

Microstructure and Mineralogy of Compressed Earth Bricks Incorporating Shea Butter Wastes Stabilized with Cement

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Abstract: The current context of sustainable development encourages the development of materials with low environmental impact, which explains the renewed interest in earthen constructions. This study is therefore a contribution to the valorization of clay raw materials from Côte d'Ivoire and agro-industrial waste in eco-construction. The aims of this study was studied the effect of shea butter wastes on the mineralogy and microstructure of Compressed Earth Bricks (CEB) stabilized with cement. To do this, two clay raw materials denoted F (Fronan) and K (Katiola) were sampled and then characterized. Various geotechnical and physicochemical tests have shown that these soils are loamy sand of class A2 and essentially contain quartz, kaolinite, mica and ferric compounds. As for shea butter wastes, it is mainly rich in lignin (32%); cellulose (28%) and hemicellulose (19%). Several samples of bricks with different percentages by mass of clay and shea butter wastes (0-10%), stabilized with 5% cement were prepared and then characterized. The results of the mechanical tests showed that the clay-cement matrix could contain 4% shea butter wastes for the formulations with clay F against 6% with clay K. The corresponding optimal formulations are F₉₁TK₄C₅ and K₈₉TK₆C₅ with clays F and K respectively. The SEM images showed a less dense microstructure for the optimal formulation F₉₁TK₄C₅ compared to that of the clay-cement matrix unlike K₈₉TK₆C₅ where the microstructure remained always dense. X-ray diffraction did not allow to observe mineralogical modifications with the incorporation of shea butter wastes into the clay-cement matrix due to their low quantities in the optimal formulations.

Keywords: Clayey Materials, Shea Butter Wastes, Compressed Earth Brick, Microstructure, Mineralogy

1. Introduction

Côte d'Ivoire, like several other countries in sub-Saharan Africa, is currently experiencing rapid population growth. The low level of housing construction, mainly linked to the soaring costs of building materials, is causing a housing crisis today.

In view of this situation, valuing local materials becomes more than necessary. Thus, clay soil, a building material which had been used for over thousands of years around the world is experiencing a resurgence of interest [1, 2] because of its many advantages, in particular its availability and its closeness to the construction site. Otherwise, this material is completely recyclable, its shaping requires little energy, and it has good thermal, water and sound properties. Despite

these many advantages, this material has some weaknesses which limit its uses. Indeed, earthen constructions sometimes suffer from a poor mechanical strength; risk of cracking due to high drying shrinkage and high-water sensitivity and durability [3-5]. In order to overcome its shortcomings, many earthen products have been developed, in particular adobe; the cob; rammed earth and compressed earth brick. The Compressed Earth Brick (CEB) technique is the most recent in the history of earthen construction. In order to improve the properties of CEB, mineral stabilizers (cement, lime,...) and fibers are often used. While it is currently confirmed that the stabilization of CEB by cement improves their mechanical properties and their resistance to water, the effect of fibers or

plant material on the mechanical properties is still not unanimous in the scientific community.

Côte d'Ivoire, an agricultural country, produces a large quantity of raw materials derived from food crops and exports, the hulls and / or plant residues of which are not sufficiently valued. The production of shea butter in the northern region of the country generates a significant quantity of unsanitary wastes. For the purpose of sustainable development, shea butter wastes which constitutes 50 to 75% of the mass of seeds could be used in CEB.

The aim of this study was to study the effect of shea butter wastes on the microstructure and mineralogy of compressed earth bricks stabilized with cement.

2. Raw Materials and Methods

2.1. Raw Materials

The clay raw materials referred to F (Fronan) and K (Katiola) which are the subject of this study were obtained from the region of Katiola in the North-central of Côte d'Ivoire. The sample collection site F is located around the coordinates (08°12'327"N, 005°07'078"W); for sample K (08°09'030 "N, 005°05'850" W). The different sampling sites are shown in Figure 1.

The shea butter wastes (TK), solid waste, was collected from the shea butter preparation sites in the city of Korhogo in the northern region of Côte d'Ivoire. The collection site is also shown in Figure 1.

The mineral reinforcement used to stabilize the compressed earth bricks is a cement of the type CEM I 42.5 R, marketed by LafargeHolcim Côte d'Ivoire.

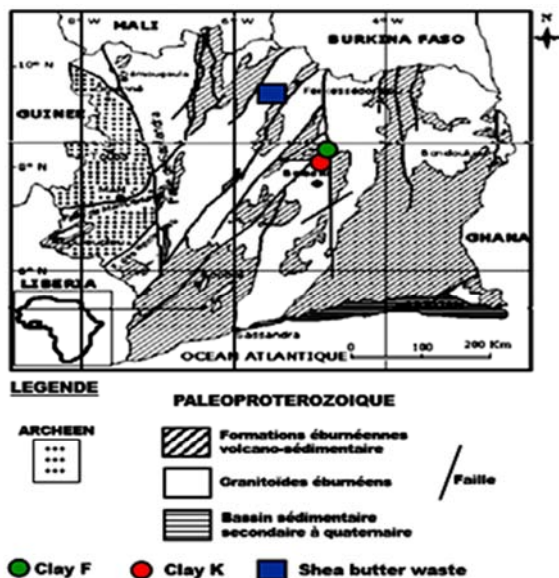


Figure 1. Collection areas for the different samples.

2.2. Methods

The chemical analysis of the clays was carried out by ICP-AES plasma emission spectrometry after sample dilution by dissolving chemically using a microwave.

The chemical composition of the shea butter wastes was determined by energy dispersive spectroscopy (EDS). As for their biochemical composition, it was evaluated through Van Soest method.

The thermograms (Differential Thermal Analysis and Thermogravimetric analysis DTA/TGA) of the clay raw materials were recorded simultaneously in air from room temperature to 1200°C, with a temperature rise of 5°C/min.

The infrared spectra were performed in diffuse reflection using a Perkin Elmer Spectrum 1000 Fourier transform spectrometer.

The FEI Quanta FEG 450 scanning electron microscope was used to observe the morphology of the clay powders and the elaborated CEB.

The particle size distribution of the clays was carried out by sieving according to the NF P 94-056 standard [6], and by sedimentometry according to the NF P 94-057 standard [7].

The Atterberg limits were determined according to the NF P 94-051 standard [8]. The liquidity limit (W_L) was determined using the Casagrande method and the plasticity limit (W_P) by the roll method. The plasticity index (I_P) is obtained by the difference between these two quantities.

The optimum water content (W_{op}) and the corresponding maximum dry density (ρ) were determined by the Proctor test modified according to the EN 13286-2 standard [9].

The organic matter content was determined by wet process of organic carbon assay with Mohr's salt.

X-ray diffractograms of CEB were obtained using a Bruker D8 ADVANCE device. The measurements were carried out on unoriented preparations of powder with a particle size of less than 100 μm in the angular range $2^\circ \leq 2\theta \leq 60^\circ$ with a step of 0.01° and a counting time of 0.25 seconds per step.

The compressive strength of the test sample was evaluated on CEB half-samples of dimension $4 \times 4 \times 8 \text{ cm}^3$ according to EN 196-1 standard [10].

2.3. Preparation of CEB

The clay samples and the shea butter wastes were pre-dried in an oven at a temperature of 105°C for 24 hours. The preparation of the $4 \times 4 \times 16 \text{ cm}^3$ prismatic test specimens was carried out according to the EN 196-1 standard [10]. To do this, the clay raw materials, shea butter wastes and cement are mixed dry for two minutes depending on the composition using an automatic mixer. Then the mixture was moistened with water with quantity determined by the Proctor test and then kneaded again for two minutes. Finally, the mixture was introduced into $4 \times 4 \times 16 \text{ cm}^3$ prismatic molds and compacted using a hydraulic press under a pressure of 40 MPa. Demolding took place 24 hours later and the bricks were stored at 20°C dry for a period of 28 days before the various tests. The different formulations studied are denoted $A_{95-x}TK_xC_5$, with A representing the clay used, TK the shea butter wastes and C the cement and X the quantity of shea butter wastes used as a partial replacement for the clay.

3. Results and Discussion

3.1. Geotechnical and Physico-chemical Characterizations of Clays

The complete particle size distribution of the clay raw materials was determined by sieving and sedimentometry. The grain size curves of samples F and K are shown in Figure 2.

It emerges from the analysis of the grain size curves that the samples are essentially made up of fine particles ($D < 80\mu\text{m}$), with 62% and 72% for F and K respectively. These large proportions of fine particles are characteristic of class A soils [11]. The particle size distributions obtained from the above curves are presented in Table 1.

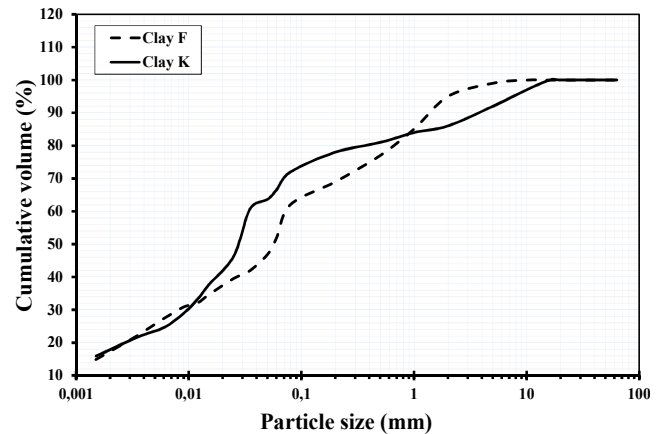


Figure 2. Particle size curves of samples F and K.

Table 1. Particle size compositions of the samples.

Sample	Gravel %	Sand %	Silt %	Clay %	Type of soil
F	5	43	36	16	Sandy- loam
K	9	24	49	18	Sandy- silt

The results of the particle size distribution show good natural cohesion for samples F and K, due to the clay content of between 5% and 30% [12]. This good natural cohesion is an asset for these samples in the making of clay bricks.

The geotechnical parameters of F and K clays are consigned in the Table 2.

Table 2. Geotechnical parameters.

Sample	Liquidity limit $W_L(\%)$	Plasticity limit $W_P(\%)$	Plasticity index $I_P(\%)$	Blue ground value (g/100g)	Organic matter content (%)	Water content $W_{OPM}(\%)$
F	44	21	23	1.35	0.9	11.6
K	39	22	17	0.5	2.8	15.4

The Atterberg limits show that the F and K samples are moderately plastic. These results, combined with those of the particle size analysis, lead to the conclusion that F and K are class A2 soils according to the road earthworks guide [11] and therefore suitable for making Compressed Earth Bricks (CEB).

The methylene blue (MBV) values for the different samples are relatively low. This highlights the low activity of the clay fraction and suggests an absence of swelling clays in the two samples. These results are in agreement with those of the particle size analysis and those of the Atterberg limits with regard to the nature and class of soils.

The organic matter content of F clay is less than 2%, so it is a very poor soil in organic matter and qualified as inorganic soil from a geotechnical point of view [13]. As for the organic matter (OM) content of K clay, it is slightly above the 2% threshold, however, it remains geotechnically acceptable. These low organic matter contents are favorable for good stabilization and good durability of the bricks produced. Indeed, a high organic matter content can delay or annihilate the effect of the treatment by consuming a greater or lesser amount of the stabilizing agent (cement or lime) to neutralize the acidity of the medium.

The results of the modified Proctor test show that F and K have identical dry densities with a greater water content for K. These two samples are qualified as fairly clayey materials with satisfactory mixing because of their density between

1.76 and 2.1 t/m^3 [14]. The identical dry density for these two samples could be related to their similar texture. The Proctor test curves of the samples are shown in Figure 3.

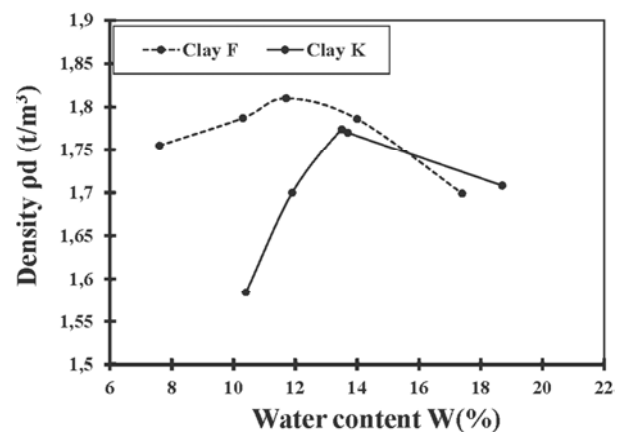


Figure 3. Curves of Proctor Modified Clay Samples.

The chemical composition of the samples is shown in Table 3. Analysis of the results shows the predominance of silica (SiO_2) and alumina (Al_2O_3) in the two clay raw materials. These high levels of SiO_2 and Al_2O_3 show that they are aluminosilicates [15].

The silica / alumina ($\text{SiO}_2 / \text{Al}_2\text{O}_3$) mass ratios of 3.37 and 2.49 for F and K respectively are high compared to that of pure kaolinite which is 1.18 [16]. This suggests the presence

of a significant amount of free silica and clay minerals of type 2:1. Indeed, for this type of clay mineral, due to the numerous substitutions, the value of the $\text{SiO}_2 / \text{Al}_2\text{O}_3$ ratio is generally between 2 and 4 [17].

Besides the major chemical elements silicon and aluminum, these samples contain a relatively large amount of iron, which suggests the presence of ferric phases. The iron present in clay raw materials can be "structural", that is to say in substitution of the Si^{4+} and or Al^{3+} cations in the tetrahedral and / or octahedral layers. It can also be "non-structural", that is to say in the form of individualized particles such as oxy-hydroxides, namely goethite ($\alpha\text{-FeOOH}$) and lepidocrotite ($\gamma\text{-FeOOH}$), and / or oxides such

as hematite ($\alpha\text{-Fe}_2\text{O}_3$) and maghemite ($\gamma\text{-Fe}_2\text{O}_3$) [18, 19]. The much higher iron content for sample K (15.57%) shows that it is a lateritic clay due to the content between 10 and 50% [20].

These clays exhibit losses on ignition lower than that expected for a pure kaolin of around 14% [21]. Sample F has a lower ignition loss compared to K, suggesting the presence of a low amount of clay mineral phases in this sample. The higher value obtained for sample K could be related to the higher organic content. These losses on ignition would be due to the dehydroxylation of the clay minerals present in the various samples and / or to the decomposition of the organic matter.

Table 3. Chemical and mineralogical compositions (% by mass of oxides).

Chemical Composition										
Sample	SiO_2	Al_2O_3	Fe_2O_3	CaO	K_2O	MgO	Na_2O	TiO_2	$\text{SiO}_2 / \text{Al}_2\text{O}_3$	PF
F	69.92	20.76	4.65	0.33	2.11	0.53	1.45	0.26	3.37	5.71
K	57.89	23.28	15.57	-	1.70	-	0.73	0.83	2.49	10.16

Mineralogical composition							
	Kaolinite	Quartz	Illite	Muscovite	Rutile	Hematite	Goethite
F	35.14	45.5	-	17.87	0.26	-	-
K	38.44	24.94	19.9	-	-	4.44	12.28

The results of the mineralogical composition show that the samples are rich in quartz and clay minerals (Kaolinite, Illite or muscovite). The large amount of quartz in the samples is therefore the source of free silica as indicated in the chemical analysis by the high values of the $\text{SiO}_2 / \text{Al}_2\text{O}_3$ ratio. These

high quartz contents will play a role of degreasing kaolinite.

Figure 4 shows the DTA/TGA thermograms of the samples, they were recorded simultaneously under dry air between room temperature and 1200°C, with a temperature rise of 5°C/min.

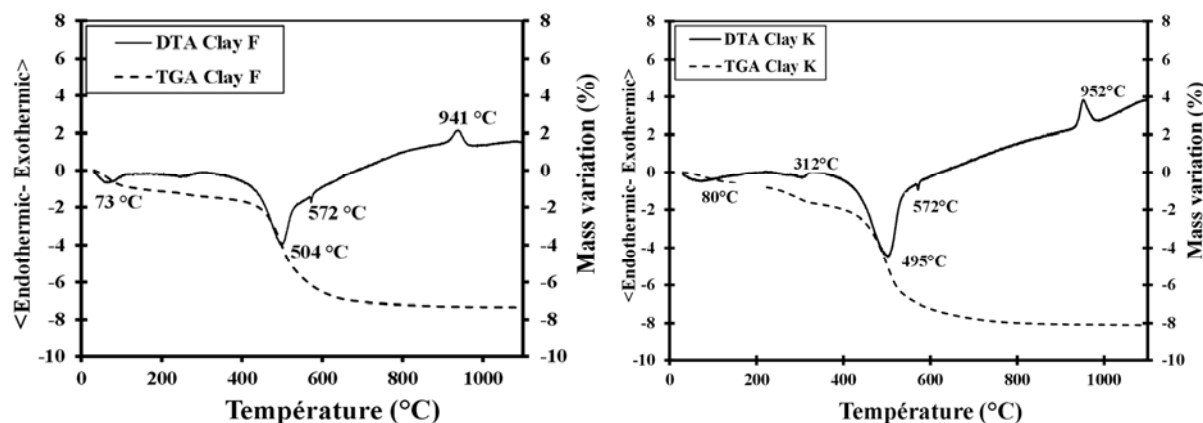
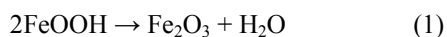


Figure 4. DTA / TGA thermograms of F and K clays.

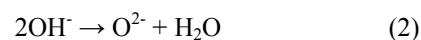
On the thermograms of the two samples, four endothermic peaks and one exothermic peak are observed.

The first endothermic peak observed between 70 and 80°C associated with a mass loss of less than 1% corresponds to the loss of hygroscopic water. This transformation has no influence on the crystal structure of the material [18].

The second endothermic peak whose maximum is found at 312°C on the thermogram of sample K with a mass loss of 1.42% is the confirmation of the transformation of goethite into hematite according to reaction 1 [22].

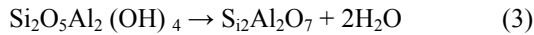


The third high intensity endothermic peak around 500°C associated with a mass loss of about 6%, corresponds to the start of structural hydroxyls. This is the dehydroxylation of kaolinite and illite, the hydroxyls of which are sensitive in this temperature range. This reaction takes place according to reaction 2.



The departure of hydroxyls disrupts the crystal lattice of these minerals. Thus, for kaolinite, dehydroxylation generates the formation of an amorphous phase called

metakaolinite [23]. The overall reaction is written using reaction 3.



The low temperatures (about 500°C) of dehydroxylation of the kaolinite of the two samples show that these kaolinites are disordered. Indeed, the dehydroxylation temperature of an ordered kaolinite is around 600°C while that of a disordered kaolinite is lower [24].

The third endothermic peak at 572°C corresponds to the allotropic transformation of α quartz into β quartz [17].

The exothermic peak observed above 930°C in the two samples without loss of mass is related to the structural reorganization of metakaolinite into spinel or mullite which are more stable compounds according to reaction 4.

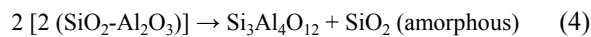


Figure 5 shows the combined infrared spectra of the two clay samples, recorded in the frequency range 4000-400 cm^{-1} with a spectral resolution of 4 cm^{-1} . The spectra generally show the same vibration bands and are identical to those generally observed in clay materials.

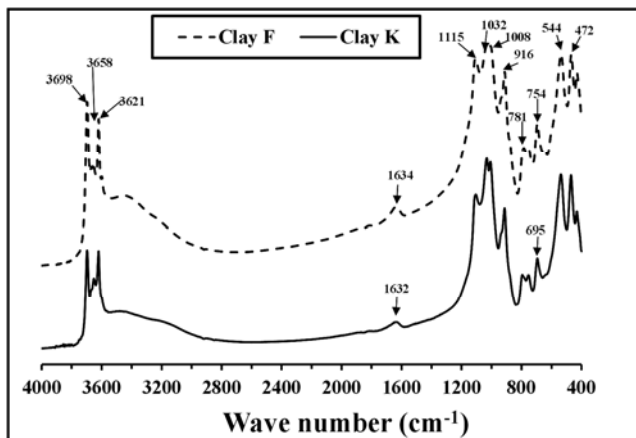


Figure 5. Infrared spectra of clay samples.

The bands observed at 3698, 3658 and 3621 cm^{-1} correspond to the vibration bands of the OH groups of kaolinite [17]. The 3698 cm^{-1} band is attributed to the vibrations of the outer hydroxyls of kaolinite [25]. As for that at 3621 cm^{-1} , it corresponds to the vibrations of the internal hydroxyls located between the tetrahedral interlayer Si_2O_5 and the octahedral sheet [26]. Also, this band can be attributed to the OH groups of muscovite in sample F and illite in sample K. The band observed in the

three samples around 1630 cm^{-1} is attributed to the hygroscopic water absorbed [27]. Then, the absorption band at 1115 cm^{-1} is due to the vibrations of the Si-O bonds of the kaolinite. That observed at 1032 cm^{-1} is associated with the vibrations of elongation of the Si-O-Si bond of kaolinite and illite [25]. As for the band at 916 cm^{-1} , it corresponds to the vibrations of deformation of the Al-OH bonds in kaolinite [28].

The bands at 754 and 544 cm^{-1} can be attributed to the Si-O-Al bonds of kaolinite.

The spectra also show the presence of the quartz vibration bands which are observed at 781, 695 and 472 cm^{-1} , which characterize the Si-O-Si bonds [15].

The two bands observed at 472 and 544 cm^{-1} in sample K may also be due to vibrations of Fe-O bonds [29].

Figure 6 shows the microstructure of the samples. Observation of the SEM image of sample F shows platelets with irregular contours similar to kaolinite particles. Also, these platelets are stacked on top of each other forming clusters. This morphology is similar to that found in general in poorly crystallized kaolinites or in illites [30]. For sample K, the platelets are less visible, which does not allow the minerals to be distinguished. This could be caused by the high level of illite in this sample.

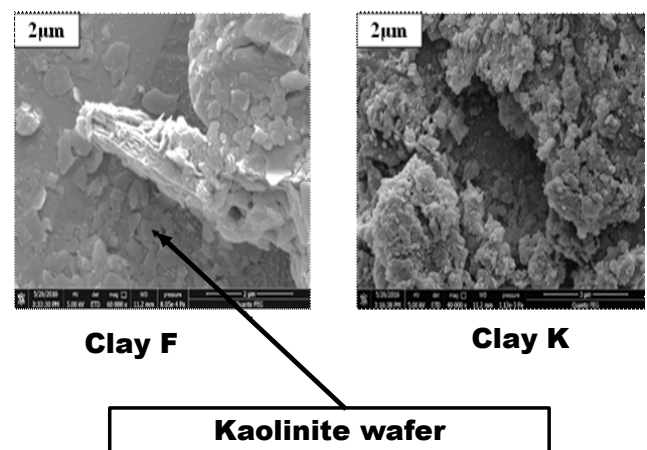


Figure 6. SEM images of clay samples.

3.2. Physico-chemical Characterization of Shea Butter Wastes

Table 4 shows the chemical and biochemical compositions of shea butter wastes (TK).

Table 4. Chemical and biochemical compositions of shea butter wastes.

Chemical Composition												
Oxide	CO ₂	K ₂ O	SiO ₂	P ₂ O ₅	CaO	SO ₃	MgO	Al ₂ O ₃	FeO	PbO	CuO	Na ₂ O
%	32.94	35.58	5.01	6.45	5.06	3.71	4.87	1.77	1.83	0.77	0.34	0.38

Biochemical Composition		
Cellulose (%)	Hemicellulose (%)	Lignin (%)
28	19	32

Carbon dioxide (CO₂) and potassium oxide (K₂O) are the major oxides. The carbon dioxide content is related to the organic matter present in the shea butter wastes because carbon is the most abundant element in plants. This carbon content must have been much higher, but the carbonization of the samples before analysis contributed to the reduction of this organic matter. As for potassium, it is always abundant in the dry matter of plants. In fact, it is absorbed by the roots in the form of the K⁺ cation and circulates in this form throughout the plant. In addition to these major oxides, phosphorus, silicon, calcium and magnesium are present in small quantities.

As for the biochemical composition, it reveals that they are made up of cellulose, hemicellulose and lignin. The low cellulose content (28%) could affect the physical characteristics of the produced CEB.

3.3. Microstructure and Mineralogy of Elaborated CEB Products

In order to study the influence of shea butter waste on the properties of CEB, different formulations were made. The composition of the different mixtures in percentage of dry mass is presented in Table 5. The amount of cement was set at 5% in the formulations because, according to some authors, a content of 5 to 6% makes it possible to obtain satisfactory results [31] and also for reasons of saving cement.

Table 5. Composition of the different mixtures.

Formulations	Clay A (%)	Shea butter wastes TK (%)	Cement C (%)
A ₁₀₀	100	0	0
A ₉₅ TK ₀ C ₅	95	0	5
A ₉₃ TK ₂ C ₅	93	2	5
A ₉₁ TK ₄ C ₅	91	4	5
A ₈₉ TK ₆ C ₅	89	6	5
A ₈₇ TK ₈ C ₅	87	8	5
A ₈₅ TK ₁₀ C ₅	85	10	5

The effect of shea butter wastes on the mechanical properties of CEB, in particular the compressive strength which makes it possible to assess the quality of the bricks is presented in Figure 7. The results show an increase in compressive strength with the addition of 5% cement which can be explained by the formation of cement hydrates (CSH) which bind the particles isolated from the soil. Subsequently, when the shea butter wastes is added to the clay-cement matrix, a decrease in compressive strength is observed both for the formulations with clay F and for the formulations with clay K. Compressive strength can be explained by the increase in the porosity of the CEB and / or by a poor distribution of the shea waste in the clay-cement matrix. These results show a weak adhesion between the shea butter wastes and the clay-cement matrix. The better strengths for lateritic clay can be explained by its good natural cohesion. In addition, by referring to the

African standard relating to construction materials [32] which requires a compressive strength of between 2 and 4 MPa for non-load-bearing walls, formulations with clay F may contain 4% of waste or a resistance of (2.88 MPa) and 6% for the formulations with K or a resistance of (3.01 MPa). The optimal formulations are therefore F₉₁TK₄C₅ with clay F and K₈₉TK₆C₅ with clay K.

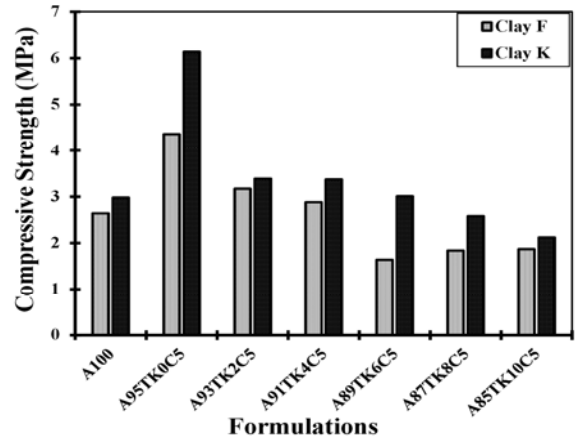
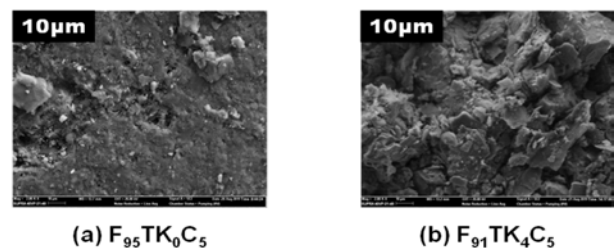


Figure 7. Effect of shea butter wastes on the compressive strength of CEB.

The optimal formulations retained based on mechanical strengths will be the subject of our next study.

Figure 8 shows SEM images of formulations with clay F, that is (F₉₅TK₀C₅; F₉₁TK₄C₅) and formulations with clay K (K₉₅TK₀C₅; K₈₉TK₆C₅). Compressed earth bricks stabilized with 5% cement F₉₅TK₀C₅; K₉₅TK₀C₅ (Figure 8a and c) exhibit a dense microstructure. This microstructure is characterized by the presence of several clear and shiny areas which would be linked to the formation of calcium compounds, in particular portlandite (CH); calcite (CaCO₃) and / or hydrated calcium silicates (CSH). The formation of these calcium compounds could explain the increase in compressive strengths after the addition of cement (Figure 7). Also, the microstructure shows the presence of a small amount of large pores. The incorporation of shea butter wastes into the clay-cement matrix (Figures 8b and d) leads to a less dense microstructure for the formulation (F₉₁TK₄C₅) characterized by an agglomeration of particles of different sizes and shapes and by the presence of more pores. This microstructure shows a weak adhesion between the shea butter wastes and the clay-cement matrix. For formulation K₈₉TK₆C₅ (Figure 8d), the microstructure is still dense, which shows good adhesion between the shea butter wastes and lateritic clay.



(a) F₉₅TK₀C₅

(b) F₉₁TK₄C₅

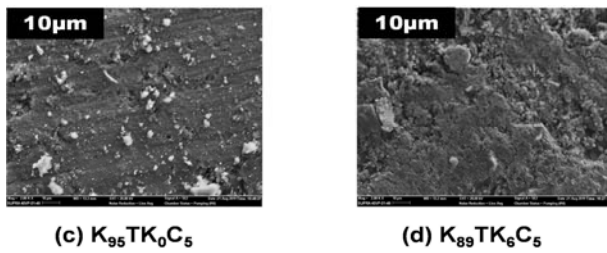


Figure 8. SEM analysis of the different formulations.

Figure 9 shows the diffractograms of the different formulations. The diffractograms of the formulations ($F_{95}TK_0C_5$ and $K_{95}TK_0C_5$) show, in addition to the peaks characteristic of the clay crystalline phases, a peak around

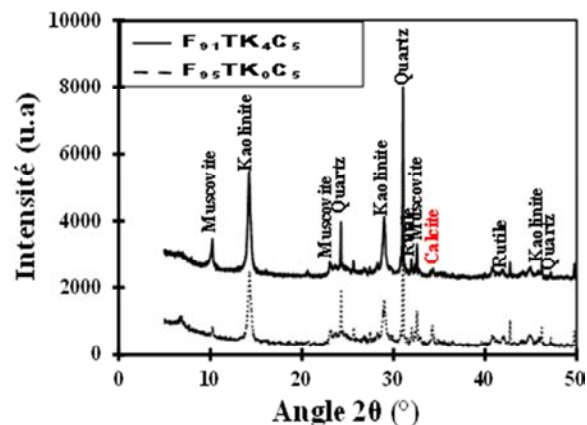
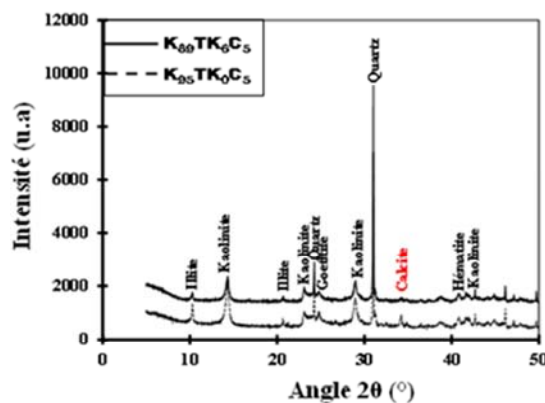


Figure 9. X-ray diffractograms of the CEB produced (a) Clay F; (b) Clay K.

4. Conclusion

This work aimed to study the effect of shea butter wastes (TK) on the microstructure and mineralogy of compressed earth bricks stabilized with cement. Two clay raw materials and shea butter wastes, wastes from the production of shea butter were used. The results of the geotechnical tests showed that the samples F and K are fine A2 class soils made up of sand and silt with an average plasticity and can therefore be used for making clay bricks. The physicochemical characterization revealed that they mainly contain quartz, clay minerals (Kaolinite, illite, muscovite) with addition of ferric compounds in sample K. The chemical and biochemical analyzes of the shea butter wastes revealed two major oxides, potassium oxide (K_2O) and carbon dioxide (CO_2) and they contain cellulose (28%); lignin (32%) and hemicellulose (19%). The results of the mechanical tests on the CEB showed that the quantity of shea butter wastes could not exceed 4% in the clay-cement matrix for the formulations with the sample F against 6% with the clay K. The optimal formulations recommended for each type of clay are therefore $F_{91}TK_4C_5$ and $K_{89}TK_6C_5$. The optimal formulation $F_{91}TK_4C_5$ has a less dense microstructure compared to that of the clay-cement matrix with an agglomeration of particles of different shapes and sizes. With the $K_{89}TK_6C_5$ formulation, the microstructure remained dense like the clay-cement matrix, which demonstrates good adhesion between lateritic clay and shea butter wastes.

34° characteristic of calcite. The latter would come from the carbonation of portlandite (calcium hydroxide) formed during the hydration of alite (C_3S) and belite (C_2S) in the presence of carbon dioxide in the air. In addition, there are no mineralogical changes with the addition of shea butter wastes in the clay-cement matrix, a decrease in the intensity of the calcite peak is observed. Indeed, the precision of X-ray diffraction (XRD) does not allow for small quantities of shea waste in the matrix, i.e. 4% in the formulation with clay F against 6% for the formulation with lateritic clay K, to produce changes.

Finally, the presence of shea wastes in the clay-cement matrix did not cause mineralogical changes due to their small quantities. Complementary studies on the hygrothermal properties and the interactions between the shea butter waste and the clay-cement matrix should be considered for the use of this new material in eco-construction.

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