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

The growing demand and use of geopolymer binders have been of high interest in recent times due to their sustainability and economic benefits. However, locally available materials must be used in the production of geopolymer binders in order to maximize their benefits. In this study, a laterite soil which is a locally available material in many parts of the world was used as the aluminosilicate precursor. In order to produce the geopolymer binders, the laterite soil was activated thermally through calcination and the resulting calcined laterite was activated with an alkali activator composed of sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3). The influence of calcination temperature and the concentration of NaOH on the properties of the developed geopolymers was investigated. Calcination temperature used ranged between 550 °C to 750 °C and the NaOH concentrations used were 8 M and 10 M. The results from this study indicate that some locally available laterite soils can be utilized in the production of geopolymer binders if they undergo calcination. Increasing the calcination temperature from 550 °C 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 compared to when laterite calcined at 550 °C was used. Increasing the concentration of the NaOH solution was also found to yield higher material performance. Microstructural investigations were also carried out to confirm the macrostructural properties.

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 earth stabilized blocks (CEBs) 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 cured at temperatures up to 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] 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, 16, 17]. 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 pretreatment. For instance, Lemougna et al. [18] utilized raw clayey laterite subjected to heat at temperatures ranging between 100°C 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 8M of alkaline solution. Lassinantti et al. [19] used alkaline solution and phosphoric acid 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°C, 600°C, 650°C, 700°C alongside sodium hydroxide concentration of 8 M and 10M 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 findings presented in this paper are anticipated to gear more research, development and application of locally available materials in the synthesis of geopolymers. 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 was collected in a huge deposit located at Ngoa Ekelle region of Yaoundé, Cameroon. The raw laterite soil (LA) has a surface area of 21.4m²/g and the chemical composition is presented in Table 1. The d₁₀, d₅₀ and d₉₀ particle size distribution were respectively 6.5 µm, 30.6 µm and 88.7 µm. After collecting the raw lateritic soil, it was oven dried for 48 hours to remove moisture and then

ball milled. The obtained powders were sieved through a mesh of 80 μm and then heated at various temperatures (i.e. 550°C, 600°C, 650°C, 700°C and 750°C) for four hours in a programmable electric furnace according to previous work [20]. 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.

Table 1
Chemical composition of raw laterite (L.O.I = loss on ignition)

Compound	Quantity (%)
Fe_2O_3	13.2
SiO_2	44
Al_2O_3	29.1
TiO_2	2.03
V_2O_5	-
P_2O_5	0.23
Cr_2O_3	0.06
K_2O	0.1
Na_2O	0.05
L.O. I	11.33

The alkaline solutions used in this study were prepared from a mixture of 8 M and 10 M of sodium hydroxide (NaOH) with commercial sodium silicate solution (Na_2SiO_3). The NaOH used was in form of pellets and were dissolved in distilled water to obtain the desired concentrations (i.e. 8 M and 10 M). The commercial Na_2SiO_3 was composed of 14.4% Na_2O , 29.5% SiO_2 and 56.1% H_2O .

2.2 Composition and preparation of geopolymer binders

All geopolymer binders are made with a NaOH to Na_2SiO_3 mass ratio of 0.5 and a solid to liquid ratio of 0.8. As mentioned earlier, 8 M and 10 M NaOH concentrations were used. For each geopolymer binder, the calcinated laterite was mixed with the alkali solution for five minutes. 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 hours, 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 8M NaOH and mixture made with LAC700 alongside 10 M NaOH, respectively.

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. [21]. The initial and final setting times of the fresh geopolymer binders were evaluated using a Vicat apparatus in accordance with EN196-1 [22]. Prismatic samples with dimensions of 40 mm x 40 mm x 160 mm were used for the mechanical properties' evaluation in accordance with EN 196-1 at 7 and 28 days.

The water absorption, bulk density and porosity were obtained in accordance with test procedures in ASTM C 373 [23]. The rate of water penetration by capillary suction into the geopolymer binders were measured by sorptivity test in accordance with ASTM C 642 [24].

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 cm^{-1} 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 $\text{CuK}\alpha$ radiation ($\lambda_{\text{Cu}} = 1.54056\text{ \AA}$, without monochromator) at a step scan of 0.02° with a time counting per step of 0.45 second operated at a voltage 40 kV and an electric current 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 $\text{K}\beta$ 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 $5^\circ\text{C}/\text{min}$ for 15min; about 30mg was introduced into a platinum crucible under air flushing. The sample and the reference were placed in two identical platinum crucibles.

3. Results And Discussion

3.1 Characterization of raw materials

The FTIR spectra of the raw laterite and calcined laterites are presented in **Fig. 1** in the range 4000 cm^{-1} – 300 cm^{-1} . The FTIR spectra are made up of bands related to hydroxyl groups (4000 cm^{-1} – 3500 cm^{-1}) and Si-O-Al bonds (1100 cm^{-1} – 950 cm^{-1}) which are the characteristic peaks of the clayey minerals. The bands located at 1009 cm^{-1} , 1032 cm^{-1} and 1116 cm^{-1} are relative 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 cm^{-1} and 936 cm^{-1} are related to the Al-OH type bonds [16]. Furthermore, the OH hydroxyl bands (between 3693 cm^{-1} and 3620 cm^{-1}) are characteristic of kaolinite [2, 16, 26]. The same is true for quartz with the doublet whose positions are respectively located at 789 cm^{-1} and 745 cm^{-1} [27].

The infrared spectra of the calcined laterites show a disappearance of the characteristic bands of kaolinite, such as OH hydroxyl bonds between 3693 cm^{-1} and 3620 cm^{-1} and Si-O-Al (750 cm^{-1}) and Al-OH (910 cm^{-1} 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 550°C to 750°C , this was further confirmed by the XRD analysis (Fig. 2).

The absorption bands appearing at the interval of 789 cm^{-1} – 745 cm^{-1} show the persistence of illite and quartz respectively [28]. The characteristic bands of the Si-O-Si links located respectively at 1009 cm^{-1} 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 [27, 29]. 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.

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°C to 750°C in the present study increases the formation of amorphous phase content requires for the geopolymer synthesis as reported by others on different clays used as solid precursors [20, 27]. The presence of halo between 15° and 30° 2θ range indicates the formation of amorphous phase after calcination which has been identified by other researchers [28, 30, 31].

The reactivity of the calcined lateritic clays in 8 M and 10 M of NaOH is presented in **Fig. 4**. It can be observed from **Fig. 4** that the dissolution of the calcined precursors was improved by increasing the molarity of NaOH solution from 8 M to 10 M as well as increasing the calcination temperature from 550°C 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 (Fig. 4a) the values 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. When applied 10 M of NaOH, it is noticed an increase in solubility and the values were 14, 18, 2; 26, 28, 3; 30, 32, 5; 44, 42, 5; 46, 45, 5, respectively (Fig. 4b). This increase quite matching with previous works that

showed increasing the molarity of activator yielded higher dissolution of the precursor. Hence, increasing the calcination temperature as well as the molarity of alkaline would expect to increase the amorphous content and favours the high dissolution of the reactive phase when activated with an activator.

3.2 Characterization of geopolymer binders

3.2.1 Setting time

The initial and final setting times of the geopolymer binders in 8 M and 10 M NaOH solution are presented in **Fig. 5**. 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°C 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 increased solubility and reactivity of the laterites (Fig. 4). 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, 35].

Similarly, increasing the concentration of the NaOH from 8 M to 10 M resulted in a decrease in the setting times. The initial setting time of geopolymer binder synthesized with laterite calcined at 550°C, 600°C, 650°C, 700°C 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 M 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 Na₂O content in the whole system yielding better dissolution and higher polycondensation between Al and Si oligomers [33, 36]. 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 [37] requires Portland cement to have an initial setting time equals to or greater than 45 minutes and a final setting time less than or equal to 375 minutes. Observing the setting times of the geopolymer binders evaluated in this study (Fig. 5), it can be observed that initial setting times ranged between 110 minutes to 183 minutes and the final setting times ranged between 191 minutes to 255 minutes. 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. 6**. The lower absorption bands ranged between 3693 cm⁻¹ 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°C and 600°C which was observed

previously by the FTIR and XRD analyses of calcined laterite powders (Fig. 1 and Fig. 2). The large bands ranged between 3374 cm^{-1} – 3396 cm^{-1} and 3363 cm^{-1} – 3394 cm^{-1} and the lower ones located 1640 cm^{-1} – 1645 cm^{-1} and 1640 cm^{-1} – 1647 cm^{-1} can be attributed to the stretching vibration of O-H of water molecules [26, 38].

By comparing the infrared spectra of calcined laterites (Fig. 1) and those of the geopolymer binders (Fig. 6), it can be observed that the important absorption bands are located at 1025 cm^{-1} , 1040 cm^{-1} , 1060 cm^{-1} , 1063 cm^{-1} 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 cm^{-1} , 1005 cm^{-1} , 989 cm^{-1} , 995 cm^{-1} and 997 cm^{-1} for GP550, GP600, GP650, GP700 and GP750, respectively made with 8 M NaOH. For geopolymer binders synthesized with 10 M NaOH, the absorption bands shifted to 1003 cm^{-1} , 1005 cm^{-1} , 992 cm^{-1} , 988 cm^{-1} and 987 cm^{-1} for GP550, GP600, GP650, GP700 and GP750, respectively.

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, 17, 39]. The bands located at 864 cm^{-1} , 867 cm^{-1} , 868 cm^{-1} and 909 cm^{-1} in Fig. 6a and those located at 858 cm^{-1} , 862 cm^{-1} , 872 cm^{-1} , 909 cm^{-1} 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 [40].

3.2.3 XRD analysis

The X-ray patterns of the geopolymer binders evaluated are presented in **Fig. 7**. It can be observed from **Fig. 7** that the reflection peaks of some mineral phases like quartz (Q SiO_2 , PDF# 00-046-1045), anatase (TiO_2 , PDF# 00-021-1272) and hematite ($\alpha\text{-Fe}_2\text{O}_3$, PDF # 04-003-2900) detected on the X-ray diffractions of calcined laterites (Fig. 2) remained after the geopolymer reaction. This implies that these mineral phases were not involved nor alter 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 [31, 41]. 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) (Fig. 2) when activated with the alkaline solution shifted to 20° and 40° (2 Theta) (Fig. 7). This displacement confirms that the geopolymerization reaction has taken place and the newly formed phases belonging to the geopolymer network [42]. The presence of kaolinite mineral in X-ray patterns of GP550 and GP600 (Fig. 2) also seen in LAC550 and LAC600 (Fig. 7) indicates that the calcination temperature of clayey laterites at both temperatures does not totally convert 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 is presented in **Figs. 8** and **Fig. 9**, respectively. It is evident from **Fig. 8** and **Fig. 9** 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 (Fig. 8) synthesized with 8 M NaOH are 19.2 MPa, 21.3 MPa, 25.5 MPa, 27.6 MPa 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 MPa, 35.8 MPa, 50.4 MPa, 56.9 MPa, 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 MPa, 2.1 MPa, 3.7 MPa, 4.3 MPa, 5.5 MPa, respectively compared to the flexural strength at 7 days (Fig. 9). 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 (Fig. 8 and **Fig. 9**) 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°C, 650°C, 700°C and 750°C is 6.5 MPa, 21.1 MPa, 27.6 MPa 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. [43] where increasing the calcination temperature from 800°C to 900°C for kaolin was found to increase the resulting compressive and flexural strength of the resulting geopolymer by 23.4 MPa and 38 MPa, respectively.

The 28 days compressive strength of geopolymer made with 8M 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 8M 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. [44] where increasing the NaOH concentration between 8 M to 18 M in the synthesis of fly ash-based geopolymer was found to yield higher compressive strength. Hamidi et al. [45] also reported approximately 20% increase in the flexural strength of fly ash-based geopolymer when the concentration of NaOH was increased from 8 M 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 M and 10 M NaOH are presented in **Fig. 10** and **Fig. 11**, 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 (Fig. 10a and **Fig. 11a**) compared to those of GP650 (Fig. 10b and **Fig. 11b**) and GP750 (Fig. 10c and **Fig. 11c**) can be associated with the weaker structure or possible lower formation of geopolymerization products which was evident in the mechanical properties (Fig. 8 and **Fig. 9**).

It is evident from the figures that increasing the concentration of NaOH from 8 M 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 NaO_2 content which favours the high dissolution of monomers from the precursors resulting in the formation of higher geopolymerization products. A higher concentration of NaOH would also ensure that there exists better cohesion between different components in the matrix resulting in higher performance. These images confirmed the higher mechanical performance exhibited by geopolymer binders synthesized with 10 M NaOH (Fig. 8 and **Fig. 9**). The sheet shapes observed on samples GP550 made with 8 M NaOH (Fig. 10a) 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 (Fig. 11a), 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. [46] showed that increasing the calcination temperature from 600°C 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 lateritic clay, 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.

3.2.6 Sorptivity test

The sorptivity of the geopolymer binders consolidated with 8 M and 10 M are presented in **Fig. 14**. From **Fig. 14**, 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 minutes 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 M 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 M and 10 M of NaOH are presented in **Figs. 15a** and **Fig. 15b**, respectively. For the geopolymer binders consolidated with 8 M NaOH, the bulk density is 1.29 g/cm³, 1.30 g/cm³, 1.31 g/cm³, 1.32 g/cm³ and 1.35 g/cm³ 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 g/cm³, 0.02 g/cm³, 0.03 g/cm³, 0.03 g/cm³ and 0.03 g/cm³, 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 (Fig. 15) 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 compared to those made with 8 M NaOH.

An increase in the bulk density was also noticed when the calcination temperature increased from 550°C 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 binder 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. [35] 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. [43] reported a 60.1% increase in bulk density when the calcination temperature of kaolin was increased from 800°C 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. 16** while that between the water absorption and mechanical properties is presented in **Fig. 17**. It can be observed from **Fig. 16** and **Fig. 17** 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.

4. 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°C 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 soils 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.

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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Figures

Figure 1

FTIR spectra of raw laterite (LA) and calcined lateritic clays

Figure 2

X-ray diffractograms of raw laterite (LA) and calcined lateritic clays

Figure 3

TG/DSC curves of raw lateritic clay

Figure 4

Dissolution rate of Al, Si and Fe from calcined lateritic clay in (a) 8M NaOH and (b) 10 M NaOH

Figure 5

Setting times of geopolymer binders

Figure 6

FTIR spectra of consolidated geopolymer binders synthesized with (a) 8 M NaOH (b) 10 M NaOH

Figure 7

X-ray pattern of consolidated geopolymer binders synthesized with (a) 8 M NaOH (b) 10 M NaOH

Figure 8

Compressive strength of geopolymer binders

Figure 9

Flexural strength of geopolymer binders

Figure 10

SEM images of geopolymer binders synthesized with 8 M NaOH (a) GP550 (b) GP650 (c) GP 750

Figure 11

SEM images of geopolymer binders synthesized with 10 M NaOH (a) GP550 (b) GP650 (c) GP 750

Figure 12

Elemental map of GP550 made with 8 M NaOH

Figure 13

Elemental map of GP750 made with 10 M NaOH

(a)

(b)

Figure 14

Sorptivity of geopolymer binders synthesized with (a) 8 M NaOH (b) 10 M NaOH

Figure 15

Bulk density and water absorption of geopolymer binders synthesized with (a) 8 M NaOH (b) 10 M NaOH

Figure 16

Correlation between bulk density and mechanical properties

Figure 17

Correlation between water absorption and mechanical properties