

Characterization and mechanical properties of one-part geopolymer based on a pure metakaolin

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Abstract

Geopolymers are amorphous silicate polymers that have been extensively studied due to their applications, scientific and technical relevance, and their potential as cement in substitution to Portland. Much of the focus has been in the use of precursors and aggregates that are rarely pure and homogeneous with the use of tailings being very common. The presence of these impurities complicates the interpretation of some spectral methods, mainly infrared spectrometry. Therefore, this research focuses on the use of a nearly pure kaolin main precursor, having only a trace of iron and titanium, along with other almost pure reagents, to achieve an ideal geopolymer. It was possible to produce metakaolinite, resulting in a material with a good size particle (8.24 μm) and surface specific area (6.57 m^2/g). The XRD result shows that the calcination process resulted in 100% of amorphous material. FTIR data revealed the presence of H_2O and NaCO_3 in the geopolymer. More important, the Si-Al-O various bands located between 1100 and 500 cm^{-1} validate the polymerization reaction effectiveness. SEM-EDS analyses have demonstrated that the reaction with Na_2SiO_3 and NaOH were nearly complete, concerning the finer metakaolinite particles that comprised the material's matrix. However, the coarser metakaolinite lamellae, in the 15-35 μm range, did not react completely; a thin border layer was enriched with sodium and most of the interior material maintained its Si-Al-O composition. The presence of traces of iron and titanium did not influence in the polymerization reaction. Compressive strength values of the geopolymer have presented good values in the range of 25 to 35 MPa.

keywords: geopolymer, pure kaolin, metakaolin, one-part, SEM, analytical standard.

1. Introduction

Geopolymers are three dimensional materials, composed of covalent bonds, linking mineral molecules in a big network. They are also described as a ceramic-like inorganic polymers. The name and concept were proposed in 1979 by Michel Davidovits (Davidovits, 2017).

Aluminosilicate inorganic polymers, or geopolymer, are materials that are interesting for various potential applications. These materials, prepared via

alkaline activation by aluminosilicate sources at low temperatures, display high mechanical properties and heat resistance (Williams *et al.*, 2011; Davidovits, 2017; Arnoult *et al.*, 2018). The geopolymer final structure depends on the nature of the oligomers formed during polycondensation reactions, which lines up with what the authors claim, that is: the geopolymers' properties are strongly affected by the raw materials used (Arnoult *et al.*, 2018).

A considerable number of academics noted that the structure of the final materials is highly influenced by the properties of the precursors, i.e., the metakaolin and the alkaline solution (Arnoult *et al.*, 2018). For example, the Si/Al ratio of metakaolin influences the dissolution of the aluminosilicate source and consequently the availability of silicium and aluminum species (Arnoult *et al.*, 2018). Because of this influence, several studies investigated the

impact of metakaolin reactivity on polymerization reaction kinetics, and network formation, along with the properties of the resulting materials (Vidal *et al.*, 2016).

The activation process of aluminosilicate materials in one-part geopolymer can be done by solid activators after adding water. Basically before the usage, the aluminosilicate material (metakaolin) and the solid activators (Na_2SiO_3 and NaOH) are mixed uniformly to form the one-part geopolymer binders, and when the usage is needed, just add water to the binders and mix. The process is very similar to Portland cement; the solid phases start to dissolve immediately when the solid phases are mixed with water (Williams *et al.*, 2011).

2. Material and methods

2.1 Materials

The precursor material selected for this research was a nearly pure metakaolin (MTK) as the alumi-

Eitel showed, as early as 1939, in his electron microscopy studies that kaolinite particles retain their hexagonal outline up to 750°C dehydration temperature (Davidovits, 2017). There are many techniques that provide important information about precursors, such as particle size distribution and specific surface area; for the latter, the BET technique is usually adopted (van Riessen *et al.*, 2017). Furthermore, to understand the extent of reaction in geopolymers, methods including x-ray diffraction (XRD), nuclear magnetic resonance (NMR) and Fourier transform infrared spectrometry (FTIR) have been used (Williams *et al.*, 2011; Vidal *et al.*, 2016; van Riessen *et al.*, 2017;

Caggiani *et al.*, 2021).

The current research studies the characterization of a material produced by calcination of a nearly pure kaolin and the respective geopolymer, using established characterization methods. In many studies on geopolymers, the main precursor, metakaolin, contains mineral impurities, inherited from the original kaolin, such as quartz, micas and feldspars (Figueiredo *et al.*, 2021). The presence of these silicates complicates the interpretation of some spectral methods, mainly infrared spectrometry, when applied to the finished geopolymer, which is also a silicate. Therefore, the use of a pure metakaolin was one of the motivations for this study.

2.2 Sample characterization techniques

Initial investigation was employed to identify the particle size distribution and the specific surface area of kaolin and MTK, using the following instruments: CILAS 1190, a laser granulometer that analyzes

nosilicate material; sodium silicate, Na_2SiO_3 , ‘SS’, and sodium hydroxide, NaOH , ‘SH’, were the activators. Pre-

cursor and activator materials were handled as solids.

particles in the range of 0.04 to 500µm in a liquid medium. The test was performed with 0.5%*m/V* sodium hexametaphosphate dispersant; BET Quantachrome NovaWin equipment was used to perform the nitrogen

gas adsorption tests. Chemical composition of the metakaolin was obtained by X-ray fluorescence spectrometry (XRF) analysis with a Philips (PANalytical) spectrometer, PW 2400 model and is presented in Table 1.

Table 1 - Chemical composition of precursor and activators.

Material	Al_2O_3	SiO_2	Na_2O	Fe_2O_3	TiO_2	CaO	MgO
Kaolin	37.98 %	43.10%	0.08%	0.48%	0.42%	0.01%	0.05%
SS		63.00%	18.00%				
SH			77.48%	10ppm		0.01%	50ppm

In order to identify the minerals presented in kaolin, determine if the dehydroxylation process was effective to obtain a good quality metakaolin and certify the geopolymer formation, X-ray diffraction (XRD) analyses were carried out with a PANalytical X'Pert APD, PW 3710/31 controller, diffractometer, using copper anode tube ($Z=29$): $\lambda \text{K}\alpha$ average = 1.54184Å - $\lambda \text{K}\alpha 1$ = 1.54056Å; readings were taken with step size of

0.060 and step time of 1.00s. To corroborate with the phase analysis and functional groups present, a Bruker device, Alpha model, with attenuated total internal reflectance (ATR), 128 scans, diamond crystal and a resolution of 4 cm^{-1} were used to perform a Fourier transform infrared (FTIR) spectroscopy analysis.

Microstructure analyses were obtained by means of a scanning electron

microscope (SEM) FEG Dual Beam Quanta 3D FEI. Energy dispersion X-ray spectrometer (EDS) was a Bruker Flash 5010, Esprit software 2.1. The preparation of kaolin and metakaolinite samples was carried out on loose particles, over a conductive carbon tape. For the geopolymer, fragments of broken specimens were obtained during the uniaxial compression test. All the SEM images in this study are by backscattered electrons.

2.3 Geopolymer synthesis

The first part of the study consisted of producing a good quality precursor material from a source of practically pure kaolinite. The process was carried out by calcination in a fixed thin bed ceramic tray, to ensure complete dehydroxylation. It was determined that 2 types of metakaolin

would be produced: the first calcined for 5 hours at 700°C (MTK-I) and the second calcined for 2 hours at 700°C (MTK-II). The objective was to verify whether the difference in calcining time would affect the material's properties (Brito *et al.*, 2017).

As the geopolymer in the study

consists only of binding material (metakaolin, NaOH and Na_2SiO_3), the product obtained was called pure geopolymer, precisely because it did not contain aggregating material, such as sand, tailings or industrial waste, in its structure.

For the preparation of the pure geo-

polymer, the following dosages were used: MTK=0.7; SS=0.24; SH = 0.06 (in parts) and the water/binder ratio=0.4, resulting in ratio of Si:Al=1.65 and Si:Na=1.79. The mixing of the material followed the specifications of one-part type geopolymers, which consists of first mixing the

dry parts, and then adding the water. The first step lasted about 2 min. The second step was the addition of the water to the dry mixture and the continuous mixing lasting over 3 min. After mixing, the cement paste samples were poured into the cylinder molds with 20mm diameter x

40mm tall. Subsequently, the mold passed by a vibration process to ensure the mixture homogeneity and eliminate air bubbles. The geopolymer samples were demolded after 24 hours of curing at room temperature and humidity (Ma *et al.*, 2019; Figueiredo *et al.*, 2021).

2.4 Compressive strength

Compressive uniaxial strength tests were carried out using an electric

press to apply a constant tension rate of 25kgf/s until failure, with curing inter-

vals of 1, 3, 9 and 14 days (Ma *et al.*, 2019; Figueiredo *et al.*, 2021).

3. Results and discussion

3.1 Particle size distribution

In Table 2, one verifies that the calcination process resulted in an increase in particle size of 29.96% for MTK-I at

D50 and 23.03% for MTK-II. The mean particle diameter increased by 28.40% for MTK-I and 21.53% for MTK-II. The con-

clusion is that metakaolin (MTK-I), corresponding to the longest calcination time (5h), had a greater increase in particles size.

Table 2 - Size distribution CILAS 1064.

	D ₅₀	D _{average}
Kaolin	6.34µm	7.57µm
MTK-I	8.24µm	9.72µm
MTK-II	7.80µm	9.20µm

3.2 Gas adsorption

From the results, as shown in Table 3, the specific surface areas were obtained.

It can be concluded from the calcination process that in the transformation of kaolin

into metakaolin, there was an increase in particle size and in the specific surface area.

Table 3 - Surface area results by BET method.

Material	Specific area
Kaolin	6.30 m ² /g
MTK-I	6.57 m ² /g
MTK-II	7.08 m ² /g

3.3 X-ray diffraction

3.3.1 Kaolin

The kaolin sample was analyzed to verify the purity level of the material. The sample presented the characteristic peaks of the phases very clearly, making it possible to identify them (Figure 1). The strong peaks

at 7.17, 3.58 and 1.49Å correspond to the kaolinite reflections d001, d002 and d060 and the bands at 1.49, 1.66, 1.99, 2.34, 2.49, 2.56 and 4.46Å are also correlated to kaolinite (Williams *et al.*, 2011). Trace

amounts of anatase and goethite were tentatively identified. This is consistent with the chemical analysis of kaolin where minor amounts of Fe₂O₃=0.48 and TiO₂=0.42 were measured (Worasith *et al.*, 2011).

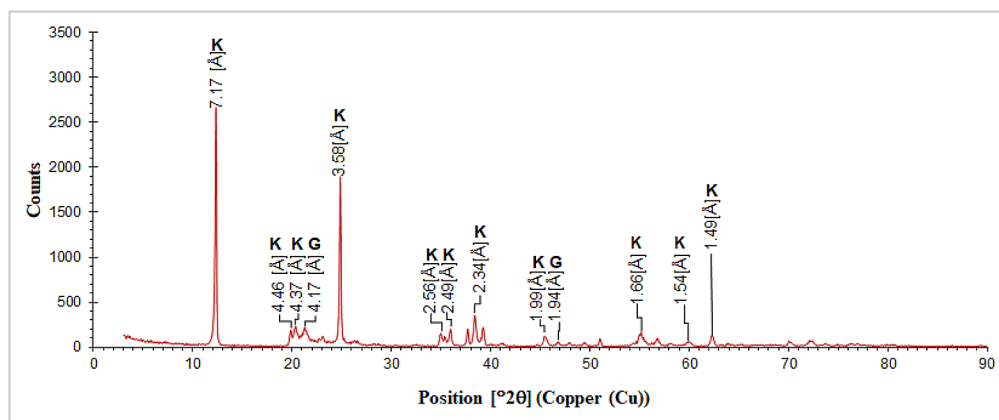


Figure 1 - XRD kaolin with identified peaks. Where: K= kaolin; G=goethite.

3.3.2 Metakaolin and geopolymer

All calcinations that resulted in MTK proved that there was complete dehydroxilation of the kaolin. The analysis of the diffractogram obtained for the pure geopolymer failed to detect any characteristic peaks of the

material; thus, it was made up only of amorphous material (Figure 2). Williams *et al.* (2011) and Scherb *et al.* (2020) point out that metakaolin and geopolymer XRD patterns exhibit diffuse scattering, occasionally

cited as large amorphous spines. For metakaolin, the amorphous spine is unsymmetrical with a centroid around $d \approx 4.02 \text{ \AA}$, while the geopolymer hump is symmetric with a centroid at $d \approx 3.2 \text{ \AA}$ ($27\text{-}28^\circ$ in 2θ) (Williams *et al.*, 2011).

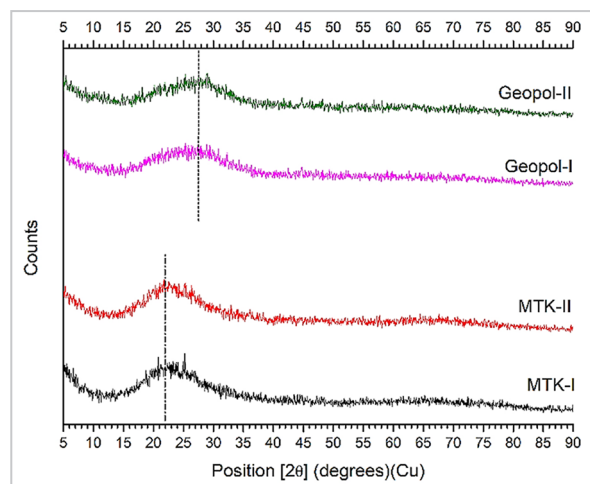


Figure 2 - Comparative XRD of metakaolin and geopolymer.

3.4 Fourier Transform Infrared Spectroscopy (FTIR)

In the region of highest wavenumbers (Figure 3), the broad bands having values at 3322 and 3377 cm^{-1} of geopolymer, are due to the symmetric and asymmetric axial vibrations of H_2O which indicate that the phase is hydrated. The peak located at 1647 cm^{-1} , in the geopolymer spectra, is related to H_2O bending vibration (Worasith *et al.*, 2011; Vidal *et al.*, 2016; Naghsh and Shams., 2017; Ma *et al.*, 2019; Scherb *et al.*, 2020; Figueiredo *et al.*, 2021).

The typical spectrum of the original kaolin sample shows the OH stretching vibrations in two well-defined peaks at 3694 and 3621 cm^{-1} flanking two weaker peaks at 3669 and 3652 cm^{-1} . Furthermore, the peaks in the

1200 to 400 cm^{-1} range are a confirmation of the presence of kaolinite in the sample (Worasith *et al.*, 2011).

In relation to the geopolymer, literature mentions the main fingerprints of the material: axial vibration bands near 3400 cm^{-1} , angular vibration close to 1637 cm^{-1} for H-OH and asymmetrical stretching vibrations of Si-O-Si and Al-O-Al centered at 987 cm^{-1} . All bands were identified in the geopolymer samples, which demonstrates that the polymerization reaction occurred effectively (Vidal *et al.*, 2016; Figueiredo *et al.*, 2021).

In the range from 1388 to 1439 cm^{-1} , FTIR presents bands for the geopolymer, which refer to the presence of asym-

metric stretching vibrations of Na_2CO_3 (Figueiredo *et al.*, 2021).

A wide band is visible in the Si-O-Si and Si-O-Al stretching vibration range between 900 cm^{-1} and 1200 cm^{-1} , due to the amorphization of the kaolinite crystal structure. As reported for Silva and Santana (2013), in the range of 1000 to 1120 cm^{-1} kaolinite presents three bands of Si-O apical deformations referring to the common plane between the tetrahedral and octahedral lamellae. For the geopolymer, the bands assigned to the Si-O vibrations are located at 982 and 987 cm^{-1} (Worasith *et al.*, 2011; Scherb *et al.*, 2020; Figueiredo *et al.*, 2021). Table 4 displays these and other important assignments.

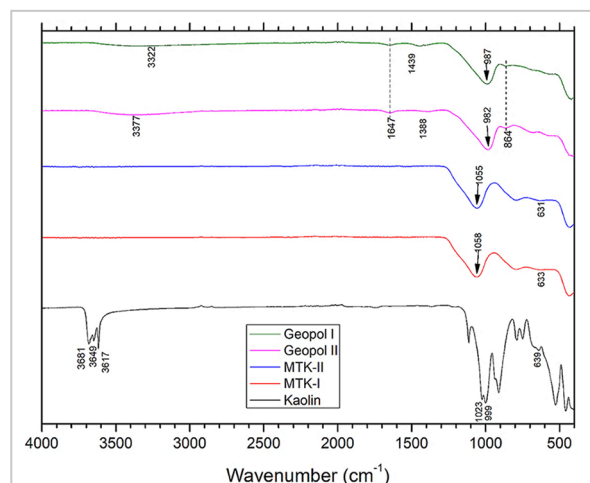


Figure 3 - FTIR spectra of kaolin, metakaolin and geopolymer.

Table 4 - FTIR band identification (values in cm^{-1}).

Assignment	Kaolin	Metakaolin		Geopolymer	
		MTK- I	MTK-II	Geopol - I	Geopol - II
O-H stretching (symmetric and asymmetric stretching vibrations of H_2O)	3681			3322	3377
	3649				
	3617				
H_2O bending vibration				1647	1647
Na_2CO_3 stretching (asymmetric vibrations)				1439	1388
Si-O-Al e Si-O-Si stretching (symmetric and asymmetric vibrations)	1113	1058	1055	987	982
	1023				
	999				
Si-OH bending vibration	910			863	865
Al-Si-O bending	525	633	631	681	540
	639				

3.5 SEM EDS

The analysis by SEM-EDS was applied in order to track the possible impacts

of the calcination and polymerization process on the kaolin morphology, as well

as to identify the reactivity of activators in the geopolymer structure.

3.5.1 Kaolin

In Figure 4 one observes the hexagonal lamellar particles, as well as the stacking of these that are plates characteristic of kaolinite. Also, small white dots are

visible that indicate the presence of Ti, in addition to the elements O, Al and Si, which are the main constituents of aluminosilicate (kaolin) (Table 5); furthermore,

there is trace Fe which indicates that it may contain goethite. The presence of these elements is confirmed with XRD analysis, which identified anatase and goethite.

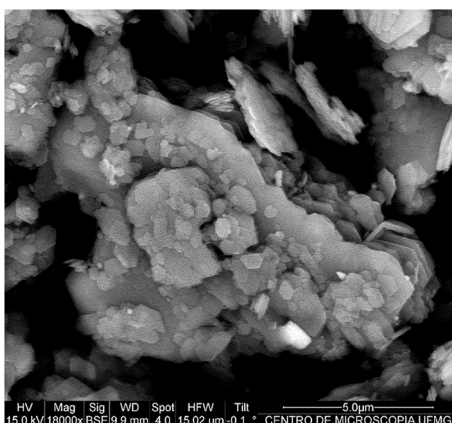


Figure 4 - SEM image of the kaolin.

Table 5 - EDS Microanalyses.

Oxygen	Aluminum	Silicon	Iron	Mineral
57.94	20.56%	20.86%	0.64%	kaolin

3.5.2 Metakaolin

Generally, the kaolinite lamellar morphology is maintained. However, at some points there has been an expansion of the lamellae stackings, like the pages of a book opening up (Figure 5). This ex-

foliation is consistent with the increase in the specific surface area after calcination. Also, small TiO_2 spots are present in the matrix of metakaolin lamellae.

In Figure 6 and the correspond-

ing chemical maps, the composition of metakaolin is made clear, with the homogeneous distribution of Si, Al, O and low Fe. Furthermore, small subhedral scattered grains of TiO_2 are clearly identified.

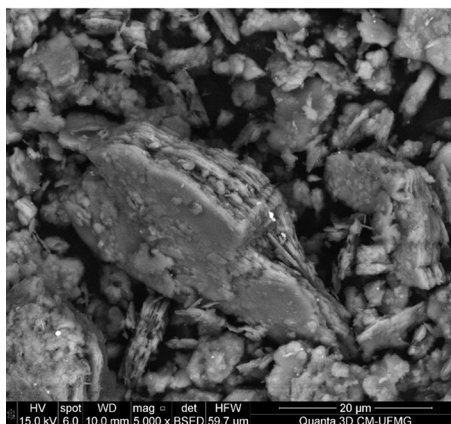


Figure 5 - SEM image of the metakaolin.

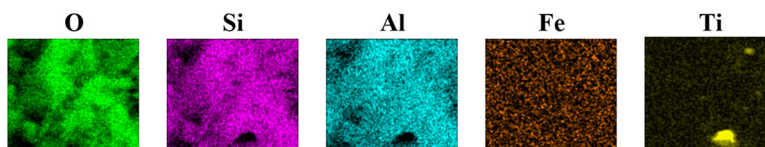
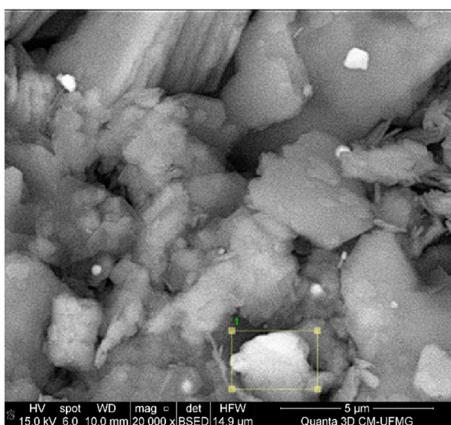


Figure 6 - SEM-EDS Metakaolin chemical analysis.

3.5.3 Pure Geopolymer

The objective of analyzing the microstructure of the geopolymer was based on monitoring the reactivity of the important elements: O, Si, Al and Na, as they are the main ones in the polymerization reaction.

Preserved metakaolin lamellae are conspicuous in the geopolymer matrix (Figure 7), along with round pores, the latter derived from air bubbles, and a

few fissures. The metakaolinite plates are well and randomly distributed, which is a positive point.

A critical indicator of the extension and homogeneity of the polymerization reaction is the location of the element sodium. In Figure 8, there is a large metakaolinite lamella at the image's center, surrounded by a matrix of much smaller plates. In the chemical

distribution maps, it is clear that Na has reacted with all the matrix components, making up a Si, Al and Na mass, which is really the geopolymer material. However, Na has reacted with only a thin border in the large metakaolinite plate; this is probably due to the fast reaction, in the short time available. This effect has been observed for most grains, in the 15-35μm range.

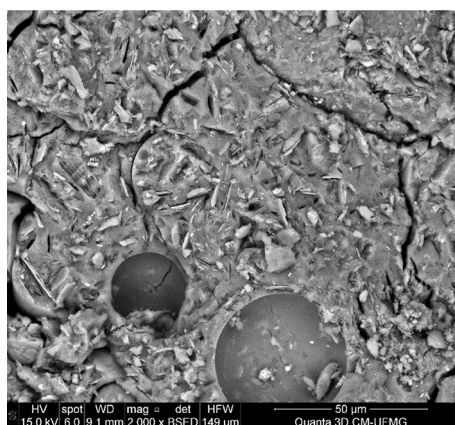


Figure 7 - SEM-EDS Pure Geopolymer.

In addition to tracking the reactivity of Na in the matrix, it was verified that the minor presence of Fe and Ti did not affect in the polymerization reaction, which justifies research that

adds industrial waste to the matrix and still obtains geopolymers with good mechanical strength.

In the same Figure 8, one notices that Si is the element with higher

concentration. This is a consequence of the addition of sodium silicate; thus, the Si concentration was increased above that already present in the metakaolin.

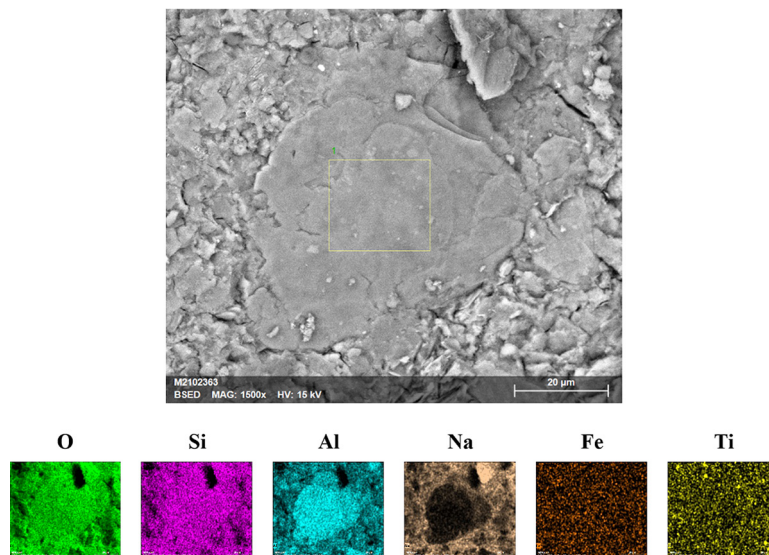


Figure 8 - SEM-EDS Geopolymer: maps of chemical microanalyses.

3.6 Compression Strength

The compressive strength test was carried out in order to verify the physical behavior of the geopolymer produced from the pure metakaolin. The material selected to perform the uniaxial tests was

the geopolymer made with MTK-I, since it presented a satisfactory size particle increase and specific surface area. Results are displayed in Figure 9. In general, there was a small increase in the values of compressive strength, but they displayed similar strength along the curing days, since the values are in the range of 25 to 35MPa. These are considered good values, mainly for a material without aggregate and filler.

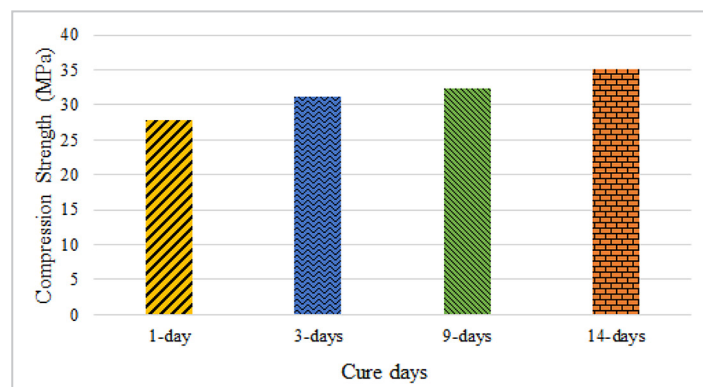


Figure 9 - Uniaxial compressive test – geopolymer.

4. Conclusions

This study was quite important for comprehending the essential parameters involved in the geopolymer's properties. It was possible to produce metakaolinite from a pure kaolin, resulting in a material with a good size particle (8.24 μm) and specific surface area (6.57 m^2/g). The XRD result shows that the calcination process occurred in 100% of the material, represented by absence of the crystalline phase, and managed to identify the presence of traces of iron and titanium in the kaolin. FTIR data revealed the presence

of H_2O and NaCO_3 in the geopolymer. More important, the various Si-Al-O-OH bands located between 1100 and 500 cm^{-1} validate the geopolymerization reaction effectiveness. In this case, the use of a pure metakaolinite was important in facilitating the proper band assignments.

SEM-EDS analyses have demonstrated that the reaction with Na_2SiO_3 and NaOH were rather complete, concerning the finer metakaolinite particles, comprising the material's matrix. However, the larger metakaolinite

lamellae, in the 15-35 μm range, do not react completely, and only a relatively thin border layer is enriched with sodium; thus, most of the interior material maintains its Si-Al-O composition. The presence of small concentrations of iron and titanium did not influence the polymerization reaction.

Compressive strength values of the geopolymer, throughout the curing ages of 1, 3, 9 and 14 days, have presented good values, in the range of 25 to 35MPa.

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