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Evaluation of the potential use of granite waste in products of the red ceramic industry in the state of Amazonas

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ABSTRACT

The increase in the exploitation of raw materials in the civil construction sector has led to an increase in damage to the environment, mainly due to the improper disposal of granitic waste. However, this fact has caused damage to the environment. Therefore, this research aims to analyze the influence of the introduction of granite waste in a clay matrix aiming its use in the red ceramic industry. For this, the clay from the ceramic pole in the city of Iranduba - AM and granite waste from a company located in the municipality of Presidente Figueiredo - AM were collected. Formulations were made containing three levels of waste (0, 10 and 20% mass) and three firing temperatures (1173, 1273 and 1373K). The materials were characterized by X-ray fluorescence spectroscopy, X-ray diffraction, scanning electron microscopy and the following technological properties were determined: water absorption; linear retraction; apparent porosity; specific mass; and 3-point flexural rupture module. The results showed that the glassy phases generated provide a better densification of the formulations with 20% of waste, sintered at 1373K, as they presented favorable mechanical properties for the manufacture of red ceramic products, providing a correct destination of the waste and providing alternatives for the social, economic context and environmental impact of the Amazon region.

Keywords: red ceramic, waste, granite, technological properties.

1. INTRODUCTION

The red ceramic industry is responsible for the manufacture of the materials most used in civil construction such as: tiles, expanded clay, bricks, among others. According to the Brazilian Institute of Geography and Statistics (IBGE), the civil construction sector ended 2019 with a 1.7% increase in the national gross domestic product (GDP) compared to the previous year and is constantly growing [1]. Despite the great importance of this sector for the country's economic development, there is an increasing need for the main raw material of the red ceramic industry, clay, in which its exploration is simultaneously intensified and, consequently, increasing environmental impacts [2]. In this way, researchers sought solutions that would reduce damage to the environment, such as the incorporation of industrial waste in the clay matrix [3, 4, 42, 43]

The red ceramic industry represents 4.8% of the civil construction industry in Brazil, comprising 6903 companies that generate an equivalent to R$ 18 billion in annual sales [5]. According to data from the Ministry of Mines and Energy, the main products developed in the North are bricks and blocks [6]. The state of Amazonas has approximately 100 industries in its red ceramic production pole, with a large part of these companies located in the municipalities of Iranduba and Manacapuru, which are close to their main consumer center, the metropolitan region of Manaus [7]. It is important to emphasize that in the last decades there has been a rapid and progressive growth in the population of the capital of the state of Amazonas, this has resulted in a greater demand in the civil construction sector [8, 9]. Consequently, the demand and need for raw

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materials in the regional ceramic sector increases.

Another raw material widely used in civil construction is granite, commonly used as ornamental stones and gravel [2]. According to data from ABIROCHAS, Brazil is the 4th largest world producer of marble and granite, in which it represents 7% of the world production of the rock sector in 2018, behind the United States, China and Italy. The main Brazilian exporter is the state of Espírito Santo, which accounts for 79.37% of national production [9, 47, 48]. The State of Amazonas presents geological areas favorable to the presence of granitic rocks, found mainly in the municipalities of Presidente Figueiredo, Barcelos and São Gabriel da Cachoeira. The city of Presidente Figueiredo stands out as an important pole of products for civil construction in Manaus, with its main product being gravel [10]. The municipality has a granite reserve of about 200 km^2, which is explored by small companies, which together generate a considerable amount of solid waste from the extraction process of granitic rocks [11, 12].

However, it appears that in all stages of the granite beneficiation process, a sediment with a high degree of fineness is generated, commonly found as granite dust or mud [2, 13, 14]. According to the NBR - 10004 standard, granite waste is an inorganic, non-biodegradable solid, classified as class IIA waste - non-inert and recyclable [15]. It is important to emphasize that in the stage of cutting granite blocks, about 20% to 25% become tailings [16]. Consequently, alternatives aimed at the technological enhancement of the introduction of this industrial residue in various products of the ceramic industry are of great interest to the scientific and industrial community [13, 16-21, 44, 45].

Research shows that these residues generate direct negative impacts on the environment, since most industries do not have proper disposal systems, nor an adequate disposal of the tailings, thus, they can reach and contaminate, for example, soil, subsoil, groundwater, rivers, streams, and the like [2, 3, 14-16, 21, 22, 47]. It is also noteworthy that the dry residue (fine powder) inspired by mammals, is deposited in their lungs, which may have harmful consequences for their health [15, 22, 46].

In view of this, the importance of adding value to unwanted waste is emphasized, seeking scientific, political and environmental measures in work processes, in which they aim to comply with the legislation that deals with the final sanitary destination of solids [46] in order to reduce the amount of waste discarded in nature, mainly avoiding the generation of effluents. It is noteworthy that the territory of Presidente Figueiredo has a hydrography formed by many waterfalls, rapids, streams, lakes and an extensive vegetation cover [23]. Consequently, when these residues are disposed of incorrectly, they possibly reach these environments, where in the form of mud it becomes abrasive, harming the fauna, flora and residents of the region.

Studies demonstrate great potential and improvements in traditional ceramic products with the incorporation of granite waste [2, 3, 25-35]. The main reason for this reuse to be possible is the compatibility and mineralogical heterogeneity between the clays used in the traditional ceramic industry and the by-product of the granite exploration activity [27, 29, 31, 35]. In addition, fluxing compounds, commonly found in granite residue, contribute to the development of a greater amount of vitreous phase during the sintering procedure, filling the pores and increasing the durability of the red ceramic pieces providing an improvement in the quality of the product [3, 35, 15].

The literature presents research with quantities of granite residues from 5% to 60% (by weight) introduced in red ceramic [3, 14, 22, 24, 26, 27, 33-35]. Since ceramic products for civil construction were the products most investigated in the studies [14, 22, 25-27, 30, 32, 34]. In most cases, the feasibility of introducing the residue was observed based on results of chemical analysis, thermal analysis, X-ray diffraction, water absorption, and mechanical resistance, where sintering temperatures in the range of 773K were used. at 1423K [3, 14, 24, 27, 32-35].

Within the context addressed, the objective of this research is to analyze the influence of the introduction of granite waste in the microstructure of a clay matrix, evaluating, based on technological properties, its use in the red ceramic industry. To achieve the results, phase transformations, changes in the morphological aspect of the materials' microstructure were investigated, as well as the following technological properties were determined: water absorption (WA); linear firing retraction (LFR); apparent porosity (AP); apparent specific mass (ASM); and 3-point flexural rupture module (FRM), in which the technical feasibility of this destination was assessed. Finally, this research aims to provide solutions to two problems pertinent to the current social, economic and environmental context of the Amazon region: the first is the technically correct destination for the waste generated in the exploitation of granite in the municipality of Presidente Figueiredo (AM), avoiding, thus, the pollution of the environment; and the second is the possible contribution to the development of a new material, providing benefits to the local economy and job creation.

2. MATERIAL AND METHODS

The ceramic mass used was clay from the ceramic pole in the city of Iranduba - AM and the granite waste (GW) collected at the company Pedreiras Canoas, located in the municipality of Presidente Figueiredo - AM.

2.1 Characterization of the raw materials

Initially to determine the chemical and mineralogical analysis, the raw materials were sieved in the 200 mesh (ABNT nº 200 - 0.074mm opening). The chemical composition of clay and GW, in the form of oxides, was determined using the X-ray fluorescence (XRF) technique, using PANanlytical equipment, model Epsilon3-XL. To identify the mineralogical composition of the clay and the GW, the X-ray diffraction technique (XRD) was used in a Shimadzu equipment, model XRD-6000. The analysis conditions were as follows: scanning field from 5-80° to 2θ, with a step of 0.02° and scanning speed 2°/min. To identify the peaks, the software X'Pert High Score, version 2.0 was used with letters compiled by the Joint Commitee on Powder Diffraction Standards (JCPDS) and registered at the International Center for Diffraction Data (ICDD). For the quantification of the present phases, the refinement of the diffraction profile was adopted by the Rietveld method [36] and the standard adjustment by the Materials Analysis Using Diffraction (MAUD) software.

2.2 Sample preparation

Table 1 demonstrates the experimental design used to produce the compositions [41]. The formulations were obtained through two-by-two factorial design (2^2) with 7 tests being three central points with the two coded parameters, the temperature and the percentage of granite powder residue. The content of granite powder in the mass was 0%, 10% and 20% in mass and the firing temperatures: 1173K; 1273K and 1373K. The samples were initially passed through a 40 mesh sieve (ABNT nº 40 - opening of 0.42 mm) and dried at 373 K for 24 hours in an oven (MARCONI model MA033). Then, the experimental formulations were pre-wetted with 10% water. For the preparation of the samples, 13g of material were used, which were obtained by single-action uniaxial pressing using a hydraulic press (MARCON) and a rigid metallic matrix with a rectangular cavity measuring 6cm x 2cm. The compaction pressure to which the samples were submitted was 25 MPa. Five samples were taken for each formulation.

Then the samples were properly identified and dried in an electric oven at a temperature of 373K for 24 hours. Subsequently, they were weighed on a digital scale (Mars, AS1000C) and measured with a Western digital caliper. Then the samples were sintered in a muffle furnace (JUNG, model 2013) and weighed again, measured and subjected to the following tests of technological properties: LFR; WA; AP; ASM; and FRM, in which a Nannetti fleximeter (Model CC/96 - 2006) was used with a load of 1000 kgf, with a speed of 0.5 mm/min, with a fixed distance between the axes of the base of 4 cm.

Table 1: Composition of sample formulations.

ESSAY	E1	E2	E3	E4	E5	E6	E7
Waste concentration (% mass)	0	20	0	20	10	10	10
Sintering tempertature (K)	1173	1173	1373	1373	1273	1273	1273

2.3 Analysis of microstructural transformations

To obtain the microstructures, a Scanning Electron Microscope (SEM) (TM 3000 from the Hitachi brand) was used, with images obtained using backscattered electrons (BSE). Subsequently, the samples were deagglomerated in a porcelain mortar and sieved in the 200 mesh to identify the new phases of the sintered samples. For this purpose, X-ray diffraction was performed on the same equipment mentioned above, with a 10-60 ° scanning field. for 2θ with a step of 0.02° and scanning speed 2°/min. To identify the peaks and quantify the phases, the same procedures described above were used.

3. RESULTS AND DISCUSSION

3.1 Chemical analysis

Table 2 shows the chemical composition of GW and clay. According to the results obtained, it is possible to verify that silicon and aluminum are the predominant elements in the studied samples. High amounts of SiO_2

are common features of igneous granitic rocks [37]. So, this fact may be associated with the presence of free silica from quartz. Therefore, indicating the possible presence of primary minerals (quartz, feldspar and mica group minerals) [24]. the result found is in agreement with the results found in previous researches [42, 43, 49]. The clay showed high compositions of SiO_2 and Al_2O_3. Thus, they are characterized by the possible presence of clay minerals, usually kaolinite. There are considerable levels of fluxing elements (K_2O, CaO and MgO) in the GW samples. The GW presented considerable K_2O contents (6.97%), an important characteristic, as this element is related to the phases that improve the vitrification of the materials [37], providing a liquid phase during the firing period and which may result in the reduction of pores in the sample. It is observed that the significant amounts of Fe_2O_3 and CaO in the GW. This is possibly related to the addition of lime and shot as abrasives and lubricants in the beneficiation process [14, 24]. The clay mass presented low content of melting agent (<2%) and high content of Fe_2O_3 (6.93%), a result that characterizes the refractoriness and reddish color of the post-burning material [37]. Finally, it is possible to observe considerable levels of titanium oxide, which indicate the possible sedimentary origin [37].

Table 2: Chemical composition of raw materials with elements in oxides (%).

RAW MATERIALS	SiO_2	Al_2O_3	K_2O	Fe_2O_3	CaO	MgO	P_2O_5	TiO_2	ZrO_2	MnO
GW	75,12	13,71	6,97	1,71	1,15	0,41	0,42	0,21	0,02	0,06
Clay	55,65	32,87	1,18	6,93	0,20	0,33	0,49	2,02	0,12	0,01

3.2 Mineralogical identification

Fig. 1 shows the diffractograms of clay and GW, which presents the crystalline phases of quartz (SiO_2), muscovite $(K,Na)Al_2(SiAl)_4(OH)_2$, albite $Na(AlSiO_3O_8)$, dolomite $CaMg(CO_3)_2$ and Microclinium $K(AlSi_3O_8)$. These phases are characteristic of granite rocks. While the clay consists of peaks characteristic of the mineralogical phases of quartz (SiO_2), hematite (Fe_2O_3), kaolinite $Al_2Si_2O_5(OH)_4$ and microcline $K(AlSi_3O_8)$. The muscovite and albite phases, present in the residue, present in their composition the element sodium (Na), which are not present in the chemical investigation, this can be explained by the limited precision of the XRF technique in detecting the element sodium [38]. These phases, as well as the microcline stage, are desirable as they assist in the formation of a glassy matrix and linear shrinkage during sintering [43, 44]. Therefore, both samples showed a high content of quartz and considerable phases present in the residue that present fusion characteristics in sintering, which may contribute to the optimization of the firing cycle.

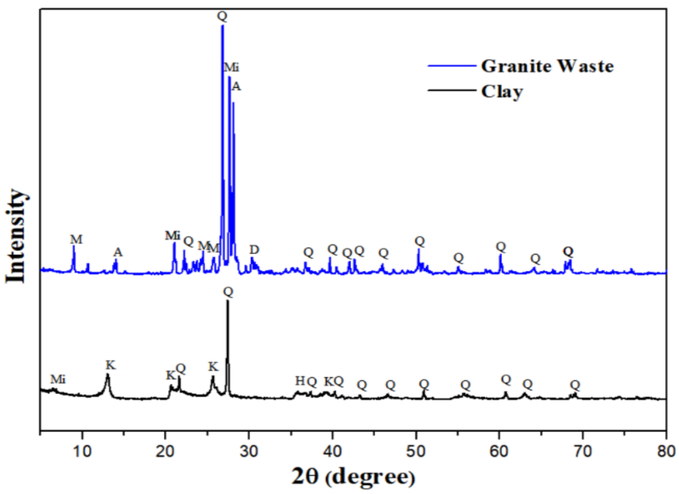

Figure 1: X-ray diffractogram of the granite waste; and diffractogram of the clay: Q - quartz, M - muscovite, Mi - microcline, D - dolomite, A - albite, H - hematite and K - kaolinite.

3.3 Mineralogical post-firing identification

The X-ray diffractograms of the formulations are shown in Fig. 2. It is possible to observe in the diffractogram of the E1 essay (0% granite waste), sintered at 1173K, that the quartz peaks remained in large quantities and that the crystalline kaolinite phases have been converted to the metakaolinite phase ($Al_4Si_4O1_{3.5}OH$). It has also been observed that the crystalline phases of hematite and microcline are transformed into amorphous phases. In E3 (0% granite waste) the sintering temperature was 1373K, in this case, only quartz peaks were identified. The diffractograms of E2 and E4 essays (20% granite waste) were sintered at 1173 K and 1373K, respectively. It was noted that in E2 the quartz phases are still present in large quantities and that the albite and microcline phases, coming from the GW, also appeared in considerable proportions. Likewise, the transformation of kaolinite into metakaolinite also occurred. In E4, only the quartz phases and a small proportion of microcline were identified. The essays E5, E6 and E7 (10% granite waste), were made to obtain the average results of the formulations and were sintered at 1273K. In these three essays, the presence of quartz, albite and microcline was observed.

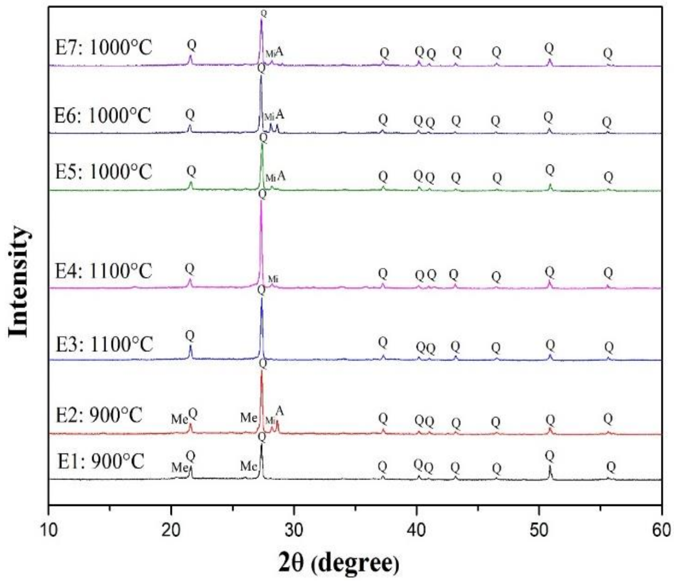

Figure 2: X-ray diffractogram of the Sintered Essays: Q - quartz, Me - metakaolinite, Mi - microcline and A - albite.

It appears that as the sintering temperature increased, there was a decrease in the crystalline phases in the formulations. The formation of the glassy phases formed during firing is justified by the presence of fluxing oxides present in granite and clay. Despite the accentuated presence of quartz in the raw materials, which possibly prevents the development of plasticity, standard clay tends to benefit from melting elements with the addition of GW. Therefore, the crystalline phases Muscovite, dolomite, and hematite probably became the glass phase in the new material and can possibly provide good compaction, mechanical strength, hardness and increased density due to decreased porosity.

3.4 Microstructural analysis

Fig. 3 shows the micrographs (SEM) of the formulations for each waste content in the clay and the sintering temperatures. It was observed in the microstructure of E1 (0% of granite waste, sintered at 1173K) different particle size and rough microstructure, the fine particles present in the form of agglomerates indicate the presence of clay minerals, a typical characteristic of the studied clay. E2, in which 20% of granite waste was added, sintered at the same temperature, showed larger and irregular grains, divided into islands, characteristics of quartz particles, some lamellar structures and the presence of white dots, however it was not constant behavior. In formulations E3 (0% granite waste) and E4 (20% granite waste) the sintering temperature was 1373K. At E3, less roughness and the presence of structural cracks in the material were observed. In E4, smooth particles were observed, which may be associated with vitreous phases formed after sintering, as confirmed in the X-ray diffractogram of the same test. It is also possible to observe the presence of voids in the structure, which are probably related to the presence of pores. In sample E5 (10% of granite waste, sintered at 1273K), the reduction of cracks and the presence of the formation of aggregates of small particles are noted.

Finally, as the GW content and the sintering temperature increased, small pores possibly closed, generated by the development of a greater amount of vitreous phase during the sintering procedure. Studies [35, 39] that investigated the influence of the addition of granite waste in ceramic mass, also observed similarities in the surface morphology found in this study.

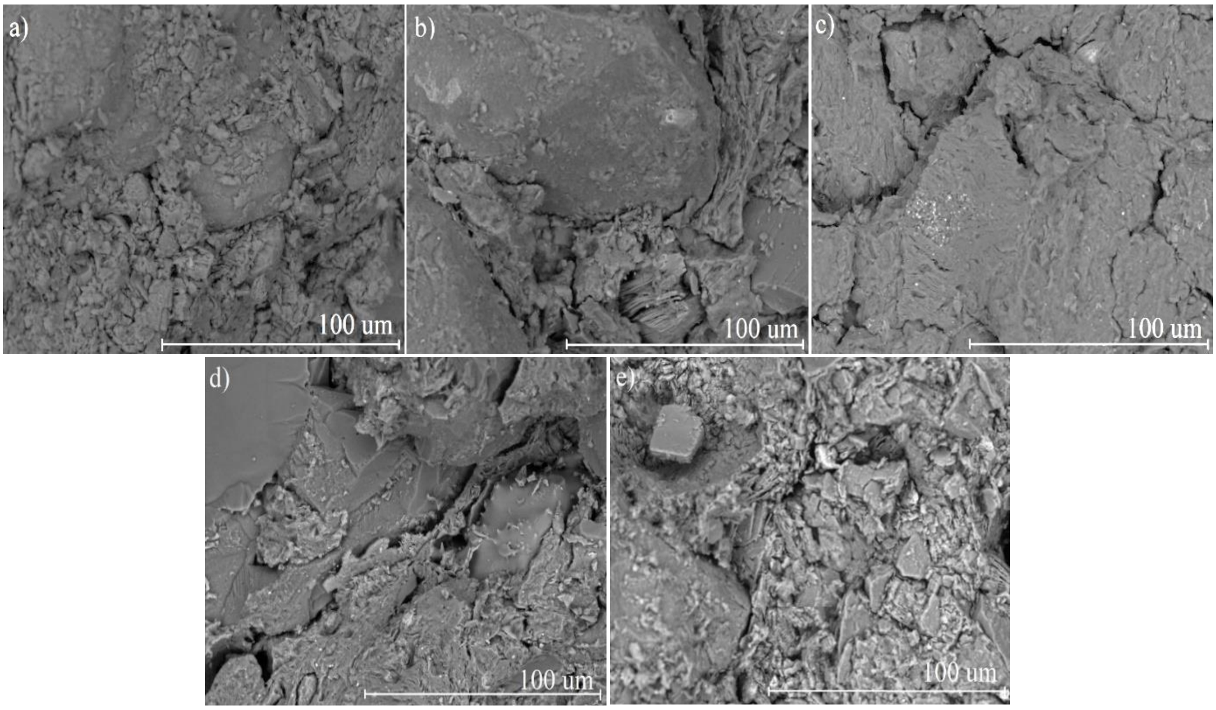

Figure 3: Scanning electron microscopy images of sintered formulations. (a) 0% of granite waste sintered at 1173K; (b) 20% of granite waste sintered at 1173K; (c) 0% of granite waste sintered at 1373K; (d) 20% granite waste sintered at 1373K; (e) 10% granite waste sintered at 1273K.

3.5 Technological properties

Fig. 4 presents the values of technological properties graphically. Fig. 4 (a) shows that the water absorption in the samples decreases with increasing temperature. It was also possible to verify that the formulations that contain quantity of granite waste presented inferior values of water absorption, mainly in the sample with 20% of GW, sintered at 1373K. The results found are in accordance with the results presented in the literature [3, 13]. All formulations showed a water absorption percentage below 20%, this is a positive fact, as the values are below the limit specified by international civil construction standards, that is, below 22% for bricks and 20% for tiles [13]. Analyzing the samples that contain GW, the values are below 14%, occurring the decrease for the samples sintered at 1373K, as expected, that is, the values are within the established standards.

The apparent porosity is shown in Fig. 4 (b), in which the samples also showed a significant decrease in their respective values with increasing temperature, the lowest value being in the sample with 20% GW, sintered at 1373K. The alteration of these properties indicates a densification, considering the increase in temperature, the presence of elements such as Magnesium, Potassium and Sodium and the phases identified in the X-ray diffractograms of the formulations, consequently, there was the formation of a vitreous phase by the fusion of these oxides, filling the pores and increasing the density of the samples [40], which can be observed and confirmed in the SEM images. In the analysis of the Apparent Specific Mass, it is observed that there was an increase as a function of the temperature, the values of the sample with GW were the most positive for the temperature of 1373 K. This result is expected when observing the behavior of apparent porosity and water absorption, the decrease in these two indicates an increase in density, as can be seen in figure 4 (c).

Linear firing retraction showed higher values at a temperature of 1373K as shown in fig. 4 (d). This increase in the linear shrinkage of the samples is related to the formation of a vitreous phase in the sintering process, mainly due to the presence of the flux elements found in the XRF analysis, in addition, the loss of kaolinite hydroxylates increases the packaging of the specimen [3]. It is important to note that the formulation with GW and the formulation that consists only of clay did not show great variations. This demonstrates that in this case the introduction of the granite residue did not cause major changes in the red ceramic matrix at this sintering temperature.

Figure 4 (e) illustrates the results of the ceramic matrix resistance module with the percentage of GW and the sintering temperatures. It can be seen that when burning at 1373K, the compositions presented lower rupture modules compared to those sintered at 1173K. The compositions with GW contents showed values of resistance to bending greater than that of the clay matrix without granite waste, and the formulation sintered at 1173K showed a greater variation in the standard deviation. The samples with GW that obtained high rupture modules, this fact indicates that the GW contributes to the strength of the ceramic matrix. This performance is related to the melting of feldspars present in the raw materials [32]. When comparing with the literature, it appears that the influence of GW shows more prominent results in contents of 40% and also with sintering temperatures of 1423K, conditions that increase the formation of the vitreous phase [32]. The formulations with GW showed results of flexural strength higher than the minimum for the manufacture of ceramic bricks (1.5 MPa) [3] and values within the recommended for tile strength (> 6.5 MPa) [13].

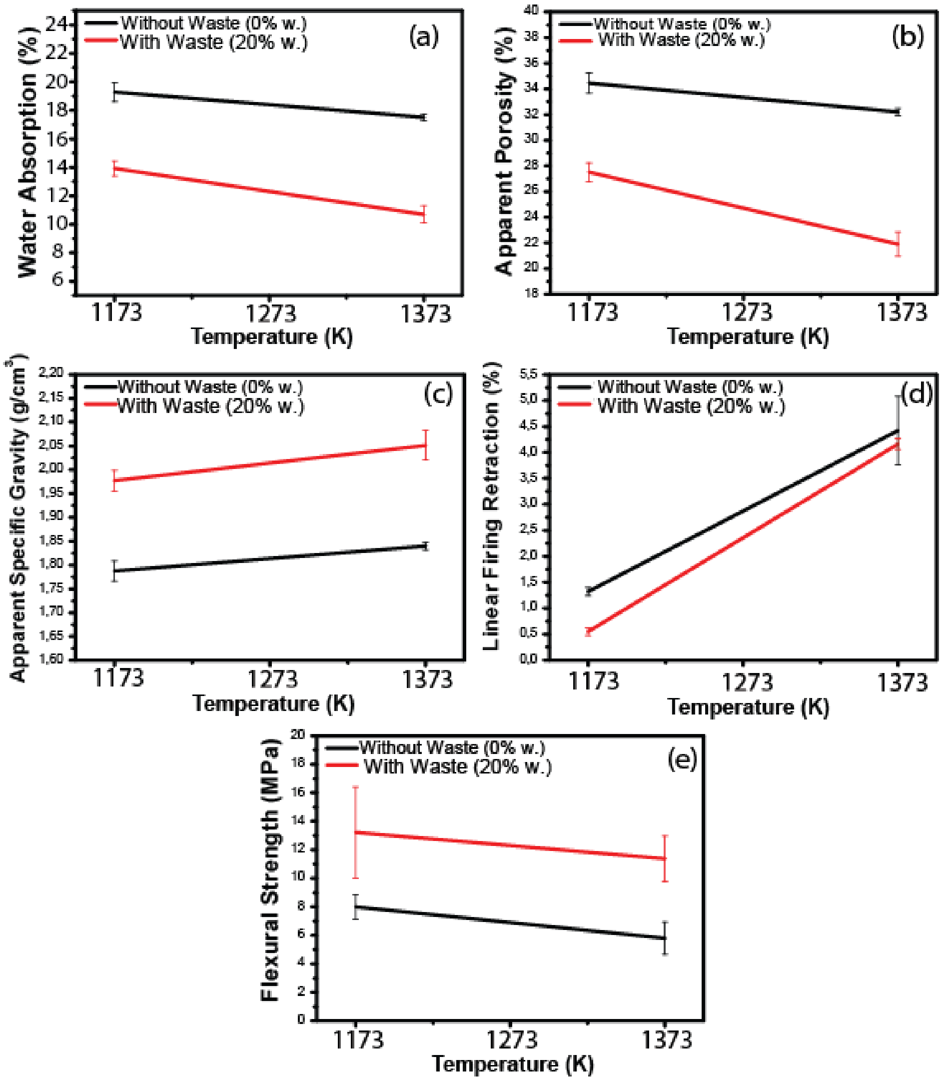

Figure 4: (a) Water absorption; (b) apparent porosity; (c) apparent specific mass; (d) linear firing retraction; (e) flexural strength module.

In short, a decrease in water absorption and apparent porosity, as well as an increase in apparent density and linear firing retraction of the samples with addition of 20% granite waste, sintered at 1373K, is a consequence of the increase in the phases which contribute to reducing porosity and promote greater densification. Therefore, consequently, there was an increase in flexural strength with the addition of granite waste for this sample.

A study presented a qualitative analysis regarding the impacts on the physical and anthropic environments generated in the productive processes of the ornamental stone processing companies in the city of Manaus and found that no company studied makes the correct destination of this material according to the National Solid Waste Policy [46]. Therefore, based on the results pointed out in this present research, it can be seen that it is possible to apply granite dust residue in the ceramic mass, aiming at the possibility of producing products from the red ceramic industry without reducing the mechanical resistance of the manufacture, in which it becomes a viable alternative from an environmental and economic point of view.

4. CONCLUSIONS

This study aimed to analyze the influence of the introduction of granite waste in clayey mass, evaluating its use in the red ceramic industry sector. Based on the results, it can be concluded that the chemical analysis showed considerable levels of flux elements in the waste granite samples. The mineralogical identification of the granite waste confirmed the presence of these elements that provide the formation of a glassy matrix at high temperatures through the muscovite, albite and microcline phases. It was found that in the mineralogical identification of the sintered formulations as the firing temperature was increased, there was a decrease in the crystalline phases in the formulations. The micrographs of formulations E4 and E5 showed better results, as smooth particles and a reduction in cracks in the clay matrix were observed. The results obtained in the mechanical properties of the samples show that all samples with granite waste obtained a decrease in water absorption and apparent porosity, an increase in apparent specific mass, linear firing retraction and flexural strength module when compared with samples without granite waste. This result reveals that a greater densification of the samples corresponding to the increase in the liquid phases that contribute to the reduction of the porosity and filling of voids between the grains. Samples with the addition of 20% granite waste, sintered at 1373K, obtained the best levels for the production of products in the red ceramic industry sector. Although formulations with 20% of granite waste sintered at 1173K, obtain results slightly higher than the ceramic matrix sintered at the same temperature, this effect was not significant. Finally, it appears that it burns at 1373K of samples with 20% granite waste from the Amazon region, has a positive influence on the increase of resistance, and can be used as a source of melting oxides for the products of the regional ceramic pole, providing benefits the local economy, consequently the generation of jobs and promoting solutions to reduce the environmental impact of the Amazon region through the development of new material.

5. ACKNOWLEDGEMENTS

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