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Characterization of materials used in the manufacture of ceramic tile with incorporation of ornamental rock waste

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Abstract. The production of ceramic tiles, such as tiles, has a great environmental impact, either in the extraction of natural raw materials or gas emissions in the burning stages. The use of industrial solid waste in ceramic materials can contribute to the reduction of these impacts, according to the characteristics of solid waste and its interaction with ceramic materials in the processing steps. Thus, this study aimed to characterize the materials needed to make a ceramic tile with incorporation of ornamental rock waste (ORW), thus evaluating its main characteristics regarding the feasibility of this incorporation. The physical characterization of the clays used in the production of ceramic artifacts was performed, and for the waste the mineralogical analyzes were performed, through x-ray diffraction (XRD), microstructure analysis from confocal optical microscopy, after sintering the prototypes and chemical analysis by X-ray spectroscopy (EDX). Soon after the raw materials went through the step of conformation and preparation of the prismatic specimens by the process of extrusion of the ceramic mass, with an incorporation of the ORW in 0% and 15% of the ceramic mass, for its subsequent The prototypes were sintered at three different temperatures (850 °C, 950 °C and 1,050 °C). The specimens were submitted to technological tests of mechanical resistance, water absorption, firing shrinkage and porosity to evaluate the incorporation viability. The results indicated the presence of quartz particles in all raw materials, and also that the clays of the study region are predominantly kaolinitic. The presence of these materials in the ceramic masses directly influences the micrographs, because they result in the formation of liquid phase, inert particles that can turn the site into a stress concentration point and when incorporated in the ORW the specimens met the technical specifications of the Brazilian standard for application on ceramic tiles. The results found in the technological tests carried out, that the incorporation of 15% of ornamental rock waste in both clays did not affect the tile properties, indicating the feasibility of incorporating this waste in civil construction, minimizing the impacts generated.

Key words: waste, reuse, rural constructions, sustainability.

INTRODUCTION

One of the major problems of industrial activity worldwide is the generation of solid industrial waste, which has been growing at significant rates in several countries. The problem of the generation of industrial solid waste is related to environmental issues, linked to the disposal of these materials in the environment, such as in landfills and the pollution of soil and groundwater, due to incorrect packaging. Another eminent factor is the economic issue, since the disposal and proper transportation of this waste involves great costs for the producing companies (Azevedo et al., 2018).

Thus, the potential of using industrial solid waste in various ways has been discussed on several fronts, the most promising being its incorporation in civil construction materials, such as cement and ceramics, which are widely used worldwide (Marvila et al., 2019; Areias et al., 2020). The incorporation of waste in construction materials, in addition to providing a more noble destination for the material, than its simple disposal in landfills, can lead to an improvement in technological properties that enhance its use and increase its added value, in addition to allowing in some reduction in consumption of other raw materials (Azevedo et al., 2020a).

The ceramic industry is known for its high potential for damage to the environment, due to the use of a large amount of natural raw material, such as natural clay, which is extracted from deposits and causes impacts before and after extraction, due to the high energy consumption in the stage of burning the parts, which leads to increased financial costs and in cases where it is poorly executed in defects that may make the use of parts by civil construction unfeasible and the direct impacts such as the generation of solid waste, called the chamotte, increased emission polluting gases in the atmosphere and others (Coutinho & Vieira, 2016). All of these factors contribute to this type of industrial activity being the result of scientific research to improve its products and the production process, in order to reduce environmental impacts (Mendoza-Cuenca et al., 2015).

The minimization of the use of natural resources for the manufacture of ceramic products has been made from the incoporation of wastes from other productive chains in the manufacture of ceramic products, as in the study of Vieira et al. (2016), in which ornamental stone wastes were incorporated in ceramics in order to obtain contribution in the properties of the materials, as well as improvements in the final technological properties.

The incorporation of rock waste in clays allows the adjustment of plasticity in the ceramic mass, due to the coarse granulometry of the residue and an increase in dry density, in addition, in the study of Amaral et al. (2019), the increase in the waste content resulted in less linear shrinkage, decreased water absorption, which improves some properties of the ceramic mass.

The chemical and mineralogical composition of the clays depends on the formation deposits where they are located, and these compositions and the amounts of quartz, calcite, dolomite, feldspar and other organic compounds, can vary according to the level of these deposits. The composition can directly interfere in the properties of the materials to be manufactured, also influencing its final production cost (Tretjakova et al., 2018).

Brazil produces a significant amount of ornamental rocks, such as marble and granite, which together generate large amounts of solid waste in the stages of processing the blocks after their extraction from nature, this waste, called ornamental rock waste, is in general transported to landfills, which raises its production costs (Areias et al., 2020).

The state of Espírito Santo - ES is one of the largest producers of ornamental stones in Brazil and close to it is the municipality of Campos dos Goytacazes - RJ, which has one of the largest production centers for ceramic artefacts in the country (Azevedo et al., 2019). Thus, there is a potential for the use of ornamental rock waste in red ceramic pieces, such as blocks and floors, however its application in tiles is still little explored in the scientific literature, limited to small percentages and limited firing temperatures (Marvila et al., 2018; Azevedo et al., 2020b).Thus, this study aimed to characterize the materials needed to make a ceramic tile with incorporation of ornamental rock waste (ORW), thus evaluating its main characteristics regarding the feasibility of this incorporation.

MATERIALS AND METHODS

The clays used in the present work come from the municipality of Campos dos Goytacazes, State of Rio de Janeiro, and were extracted from a local ceramic industry, called Arte Cerâmica Sardinha. The ornamental stone waste was collected in the municipality of Cachoeiro do Itapemirim, in Espírito Santo, from the company Decolores Marmores e Granitos do Brasil, in the form of mud, with natural humidity (in the range of 12 to 26%).

The industrial mass was made using clays locally called 'strong' (AF) clay, that is, a more plastic clay and a mixed clay (AM), containing 'strong' and 'weak' clay. Both the mass, the clays and the rock waste were previously subjected to drying.

For the preparation of the ceramic mass, 15% of ornamental rock waste was added for the weight of each clay. The amount of 15% is already known in other studies in the literature that evaluated the use of this waste for the production of other ceramic artefacts, such as tiles and blocks, which is different from this work, but we opted to start from this percentage called 'excellent' and to evaluate the influence of sintering temperatures for economic reasons (the cost of the sintering step is high) and environmental (maximal waste and reduction of gas emissions in the atmosphere with sintering).

The chemical characterizations of the clay and the waste were determined by X-ray fluorescence spectroscopy, with the SHIMADZU EDX-700 equipment, performed at the Civil Engineering Laboratory - LECIV at UENF.

X-ray diffraction determines the atomic and molecular structure of a crystal, it was performed using Rigaki MiniFlex 60 equipment at the Civil Engineering Laboratory - LECIV at UENF.

The prototypes were made using an extruder of the Verdés brand, model BR-051. After extrusion, the prototypes went through a natural and artificial drying process. The prototypes were sintered at three different temperatures (850 °C, 950 °C and 1,050 °C). The heating rate was 2 °C min^{-1} with 120 minutes of stay at the threshold temperature. The cooling carried out natural convection until room temperature, when the oven was turned off. These sintering temperatures (850 °C, 950 °C and 1,050 °C) are the ones commonly used in the ceramic industry for this type of piece, very low temperatures compromise the transformation of mineral phases that exist in the clays and negatively influence the final properties of the tiles, since too high temperatures incur high costs and possibility of increased defects in parts, such as high linear shrinkage, so the evaluation of temperature ranges becomes important, and the choice of these in a specific way was given by the literature.

Water absorption was analyzed. The water absorption test was carried out according to ASTM C373 (1972), in which the specimens were dried in an oven at ± 110 °C for 24 hours, and their masses were measured. Afterwards, they were placed in a container containing water for 24 hours and the excess surface water was removed for the new mass weighing.

The analysis of the linear shrinkage of the prototypes was carried out after sintering, being carried out according to ABNT-MB-305, using a 0.01 mm resolution pachymeter.

The three point flexural strength of the prototypes was analyzed. The flexural strength was determined according to ASTM C674 (1977) with the aid of an EMIC hydraulic press, model CL 3000. The load application speed was 0.1mm min^{-1} and the distance between the cleavers was of 8.0 cm.

The confocal micrograph was performed in order to obtain enlarged images of the specimens and to distinguish details of the surface, using an Olymlpus microscope and LAMAV / UENF CGA model.

RESULTS AND DISCUSSION

The chemical compositions of the raw materials used were determined (Table 1).

Table 1. Chemical composition of the raw materials used to make the prototypes.

Raw material	SiO_2	Al_2O_3	Fe_2O_3	K_2O	TiO_2	SO_3	CaO
AF	60.35	31.33	2.88	2.20	1.17	1.35	0.57
AM	56.46	34.01	3.31	2.26	1.42	1.78	0.66
ORW	86.22	7.15	0.26	2.15	0.10	1.59	2.34

It was possible to observe that both clays are predominantly made up of SiO_2 and Al_2O_3 (91.68% for AF and 90.47% for AM), and according to Coutinho & Vieira (2016) these form the clay silicates like mica and kaolinite, also indicate high percentage of clay minerals. Also according to these authors, the high percentage of Fe_2O_3, between 1 and 5%, gives the beige-rosacea color after the burning of the clays.

However, the ornamental rock waste showed a high percentage of alkaline oxides ($K_2O + Na_2O$), and according to Vieira et al. (2004) this indicates that these oxides form eutectic with silica at sintering temperatures above 700 °C.

With the x-ray diffractograms of the clays (Figs 1 and 2) it was possible to observe that both have higher diffraction peaks corresponding to kaolinite, and according to Babisk et al. (2019), this mineral clay is responsible for the plasticity of the clays. In addition, the presence of kaolinite proves that the clays are typically from the Campos region (Areias et al., 2017; Vieira et al., 2000).

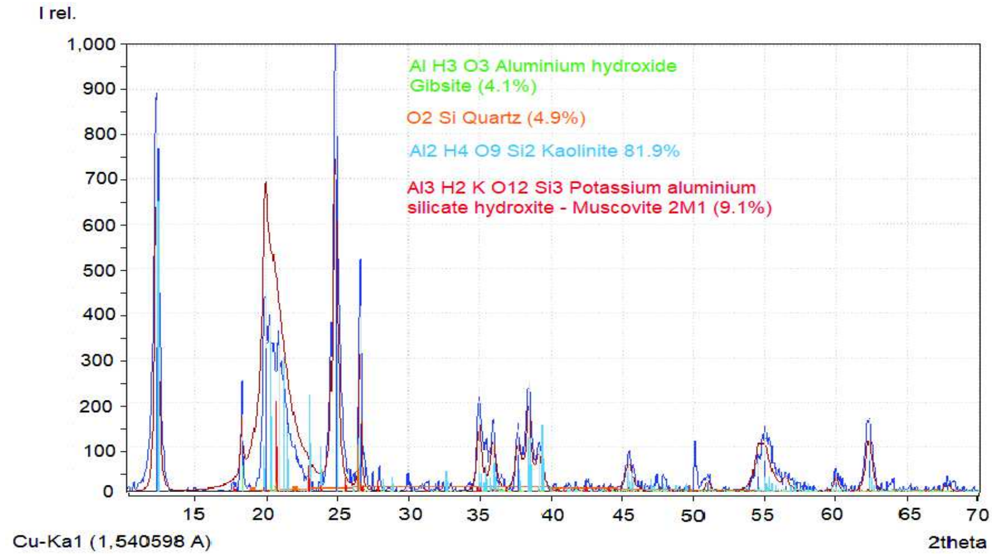

Figure 1. X-ray diffractogram - mixed clay (AM).

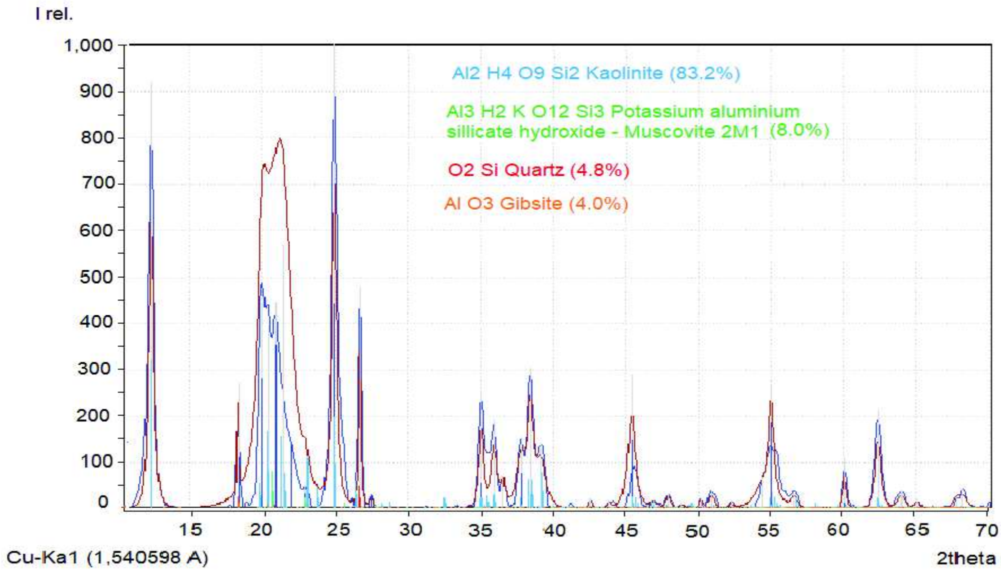

Figure 2. X-ray diffractogram - strong clay (AF).

The analysis performed by DRX for ORW (Fig. 3) indicates that the waste is rich in Ca due to the presence of this substrate in the form of dolomite, according to Neves et al. (2019), due to the sawing of blocks and also by the presence of marble waste in the composition of the ORW. The presence of quartz in the ORW indicates, according to Coutinho & Vieira (2016) impurities that act as non-plastic raw material inert substances during the sintering process.

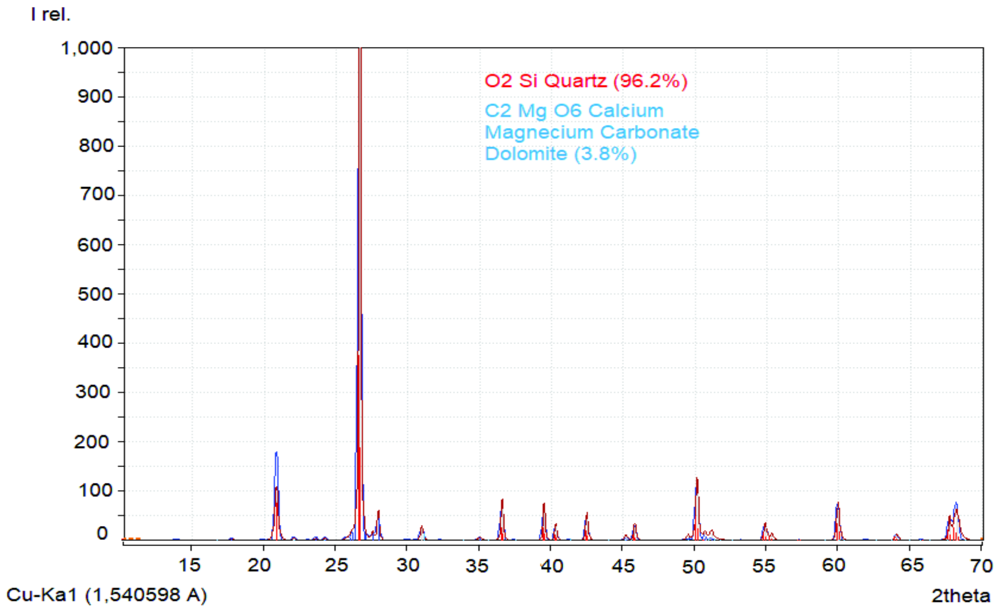

Figure 3. X-ray diffractograms - ornamental rock waste (ORW).

The average water absorption values after the sintering process are shown in Table 2. The sintered prototypes 1,050 °C showed lower water absorption values in all ceramic mass compositions. According to Vieira et al. (2016), this decrease is associated with the presence of fluxing oxides that contribute to the formation of a liquid phase and closing porosity. This change in behavior between temperatures of 850 to 1,050 °C is associated with the refractory behavior of a kaolinitic clay body that has low amounts of fluxing oxides and a high amount of alumina in its chemical composition (Vieira et al., 2016).

The reduction in the absorption value at a temperature of 1,050 °C, is associated with the mechanisms of sintering, diffusion in the solid state and formation of the liquid phase that act effectively in the clays of Campos dos Goytacazes, so the temperatures of 850 and 950 °C were also selected, due to the fact that they are not among some critical temperature ranges (Azevedo et al., 2018; Amaral et al., 2019).

Table 2. Average water absorption values observed in each ceramic mass composition

Temperature	Water absorption (%)			
(°C)	AM0	AM15	AF0	AF15
850	15.4	15.8	20.3	18.1
950	20.4	19.4	18.4	14.9
1,050	11.3	11.4	14.3	13.3

AM0: Ceramic clay made with mixed clay and 0% addition of ornamental rock waste; AM15: Ceramic clay made with mixed clay and 15% addition of ornamental rock waste; AF0: Ceramic clay made with strong clay and 0% addition of ornamental rock waste; AF15: Ceramic clay made with strong clay and 15% addition of ornamental rock waste.

According to the ABNT 15270-2 standard (ABNT, 2017) the maximum water absorption index indicated for ceramic blocks is 22%, which indicates that in this study all the ceramic masses tested fall within the standard's determination. The NBR 13582 (ABNT, 2005) for Roman ceramic tiles mentions that the ideal and maximum value forwater absorption must not exceed 18%, which was not met by all ceramic masses, at all temperatures. However, the ceramic masses sintered at 1,050 °C showed values that meet the standard for ceramic tiles. At 950 °C, only the AF15 mass met the requirements of the standard for tiles (Fig. 4).

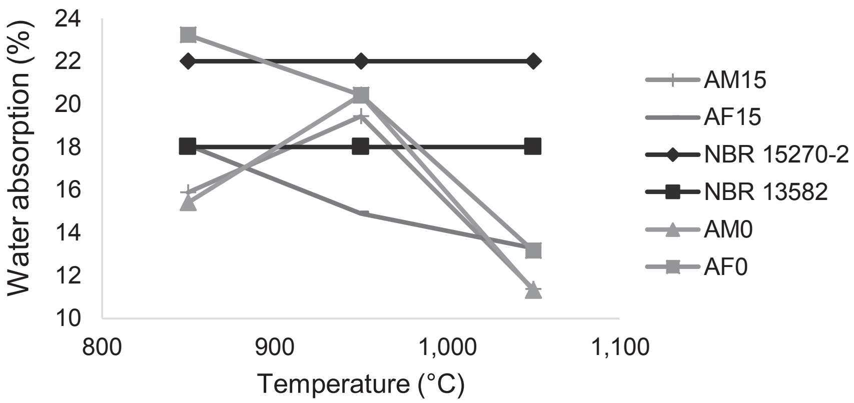

Figure 4. Average water absorption values for ceramic masses compared to standards.
AM0: Ceramic clay made with mixed clay and 0% addition of ornamental rock waste; AM15: Ceramic clay made with mixed clay and 15% addition of ornamental rock waste; AF0: Ceramic clay made with strong clay and 0% addition of ornamental rock waste; AF15: Ceramic clay made with strong clay and 15% addition of ornamental rock waste; NBR 15270-2: Standard NBR 15270-2; NBR: 13582: Standard NBR: 13582.

The values regarding the linear shrinkage observed for each ceramic mass composition, at each sintering temperature can be seen in Table 3.

The increase in linear shrinkage is associated with the formation of a liquid phase, as well as the recrystallization of the ceramic phases at high temperatures, greater densification which is caused by physical changes and a reduction in the volume of the specimens, moreover, this increase may also be related the higher content of fluxing oxides which justifies the higher values in the ceramic mass with incorporation of 15% ORW (Vieira et al., 2015; Gaspareto & Teixeira, 2017; Silva et al., 2018).

Table 3. Average values of linear shrinkage observed at home ceramic mass composition

Temperature (°C)	Linear shrinkage			
	AM0	AM15	AF0	AF15
850	1.39	1.02	1.42	1.54
950	2.11	2.01	2.36	2.52
1,050	3.50	3.17	3.99	4.17

AM0: Ceramic clay made with mixed clay and 0% addition of ornamental rock waste; AM15: Ceramic clay made with mixed clay and 15% addition of ornamental rock waste; AF0: Ceramic clay made with strong clay and 0% addition of ornamental rock waste; AF15: Ceramic clay made with strong clay and 15% addition of ornamental rock waste

Table 4 shows the flexural strength values at three points for each of the ceramic masses.

The mean of the rupture module increased as the sintering temperature increased, with the AM15 mass sintered at 1050 showing the highest mean value of resistance to rupture. According to Brito et al. (2015), this occurs due to the presence of the lowest silica content between clays and the highest ratio Al_2O_3/SiO_2, these are important characteristics for the compaction and densification of the masses, which favor the physical-mechanical properties.

In general, most of the evaluated ceramic masses presented values greater than or equal to 6.5 Mpa, established by the standard ABNT NBR 15310 (ABNT, 2005) for ceramic tiles. The prototypes sintered at 1050 °C showed higher values compared to the other temperatures for all tested compositions. The decrease in flexural strength may be related to the presence of quartz in the structure of the ceramic masses, which acts as fracture initiation sites.

Table 4. Average flexural strength values found in each ceramic mass composition

Temperature (°C)	flexural strength (Mpa)			
	AM0	AM15	AF0	AF15
850	5.21	7.14	6.73	9.35
950	5.19	7.16	6.04	5.87
1,050	8.35	12.5	8.58	9.40

According Viera & Pinheiro (2013) the microstructures of the ceramic masses showed different colors due to the dehydration of the iron hydroxide and hematite that are present in the clays, that can be seen in the present study, whereas the black particles indicate the presence of the iron compounds (Figs 5, 6, 7 and 8 (arrows 1)), the red ones are those of hematite and the white circle indicates muscovite mica (Figs 5 and 7 (arrows 2)), which it is present in the composition of the rock waste.

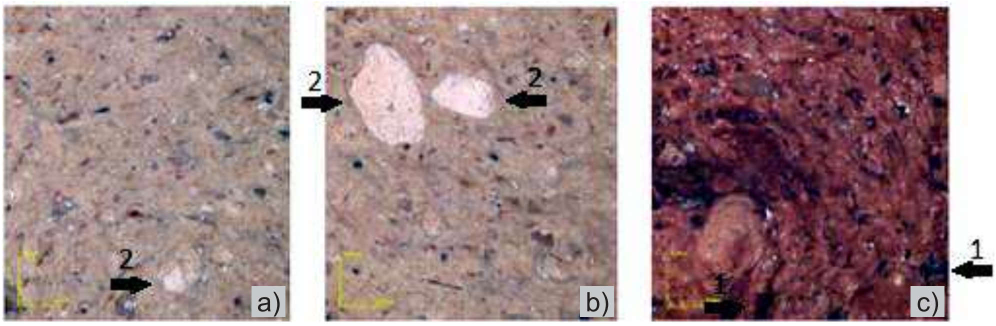

Figure 5. Confocal optical microscopy of ceramic masses with 0% incorporation of ORW in mixed clay (AM0) sintered at 850 °C (a), 950 °C (b) e 1,050 °C(c).

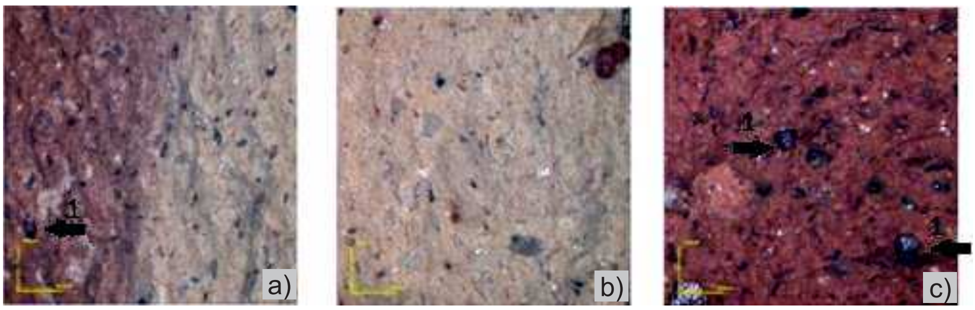

Figure 6. Confocal optical microscopy of ceramic masses with 15% incorporation of ORW in mixed clay (AM15) sintered at 850 °C (a), 950 °C (b) e 1,050 °C(c).

The micrographs of the ceramic masses with 15% ORW burned at 1,050 °C when enlarged in 108x have free quartz grains and some cracks around the quartz grain. It is also possible to observe in Fig. 7, c, according to the analysis of the Campos dos Goytacazes clays, that the presence of quartz grains separated from the matrix occurs only in the porous microstructure at temperatures above 1,050 °C due to the composition of the raw materials, in agreement with Vieira et al. (2000).

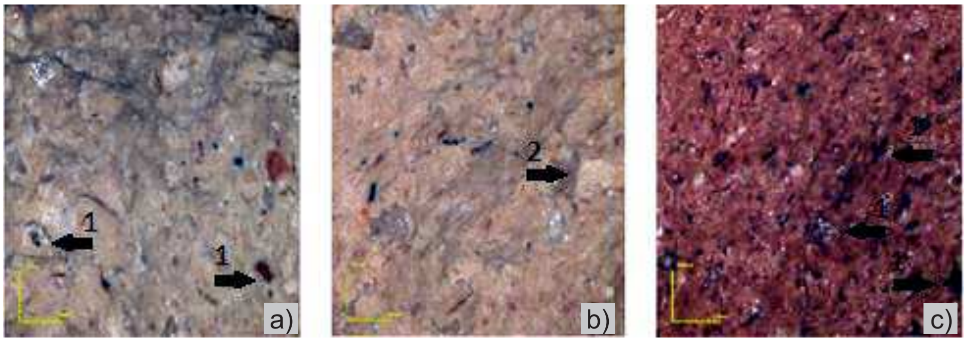

Figure 7. Confocal optical microscopy of the ceramic masses with 0% ORW incorporation in strong clay (AF0) sintered at 850 °C (a), 950 °C (b) e 1,050 °C(c).

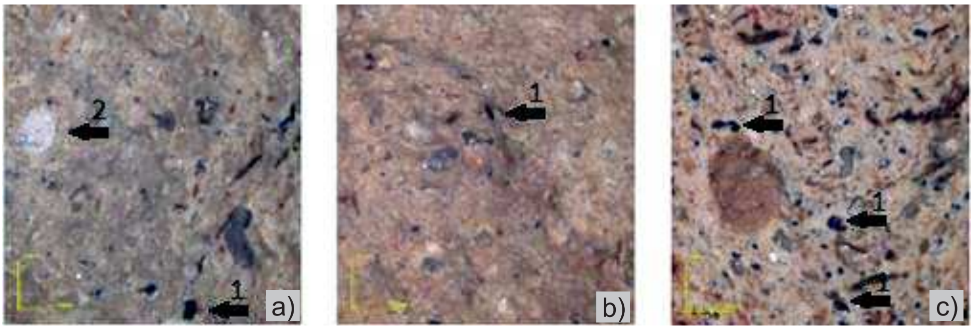

Figure 8. Confocal optical microscopy of the ceramic masses with 15% ORW incorporation in strong clay (AF15) sintered at 850 °C (a), 950 °C (b) e 1,050 °C(c).

CONCLUSIONS

The ceramic mass composed of the clays and the waste present after the sintering properties expected from the raw materials due to the chemical composition and the x-ray analyzes, which indicated the reddish color, the presence of free quartz inert to sintering and the increased formation liquid phase which contributes to the densification of prototypes.

The sintering temperature that resulted in the best results was 1,050 °C.

It is possible to conclude from the results found in the technological tests carried out, that the incorporation of 15% of ornamental rock waste in both clays did not affect the tile properties, indicating the feasibility of incorporating this waste in civil construction, minimizing the impacts generated.

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