

Study of the Curing Temperature of 600°C, 700°C and 800°C of Mouyondzi Clay on the Mechanical, Physical and Microstructural Properties of Geopolymer Obtained

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Abstract

This work consists of determining the right curing temperature for Mouyondzi clay, with a view to obtaining a very reactive metakaolinic amorphous phase, which will give us a geopolymer with good physical and mechanical performance. The kaolin-dominant Mouyondzi clay was calcined at 600°C, 700°C and 800°C with a heating rate of one degree per minute. In order to achieve the objective of this work, the performance of geopolymer was measured by compressive strength on geopolymer prisms of 28 days of age, by XRD and IRFT of geopolymer powders, calcined clay and raw clay, and by SEM of geopolymer blocks. Analysis of the results shows that the resistance value increases with the curing temperature of the clay and reaches a maximum of 49 MPa at 800°C. At 600°C we already have 31 MPa, the equivalent of Portland cement with the addition. The XRD confirms the disappearance of clay species from 600°C. At 800°C there is not yet the appearance of a new crystalline phase. Quartz is the only mineral species present. We can therefore confirm that at 800°C, the geopolymer obtained exhibits higher physical and mechanical performance than the other curing temperatures studied for Mouyondzi clay. This is confirmed by the appearance of a new aluminosilicate phase in the IRFT spectra and in the SEM images appearing as a continuous plate.

Keywords

Geopolymer, Compressive Strength, Mouyonszi Clay, Amorphous Phase

1. Introduction

Environmental problems are a major problem for mankind today. These environmental imbalances are caused by the emission of greenhouse gases released in large part by the cement companies. Portland cement is the most used building material by humans at 1 m³ per person per year [1], its production consumes a lot of energy from the extraction of raw materials to the final product [1]. The production of one tonne of clinker consumes 3000 to 8000 KJ of fuel and 70 to 160 KWh per tonne of cement depending on the country [1]. The search for an alternative to Portland cement is therefore essential. It has been reported in the literature that geopolymers are able to replace Portland cement [2].

Geopolymers are reciprocals of organic polymers whose matrix is of the aluminosilicate type. They are obtained by alkaline activation at room temperature of low calcium aluminosilicates with an alkali hydroxide solution [2]. These inorganic polymers are obtained from a diverse range of aluminosilicate materials such as fly ash, blast furnace slag and clay minerals [3]. It should be noted that the physical properties of clays such as specific surface area and cation exchange capacity make them the most used in this field. Kaolinite and its derivatives are the most widely used clay minerals due to their 1:1 type structure, kaolinite is transformed into metakaolinite [3]. Previous studies have revealed that the clay collected in the locality of Mouyondzi in Congo Brazzaville is predominantly kaolinitic and therefore could be used for the development of a geopolymer [4].

This study enters into the valuation of Mouyondzi clay in the field of geopolymer production. The clay will be heated to 600°C, 700°C and 800°C in order to find the temperature where the resulting amorphous phase will be more reactive.

The performance of the formulated geopolymers will be determined by X-ray Diffraction (XRD), Infrared Spectroscopy (IR), Scanning Electron Microscopy (SEM) and compressive strength. The effectiveness of the heat treatment will be determined by XRD and IR.

2. Experimental Equipment and Methods

2.1. Aluminosilicate Materials

The aluminosilicate material used in this work is a clay taken in the department of Bouenza (Republic of Congo), in the district of Mouyondzi (Figure 1) [4]. Moutou and all have shown that kaolinite, illite and quartz constitute the mineral phases of this clay. Kaolinite is the most dominant clay species. Table 1 gives the results of the chemical oxide analysis. Observation of this table shows that

Table 1. Chemical composition of the raw materials used.

Materials	H ₂ O	SiO ₂	Al ₂ O ₃	K ₂ O	Na ₂ O	Fe ₂ O ₃	TiO ₂	CaO	MgO	PF
Clay	-	61.45	20.96	1.5	0.05	1.5	2.36	0.31	0.59	10.5
Soda silicate	56.2	31	-	-	12.8	-	-	-	-	-
Soda (12 M)	91.2	-	-	-	28.48	-	-	-	-	-

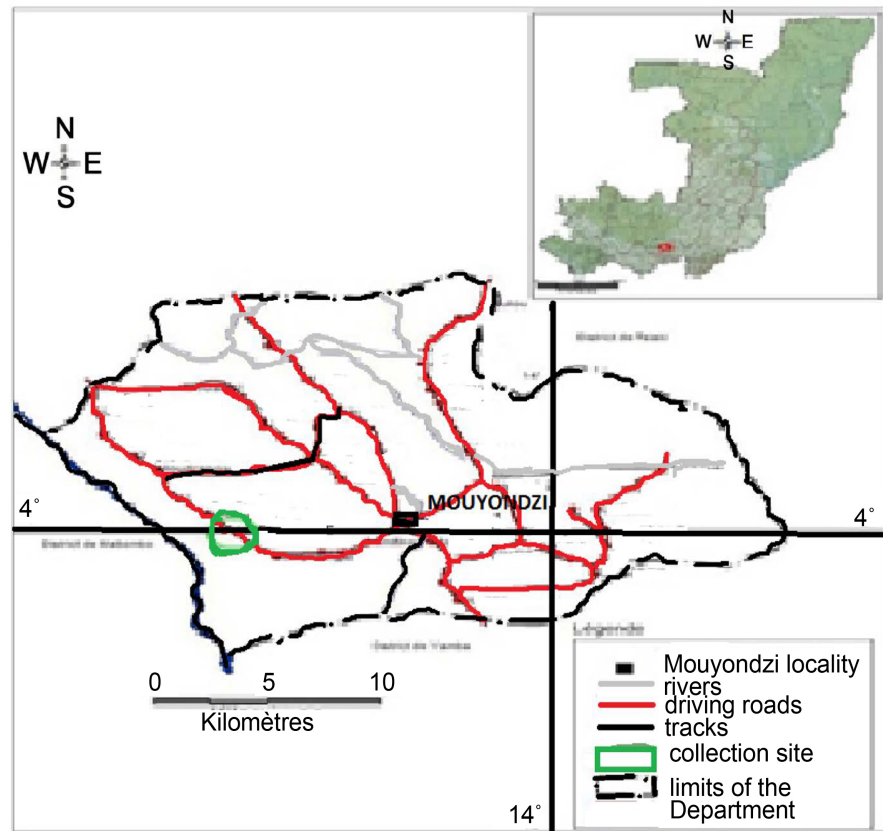


Figure 1. Location of the sampling site [4].

SiO_2 is the oxide with the highest percentage followed by Al_2O_3 . The $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio gives 2.9, making Mouyondzi clay favorable for the development of geopolymers.

The sample was taken from a depth of one meter, dried in the open air on the bench in the laboratory, crushed using a porcelain mortar, sieved at $96\ \mu\text{m}$ then stored in plastic jars to prevent any contamination. This powdered sample referenced Mou was heated to 600°C , 700°C and 800°C , with a heating rate of $1^\circ/\text{min}$. The calcined clay is stored in waterproof plastic pots, and referenced respectively Mou6, Mou7 and Mou8.

The transformation of kaolinite into metakaolinite begins very slowly from 450°C , it continues up to 800°C [4], hence the choice of processing temperatures of 600°C , 700°C and 800°C .

2.2. Alkaline Solution

The activator solution consisted of a 12 M sodium hydroxide solution and a sodium silicate solution. The soda solution was prepared by dissolving 99% by mass purity caustic soda in distilled water. The sodium silicate was composed of (mass%): SiO_2 (31.00), Na_2O (12.8) and H_2O (56.2). This prepared activator solution was prepared at room temperature stored for 24 hours before use to achieve equilibrium.

2.3. Preparation of Study Specimens

The geopolymer paste was formulated by mixing the calcined clay powder at different temperature (Mou6, Mou7 or Mou8) with the alkaline solution. The mixture was kneaded with an electric mixer for 10 minutes with a one minute pause after 5 minutes, to promote good diffusion throughout the mixture.

The resulting paste was poured into the $4 \times 4 \times 16$ cm steel prism molds. The molds were vibrated after casting for 10 minutes to remove air bubbles trapped during the casting process. Demoulding was carried out after 24 hours and the freshly demolded test pieces were packaged in polyethylene. These prismatic specimens were used for compressive strength measurements after 28 days of age. Part of the debris from this measurement was used for SEM and another part was crushed and sieved at $50 \mu\text{m}$ for performing XRD and IR.

Table 2 shows the samples and their respective labels.

2.4. Characterization Techniques

The characterization techniques of formulated geopolymers and calcined clay powders used in this work are:

1) DRX

X-ray diffraction was used for the determination of the mineral phases of Mou, Mou6, Mou7, Mou8, GMou6, GMou7 and GMou8. The measurements were carried out using a Bruker D8 type diffractometer in Bragg-Brentano geometry using $\text{CuK}\alpha$ radiation. The data was recorded in a domain between 10°C and 70°C with a step of 0.006°C and a fixed divergence slit of 0.3°C .

2) IRFT

Fourier transform infrared spectroscopy measurements were carried out on a FTIR Thermo is 10 spectrometer. The Mou, Mou6, Mou7, Mou8, GMou6, GMou7 and GMou8 samples (in the solid state and sol-gel) were characterized by Attenuated Total Reflection (ATR) by mounting an ATR unit in the sample compartment of the spectrometer. The spectra were recorded in a range between 500 cm^{-1} and 4500 cm^{-1} with a spectral resolution of 0.5 cm^{-1} .

3) MEB

The SEM was performed at the AARON KLUG CENTER for Imaging and

Table 2. Label per sample.

Label	Sample
Mou	Mouyondzi clay (raw)
Mou6	Mou heated to 600°C
Mou7	Mou heated to 700°C
Mou8	Mou heated to 800°C
GMou6	Geopolymer based on Mou6
GMou7	Geopolymer based on Mou7
GMou8	Geopolymer based on Mou8

Analysis Electron Microscope Unit at the University of Cape Town in South Africa. The device used is FEI Nova NanoSEM 230 type. The Nova NanoSEM is a high resolution field emission SEM, combining low kV imaging and analysis capabilities with unique low vacuum performance.

The preparation of the specimens and the mechanical resistance were carried out in the physics laboratory of the Société Nouvelle des Ciments du Congo (SO.NO.C.C) in Congo Brazzaville.

3. Result

3.1. Compressive Strength

The compressive strength of the samples of prismic geopolymer cement paste hardened after 28 days as a function of the calcination temperature of the clay fractions is shown in **Figure 2**.

It is observed that the compressive strength increases with the curing temperature of the clay. This increase is attributed to the increase in the amorphous phase in the aluminosilicate material [5] [6]. It is evident that when the dehydroxylation of kaolinite clays takes place, the structure of the resulting metakaolinite is in a state of disorder which increases with temperature up to 800°C. The maximum of this disorder is observed at 800°C. The increase in disorder leads to better characteristics of the geopolymer product [6]. Calcination of kaolinite clays above 800°C decreases the tendency for disorder in the structure of metakaolinite, giving rise to another crystalline phase [7].

3.2. Microstructure

The SEM images of geopolymer mortar GMou6, GMou7 and GMou8 are presented in **Figure 3**.

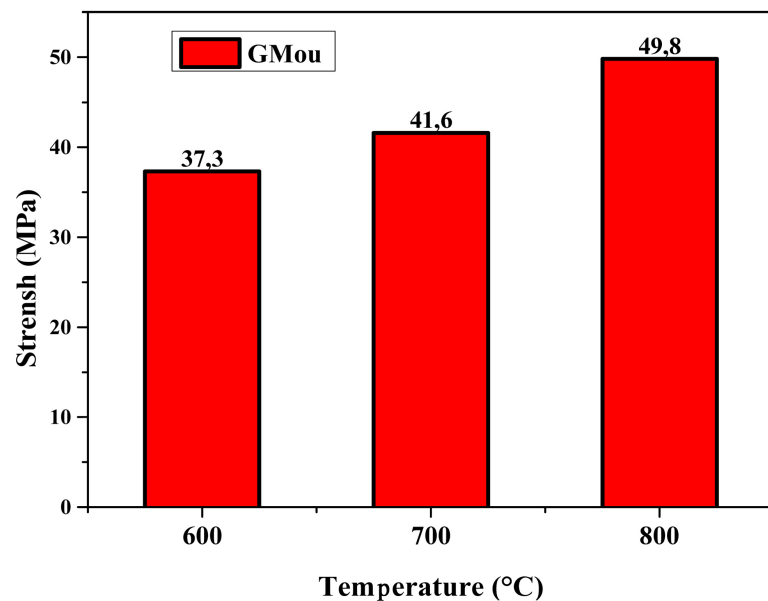
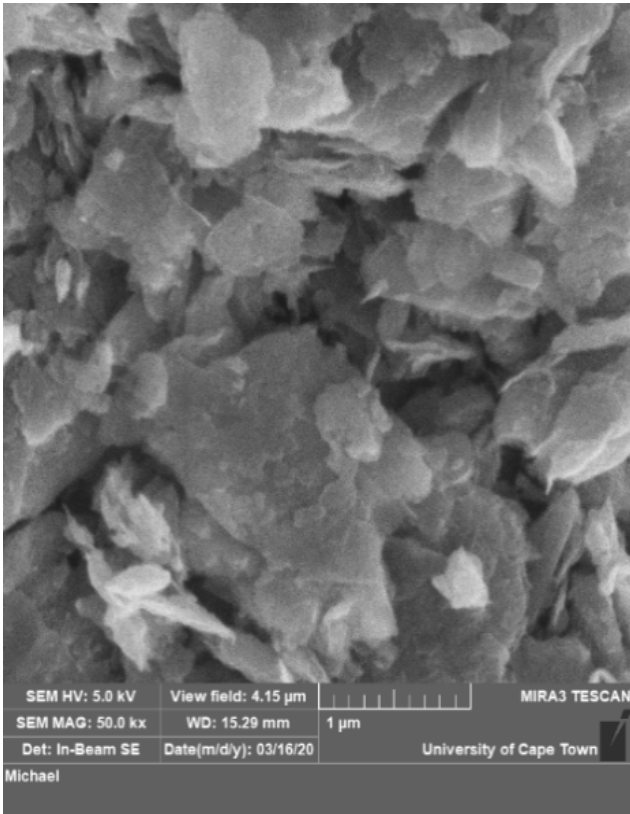
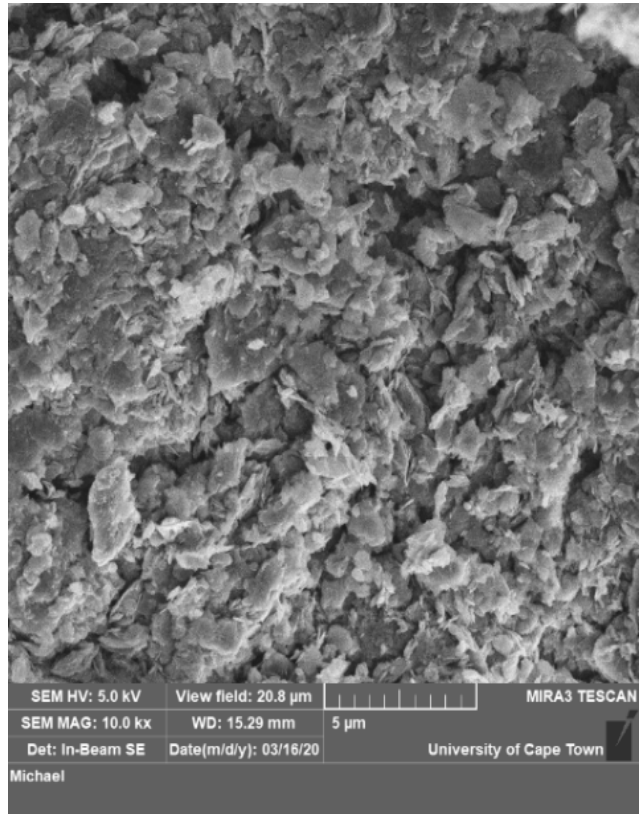


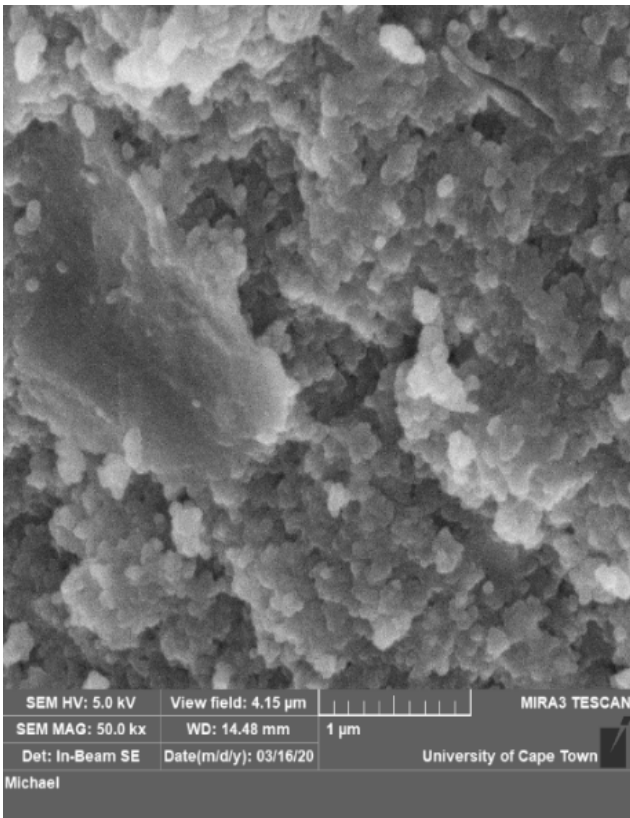
Figure 2. Compressive strength of geopolymer mortar.



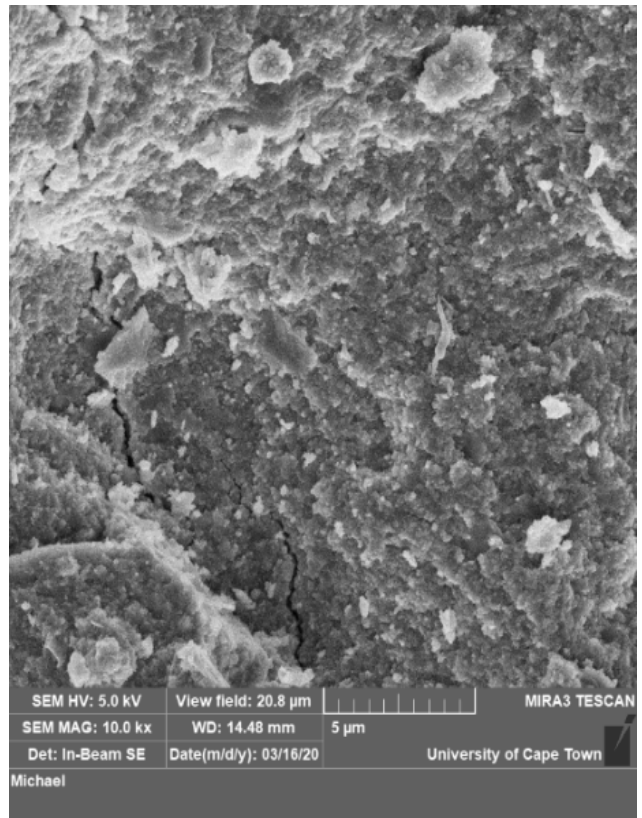
(a)



(b)



(c)



(d)

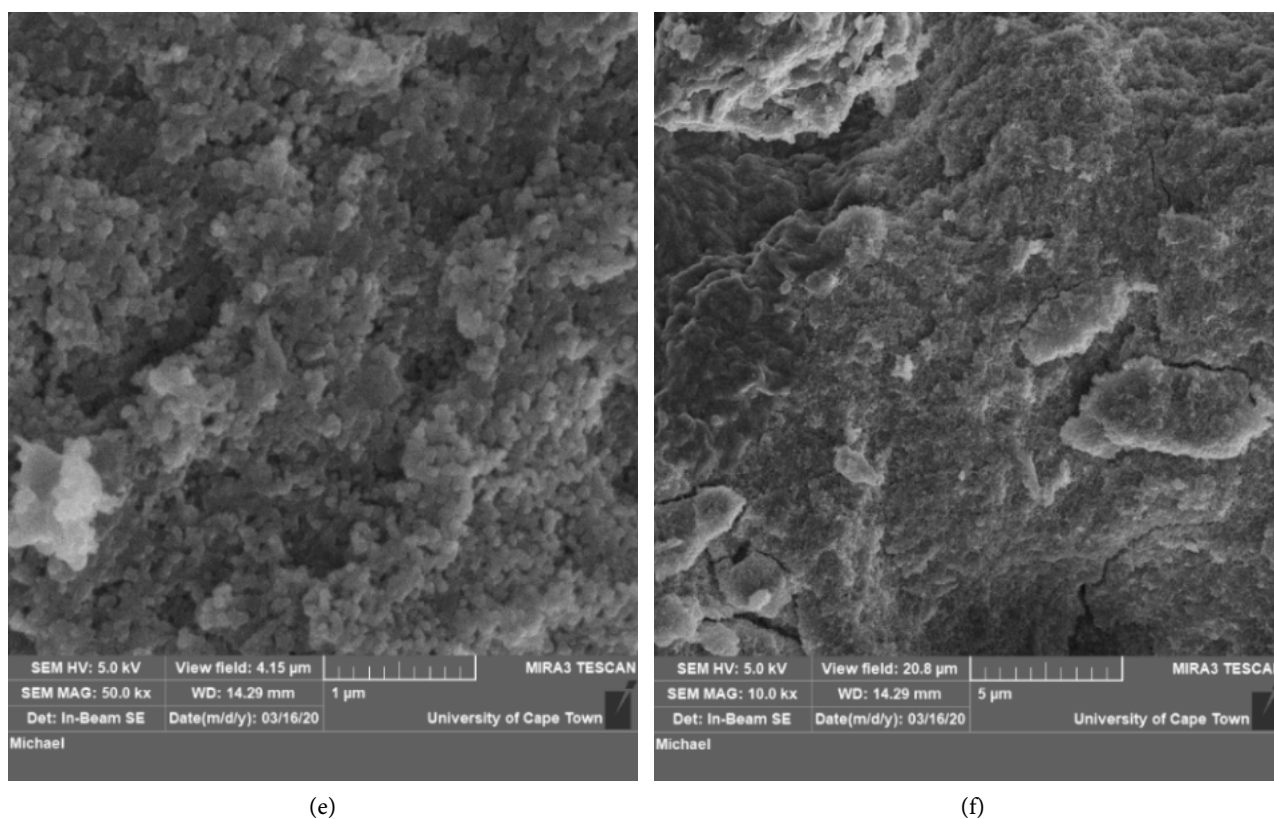


Figure 3. Micrograph of GMou6, GMou7 and GMou8. (a) GMou6 1 μm ; (b) GMou6 5 μm ; (c) GMou7 1 μm ; (d) GMou7 5 μm ; (e) GMou8 1 μm ; (f) GMou8 5 μm .

On these images, we observe undissolved particles associated with binding phases of amorphous aluminosilicate type characteristic of geopolymers [7].

In **Figure 3(a)** and **Figure 3(b)**, the image appears as an assembly of aggregates with high porosity. **Figure 3(c)** and **Figure 3(d)** shows continuous structures with some undissolved particles, indicating low porosity compared to others.

These continuous structures are of amorphous aluminosilicate type material characteristic of a geopolymer [8]. The cracks observed are caused by the impact of the mechanical study.

In **Figure 3(e)** and **Figure 3(f)** there is a considerable decrease in undissolved particles, some of which are visible in **Figure 3(e)**.

The SEM results indicate the formation of a new product with a structure different from that of metakaolin. These observations are in agreement with the results of the compressive strength because the strong presence of undissolved particles and the gathering in aggregates of GMou6 makes it less resistant than the other matrixes. GMou7 has a lower level of geopolymerization than GMou8 due to the fact that GMou7 carries more undissolved particles therefore not having reacted, so it is less resistant than GMou8.

3.2.1. Infra Red

Figures 4-6 show the IR spectra of the different matrices at different curing

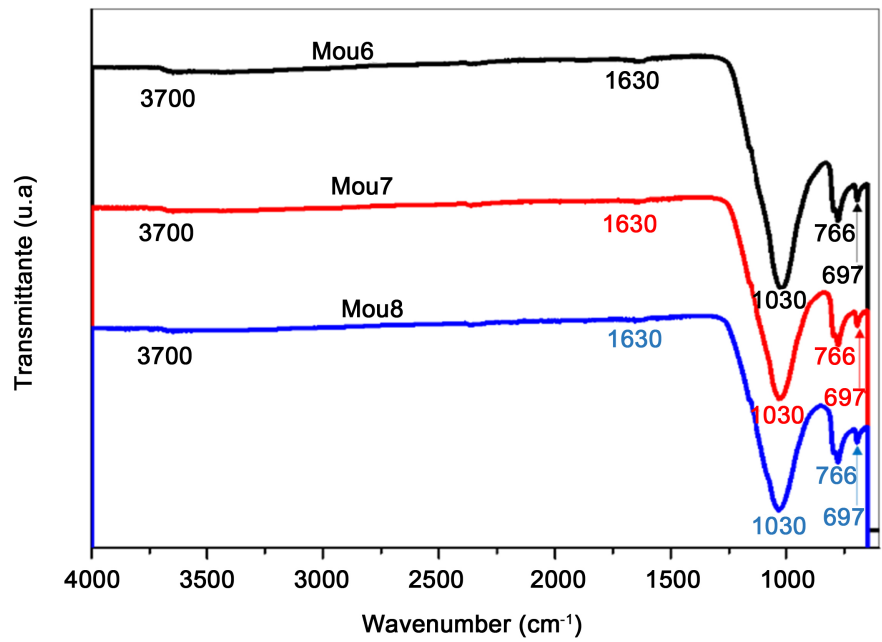


Figure 4. IR spectrum of geopolymers obtained from GMou6 GMou7 GMou8.

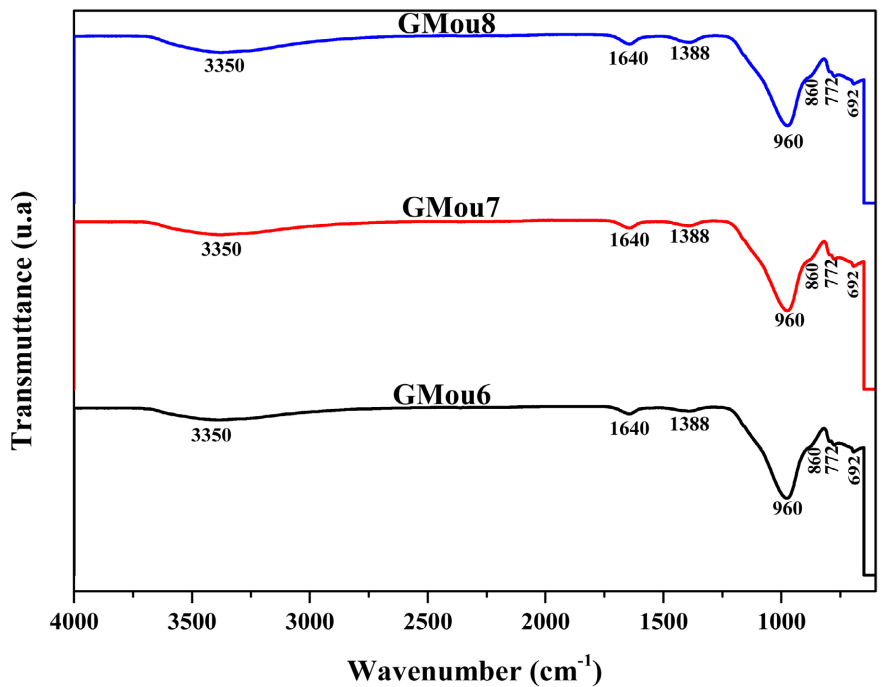


Figure 5. IR spectrum of geopolymers obtained from GMou6 GMou7 GMou8.

temperature, *i.e.* calcined clays, geopolymers hardened after 28 days of age depending on the curing temperature and the raw clay.

Recall that the IR spectrum of raw clay is characterized by different vibrations attributed to the Si-O, Al (VI)-OH and Si-O-Al bonds of kaolinite and quartz [8] [9]. Frequencies from 3700 to 3100 cm⁻¹ are assigned to the stretching mode of Al (VI)-OH bonds, the range of bands from 1200 to 600 cm⁻¹ are attributed

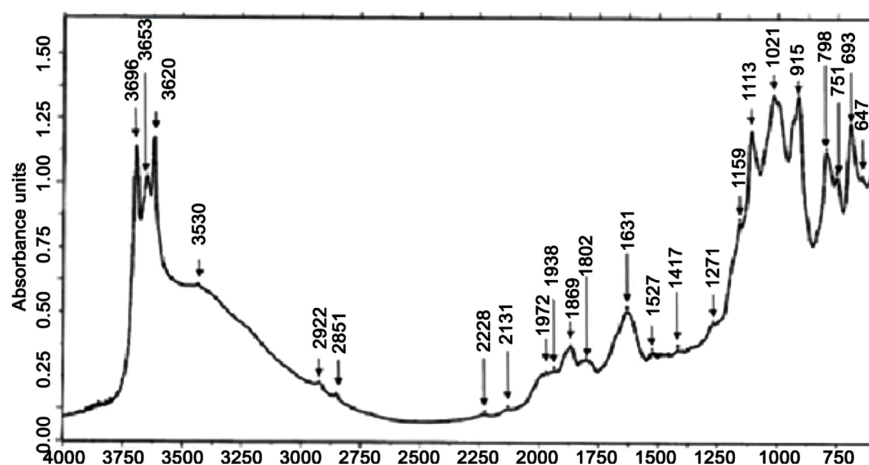


Figure 6. IR spectrum of Mo [4].

to frequencies related to the mode of deformation of the Al (VI)-OH bond of kaolinite. The Si-O bond elongation vibration mode is observed by a band at 1021 cm^{-1} . The presence of water is characterized by the strong bands at 3420 and 1631 cm^{-1} . Quartz is manifested by bands at 798 cm^{-1} (stretching mode of Si-O) and at 696 cm^{-1} (straining mode of Si-O) [4] [8].

The IR spectrum of metakaolinite (Figure 4) shows the disappearance of the characteristic bonds of kaolinite from 3700 to 3100 cm^{-1} and the band range from 1200 to 600 cm^{-1} [6] [8] [10] attributed respectively to the mode Al-OH bond stretching and elongation. The vibrations attributed to the mode of elongation of Si-O have also disappeared. The characteristic bands of hygroscopic water are greatly reduced and are marked by the vibrations of stretching and deformation of O-H and H-O-H groups [9] [10]. The band at 1021 cm^{-1} in the IR spectrum of raw clay corresponding to the Si-O-Si or Si-O-Al bond has become wider in the spectrum of calcined clays, which is explained by the decrease the crystallinity of kaolinite during calcination and metakaolin formation [8] [9] [10].

In IR spectra of geopolymers, a broad band is observed between 3000 and 3600 cm^{-1} with a maximum at 3360 cm^{-1} , a band at 1650 cm^{-1} and 1390 cm^{-1} attributed to OH stretching and bending vibrations of HOH water molecules absorbed or trapped on the surface in the large cavities of the geopolymer framework [6] [8].

The biggest difference between raw clay, calcined clay and geopolymers is the asymmetric stretch vibration bands of Si-O-Si and Al-O-Si. The band at 1021 cm^{-1} on raw clay and at 1030 cm^{-1} on calcined clay correspond to the asymmetric stretching mode of Si-O in tetrahedron and this band is slightly offset at 980 cm^{-1} on geopolymers [9]. This band is characteristic of the formation of aluminosilicate gel. The 860 cm^{-1} band present only on geopolymers is attributed to the bending vibration of Si-OH. The presence of this bond in geopolymeric products causes a decrease in the degree of polycondensation reaction thus a reduction in mechanical strength. The intensity of this band is very weak in our

case. This confirms a good aluminosilicate polycondensation reaction [8].

The bands at 915 cm^{-1} , 798 cm^{-1} and at 751 cm^{-1} on the raw clay spectrum; 766 cm^{-1} on calcined clay are attributed respectively to the vibration stretch of Al (VI)-OH and Al (VI)-O these bands underwent a shift on the IR spectrum of geopolymers after polycondensation. The 692 cm^{-1} band found on geopolymers, around 697 cm^{-1} on calcined clays, is attributed to the symmetrical stretching of Si-O [7].

3.2.2. X-Ray Diffraction

Figure 7 recalls the DRX spectrum of raw Mouyondzi clay. **Figures 8-10** give the XRD of geopolymer paste powders cured after 28 days of age.

Analysis of the diffractogram of the raw clay shows the presence of the following

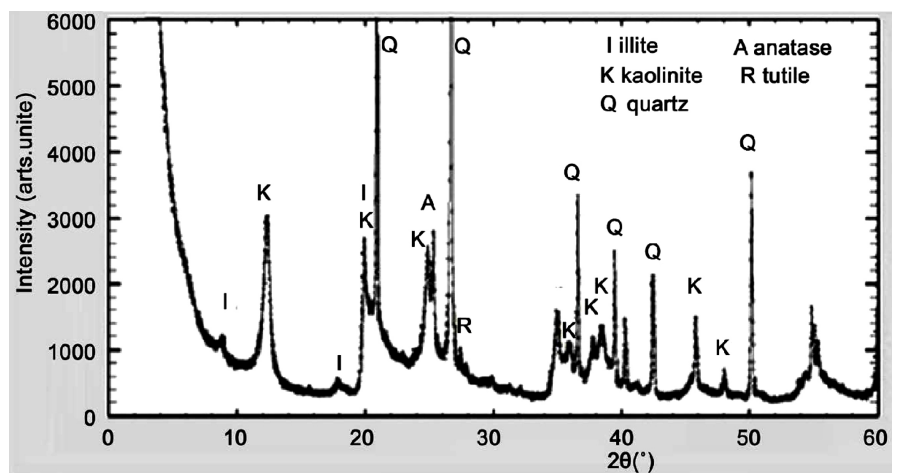


Figure 7. XRD spectrum of Mou [4].

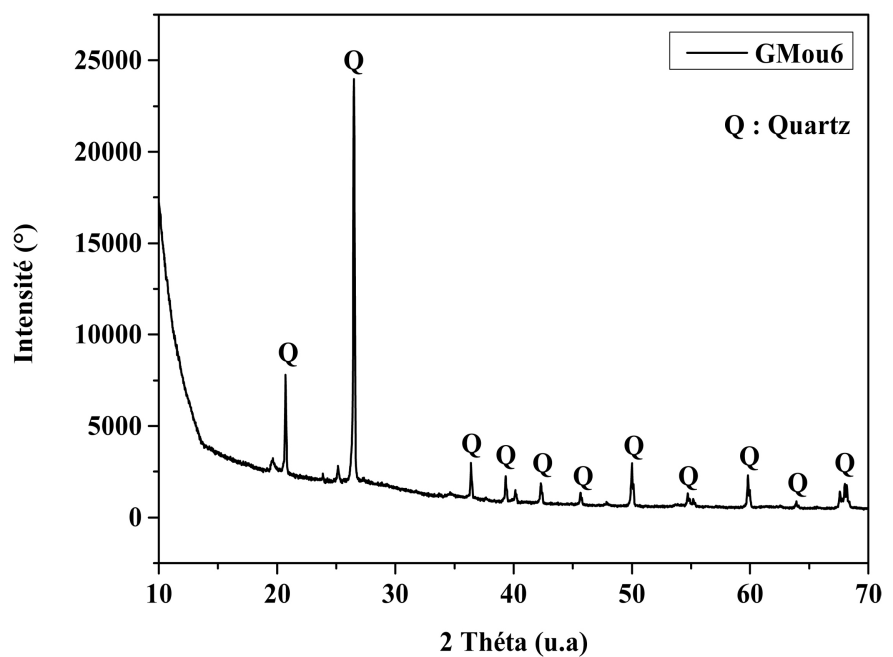


Figure 8. XRD spectrum of GMou6.

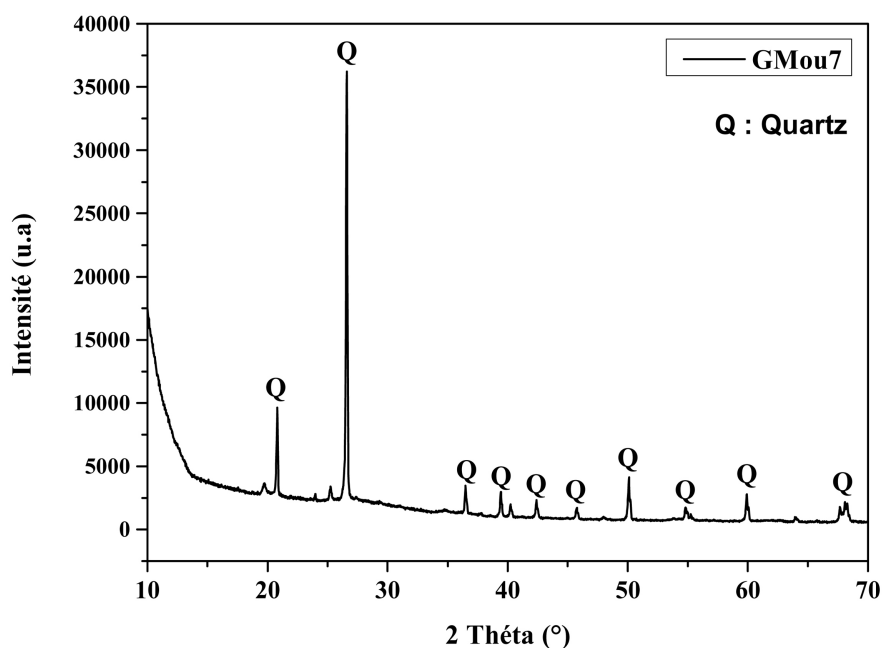


Figure 9. XRD spectrum of GMou7.

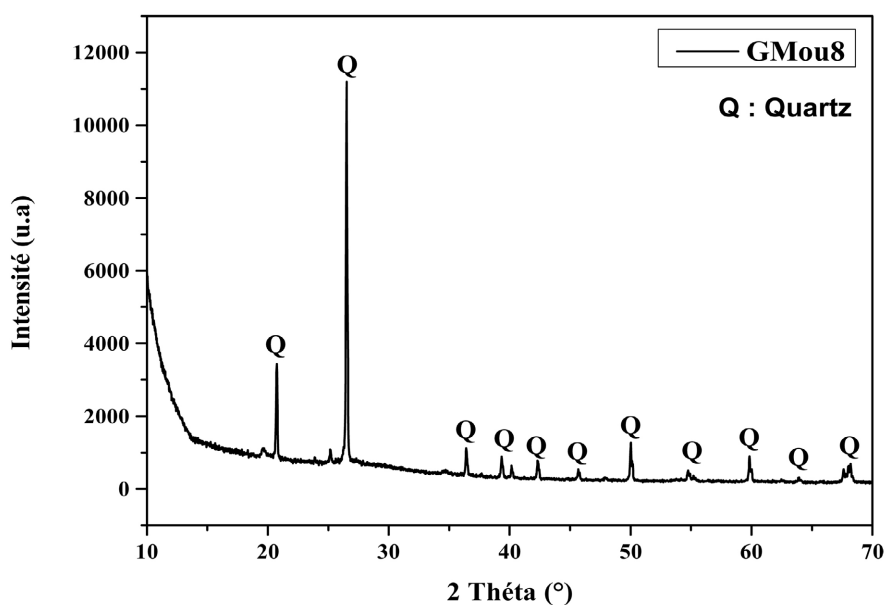


Figure 10. XRD spectrum of GMou8.

main minerals: Illite, quartz and kaolinite. Next to that we also have anatase and rutile which turn out to be in very low quantities. The main clay species of Mou is kaolinite which is in a high state of disorder. The transformation of kaolinite begins at 450°C according to the results of thermal analysis of this clay [4]. This allowed the cure to start at 600°C to maximize the degree of amorphization.

Analysis of the diffractograms of GMou6, GMou7 and GMou8 revealed the disappearance of the peaks characteristic of clay minerals, in particular kaolinite. Quartz is the only mineral species crystallized in these geopolymers. This phase

constitutes the undissolved phase of the geopolymers. This result shows us that at 600°C the kaolinite is no longer present, it has completely amorphized into metakaolinite, a very reactive phase and a search for geosynthesis. The halo dome located between 20°C and 40°C for geopolymer-type materials is not intense enough due to the process of eliminating background noise after recording diffractograms [8].

4. Conclusions

The use of metakaolinite as the source of aluminosilicate in this study provided the following confirmations:

- The use of raw clay did not allow polycondensation of tetrahedron SO_4 and AlO_4 in geopolymer due to insufficient degree of disorder.
- The calcination technique (very slow at 1°C/min) adopted in this study leads to a high amorphization rate from 600°C.
- The compressive strength of geopolymer pastes hardened after 28 days increases with the clay processing temperature between 600°C and 800°C. Over the temperature range studied, the percentage of metakaolinite increases until it reaches a peak at 800°C.
- SEM results reveal the presence of undissolved unreacted particles, which consists entirely of quartz and a gel phase presented as a continuous plate. This structure is characteristic of geopolymer-type materials; it is sharper for clay calcined at 800°C. The temperature of 800°C is the best temperature for the synthesis of geopolymers from Mouyondzi clay.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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