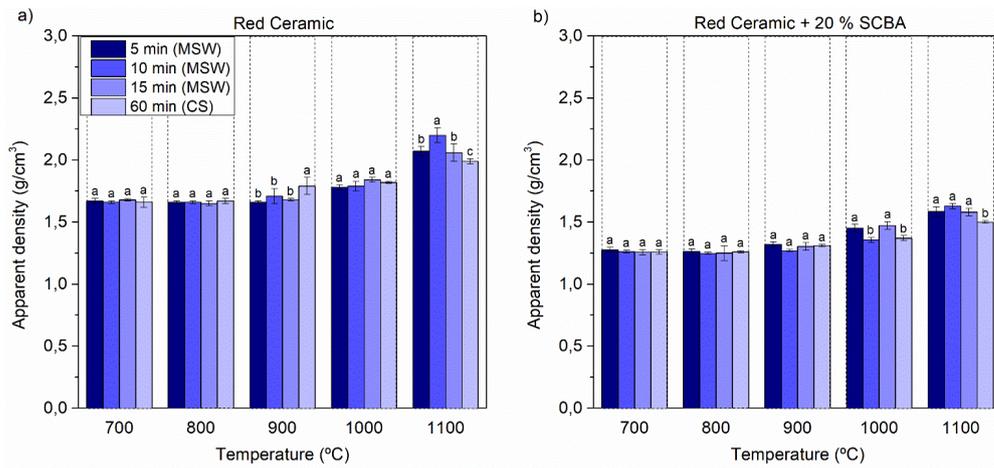


Figure 7. Apparent-density trend curves (g/cm^3) of a) red clay ceramics and, b) SCBA-added red clay ceramics.

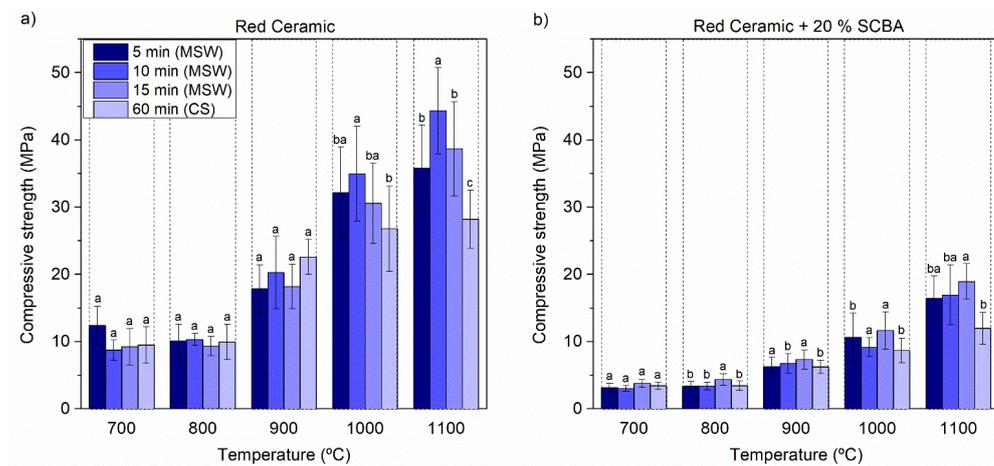


Figure 8. Compressive-strength trend curves (MPa) of a) red clay ceramics and, b) SCBA-added red clay ceramics.

specimens with added SCBA sintered at 1100 °C in a microwave oven did not differ significantly from those of the red ceramic without additions, thus microwave oven sintering and an increase in sintering temperature compensated for the addition of SCBA.

According to the characteristic peaks of diffraction, it is possible to note that there was no difference in their composition, regardless of the addition of SCBA and the type of sintering, this occurs because the composition of SCBA is very close to the clay, thus not changing the composition of the ceramic material. All compositions for both types of sintering presented as crystalline phase Bernalite ($\text{Fe}(\text{OH})_3$), hedenbergite ($\text{FeCa}(\text{Si}_2\text{O}_6)$), hematite (Fe_2O_3), muscovite ($\text{KA}l_2(\text{AlSi}_3\text{O}_{10})(\text{F},\text{OH})_2$), quartz (SiO_2) and rutile (TiO_2). With predominance of the quartz peaks, since it predominates in the raw materials used.

In Figure 10a and 10c, it is possible to observe the difference in pore distribution for the red ceramics sintered by the two methods. In the specimens sintered in the microwave oven the pores are isolated, there was a greater densification of the microstructure, which contributed to the reduction of water absorption and apparent porosity and increased specific mass and compressive strength, according to the results already presented.

In both sintering methods studied for the red ceramics with the addition of SCBA, an increase in the number of interconnected pores is observed, which is confirmed with the increase in the values of water absorption and apparent porosity, and the decrease in specific mass and compressive strength, which was also observed in the microtomography's obtained.

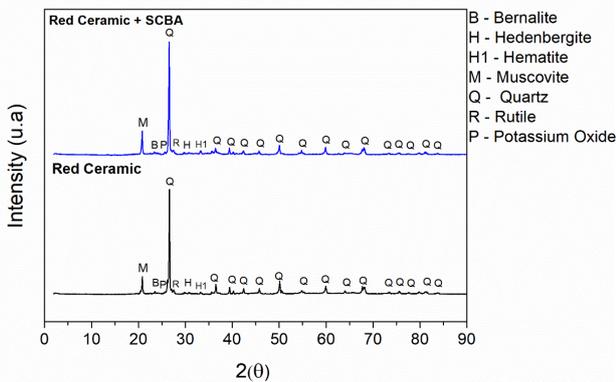


Figure 9. X-ray diffractogram of specimens sintered at 1100 °C (XRDs of different sintering durations, temperatures, and techniques followed the same pattern).

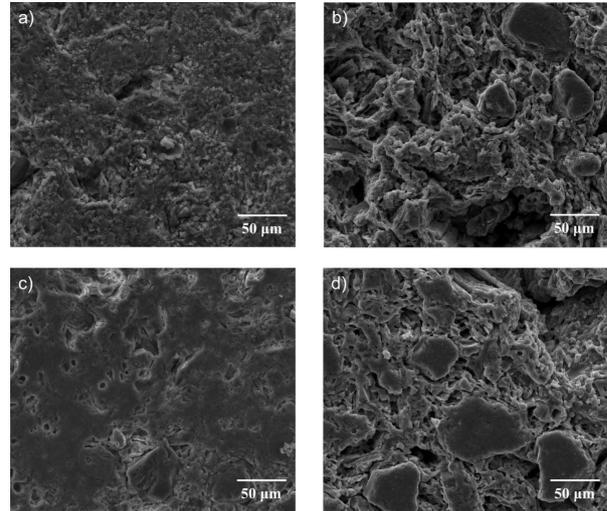


Figure 10. Micrographs of specimens sintered at 1100 °C in conventional a) red ceramic and, b) SCBA-added red ceramics; in a microwave oven c) red ceramic and, d) SCBA-added red ceramics.

4. Conclusions

The reuse of the SCBA in red ceramics, contributes to give a destination to this waste, besides reducing the consumption of natural raw material, extracted from deposits.

The sugarcane ash for having a chemical composition close to the clay does not cause major changes in the final composition. However, the high content of organic matter contributes to the increase of water absorption and decrease of mechanical resistance and apparent specific mass.

The decrease in the specific mass due to the existence of pores, provides the production of lighter ceramic materials, which facilitates their transport and contributes to a lower cost in structures. Besides improving thermal and acoustic insulation due to the presence of air inside. This can be useful in the production of blocks and ceramic tiles.

The decrease of the mechanical resistance of the materials with addition of SCBA is partially compensated by the sintering in microwave oven and the increase of the temperature. This fact can be observed in the values found for specimens with added SCBA, sintered in a microwave oven at 1100 °C, very close to those found for red ceramics sintered at 900 °C in a conventional oven.

Thus the use of SCBA in ceramic mass and sintering in microwave oven, presents positive results, when talking about sustainability and environmental protection, providing reuse of waste, savings in consumption of raw materials and energy.

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