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Influence of processing parameters variation on the development of geopolymeric ceramic blocks with calcined kaolinite clay

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ABSTRACT

The cement industry is responsible for a considerable part of the emission of gases that cause the greenhouse effect. In this context, geopolymer materials have been the subject of extensive studies, as they are an ecologically sustainable viable alternative to Portland cement. The geopolymer consists of a part rich in silica and alumina and another alkaline activating fraction, not having burning steps in its production chain, which consequently reduces the emission of CO_2. Geopolymers also allow the incorporation of specific residues in its precursor part, such as calcined clays. The specimens for this study were produced by the molding process, using commercial metakaolin as the basis for the precursor fraction. After demolding, the test cups were subjected to ambient curing at 25 °C and thermal curing at 80 °C, for periods of 7, 14 and 28 days. The objective of this work was to analyze the behavior of geopolymers specimens against the variation of several parameters such as percentage of calcined clay (CC) incorporation into metakaolin in 10% and 20%, its granulometry in 100 and 200 Mesh, the molar ratio of SiO_2/Al_2O_3, type and period of cure to evaluate technological properties such as resistance to bending, open porosity, water absorption, linear shrinkage and bulk density, aiming for future applications in the production of ceramic bricks. The results showed that an increase in the percentage of CC incorporation contributes to an improvement in the flexural strength, reaching 4.88 MPa to 20%. The molar SiO_2/Al_2O_3 ratio of 3.0 was the most ideal. The importance of this study is linked to the evaluation of several parameters for the incorporation of calcined clay, in order to obtain properties required for future applications in ceramic bricks.

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Table 1
Compositions studied.

Composition	Metakaolin (g)	Calcined kaolinite clay (g)	Sand (g)	Sodium hydroxide (g)	Sodium silicate (g)	Water (g)
0% – 3 M	460.0	0.0	690.0	144.0	6.0	325.0
10% – 3 M	414.0	46.0	690.0	144.0	10.0	325.0
20% – 3 M	368.0	92.0	690.0	144.0	12.5	325.0
0% – 3.5 M	423.2	0.0	634.8	140.0	72.0	340.0
10% – 3.5 M	380.9	42.3	634.8	140.0	75.0	340.0
20% – 3.5 M	338.6	84.6	634.8	140.0	80.0	340.0

1. Introduction

Ordinary Portland cement (OPC) is one of the construction industry materials that most negatively impact the environment. It is responsible for a considerable fraction of CO_2 and other greenhouse gas (GG) emissions, that is, from 7% to 10% of total GG emissions. In addition to releasing a lot of carbon dioxide, conventional cement requires a large amount of energy and natural raw material in its production processes [5,18].

As an environmental and technological alternative to the widespread use of Portland cement, geopolymer materials have some positive characteristics, both environmental and technological [20]. Geopolymer materials release a smaller amount of CO_2 and can use industrial waste and CC as raw material, in addition to showing good behavior to mechanical efforts and good durability [6,19]. In alternative applications to ceramic materials, there is still the advantage of energy savings, as there is no need for sintering the products to gain strength. With production steps in general, below 100 °C [16,22]. However, these materials have some disadvantages, such as the high acquisition cost of some inputs and limited studies related to their behavior against some aggressive agents, which currently restricts their application on a commercial scale [35].

The geopolymeric material is generally composed by the activation of a precursor portion by an activating alkaline solution [15]. The precursor fraction is rich in alumina and silica that, when in contact with the alkaline activating solution, previously prepared, initiates the geopolymerization reaction [17]. In a first stage of contact between the parts, the aluminates and silicates present in the precursor dissolve, giving rise to free tetrahedral units of AlO_4 and SiO_4, which compose a first gel [34]. In a second stage of the reaction, by condensation of the gel components, a second gel is formed, that is, an inorganic polymer, more stable [29]. In the last step of the reaction, crystallized zeolite is formed, giving rise to a hardened geopolymer mass [12].

Geopolymer can be produced with precursors such as metakaolin, fly ash, granulated blast furnace slag, ferrosilicon slag with alumina or CC in many cases. Clay minerals and volcanic ash have great potential [3,20,23,33] as a raw material to produce geopolymers, as they are a low-cost resource and present in abundance worldwide [4,32,36]. In general, research uses kaolin clays, calcined at different temperatures such as Abbas et al. [1] which heated up to 850 °C in a rotary kiln and Ounissi et al. [28] which used temperatures around 800 °C in electric oven. According to Marvila et al. [21] these temperatures are adjusted according to the degree of purity and crystallization of the material, which can generally alternate between 500 and 800 °C.

Kaolin clays have concentrations of SiO_2 and AlO_3 that vary according to the location of extraction [8-9]. Once extracted, they are submitted to the calcination process, aiming to produce a material with pozzolanic properties and high reactivity. During the process, water is removed from the structure and the material is dissociated into aluminosilicates, that is, metakaolin. At temperatures outside the indicated range, other glassy phases such as mullite are formed [27].

Limiting factors regarding the use of clay minerals in the production of geopolymers are the variation in thermal parameters required for activation and the variation in the chemical composition of these minerals [11].

This work aims at the production of geopolymer, to evaluate its potential application in the production of ceramic bricks. The main innovation of the work is related to the parameters that were evaluated for the application of CC, in which the published studies do not proceed with this correlation, so this research evaluated: (i) Percentage of CC used as a substitute for the commercial precursor (0%, 10% and 20%); (ii) Molar ratio between SiO_2/Al_2O_3 (3.0 and 3.5); (iii) Granulometry of calcined clay (through sieving at 100 mesh and 200 mesh); (iv) Type of cure performed (at 25 °C ambient temperature and at 80 °C thermal cure); (v) Curing period (in 7, 14 and 28 days).

With all the variables mentioned above, the technological properties of flexural strength, open porosity, bulk density, linear shrinkage and water absorption were evaluated.

2. Materials and methods

To carry out this study, specimens of prismatic geometry were made, as other studies aimed at application in ceramic materials already use this geometry [30,31]. The sieve used to process the calcined clay was varied, aiming at its incorporation as a substitute for commercial metakaolin, using manual sieving with a 100 and 200 Mesh sieve. The CC selected for this work was obtained from kaolinitic clay extracted from a mineral deposit in the city of Campos dos Goytacazes, Rio de Janeiro, Brazil. The extraction site is commonly used by the local ceramic industry to collect raw material. The metakaolin used as a precursor element in the preparation of the samples was the commercial Metakaolin HP Ultra. This metakaolin is one of the most consumed in the Brazilian market, and presents some problems related to color variability depending on the period of the year and deposit, which can be a problem for its application in geopolymeric materials, justifying even the search for alternative precursors. The calcination of the clay was carried out

Fig. 1. Three-point flexural strength test.

in a laboratory muffle furnace using a rate of 5 °C/min up to a temperature of 600 °C, since the transformation of kaolinite into metakaolin occurs at approximately 550 °C. After reaching the maximum temperature, the clay was kept at this level for 1 h, to allow the transformation of the material phases.

The sand used in the aggregate function was submitted to manual 35 Mesh sieving, being the same widely used in the civil construction industry and acquired in the commerce of the city of Campos dos Goytacazes, RJ, Brazil.

Based on the requirements of the standard [2], which presents parameters such as the amount of water to be used, in order to obtain satisfactory workability for molding the specimens, the activating alkaline solution was prepared. The solution was made with Sodium Hydroxide (NaOH), Sodium Silicate (Na_2SiO_3 – 18% Na_2O – 63% SiO_2). The solution was prepared in a magnetic mixer 24 h before molding and stored in a glass beaker.

The compositions evaluated in the research are presented in Table 1. It is observed that the amount of material in mass is not the same for the compositions studied. However, this does not interfere with the analysis of the results because the evaluation of the geopolymerization process is performed through molar ratios of SiO_2/Al_2O_3, which is a key parameter in the geopolymerization process, and not through mass ratios. These ratios were fixed at 3 M and 3.5 M in this study, as seen in Table 1.

The materials were dry mixed with the aid of an automatic mixer, and after the homogenization of the dry components, the alkaline solution was added, proceeding with automatic mixing again for about 15 min, until the formation of the geopolymer mass. After the mixing period and with the mass with adequate consistency (visual analysis), it was placed in small layers in acrylic molds with the dimensions of the specimens of 115 × 20 × 3 mm, together with a manual densification process with socket, preventing the formation of pores due to molding errors. Four specimens were made for each mixture shown in Table 1.

After the demolding process, the specimens were subjected to two types of curing, curing at room temperature, that is, 25 °C, and thermal curing, at 80 °C. The curing processes were carried out in 7, 14 and 28 days. After each curing period, the specimens were submitted to technological tests. Both water absorption and flexural strength are properties of greater relevance for the purpose of this work, which is to evaluate the feasibility of producing ceramic bricks. Water absorption (WA) and flexural strength were obtained through tests based on the standards NBR 15310:2005 [2] and ASTM C674 – 13. respectively. To obtain water absorption, the masses of the dry specimens were measured and after submitting them to 24 h of complete immersion in water. The mechanical resistance to bending at three points was obtained by means of a universal testing machine brand INSTRON, model 5882, at a load rate of 1 mm/min and 90 mm between the cutlasses, as shown in Fig. 1.

For the calculation and study of apparent density and open porosity, the Standard Test Method ASTM C 373 – 88: 1999 was used. Bulk density is the ratio between the dry mass and the outer volume of the sample. Open porosity was determined by dividing the difference between water-saturated mass and dry mass of the samples by their respective volumes. A hydrostatic balance was used to determine the mass suspended in water of the samples.

To determine the linear shrinkage, a caliper with a resolution of 0.01 mm was used, and the measurements required before and after the normal and thermal curing processes were measured. Finally, an analysis by scanning electron microscopy was performed on the compositions with the best performance evaluated. The equipment used was SSX550 Shimadzu SEDX microscope.

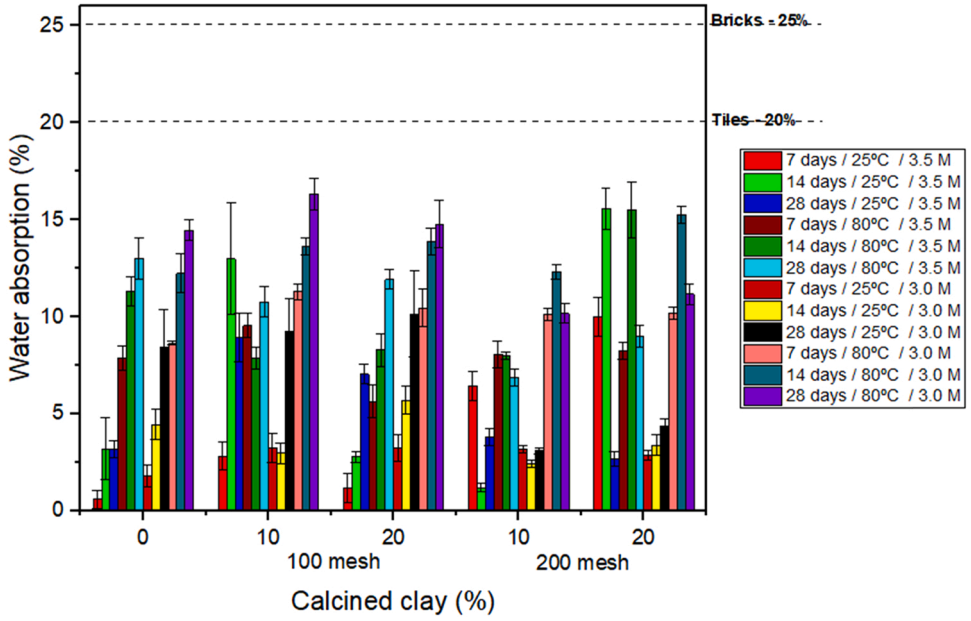


Fig. 2. Results of water absorption.

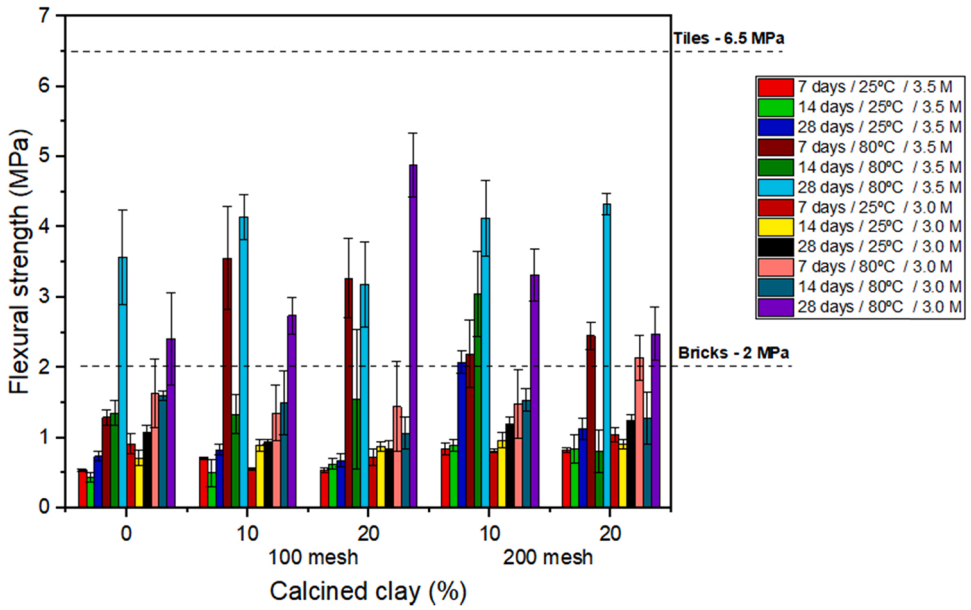


Fig. 3. Results of flexural strength.

3. Results and discussions

As already highlighted, the absorption of water has a great impact on the viability or not of a ceramic material intended to produce bricks. The water absorption results obtained are shown in Fig. 2.

The water absorption was higher in the samples in which the 100 Mesh sieving was used, with thermal curing being applied for a period of 28 days, with a molar ratio of silica to alumina of 3.0. It is noteworthy that the highest value obtained from this property was 16.34% with 10% incorporation of CC. Percentages close to those reached by De Azevedo et al. [6], in which a water absorption of 13.2% was obtained with the incorporation of glass polishing residue.

There was a considerable drop when incorporating the CC, as in Eliche-Quesada et al. [10], due to the increase in the curing time from 7 to 28 days and the molar ratio of silica and alumina from 3.0 to 3.5. The higher molar ratio leads to the formation of a smaller

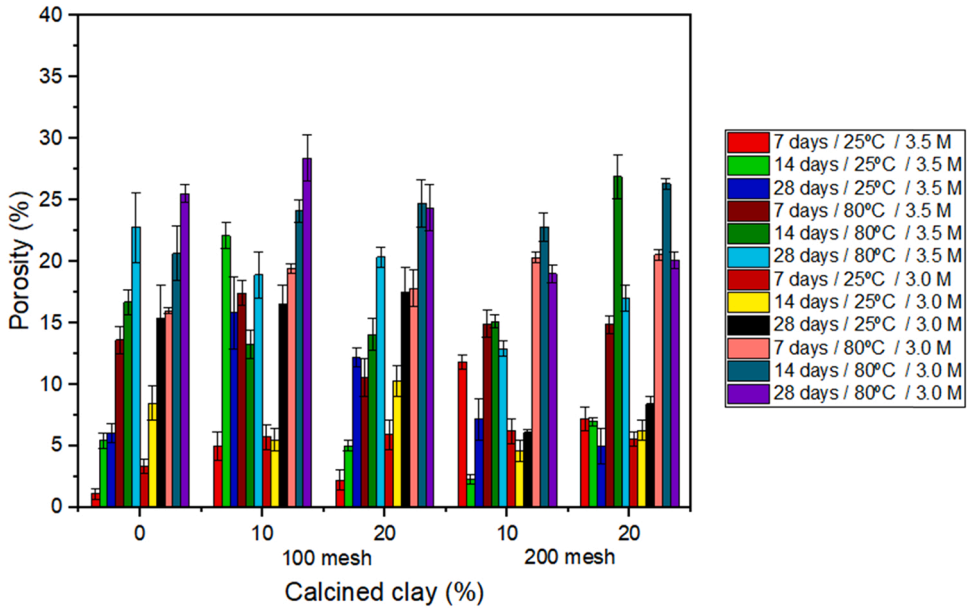


Fig. 4. Results of porosity.

number of pores, that is, it forms a denser geopolymer matrix, resulting in reduced water absorption [13].

The higher values of this property in sieving at 100 Mesh can be explained by the particle size, which the more refined, the lower the absorption tends to be, pointing to a considerable reduction in empty spaces and a higher cohesion of the structure.

The thermally cured compositions showed greater water absorption than the normal cured compositions due to evaporation of the water used in the alkaline activation process. This water, however, is free water and not chemically bound responsible for the formation of resistant products [39]. In general, the higher the curing age, the greater the water absorption of the compositions.

In view of the tested samples, none showed unsatisfactory results for application on ceramic bricks, which should not have a water absorption greater than 25%. The same happened with the water absorption limits for tiles, where all the evaluated compositions presented water absorption lower than 20%.

Another outstanding property for the feasibility of applying the geopolymeric material in question in the production of ceramic bricks is its resistance to bending. The results obtained are shown in Fig. 3.

The minimum bending strength required for ceramic bricks is 2 MPa. All specimens subjected to a cure temperature of 80 °C for a period of 28 days, both those with 3.0 and 3.5 molar ratio of SiO_2/Al_2O_3 showed satisfactory results. The higher temperature in the curing process can accelerate the dissolution of aluminosilicates, leading to a greater availability of Si in the process, forming silica bonds with oxygen, increasing flexural strength [6].

In the processing at 100 Mesh and molar ratio of 3.0, the flexural strength of the specimens increased with the increase in the percentage of CC incorporation, with the highest value being obtained, 4.88 MPa, corroborating with an improvement of this property with the presence of CC. This is because the clay calcined in the laboratory has controlled burning rates, showing greater reactivity than commercial metakaolin. When 200 Mesh refining was applied, all results in these parameters, that is, 3.0 molar ratio and 28 days of thermal cure, were higher than required, however, the behavior was not linear with the increase in the incorporation of CC, with the best resistance being 3.32 MPa, for 10% clay.

After 28 days of curing, at a temperature of 80 °C, with a molar ratio of 3.5, the pattern of results was inverted, with a non-linear increase as the CC content increased by 100 Mesh, reaching a maximum of 4.14 MPa, at 10% incorporation. When the raw material was processed in 200 Mesh, the increase in this property was proportional to the increase in the fraction of metakaolin replaced by clay, reaching its maximum value of 4.33 MPa with 20% replacement metakaolin for CC.

Only one set of parameters reached the 2 MPa of flexural strength of the specimens treated with 200 Mesh, submitted to the thermal curing process at 80 °C for 14 days, in a molar ratio of 3.5. The degree of incorporation in these specimens was 10%, reaching approximately 3.05 MPa of flexural strength, having declined sharply when the replacement was expanded to 20%. A possible explanation for the sharp drop in this property with the addition of a higher percentage of calcined clay to a high proportion of silica, has increased the reactivity, contributing to the formation of pores and a weakened geopolymer structure [20].

Of the tested specimens, only one subjected to normal cure, at 25 °C, sieved at 200 Mesh, reached the minimum requirements for applications in ceramic bricks. Being used in its production a curing period of 28 days and a molar ratio of 3.5. The other test specimens tested did not obtain the required minimum flexural strength.

Under the influence of the molar ratio, it is observed that compositions with a molar ratio SiO_2/Al_2O_3 equal to 3.5 presented a lower behavior than compositions with a molar ratio equal to 3.0. This happens because silica, being more reactive, promotes the formation of a more porous structure, causing an increase in water absorption and a reduction in resistance properties [24].

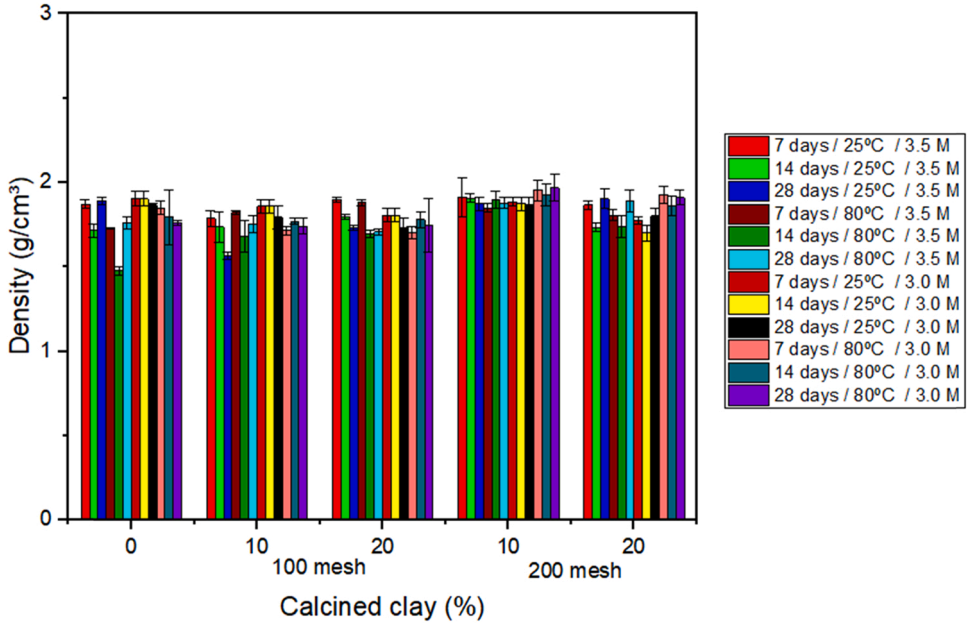


Fig. 5. Results of density.

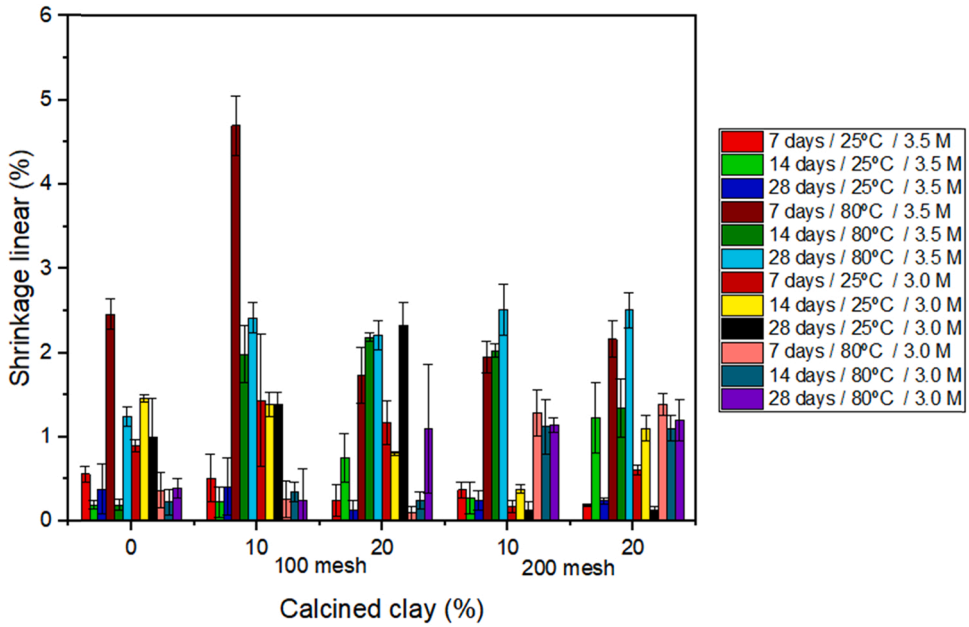
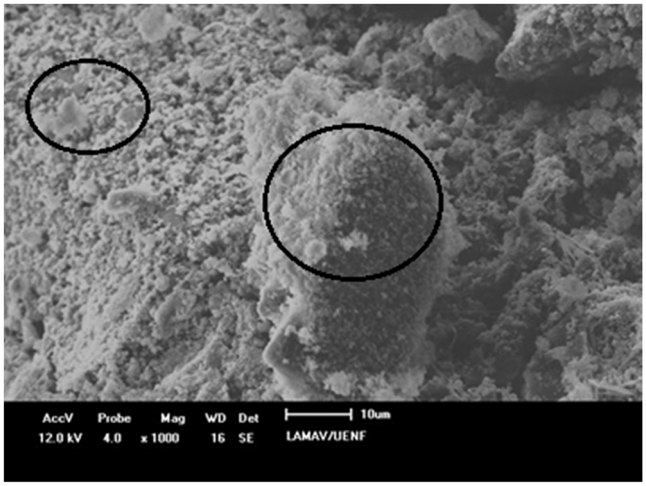


Fig. 6. Linear retraction for materials studied.

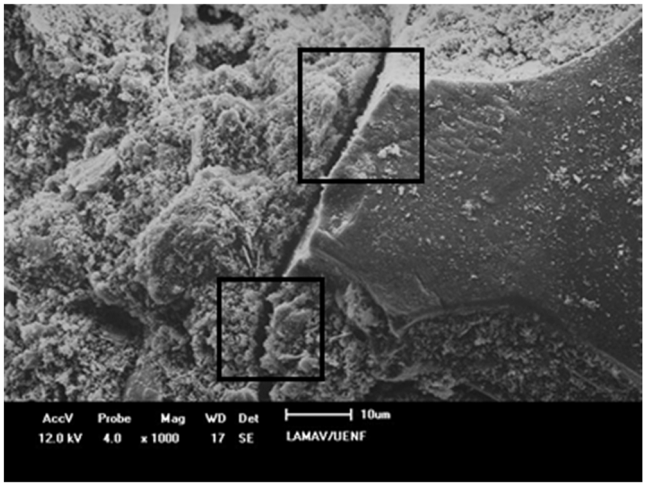
Open porosity mirrors water absorption, where in general, the more water is absorbed, the greater the porosity tends to be, as shown in Fig. 4. Pores perform the function of sites, where water is retained. The behavior was not similar only for the specimens exposed to normal curing, for a period of 7 and 14 days, with a molar ratio of 3.5, in the processing at 200 Mesh with 20% incorporation of CC. In these specimens, water absorption levels considerably higher than porosity were achieved. This happens as a result of the high content of silica, which, being more reactive, promotes an increase in the porosity of the material [26].

Fig. 5 presents the results of apparent density, which is an important property in the study of geopolymers. As this property increases, the number of pores decreases and the opposite also occurs, that is, these properties are interrelated [14]. As the bonds that make up the structure are formed, its mechanical properties improve.

The apparent density underwent a slight increase depending on the sieve used, both for incorporation of 10% and 20% of CC,



(a)



(b)

Fig. 7. SEM of the compositions cured at a temperature of 80 °C, for 28 days, 100 mesh, molar ratio $SiO_2/Al_2O_3 = 3.0$: (a) 20% calcined clay; (b) 0% calcined clay. Legend: O represents strengths phases formed by geopolymerization; □ represents pores.

because, when the raw material was benefited with a finer granulometry, that is, 200 Mesh, the structure presented a denser characteristic than that refined at 100 Mesh. It is noteworthy that the specimens subjected to thermal curing at 80 °C had a greater growth of this property, both for those produced with a SiO_2/Al_2O_3 ratio of 3.0 and 3.5. The highest apparent density obtained was approximately 1.87 g/cm^3, for 28 days of thermal cure, 3.0 molar ratio with 10% replacement of metakaolin by CC, at 200 Mesh.

When using a 100 Mesh sieve, the density changed little with the incorporation of clay to the precursor, with a considerable increase only for specimens subjected to thermal cure for 14 days, with a molar ratio of 3.5. There was a jump from 1.48 g/cm^3 of the parameter specimen to 1.68 g/cm^3 in 10% and 1.70 g/cm^3 in approximately 20% of incorporation.

Linear shrinkage is an important property, as it is possible to project the final dimensions of the geopolymers produced after the curing processes, both thermal and normal, based on it. In addition, the reduction of linear retraction is essential to provide durability to the geopolymer material [7]. Fig. 6 below presents the results of this technological behavior.

In samples exposed to normal curing for a period of 7 days with a molar ratio of 3.5, the incorporation of CC influenced a decrease in linear shrinkage, however, in most cases with the progression of curing time, this property increased, something to be expected given the longest period of water loss in the specimens [37].

In general, processing at 100 mesh contributed to an increase in shrinkage, in specimens with a molar ratio of 3.5, subjected to thermal cure at 80 °C, obtaining, as expected, higher shrinkage values. Be aware of curing processes carried out at high temperatures that lead to high shrinkage, as this can lead to water loss during the curing reaction, leading to the formation of small cracks and harmful pores that compromise the structure and thus its mechanical properties [38]. The highest percentages of this characteristic were obtained by samples submitted to the curing process for 7 days, reaching approximately 4.7% of average, with peaks reaching the mark of 5.0%.

Considering the set of analyzed samples, the results referring to linear retraction were satisfactory for the analysis parameters of this study. The property did not reach very high levels, remaining in general between 2.5% and 3.0%, with emphasis on the highest values achieved with the incorporation of 10% CC.

Fig. 7 presents the SEM results of the compositions chosen for analysis. Fig. 7(a) illustrates the result of the 100 mesh composition, with 28 days of cure at a temperature of 80ºC, for a molar ratio SiO_2/Al_2O_3 equal to 3.00% and 20% CC. The strength results for this composition were 4.88 MPa, the highest value obtained in the experimental program. It is observed that this composition presents a compact structure formed by an abundant whitish phase. This phase is the main responsible for the strength of this composition. Fig. 7 (b), on the other hand, illustrates this same composition containing 0% CC. There is a clear presence of pores in the composition of the material, which is one of the reasons for the lower strength of this composition, which only reaches a strength of 2.41 MPa.

4. Conclusion

With the results obtained in this research, it is possible to conclude that:

- The increase in the percentage of calcined clay in the production of geopolymers favors the increase in strength and improves the other properties evaluated. This is because calcined clay has controlled burning, unlike commercial metakaolin. The best strength results were obtained using 20% calcined clay, reaching a flexural tensile strength of 4.88 MPa.
- The SiO_2/Al_2O_3 molar ratio equal to 3.0 is ideal to produce the geopolymers evaluated in this study. This happens because the increase in silica content increases the geopolymer's reactivity, causing a more porous structure and consequently increasing water absorption and reducing strength.
- Calcined clays with a particle size of 100 mesh favor the increase in water absorption, due to the increase in porosity. It was expected that there would be an increase in the flexural strength of geopolymers with the use of finer calcined clay (200 mesh), but this was not verified. About strength, it cannot be said that there are differences in the behavior of geopolymers as a function of particle size.
- Regarding the type of cure, it was observed that thermal cure increases the water absorption of geopolymers, but this does not affect the material's strength gain. The justification for this is that the geopolymerization mechanisms are favored with an increase in the degree of contact between the precursor and the activator, which is favored with the increase in temperature. Therefore, although the porosity is greater, the strength is also greater due to the formation of more resistant products due to the increase in temperature.
- In general, 28 days cure increases endurance compared to 7 and 14 days. But some compositions had a different effect. This happens because compositions in normal cure, for example, are susceptible to efflorescence and other pathologies, which compromise their strength.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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