



Engineering and structural properties of compressed earth blocks (CEB) stabilized with a calcined clay-based alkali-activated binder

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Abstract

The chemical stabilization of compressed earth bricks (CEB) by geopolymer binders appears nowadays as an ecological and efficient technique for the production of durable earth bricks. This work aims to evaluate the feasibility of stabilizing clayey soil-based compressed earth blocks with calcined kaolinite-based alkali-activated binder. The alkali-activated binder was synthesized from a mixture of calcined kaolinite, sodium hydroxide (8 M) and sodium silicate solutions. Clayey soil corrected by sand formed the principal matrix of the CEB. Compressed Earth Bricks (CEBs) stabilized with 5, 10, 15 and 20% of geopolymer were produced at 25 and 70 °C. After a cure during 14 and 28 days, the bricks were characterized by bulk density, dry and wet compressive strengths and flexural strength, mineralogical analysis via X-ray diffraction and structural analysis via Fourier Transfer Infrared (FTIR). The flexural strength values increase with the incorporation of the stabilizer from 0.3 to 3.0 MPa and 1.2 to 3.6 MPa at 25 °C and 70 °C, respectively. The compressive strength values increase also with the addition of the stabilizer from 5.6 to 18.1 MPa and 7.7 to 20.0 MPa at 25 °C and 70 °C, respectively. In the humid environment, the loss of resistance of CEBs compared to the values obtained in the dry environment is about 55%. Calcined clay-based alkaline binder appears thus, as an energy-efficient and sustainable solution for the stabilization of CEBs. The CEBs with 15 and 20% of stabilizer display interesting engineering properties for development of building bricks applicable in area with severe weathering and the other bricks as load bearing masonry units.

Keywords Calcined clay · Alkali-activated binder · Stabilizer · Compressed earth blocks · Engineering properties · Structural properties

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Introduction

In its quest for modernity, African buildings, especially those of the sub-Saharan part, have unfortunately cut themselves off from its age-old bioclimatic tradition, a set of know-how that has allowed the residents to face extreme climates in the past. Today, on the continent, most of the constructions are poorly adapted to the hot climate, thermally uncomfortable and energy-consuming. Because the concrete presents in the majority of urban constructions is not a worthy humidity regulator. But the good news is that there is still time to do better because according to UN-Habitat, ~80% of the buildings that will be inhabited in 2050 are not yet built. Engineers, architects, international organizations and researchers are working passionately on this issue, including research into innovative building materials [1–7]. Several of these studies have shown the possibility to build houses that are better adapted to the African climate, thermally comfortable, economically affordable and environmentally friendly, or esthetically pleasing and modern by using local materials. The results of these researches have shown that the use of earth bricks appears as the one of most suitable materials [8]. In the quest to perform the properties of local construction materials, the mud-bricks have been replaced by the terracotta bricks. However, the production of terracotta bricks is energy-consuming, making the products expensive. Nowadays, stabilized compressed earth bricks are emerging as a cheaper building material with suitable properties.

Unfortunately, the production of the binder commonly used as stabilizers i.e., ordinary Portland cement is energy intensive and contributes significantly to the destruction of the ozone layer. Toward to reduce the consumption of OPC cement in soil and earth bricks stabilization, researchers are using cementitious materials from solid wastes such as rice straw Ash, sawdust ashes, etc., and alternative binders to Portland cement [4, 9–13]. Currently, the geopolymer binder which is a new class of binder appears as the eco-efficient alternative to ordinary Portland cement. The production of this binder is less energy-consuming and emits less greenhouse gases. The synthesis of this binder allows to valorize some geomaterials (clay, laterite, bauxite, feldspathic rocks, etc.), industrial wastes (fly ash, blast furnace slag, red mud, etc.) and volcanic wastes (pozzolans, volcanic ashes, pumices, etc.) [14]. Geopolymerization technology has received a lot attention in recent decades and is one of the solutions proposed today to improve the quality of earth blocs. It is the chemical reaction that occurs at room or slightly elevated temperature between an amorphous aluminosilicate phases and an activating solution (alkaline or phosphoric acid) [15, 16]. This synthesized binder has properties similar or greater than those of Portland cement and contributes positively to the reduction of greenhouse gases [17]. The geopolymer

binder can exhibit a variety of outstanding properties and characteristics, depending on the raw materials used, processing and synthesis conditions such as: aqueous resistance, low linear shrinkage, high compressive strength [18] and low thermal conductivity [19].

Among the geopolymerization precursors mentioned above, kaolinitic clay is one of the materials available in almost all countries of the world. When thermally activated between 550 and 750 °C, it presents a good reactivity in alkaline medium [1, 16, 20, 21]. Several research works have shown that calcined kaolinite is a good precursor for the synthesis of geopolymer binder. Recent works also valorize these binders in the chemical stabilization of soils and compressed earth bricks based on lateritic soils [13, 22–24]. Limited works show its use in the stabilization of clayey soil-based compressed earth bricks, evaluate its maximum percentage of incorporation in stabilized compressed earth bricks as well as the effect of temperature and age of the stabilization process.

This work, thus, investigates the stabilization of earth bricks-based clayey soil using a metakaolin-alkali-based geopolymer binder while investigating the effect of the percentage of geopolymer binder (0, 10, 15 and 20 wt%), curing temperature (room temperature and 70 °C) and time (14 and 28 days). It is also hoped that the outcomes of this study would gear more research in the use of the sustainable and alternative binder for the stabilization of compressed earth block in the construction applications in tropical areas.

Materials and methods

Mix-design of the compressive earth blocks

The bricks labeled CSEB0, CSEB5, CSEB10, CSEB15 and CSEB 20 were obtained by mixing clayey soil (CS) corrected with sand, calcined clay (CCL) and alkaline solution (AS). Based on previous research [7], the alkaline solution/calcined clay mass ratio was kept constant to 0.8.

In order to initiate stabilization, the mixture obtained is left to rest for 5 min, then, tap water is added until a semi-plastic paste is obtained. The resulted mixtures were uniaxially compacting in both steel molds ($40 \times 40 \times 160 \text{ mm}^3$ and $40 \times 40 \times 40 \text{ mm}^3$) at a pressure of 8 MPa using a pressing machine (MANNETI Press, laboratory scale). After demolding, samples were sealed in plastics bags and store at room temperature for 2 days then removed and left in the open air for 7 days. Subsequently, one part is hardened at 70 °C for 24 h and the other kept at room temperature before the characterization. Table 1 depicts the weight composition of different compressed stabilized earth blocks (CSEB).

Table 1 Composition of different compressed stabilized earth blocks (CSEB)

Formulations	CS (wt%)	MK (wt%)	Sand (wt%)	H ₂ O (wt%)	Alkaline solution (wt%)
SCEB0	67.1	/	16.8	7.8	8.4
SCEB5	64.7	4.2	16.2	11.4	3.4
SCEB10	59.5	8.2	14.9	10.7	6.6
SCEB15	55.3	12.4	13.8	8.8	9.7
SCEB20	51.5	16.1	12.9	6.7	12.8

Materials and activator

In this work, the main raw material was clayey soil. The clayey soil from DIBAMBA locality, littoral region of Cameroon (situated between 03°58' and 35°4' North latitude and 09°53 and 31°2' East longitude) was used as main raw material. Two samples were collected: one at the surface, coded CS, which is used as the base raw material, and the other, coded CI, which was taken a little deeper in order to have a good content of clay minerals and acts as a precursor for the binder geopolymers. The CS is dried in an oven at 105 °C for 48 h before being crushed and sieved at 800 µm. To obtain amorphous aluminosilicate material, the CI sample was dried, crushed, sieved at 200 µm, and calcined at 700 °C for 4 h at a heating speed of 5 °C/min.

To adjust the particle size distribution (interval 15 to 30% of fine particles) and complete the percentage of sand, 30 wt% of river sand (SA) sieved at 2 mm was incorporated within the raw precursor regarding the requirements in terms of the physical properties of the clayey soil for earth bricks found in the literature [25].

The activating solution was made from a 2:1 volumetric mixture of commercial sodium silicate (SiO₂/Na₂O ratio 3.1, LOI=60 wt%) and sodium hydroxide (8 M) obtained by the dilution of commercial soda (17 M and 99.9% of purity).

Experimental procedures

The elemental composition of PZ and CS was determined using XRF analysis. The method of sedimentometry has been applied to determine the distribution of particles of CS according to the mass of samples in the medium. The particle sizes were done using dry sieving and sedimentation methods for fine particles according to ASTM D7928's standard. The plasticity analysis was determined through ASTM D4318's

standard. The mineralogical composition of both feedstocks was determined with a Bruker D2 Phase powder X-ray diffractometer (Bruker, Germany), equipped with a flat sample holder, using CuKα radiation with λ = 1.5418 Å at 30 kV at a scan speed of 2°/min ranging from 2θ angles of 5 to 70°. The functional groups of CS and CC were determined through Fourier transform infrared (FTIR) spectroscopy (Bruker Tensor 37, Bruker, Germany) as KBr pellets in the range of 400–4000 cm⁻¹.

The engineering properties were consisted to determine the following parameters: bulk density, flexural strength, dry and wet compressive strengths of the resulting products aged at 14 and 28 days according the protocol of the French Standard P.61–503. These properties were assessed after 14 and 28 days in order to appreciate the kinetics of curing. The water stability was evaluated during wet compressive strength after two days of immersion. In many research studies, it has been reported that the quality of water can also affect the properties of cementitious materials [26–29]. Therefore, distilled water was used in this research for the characterization tests and tap water four molding the samples.

Results and discussion

Characterization of raw materials

The raw materials underwent to several characterizations before the formulation of earth blocs. The results of the chemical analysis are recorded in Table 2. It was found that CS and CI consist mainly of SiO₂, Al₂O₃ and Na₂O with around 47–79, 25 and 9%, respectively. Iron oxide, Fe₂O₃ (1.87%) is responsible for the characteristic red color of these clayey soils.

Table 2 Chemical analysis of the raw materials

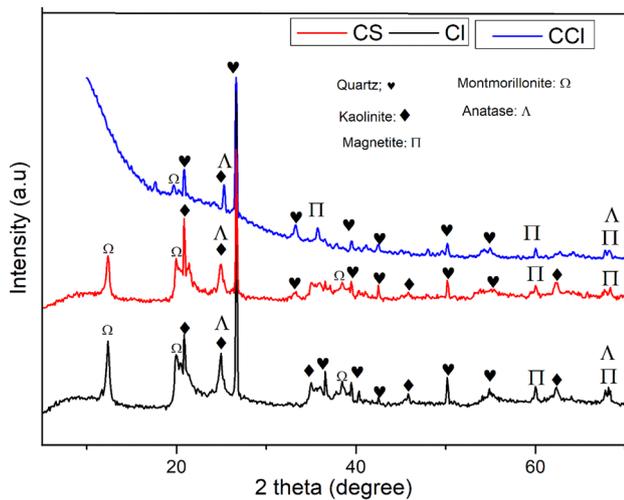
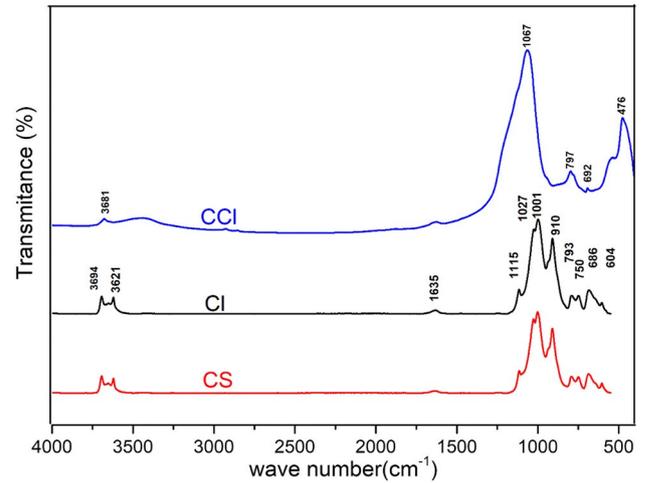
Oxides	SiO ₂	Al ₂ O ₃	CaO	Na ₂ O	K ₂ O	Fe ₂ O ₃	MgO	TiO ₂	LOI
CS (wt%)	47.4	25.9	2.36	9.27	2.5	1.87	1.7	1.78	5.6
CI (wt%)	49.1	25.1	2.35	9.24	2.52	1.66	1.7	1.72	6.61
CCl (wt%)	47.8	41.5	0.1	0.1	0.4	5.3	0.1	0.6	0.7

Table 3 Geotechnical characteristics of raw materials

Geotechnical characteristics	CS	CI	Cameroonian standard for CEB (NC102-114,2002–2006)
<i>Particle size distribution (%)</i>			
Gravel	13.33	1.08	36 ≤
sand	21.63	16.61	34–46
silt	32.70	31.32	20–24
clay	32.80	50.99	10–30
<i>Atterberg limits</i>			
W _L	70.44	103.23	25–50
W _P	53.90	67.56	20–35
I _P	16.54	35.67	2–30

Geotechnical characteristics of the raw materials are recorded in Table 3. The results of particle size analysis show that these materials are clayey soils. They are constituted of gravel, sand, silt and clay at different proportions. These are soils of silty-clayey aspect. Their comparison with the values recommended by the local standard (NC 102-114, 2006) shows that these soils are not appropriated for earth blocks. They are clearly too clayey and poor in sand. The results of Atterberg limits show that the samples are plastic. According to the above results, the clay soil (CS) needs the incorporation of coarse particles such as sand in order to correct plasticity which will allow the manufacture compressed bricks.

The XRD patterns of clay soil (CS), clay (CI) and calcined clay (CCI) are depicted on Fig. 1. It can be observed that the samples CS and CI are composed of clayey minerals such as kaolinite ($\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$), montmorillonite ($(\text{Na},$

**Fig. 1** XRD patterns of clay soil and calcined clay**Fig. 2** FTIR spectra of clayey soil (CS), clay (CI) and calcined clay (CCL)

$\text{Ca})_{0.3}(\text{Al}, \text{Mg})_2(\text{Si}_4\text{O}_{10}(\text{OH})_2 \cdot n\text{H}_2\text{O})_2$ and illite ($\text{K}, \text{H}_3\text{O}) (\text{Al}, \text{Mg}, \text{Fe})_2(\text{Si}, \text{Al})_4\text{O}_{10}[(\text{OH})_2, (\text{H}_2\text{O})_2]$. They also contain the mineral phases of quartz (SiO_2), anatase (TiO_2) and magnetite (Fe_2O_3). The quasi-disappearance of clayey phase peaks after calcination shows that the transformation of crystalline aluminosilicate structures onto amorphous aluminosilicate structure has been effective [20, 30]. This alteration is consistent with the appearance of a halo located between 15° and 35° (2 Theta) on CCL pattern [31].

Figure 2 shows the infrared spectra of CS, CI and CCI samples. Comparing these spectra, it can be observed that the spectra of CS and CI are quasi-similar. This is probably due to similar chemical and mineral compositions. These spectra show few absorption bands with weak and narrow peaks. The absorption bands at $\sim 3694 \text{ cm}^{-1}$ that correspond to the stretching vibrations of OH groups [20] and at $\sim 1635 \text{ cm}^{-1}$ are assigned to the H–O–H bending of the water molecules [32]. The strongest peak appears at $\sim 1024 \text{ cm}^{-1}$ is the one assigned to Si–O–T (T = Al or Si) bending [33]. The absorption bands observed at 1115 and 1027 cm^{-1} are assigned to the stretching vibrations of elongation of the Si–O of quartz network. Similarly, those centered between 686 and 604 cm^{-1} are assigned to the stretching vibrations of elongation of Si–O–Si of quartz network [34]. At ~ 910 and 1001 cm^{-1} , the bands observed here characterize the Al–OH bond of the kaolinite or the montmorillonite. The absorption bands located between 792 and 749 cm^{-1} are attributable to the bending vibration of Si–O–Al^{IV} bonds [35]. The spectrum of CCI shows the absence of the bands at ~ 3621 , 1115 and 910 cm^{-1} of OH and Al–OH bonds, respectively, of kaolinite confirms its transformation into metakaolin [36]. The presence of bands related to Si–O bonds reveals that they are insensitive to calcination.

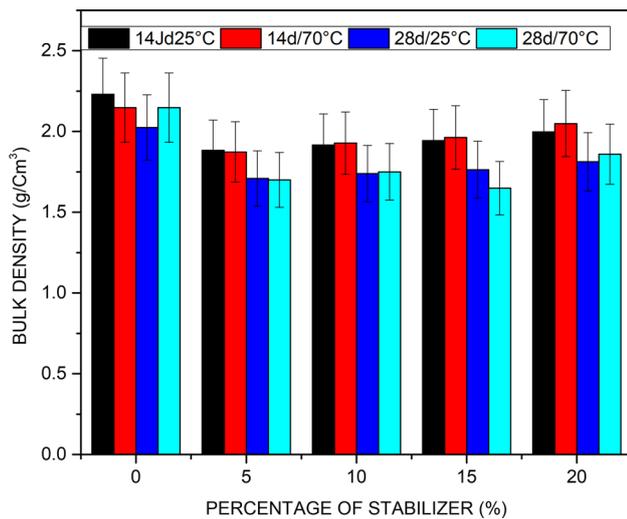


Fig. 3 Bulk density of compressed stabilized earth blocks

Engineering properties of compressed stabilized earth blocks

Bulk density of CSEBs

The variation of bulk density of the stabilized compressed earth blocks with the incorporation of geopolymer binder is illustrated in Fig. 3. The values of samples without addition of geopolymer binder are 2.23 and 2.14 g/cm³ at 25 °C and 2.12 and 2.04 g/cm³ at 70 °C for samples after 14 and 28 days, respectively. These values decrease slightly with the rise of curing temperature and the days of cured. This could be probability due to the water loss at high temperature and during long time of curing. The addition of 5 wt% of geopolymer binder affects the bulk density by reducing their values near to 1.90 at 25 °C and 1.80 g/cm³ at 70 °C for specimens cured during 14 and 28 days, respectively. These can be explained by the fact that the calcined clay is lighter than clay soil. These values increase slightly with the incorporation of geopolymer binder and reach 2.01 and 2.04 after 14 days of curing at 25 and 70 °C, respectively, and 1.81 and 1.86 after 28 days of curing at 25 and 70 °C, respectively. The addition of geopolymer binder allows the development of chemical bonds which maintains the compactness of structure. In fact, the addition of calcined clay (sieved at 100 μm), which consists of fine particles has reduced the number of cavities existing after its polycondensation/polymerization by the formation of aluminosilicate polymer and thus confers high apparent density to CSEBs. The variation with temperature can be justified by the fact that the curing temperature i.e., 70 °C used is not sufficient to cause residual dehydration capable to amplify the pores. This temperature which is less than 100 °C is appropriate for the improvement of geopolymerization. These values remain

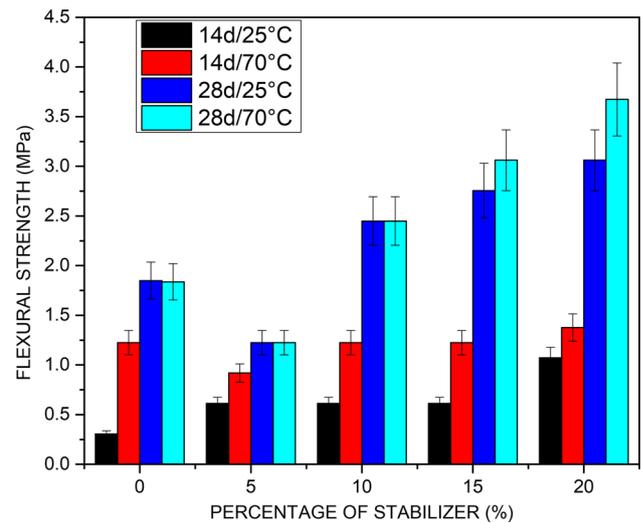


Fig. 4 Flexural strength of stabilized compressed earth blocks

within the interval [1.6–2.2] g/cm³ recommended for mud bricks [37].

Flexural strength of CSEBs

Figure 4 shows the evolution of flexural strength of CSEBs with the temperature of curing and addition of geopolymer binder. Generally, it can be observed that the values increase with the rise of calcined clay, temperature and curing time. After 14 days of curing at 25 °C, the flexural strength of specimen without addition of geopolymer binder presents the lower value at 0.30 MPa. This value increases with the temperature (70 °C) of curing at 1.25 MPa. Also, at 28 days of curing, the values reach ~2.85 MPa for the samples cured at 70 °C. The addition of 5 wt% of binder has not mostly affect the strength. At 10 wt%, the values of flexural strength increase considerably and particular for the samples cured at 70 °C. These values increase with the addition of geopolymer binder and the higher values (1.10 and 3.10 MPa after 14 and 28 days, respectively, cured at 25 °C and 1.50 and 3.67 MPa after 14 and 28 days, respectively, cured at 70 °C) were obtained at 20 wt.% of geopolymer binder incorporated. The better values observed are mainly due to the temperature curing. This showed that the temperature is a kinetic parameter for the polycondensation/polymerization of the reactive species [38]. Therefore, the formation of bonds between the geopolymer phases and inert particles (clay soil and sand) leading to an improvement in the resistance of the products. The increase of flexural strength values also reveals that the metakaolin-based geopolymer binder improves the ductility of CSEBs. It varies from 0.30 to 3.67 MPa.

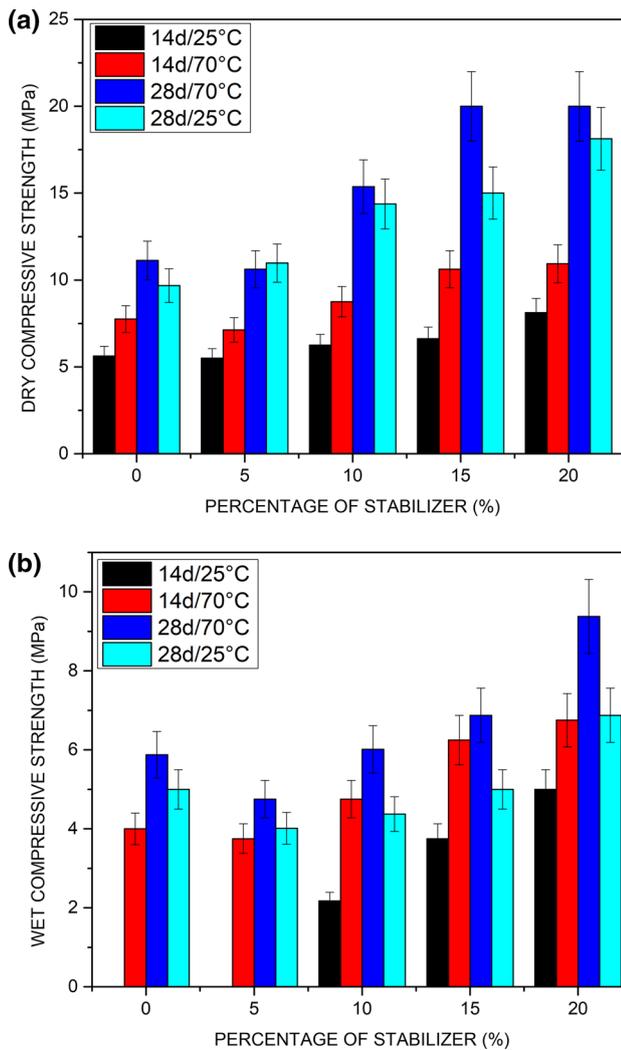


Fig. 5 Dry and wet compressive strength of stabilized compressed earth blocks cured at 14 and 28 days. **a** Dry compressive strength; **b** Wet compressive strength

Dry, wet compressive strengths and water stability of compressed stabilized earth blocks

The dry and wet compressive strengths of the compressed stabilized earth blocks are plotted in Fig. 5. The dry compressive strength (Fig. 5a) values range from 5.6 to 8.1 MPa and 7.7 to 10.9 MPa for the specimens cured at 25 and 70 °C, respectively, after 14 days. After 28 days, compressive strength values vary from 11.0 to 20.0 MPa and 9.6 to 18.1 MPa for the specimens cured at 25 and 70 °C, respectively. These values increase with the rise of calcined clay, temperature and curing time. Samples aged 14 and 28 days after the incorporation of 20 wt% of calcined clay gain around 45 and 58% of strength, respectively. This observation shows that the metakaolin-based geopolymer binder is good material for stabilization of compressed earth blocks.

In fact, the geopolymer binder in the matrix interacts with unreacted solid phases such as sand to form a strong bond and this interaction emerges with the rise of geopolymer binder. The increase of the compressive strength from 14 to 28 days confirms that the geopolymerization is an ongoing reaction. Omar et al. [39] also use calcined clay-based geopolymer binder for the stabilization of laterite soil-based compressed earth blocks. The highest dry compressive strength obtained was 8.9 MPa for CEBs stabilized with 20 wt% of geopolymer. The high values of compressive (18 MPa at 25 °C and 20 MPa at 70 °C) obtained here are attributed to the addition of sand which performed the consolidation of CEBs and the increase of temperature with promoted the polymerization/polycondensation. The considerable values of geopolymer without calcined clay (5.6 MPa at 25 °C and 7.9 MPa at 70 °C) illustrate that alkaline solution obtained by the mixing of sodium hydroxide and sodium silicate is also a stabilizer material for the compressed earth block. The increase of strength with the rise of temperature from 25 to 70 °C confirms that the reactivity of calcined clay in alkaline solution emerges with the temperature [40]. These values of dry compressive strength are well above the minimum of 2 MPa required by the local standard (NC 102, 2006). Thus, all the earth blocks are appropriated for the construction and the solicitation of each formulation for the construction must depend on the compressive strength of CSEB to achieve.

The wet compressive strength values (Fig. 5b) increase also with the percentage of geopolymer binder such as flexural and dry compressive strength values. In the humid environment, the loss of resistance of CSEBs compared to the values obtained in the dry environment is about 55%. This considerable loss is assigned to the weakening of geopolymer network which assures link between unreacted particles due to its hydrolysis. This observation is consistent with the literature review which showed the loss of mechanical properties of geopolymer materials in water [4, 39, 41].

The behavior of CSEBs in water is shown in Fig. 6 and Table 4, the perception of this stability after 4 weeks. The breakdown of CSEB0 and CSEB5 aged 14 days at 25 °C shows that these conditions are not appropriated for the formulation of stable CSEB. In fact, the stabilization requires an optimum quantity of binder for the consolidation of the non-reactive species which has not been reached at 5 wt%. This water stability is markedly more improved with the increase of the temperature after 14 days of curing. The optimal values are reached for a cure during 28 days. According to the literature [42], these mechanical strengths values which already satisfying the standard values may improve with a prolonged curing time beyond 28 days.



Fig. 6 Water stability of stabilized compressed earth blocks. **a** CSEB0, CSEB5 and CSEB10 aged 14 days, cured at 25°; **b** CSEB 10 aged 14 and 28 days cured at 70 °C; **c** CSEB15 and CSEB20 aged 14 and 28 days cured at 25 and 70 °C

Table 4 Perception of stabilized compressed earth blocks immerse in water after 4 weeks

	SCEB0		SCEB5		SCEB10		SCEB15	SCEB20
	14 d	28 d	14 d	28 d	14 d	28 d		
Room temperature	Poor	Poor	Poor	Poor	Poor	Good	Very good	
70 °C	Poor	Poor	Poor	Poor	Good	Good		

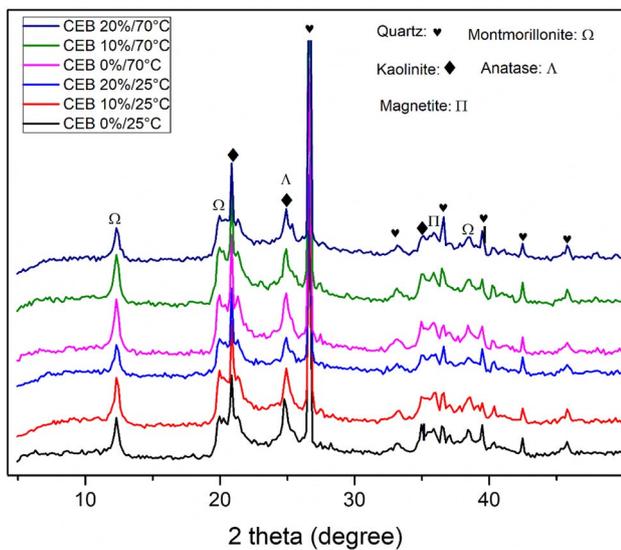


Fig. 7 XRD patterns of 28 days aged compressed stabilized earth blocks

Microstructural properties

X-ray diffraction

The X-ray diffractograms of stabilized earth blocks are shown on Fig. 7. More crystalline phases present in the diffractograms of products are similar to those obtained on the graph of raw materials (Fig. 1). The minerals observed are quartz, kaolinite, anatase, magnetite and montmorillonite.

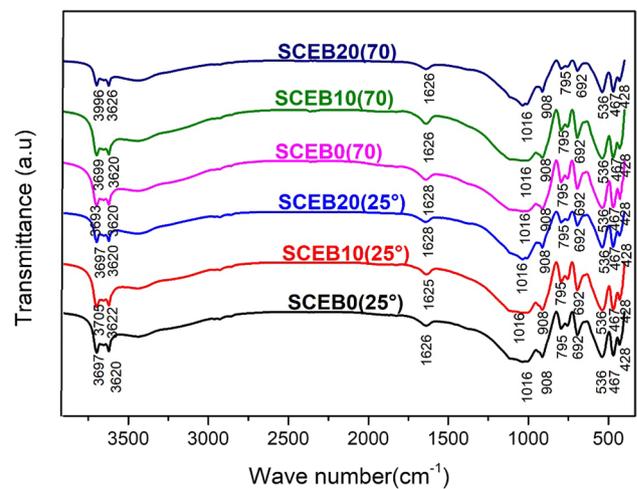


Fig. 8 FTIR spectra of stabilized compressed earth blocks

Comparing the diffractograms of selected CSEBs to the XRD pattern of clay soil (Fig. 1), all the crystalline phases originally in CS are present. This observation discloses that these phases are quasi-inert in alkaline medium [43]. Besides, no major difference is observed between the diffractograms of the products obtained with different percentage of stabilizer or haven undergone different treatments. This can be justified by the high crystallinity of clay soil and sand which dominated the amorphous phase i.e., geopolymer network obtained after alkalization.

Fourier transformed infrared

Figure 8 displays the infrared spectra of compressed earth blocks, stabilized at different percentage of geopolymer and treated at different temperatures, after 28 days of curing. It is observed that all the bands of CS are present on the spectra of CSEB. However, it is also observed that the intensity of absorption bands located between 1200 and 900 cm^{-1} corresponding to the various Si–O–T (T = Si, Al) increases with the percentage of stabilizer. This can be explained by the rearrangement of O–H, Al–OH and Si–O bonds due to their deformation during geopolymerization [44]. It is also noted that the Si–O–T bonds of CS have shifted from 1120 cm^{-1} to a lower wave number (1016 cm^{-1}). This is the consequence of the modification of the chemical environment around the chain structures due to the formation of new Si–O–T bonds [43] highlighting the geopolymerization reaction which has taken place. Moreover, the appearance of new peaks around 467 and 428 cm^{-1} was caused by the formation of Si–O bonds.

Conclusion

This study consisted to valorise the clayey soil via the synthesis of a durable construction material. It promotes stabilization of the CEBs by a geopolymer binder with is more eco-friendly than ordinary Portland cement which is commonly use as stabilizer. The effects of the percentage of stabilizer from 0 to 20 wt%, curing time (14 and 28 days) and curing temperature (25 and 70 °C) were investigate on the engineering and structural properties of the products. The following conclusions can be drawn:

- The clayey soil used must be corrected by the addition of material like sand before its use as raw material for the CEB.
- The flexural strength values increase with the incorporation of the stabilizer from 0.3 to 3.0 MPa and 1.2 to 3.6 MPa at 25 °C and 70 °C, respectively.
- The compressive strength values increase also with the addition of the stabilizer from 5.6 to 18.1 MPa and 7.7 to 20.0 MPa at 25 °C and 70 °C, respectively.
- In the humid environment, the loss of resistance of CSEBs compared to the values obtained in the dry environment is about 55%.

These results indicated that calcined clay-based alkaline geopolymer binder can be used as an efficient stabilizer for the development of structural and functional CSEBs. Calcined clay-based alkaline binder appears thus, as an energy-efficient and sustainable solution for the

stabilization of CEBs. In the view of the flexural and compressive strength values achieved, the CEB₁₅ and CEB₂₀ at 70 °C can be used as building bricks in area with severe weathering and the other bricks as load bearing masonry units. However, before their uses, it is required to evaluate the environmental impact through life cycle analysis, durability and the economic aspects of these materials.

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Author contributions ST: Validation, Methodology, Writing—review and editing, Visualization, original draft, TKRA, CJ, EI: Conceptualization, Methodology, Investigation, Writing—original draft. ST, AS, MNAF: PXRD and SEM/EDX analysis, Validation, Writing—review and editing, Visualization, AN, JGDN: Methodology, Writing review and editing, original draft, CJ and EI: Supervision, Methodology, Resources, CJ, M-AE: Resources, Supervision.

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Declarations

Conflict of interest The authors declare that they have no conflict of interest.

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