Influence of Thermal Activation and Silica Modulus on the Properties of Clayey-Lateritic Based Geopolymer Binders Cured at Room Temperature

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Abstract
In this study, a laterite soil which is a locally available material in many parts of the world was used as the aluminosilicate precursor. The main objective of this study is to investigate the effect of calcination temperature on physicochemical properties of the resulting geopolymers synthesized from calcined laterite soils. In order to produce the geopolymer binders, the laterite soil was activated thermally through calcination (from 550 to 750 °C) and the resulting calcined laterite was activated with an alkali activator composed of 8 and 10 M of sodium hydroxide (NaOH) and sodium silicate (Na2SiO3) in mass of 0.5. Then, the calcined laterite soils and synthesized geopolymer products were analyzed using X-ray diffraction (XRD), Brunner-Emmet-Teller (BET), Fourier Transform Infra-Red (FTIR), X-Ray Fluorescence (XRF), thermogravimetry (TG), scanning electron microscopy (SEM/EDX), and differential scanning calorimetry (DSC). The results from this study indicate that increasing the calcination temperature from 550 to 750 °C resulted in the transformation of phases and an increase in the reactivity of the laterites, resulting in material with improved properties. The use of laterite calcined at 750 °C and activated with 8 M NaOH solution resulted to an increase in the 28 days compressive strength by 35.3 MPa when compared to laterite calcined at 550 °C. Increasing the concentration of the NaOH solution was also found to yield higher material performance. Microstructural investigations showed a heterogeneous compact and dense structure resulting from high polycondensation much pronounced with the rise of calcination temperature from 550 to 750 °C.

Keywords Laterite soil · Calcination temperature · Silica modulus · Setting time · Mechanical properties · Microstructure

1 Introduction
Lateritic soils are formed in tropical and subtropical regions around the world and are mainly composed of aluminum, silicon and iron oxides as major elements [1, 2]. In parts of the world where lateritic soils are widely available, they are usually mixed with Portland cement in certain proportions to produce compressed stabilized earth blocks (CSEBs) for building houses or pavements [3–5]. These lateritic soils are formed from the alteration of kaolinite by iron minerals in which some aluminum atoms occupying the octahedral site are replaced by iron atoms from iron minerals like ilmenite,
goethite and hematite [6, 7]. The chemical composition of laterite makes it a suitable locally available material to be utilized as the aluminosilicate precursor in the synthesis of geopolymers [7].

Geopolymers are alkaline binders obtained from a mixture of aluminosilicate powder(s) with an alkaline solution at relatively low temperature (≤100 °C) [8–10]. This new cementitious materials have high strength [11], good durability [12] and fire-resistance properties [13]. Compared to ordinary Portland cement, it opens a pathway for the utilization of large volumes of various wastes/by-products such as fly ash [14], slag [15], construction and demolition waste [16–19] which are the major aluminosilicate precursor used in the synthesis of geopolymers. However, with varying availability of the conventional precursors (i.e. fly ash and slag) in various parts of the world, the use of locally available laterite soils as precursors would result in more sustainable and economical development of geopolymers.

Due to their local availability and large quantities, increasing interest was recently observed in the use of lateritic soils as precursors in the synthesis of geopolymers [2, 20, 21]. The main studies in this area indicated that, the characteristics of the end products are governed by the synthesis conditions and the starting material composition and pre-treatment. For instance, Lemougna et al. [22] utilized raw clayey laterite subjected to heat at temperatures ranging between 100 and 550 °C. The findings from the study showed that raw clayey laterite heated at a temperature of 450 °C yielded the highest strength. A recent study by Kaze et al. [7] showed that the use of calcination temperature of 500 °C for laterite soils resulted in the highest flexural strength of geopolymer synthesized with 8 M of alkaline solution. Lassinantti et al. [23] used alkaline solution (composed of 9.5 NaOH:10.7 3SiO₂Na₂O:29.8 H₂O) and phosphoric acid (at different concentration, 44, 36 and 28 wt.%) to produce geopolymer binder by using iron-rich laterite calcined at 700 °C. The results of the study indicated that the resulting materials are suitable for various construction applications.

It was evident from these previous studies that the thermal activation conditions of the precursor, composition of the activator and the curing conditions used would influence the resulting properties of the laterite-based geopolymer. Thus, understanding the influence of these factors on the properties of laterite based geopolymers would aid the valorization of lateritic soils through geopolymer technology. Hence, this study aims to valorize lateritic soils available in Cameroon through geopolymer science as a construction material for various infrastructural development applications.

In this study, the influence of the thermal activation temperature of the laterite and the effect of the composition of the activator on the properties of geopolymer binders was investigated. The thermal activation temperatures investigated are 550, 600, 650, 700 and 750 °C alongside sodium hydroxide concentration of 8 and 10 M was used to determine the influence of the silica modulus on the properties of the geopolymer binders. The resulting properties of the geopolymer binders were evaluated in terms of the mechanical and durability properties. The influence of the thermal activation temperature on the laterites was also carried out. The phase composition of the geopolymer binders was investigated using Fourier infrared spectroscopy (FTIR), X-ray diffractometry (XRD) and scanning electron microscopy (SEM). The mechanical and physical properties were obtained by means of flexural and compressive strengths, water absorption, bulk density and apparent porosity. The obtained results are interpreted by correlating the calcination temperature, alkaline activator, microstructure and mechanical strength. The findings presented in this paper will contribute to enrich the limited available literature from geopolymer materials using laterite soils. It is also hoped that the results presented in this study would open a pathway to produce low cost and sustainable infrastructures.

2 Materials and Methods

2.1 Materials

The raw lateritic soil as shown in Fig. 1a was collected in a huge deposit located at Ngoa Ekelle region of Yaoundé, Cameroon. The raw laterite soil (LA) has a surface area of 21.4 m²/g and the chemical composition is presented in Table 1. After collecting the raw lateritic soil, it was oven dried for 48 h to remove moisture and then ball milled as shown in Fig. 1b.

Fig. 1 Raw laterite soil a before collection and b after milling, sieving and calcination.
The obtained powders were sieved through a mesh of 80 μm and then heated at various temperatures (i.e. 550, 600, 650, 700 and 750 °C) for 4 h in a programmable electric furnace according to previous work [24]. The calcined lateritic soils were denoted LAC550, LAC600, LAC650, LAC700 and LAC750, where 550, 600, 650, 700 and 750 represent the calcination temperatures used for each powdered sample.

The alkaline solutions used in this study were prepared from a mixture of 8 and 10 M of sodium hydroxide (NaOH) with commercial sodium silicate solution (Na₂SiO₃). The NaOH used was in form of pellets and were dissolved in distilled water to obtain the desired concentrations (i.e. 8 and 10 M). The commercial Na₂SiO₃ was composed of 14.4% Na₂O, 29.5% SiO₂ and 56.1% H₂O.

### 2.2 Composition and Preparation of Geopolymer Binders

All geopolymer binders are made with a NaOH to Na₂SiO₃ mass ratio of 0.5 and a solid to liquid ratio of 0.8 were selected from the findings of Kaze et al. [25]. As mentioned earlier, 8 and 10 M NaOH concentrations were used according to previous work [26]. For each geopolymer binder, the calcinated laterite was mixed with the alkali solution for 5 min. When the mixing was completed, the fresh properties of the mixtures (i.e. setting time) were evaluated followed by placing the fresh mixtures in various moulds for the tests to be carried out. The surface of the moulds was covered with plastic bags to prevent loss of moisture from the samples. After approximately 24 h, all samples were demoulded and cured at laboratory environment conditions until the testing ages. The geopolymer binder mixtures were named considering the precursor calcination temperature and the NaOH concentration used. For instance, GP600-8 and GP700-10 represents geopolymer binder mixtures made with LAC600 alongside 8 M NaOH and mixture made with LAC700 alongside 10 M NaOH, respectively.

| Table 1 Chemical composition of raw laterite (L.O.I = loss on ignition) |
|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| Compound (wt.%) | LAT  | LAC550 | LAC600 | LAC650 | LAC700 | LAC750 |
| Fe₂O₃           | 13.2 | 17.23  | 17.40  | 17.80  | 17.90  | 17.91  |
| SiO₂            | 44.02 | 46.63  | 47.13  | 47.50  | 47.64  | 47.68  |
| Al₂O₃           | 29.1  | 30.2   | 31.20  | 31.40  | 31.54  | 31.60  |
| TiO₂            | 2.03  | 1.86   | 1.98   | 1.97   | 1.96   | 1.90   |
| V₂O₅            | –     | –      | –      | –      | –      | –      |
| P₂O₅            | 0.23  | 0.23   | 0.21   | 0.20   | 0.18   | 0.14   |
| Cr₂O₃           | 0.06  | 0.05   | 0.05   | 0.03   | 0.02   | 0.02   |
| K₂O             | 0.1   | 0.1    | 0.1    | 0.04   | 0.05   | 0.03   |
| Na₂O            | 0.05  | 0.04   | 0.04   | 0.04   | –      | 0.05   |
| L.O.I           | 11.33 | 7.02   | 5.24   | 4.45   | 3.55   | 2.45   |

### 2.3 Characterization Methods

The reactivity of the calcined iron-rich laterite powders (i.e. LAC550, LAC600, LAC650, LAC700 and LAC750) was performed using procedures similar to that of Kaze et al. [27]. Three (3) grams of each calcined laterite soil powder (at 550, 600, 650, 700 and 750 °C) was poured into a polypropylene container containing 35 mL of sodium hydroxide (NaOH) 8 M solution. Afterwards, the different samples contained in the beaker were preheated for 30 min with an electric heating plaque at 20 °C. The mixes were then stirred at 1500 rpm for 6 h at 20 °C using a centrifuge. The centrifuge was used to separate the residue and the left solution. Each sample was filtered using a 0.50 mm paper filter. Before carrying out the main elemental composition some drops of nitric acid were added to lower the filtrate pH below 2 based on the observation. For each sample, the elemental composition in the obtained filtrate after the dissolution was analyzed using inductively coupled plasma optical emission spectroscopy (ICP-OES) equipment Optima 2000 DV (Perkin Elmer, Waltham, MA, USA). This analysis would give the percentage of dissolved Si, Al and Fe from each calcined laterite soil powders.

The initial and final setting times of the fresh geopolymer binders were evaluated by using a Vicat apparatus in accordance with EN196-1 [28]. The initial and final setting times were measured on the fresh pastes of each calcined laterite (at 550, 600, 650, 700 and 750 °C) geopolymers using Vicat apparatus. The initial setting is verified at 10 min just after the preparation of the paste and any minute when the first resistance to Vicat is detected. This was done in the laboratory with 54% of relative humidity and temperature of 21 ± 2 °C. The needle used was 1.00 ± 0.005 mm in diameter.

Prismatic samples with dimensions of 40 mm × 40 mm × 160 mm were used for the mechanical properties' evaluation in accordance with EN 196-1 at 7 and 28 days.
The test shall always be carried out transversely to the direction of filling. The faces of the specimens to which the load is applied shall be ground or primed. Prismatic specimens are subjected to a bending moment by applying a load by means of upper and lower rollers. The maximum load recorded during the test is noted and the bending strength is calculated. The bending strength results of the 40 × 40 × 160 mm prismatic specimens are derived from the average of three (03) specimens.

\[ F_{cf} = \frac{3}{2} \times \frac{F_l}{d_1 \times d_2} \]

Where:

- \( F_{cf} \) Flexural strength in MPa
- \( F \) Maximum force in N.
- \( l \) Span in mm.
- \( d_1 \) and \( d_2 \) Lateral dimensions of the sample in mm, where \( d_2 \) is the height and \( d_1 \) is the width.

The test on cubes or prisms shall always be carried out transversely to the direction of filling. The faces of the specimens to which the load is applied shall be ground or primed with mortar if they are not flat or parallel. The priming shall be as thin as possible and shall not burst during the test. The half-sheets from the flexural strength tests are used for the compressive strength tests. These compressive strength results are obtained from the average of six (06) samples.

\[ F_C = \frac{F}{A} \]

Where:

- \( F_C \) Compressive strength in MPa
- \( F \) Maximum force in N.
- \( A \) Cross-sectional area on which the compressive force is exerted in mm.

The water absorption, bulk density and porosity were obtained in accordance with test procedures in ASTM C 373 [29]. The rate of water penetration by capillary suction into the geopolymer binders were measured by sorptivity test in accordance with ASTM C 642 [30].

Infrared spectroscopy measurements were performed on a Thermo Fisher Scientific Nicolet 380 instrument in transmission mode, operating in attenuated total reflectance (ATR) mode using a diamond crystal. The evaluation was done on powders obtained from the raw laterites, calcined laterites and hardened geopolymer binder samples. The FTIR spectra were recorded over the range 4000 to 400 cm\(^{-1}\) at a resolution of 4 cm\(^{-1}\) with 64 scans.

X-ray diffraction (XRD) was used to identify the phases in the precursor and the resulting geopolymer binders. A Bruker D8 Advance X-ray diffractometer with Cu\( K\alpha \) radiation (\( \lambda_{Cu} = 1.54056 \) Å, without monochromator) at a step scan of 0.02 ° with a time counting per step of 0.45 s operated at a voltage of 40 kV and an electric current of 40 mA was used. The sample was rotated during data collection at 15 rpm in order to increase the particle statistics. The diffractometer was equipped with an energy-dispersive LYNXEYE XE-T detector for the filtration of fluorescence and \( K\beta \) radiation.

Fractured samples obtained after the mechanical testing of the geopolymer binders were examined using a JEOL IT300LV scanning electron microscope (SEM) coupled with EDS. The acceleration voltage used was 40.0 kV. The samples were coated with a thin platinum layer to improve the surface conductivity.

Thermal analysis was carried out using an SDT Q600 from TA instruments. The material analysis was carried out under the following conditions: between 30 and 900 °C at a speed of 5 °C/min for 15 min; about 30 mg was introduced into a platinum crucible under air flushing. The sample and the reference were placed in two identical platinum crucibles.

The specific surface area of unheated and calcined laterites was determined according to the Brunauer–Emmet–Teller (B.E.T) method based on nitrogen absorption (on a Micromeritics GEMINI 2360 instrument). The particle size distribution (cumulative and density distribution) was determined with Malvern instruments Ltd. (Mastersizer 2000 Ver. 5.22). For the granulometry analysis, the experimentation was performed under ultrasonic condition to compare with those done without ultra-sound.

### 3 Results and Discussion

#### 3.1 Characterization of Raw Materials

The FTIR spectra of the raw laterite and calcined laterites are presented in Fig. 2 in the range 4000 - 300 cm\(^{-1}\).

The FTIR spectra are made up of bands related to hydroxyl groups (4000 - 3500 cm\(^{-1}\)) and Si-O-Al bonds (1100 - 950 cm\(^{-1}\)) which are the characteristic peaks of the clayey minerals. The bands located at 1009, 1032 and 1116 cm\(^{-1}\) are related to the Si-O-Al and Si-O-Fe type bonds characteristic of the presence of the kaolinite and goethite phases contained in this laterite [2, 25]. The absorption bands located at 910 and 936 cm\(^{-1}\) are related to the Al-OH type bonds [20]. Furthermore, the OH hydroxyl bands (between 3693 and 3620 cm\(^{-1}\)) are characteristic of kaolinite [2, 20, 31]. The same is true for quartz with the...
doublet whose positions are respectively located at 789 and 745 cm\(^{-1}\) [32].

The infrared spectra of the calcined laterites show a disappearance of the characteristic bands of kaolinite, such as OH hydroxyl bonds between 3693 and 3620 cm\(^{-1}\) and Si-O-Al (750 cm\(^{-1}\)) and Al-OH (910 and 936 cm\(^{-1}\)) type bonds compared to the spectra of uncalcined laterite (LA). This disappearance is likely due to the dehydroxylation of kaolinite mineral into amorphous metakaolin after heating from 650 to 750 °C, this was further confirmed by Fig. 5 representing the XRD analysis. The absorption bands appearing at the interval of 789-745 cm\(^{-1}\) show the persistence of illite and quartz respectively [33]. The characteristic bands of the Si-O-Si links located respectively at 1009 and 1032 cm\(^{-1}\) in a Q\(^2\) and Q\(^3\) configuration seem to transform into a single wider band located around 1116 cm\(^{-1}\) characteristic of Q\(^4\) type silicon sites. This correlates with the formation of amorphous metakaolinite which results in the transition from the Q\(^3\) tetrahedral environment of silicon to a Q\(^4\) type environment corresponding to sites characteristic of silica [32, 34]. The change in the Si-O band and the disappearance of the Si-O-Al band suggest a distortion of the tetrahedral-octahedral layers and therefore suggests some amorphization within the material.

Figure 3 exhibits the micrographs of raw (LA) and calcined laterite soils (LAC550, LAC600 and LAC750). It is noticed that increasing the calcination temperature from 25 to 750 °C led appear with less coarse particles on micrographs. This indicates that applying the thermal activation allows the dehydroxylation of compounds such as goethite and kaolinite into hematite and metakaolinite, respectively, resulting in formation of amorphous phase due to physicochemical transformation occurring during the calcination process. Hence the formed fine particles observed on different calcined laterite samples would be beneficial during the geopolymerization reaction, because the fine particles have a large surface area and could more rapidly dissolve in an alkaline solution. With this kind of particles one can expect a completely reacted solid phase, with a dense microstructure and good mechanical properties of the end products.

Figure 4 highlights the particle size distribution of raw and calcined laterite at different temperature (550, 600, 650, 700 and 750 °C). The average d\(_{10}\), d\(_{50}\) and d\(_{90}\) particle size distribution were 4.01, 40.76 and 122.43 μm; 3.39, 34.85 and 106.20 μm; 1.19, 14.72 and 78.29 μm; 2.61, 24.08 and 64.94 μm; 2.25, 19.74 and 61.91 μm; 2.01, 22.13 and 64.68 μm respectively for raw laterite LA and calcined laterites LAC550, LAC600, LAC650, LAC700 and LAC750. Whereas the slightly increase of particle sizes d\(_{0.5}\) and d\(_{0.9}\) observed on sample calcined at 750 °C could be justified by the beginning sintering of particles which became coarse. The reduction in particle size with the rise of calcination temperature is likely due to the dehydroxylation of goethite and kaolinite allowing the formation of fine particles calcined laterite powders at different temperatures. Whereas the specific surface area increases with the rise of calcination temperature. The values of specific surface areas are 21.40, 23.42, 25.32, 28.41, 29.51 and 30.41 m\(^2\)/g. the increase of specific surface area quite matches with the progressive formation of reactive or amorphous phase during the calcination process. This trend is in agreement with the previous studies carried out on the thermal activation of corroded laterite [7].

Figure 5 presents the main minerals in raw (LA) and calcined laterite soils (LAC550, LAC600, LAC650, LAC700 and LAC750). From Fig. 5, it can be noticed that without calcination, the identified reflections peaks of kaolinite (\(\text{Al}_2\text{Si}_2\text{O}_5(OH)_4\)), PDF# 01-083-4643), quartz (\(\text{SiO}_2\), PDF# 00-046-1045), illite ((K, H\(_3\)O)(Al, Mg, Fe\(_2\))(Si, Al\(_4\))O\(_{10}\)(OH\(_2\))(H\(_2\)O)), PDF# 04-017-0523), anatase (TiO\(_2\), PDF# 00-021-1272) and hematite (\(\text{α-Fe}_2\text{O}_3\), PDF # 04-003-2900) were observed on diffractogram of the raw laterite. For calcined laterites at 550 and 600 °C, it can be observed that the decrease in reflection intensity peaks of kaolinite mineral on their diffractograms suggesting their non-total transformation. Whereas the disappearance of the kaolinite reflection peaks from samples calcined between 650 and 750 °C as shown in Fig. 2 suggests their total transformation into metakaolinite as also evident in the FTIR analysis.

With the exception of the kaolinite mineral, the rest of the mineral phases were present in calcined laterites. In addition, increasing the calcination temperature from 25 to 750 °C in the present study increases the formation of amorphous phase content required for the geopolymer synthesis as reported by others on different clays used as solid precursors [24, 32].
30° 2θ range indicates the formation of amorphous phase after calcination which has been identified by other researchers [33, 35, 36]. This halo peak belonging to reactive or amorphous formed after thermal activation is not well pronounced as in case of calcined standard kaolin. This could be linked to intrinsic properties of this latter such as mineralogy complexity.

Figure 6 highlights the simultaneous curves of DTA and TG of raw laterite (LA). It can be observed from Fig. 6 that there exist two endothermic phenomena. The first endothermic peak located around 213 °C can be linked to the dihydrorylation of goethite into hematite. This was accompanied by a 2% weight loss recorded in the TG curve. The second one appearing at about 513 °C indicates the dehydroxylation of kaolinite into reactive metakaolinite with 9% of weight loss recorded in the TG curve. The exothermic peak located around 982 °C indicates the formation of mullite due to the structural reorganization of metakaolinite into spinel alumina [37–39].

The reactivity of the calcined laterite soils in 8 and 10 M of NaOH is presented in Fig. 7. It can be observed from Fig. 7 that the dissolution of the calcined precursors was improved by increasing the molarity of NaOH solution from 8 to 10 M as well as increasing the calcination temperature from 550 to 750 °C. It can also be noted that the chemical composition is made up of mostly Al and Si (as major) and Fe (as minor) oxides. In 8 M of NaOH as shown in Fig. 7a, indicates that the values of Al, Si and Fe were 12, 18, 2; 18, 22, 3; 23, 28, 4; 39, 42, 5; 42, 45, 5, respectively. For LAC550, LAC600, LAC650, LAC700 and LAC750 samples, application of 10 M of NaOH, reveals an increase in solubility with values; 14, 18, 2; 26, 28, 3; 30, 32, 5; 44, 42, 5; 46, 45, 5, respectively as represented in Fig. 7b. This increase quite matching with previous works showed that increasing the molarity of activator yielded higher dissolution of the precursor. Hence, increasing the calcination temperature as well as the molarity of alkaline activator is expected to increase the amorphous content and favours the high dissolution of the reactive phase when activated with an activator [26, 40–43].
3.2 Characterization of Geopolymer Binders

3.2.1 Setting Time

The initial and final setting times of the geopolymer binders in 8 and 10 M NaOH solution are presented in Fig. 8. It is evident from the results that increasing the calcination temperature resulted in a decrease in the setting times regardless of the composition of the activator. The final setting time of geopolymer binder made with 10 M NaOH reduced by 12.2, 23.9, 34.1 and 43.5% when the calcination temperature was increased from 550 to 600 °C, 650 °C, 700 °C and 750 °C, respectively. The reduction in the set times with higher calcination temperature can be associated with the increased solubility and reactivity of the laterites as reported in Fig. 7. This observation corresponds with other studies where increasing the calcination temperature of halloysite and kaolinite was found to increase the reactive or
amorphous phase within these materials resulting in lower setting times [7, 40].

Similarly, increasing the concentration of the NaOH from 8 to 10 M resulted in a decrease in the setting times. The initial setting time of geopolymer binder synthesized with laterite calcined at 550, 600, 650, 700 and 750 °C reduced by 10.4, 17.8, 18.1, 15.6, and 14.1% when the concentration of NaOH was increased from 8 to 10 M. This reduction in the setting times can be ascribed to the higher dissolution of the monomers in the precursors with a higher concentration of the NaOH. Increasing the NaOH concentration would result in an increase in the Na$_2$O content in the whole system yielding better dissolution and higher polycondensation between Al and Si oligomers [26, 38]. Hence, increasing the calcination temperature of laterite soils in this current study improved the reactivity of the laterite and increasing the NaOH concentration yielded higher dissolution of the monomers in the precursors.
ASTM C 150 [44] requires Portland cement to have an initial setting time equals to or greater than 45 min and a final setting time less than or equal to 375 min. Observing the setting times of the geopolymer binders evaluated in this study as represented in Fig. 8, it can be observed that initial setting times ranged between 110 to 183 min and the final setting times ranged between 191 to 255 min. Thus, these binders satisfy the Portland cement setting time requirements and can be used for applications where Portland cement is used as the binder provided other requirements are met.

### 3.2.2 FTIR Spectra

The infrared spectra of the consolidated calcined laterite based geopolymers binders are presented in Fig. 9. The lower absorption bands ranged between 3693 and 3694 cm⁻¹ on geopolymer GP550 and GP600 are linked to vibrational modes of O-H bonds belonging to residual kaolinite mineral. These vibrational modes are due to the total dehydroxylation at 550 and 600 °C which was observed previously by the FTIR and XRD analyses of calcined laterite powders as shown in Figs. 2 and 5. The large bands ranged between 3374 and 3396 cm⁻¹ and 3363 - 3394 cm⁻¹ and the lower ones located at 1640 - 1645 cm⁻¹ and 1640 - 1647 cm⁻¹ can be attributed to the stretching vibration of O-H of water molecules [31, 45].

By comparing the infrared spectra of calcined laterites in Fig. 2 and those of the geopolymer binders in Fig. 9, it can be observed that the important absorption bands are located at 1025, 1040, 1060, 1063 and 1064 cm⁻¹ for LAC550, LAC600, LAC650, LAC700 and LAC750, respectively. These absorption bands infrared showed that the vibrational modes of Si-O-Al bonds after geopolymerization reaction has occurred shifted to lower wavenumbers of 1001, 1005, 989, 995 and 997 cm⁻¹ for GP550, GP600, GP650, GP700 and GP750, respectively. For geopolymer binders synthesized with 10 M NaOH, the absorption bands shifted to 1003, 1005, 992, 988 and 987 cm⁻¹ for GP550, GP600, GP650, GP700 and GP750, respectively. In addition, this shift observed from infrared spectra after activation will favour the rise of concentration of Q² (2Si,2OH) as reported in literature [34, 46].

The displacement of the bands through the geopolymer reaction justifies the formation of the newly formed amorphous phase as previously reported by other studies where different aluminosilicate precursors are used [3, 21, 41]. The bands located at 864, 867, 868 and 909 cm⁻¹ in Fig. 6a and those located at 858, 862, 872, 909 and 910 cm⁻¹ in Fig. 6b can be linked to the vibrational modes of Al-O-Si, Si-O, Fe-O and Si-O-Al (Fe) bonds [47].

### 3.2.3 XRD Analysis

The X-ray patterns of the geopolymer binders evaluated are presented in Fig. 10. It can be observed from Fig. 10 that the reflection peaks of some mineral phases like quartz (SiO₂, PDF# 00-046-1045), anatase (TiO₂, PDF# 00-021-1272) and hematite (α-Fe₂O₃, PDF # 04-003-2900) detected on the X-ray diffractions of calcined laterites in Fig. 5 remained after the geopolymer reaction. This implies that these mineral phases were not involved or altered during the geopolymerization reaction. These observations correspond to that of other studies where it was observed that the mineralogical phases within the solid precursor for the geopolymer synthesis were not totally involved in the geopolymerization reaction [36, 48]. The presence of these mineralogical phases in the geopolymer matrix could be useful when these phases act as micro aggregates within the matrix.

The hump describing the formed amorphous phase after calcination of laterite appearing between 15 and 30°
(2Theta) in Fig. 5 when activated with the alkaline solution shifted to 20 and 40° (2 Theta) in Fig. 10. This displacement confirms that the geopolymerization reaction has taken place and the newly formed phases belonging to the geopolymer network [49]. The presence of kaolinite mineral in X-ray patterns of GP550 and GP600 in Fig. 5 also seen in LAC550 and LAC600 in Fig. 10 indicates that the calcination temperature of clayey laterites at both temperatures does not totally covert the kaolinite into reactive metakaolin. Thus, the use of calcination temperature higher than 600 °C is recommended for the total conversion of the kaolinite in the raw laterite to reactive metakaolin.

3.2.4 Mechanical Properties

The compressive and flexural strength of the geopolymer binders at various ages are presented in Figs. 11 and 12, respectively. It is evident from Figs. 11 and 12 that the mechanical properties of the geopolymer binders increased with age regardless of the calcination of the laterite and concentration of NaOH utilized. The compressive strengths of geopolymer binders from Fig. 11 synthesized with 8 M NaOH are 19.2, 21.3, 25.5, 27.6 and 33.80 MPa at 7 days for GP550, GP600, GP650, GP700 and GP750, respectively. At 28 days, the compressive strength of GP550, GP600, GP650, GP700 and GP750 made with 8 M NaOH increased to 29.3, 35.8, 50.4, 56.9, and 64.6 MPa, respectively. Similarly, the flexural strength at 28 days of GP550, GP600, GP650, GP700 and GP750 made with 8 M NaOH increased by 1.5, 2.1, 3.7, 4.3, 5.5 MPa, respectively compared to the flexural strength at 7 days as shown in Fig. 12. These results indicate the progression of the geopolymerization reaction with age resulting in the formation of additional products resulting in strength gain. It is evident from these results of Figs. 11 and 12 that increasing the calcination temperature and concentration of the NaOH increased the mechanical performance of the geopolymer binders. The 28 days compressive strength of
geopolymer synthesized with 8 M NaOH alongside laterite calcined at 600, 650, 700 and 750 °C is 6.5, 21.1, 27.6 and 35.3 MPa higher than that made with laterite calcined at 550 °C. This behaviour can be linked to higher calcination temperature resulting in higher reactivity of the laterites. These results are in agreement with that of Wang et al. [42] where increasing the calcination temperature from 800 to 900 °C for kaolin was found to increase the resulting compressive and flexural strength of the resulting geopolymer by 23.4 and 38.0 MPa, respectively.

The 28 days compressive strength of geopolymer made with 8 M NaOH and laterite calcined at 750 °C (LAC750) was further increased by 30.9% when the concentration of the NaOH was increased to 10 M. Similarly, the 28 days flexural strength of geopolymer synthesized with 8 M NaOH and LAC750 was increased by 28.1% when 10 M NaOH was used. The increase in the mechanical performance of the geopolymer binders with a higher concentration of NaOH can be associated with a higher content of Na₂O in the pore solution resulting in the higher dissolution of the monomers and a corresponding formation of a higher amount of geopolymer products. These results are in agreement with that of Hanjitsuwan et al. [50] where increasing the NaOH concentration between 8 to 18 M in the synthesis of fly ash-based geopolymer was found to yield higher compressive strength. Hamidi et al. [43] also reported approximately 20% increase in the flexural strength of fly ash-based geopolymer when the concentration of NaOH was increased from 8 to 10 M. Thus, the use of higher calcination temperature for laterite and concentration of NaOH is recommended in order to have geopolymer binders with higher mechanical performance.

3.2.5 SEM Analysis

The SEM images of selected geopolymer binders (i.e. GP550, GP650 and GP750) synthesized with 8 and 10 M NaOH are presented in Figs. 13 and 14, respectively. It can be observed from the figures that all geopolymer binders are homogenous, dense and compact. However, there also exist micro-fissures, open voids and pores within the matrix of the geopolymer binders. The larger width of micro-fissures observed in GP550 from Figs. 13a and 14a compared to those of GP650 as shown in Figs. 13b and 14b and GP750

![Fig. 13 SEM images of geopolymer binders synthesized with 8 M NaOH](image-url)
from Figs. 13c and 14c can be associated with the weaker structure or possible lower formation of geopolymerization products which was evident in the mechanical properties as shown in Figs. 11 and 12.

It is evident from the figures that increasing the concentration of NaOH from 8 to 10 M resulted in more refinement of the microstructure of the geopolymer binders. This behaviour is justified by the fact that increasing the concentration of the NaOH would yield higher NaO₂ content which favours the high dissolution of monomers from the precursors resulting in the formation of higher geopolymerization products. Thus, using a higher concentration of NaOH solution would favor a high dissolution of reactive or amorphous phase contained in calcined laterite soils related to an important formation of geopolymer binder that would ensure better cohesion between different components in the geopolymer matrix resulting in higher performance. These images confirmed the higher mechanical performance exhibited by geopolymer binders synthesized with 10 M NaOH as shown in Figs. 11 and 12. The sheet shapes observed on samples GP550 made with 8 M NaOH in Fig. 12a could be a result of the residual kaolinite that has been not totally transformed into metakaolinite after calcination. When GP550 was synthesized with 10 M NaOH as shown in Fig. 14a, no presence of these sheet shapes was observed.

The use of higher calcination temperature for the laterite resulted in a higher content of amorphous or reactive phases. This improvement in the reactive content of the laterites allows the extension of the geopolymer binder network required to tie other particles in the whole system leading to the formation of a strong and compact structure. Thus, increasing the reactive or amorphous content in laterite soils by applying thermal activation would ensure a good matrix. The study by Elimbi et al. [51] showed that increasing the calcination temperature from 600 to 800 °C for kaolinite clay used in the synthesis of geopolymer binders resulted in a more compact microstructure. Conclusively, increasing the calcination temperature of laterite soil, as well as the molarity of the alkaline solution is of primary importance for the development of dense and compact matrix with high mechanical performances.
Figures 15 and 16 show the elemental map indicating the distribution of elements within the matrices of GP550 and GP750, respectively consolidated with 10 M of NaOH. From Fig. 16, it can be observed that there exists a homogeneous distribution of the main elements such as Na, Si, Al, O, Fe in the micrograph of GP750 compared to that of GP550 as shown in Fig. 15. This homogeneous distribution can be linked to the higher reactivity of LAC750 compared to that of LAC550. This was also evident in the mechanical properties results where geopolymer binders synthesized with LAC600 exhibited higher performance. The X-ray analysis results from Fig. 5 also confirmed this as it showed that the use of a calcination temperature of 550 °C for raw laterite does not result in the total transformation of kaolinite into metakaolinite. Thus, the heterogeneous distribution of Na, Si, Al, O, Fe elements in the matrix of GP550 as shown in Fig. 15 can be ascribed to the low geopolymerization reaction. From this mapping, the geopolymer binders are composed of mainly amorphous sodium-polysialiate or polyferosialate [52].

3.2.6 Sorptivity Test

The sorptivity of the geopolymer binders consolidated with 8 and 10 M are presented in Fig. 17. From Fig. 17, it can be observed that there was a rapid increase in the rate of water penetration at the start of the test up to 200 min in all the geopolymer binders regardless of the calcination temperature used for the laterite or the concentration of NaOH used. Towards the end of the test, the rate of water penetration was slow and almost constant as evident in the slopes of the lines.

It is also evident from these results that the increase in the calcination temperature used for the laterites resulted in a decrease in the sorptivity of the geopolymer binders for all concentrations of NaOH. However, increasing the concentration of the NaOH from 8 to 10 M resulted in more reduction in the sorptivity of the geopolymer binders. The reduction in the sorptivity of the geopolymer binders with higher calcination temperature can be ascribed to the improvement of the reactivity of laterites. These results are in agreement with the trend of mechanical strength recorded for both geopolymer series synthesized with different concentrations of NaOH.

3.2.7 Bulk Density and Water Absorption

The bulk density and water absorption recorded on geopolymer binders consolidated with 8 and 10 M of NaOH are presented in Fig. 18a and b, respectively. For the geopolymer binders consolidated with 8 M NaOH, the bulk density is
1.29, 1.30, 1.31, 1.32 and 1.35 g/cm$^3$ for GP550, GP600, GP650, GP700 and GP750, respectively. When NaOH at a concentration of 10 M was used, the bulk density of GP550, GP600, GP650, GP700 and GP750 increased by 0.01, 0.02, 0.03, 0.03 and 0.03 g/cm$^3$, respectively. The increase in the bulk density with a higher concentration of NaOH can be associated with the increase in the dissolution of monomers from the laterites resulting in more formation of geopolymer products. This is evident from the water absorption results of Fig. 18 which indicates geopolymer binders synthesized with 10 M NaOH exhibited a lower water absorption compared to those synthesized with 8 M NaOH. The water absorption of GP550, GP600, GP650, GP700 and GP750 made with 10 M NaOH is 16.7, 14.1, 14.6, 23.5 and 23.9% lower compare to those made with 8 M NaOH.

An increase in the bulk density was also noticed when the calcination temperature increased from 550 to 750 °C. This increase in the bulk density with higher calcination temperature can be linked to the improvement of reactive or amorphous phases in the laterites resulting in better geopolymerization reaction and a corresponding dense matrix. A similar trend was observed in water absorption of the geopolymer binders as the water absorption reduced with higher calcination temperature. The reduction in the water absorption of the geopolymer binders with the increase in the calcination temperature can be ascribed to the high degree of polymerization that favoured the extension of geopolymer networks within the matrix. The formation of geopolymer products from the high degree of polymerization would ensure high connectivity between different particles and a corresponding reduction in open porosity. These observations are in agreement with the findings of Kaze et al. [40] where it was reported that increasing the calcination temperature improved the geopolymerization reaction leading to the formation of strong structures with lower porosity. Wang et al. [42] reported a 60.1% increase in bulk density when the calcination temperature of kaolin was increased from 800 to 900 °C. These results also correspond to the mechanical properties of the geopolymer binders. A linear correlation between bulk density and mechanical properties is presented in Fig. 19 while that between the water absorption and mechanical properties is presented in Fig. 20. It can be observed from Figs. 19 and 20 that there is a good linear relationship between the properties further confirming the mechanical behaviour of the geopolymer binders. Thus, increasing the calcination temperature as well as the concentration of NaOH can be used to improve the geopolymerization reaction which would favour the development of dense and strong matrices with few accessible voids and pores.
Conclusions

Locally available laterite soils were used in the production of geopolymer binders as an alternative to the conventional Portland cement. The influence of the laterite calcination temperature in the range of 550 to 750 °C and the concentration of NaOH on the properties of the geopolymer binders was investigated. The findings from this study showed that the thermal activation of laterite through calcination resulted in the transformation of the phases and an increase in the reactivity of the laterites. The use of calcination temperature higher than 550 °C is recommended as the thermal activation of laterite at 550 °C does not result in the total transformation of the kaolinite to metakaolinite.

Mechanical properties evaluation of the geopolymer binders showed that increasing the calcination temperature and concentration of NaOH resulted in higher performance. SEM images of the geopolymers confirmed the mechanical performance results as geopolymer binders made with higher calcination temperature and NaOH concentration exhibited denser and compact microstructure. The elemental composition of the geopolymer binders also showed that there is a
homogenous distribution of elements in geopolymer binders made with 10 M NaOH compared to those made with 8 M NaOH indicating the higher dissolution of monomers in the laterites.

Similar to the mechanical properties, it was also found out that increasing the NaOH concentration and calcination temperature also resulted in an increase in the bulk density and reduction in the water absorption and sorptivity of the geopolymer binders. The water absorption of geopolymers made with laterite calcined at 750 °C and 10 M NaOH is 16.86% lower compared to those made with the same laterite and 8 M NaOH.

The findings from this study have shown the viability of producing geopolymer binders with locally available laterite alongside the influence of the calcination temperature and NaOH concentration. However, to propel more applications of laterite-based geopolymers; it is recommended that more studies be carried out in evaluating the resistance of these binders in aggressive environments such as freeze-thaw, wet-dry and applications where they are subjected to abrasive forces. Future studies on the influence of various compounds in the laterites would also provide more understanding of various ways to further enhance the performance of laterite-based geopolymers for various applications.

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Declarations
This manuscript has been published elsewhere in any form or language and has not been submitted to more than one journal for simultaneous consideration.

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