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Mechanical and Microstructural Properties of Compressed Earth Bricks (CEB) Incorporating Shea Butter Wastes and Stabilized with Cement

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Abstract This study is part of the development of eco-building materials based on clay, cement, and agro-industrial wastes. The main objective was to study the mechanical and microstructural properties of compressed earth bricks (CEB) incorporating shea waste and stabilized with cement. To do this, three clayey raw materials denoted F (Fronan), K (Katiola) and Y (Yaou) consisting essentially of kaolinite, quartz, micas and ferric phases and the shea butter waste mainly composed of lignin (32%); cellulose (28%) and hemicellulose (19%) were used. Several samples of bricks with different compositions by mass percentage of clay, shea wastes (0 to 10%) and 5% cement were developed and characterized. The addition of shea butter wastes generates of porosity within of the compressed earth bricks. Results of the mechanical tests showed a possible substitution of 4% of clay F against 6% of clays K and Y by shea wastes. Thus, with these substitution rates, compressive strengths of 2.88 MPa, 3.01 MPa, and 2.49 MPa were obtained for F, K and Y, respectively. Also, the calcium silicates formed due to the addition of 5% of cement, allowed to keep mechanical performances despite the poor adhesion between the organic material and the clay-cement matrix linked to the low crystallinity of the shea wastes. Adding shea wastes to the clay-cement matrix therefore led to a less homogeneous microstructure.

Keywords: *clayey materials, Compressed Earth Brick (CEB), shea wastes, mechanical properties, microstructure*

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1. Introduction

The soil, like wood and stone, has been used as a building material by humans for thousands of years. Nowadays, it is estimated that the habitat of one third of humanity is still earthen [1]. Longtime abandoned for the benefit of materials such as concrete and steel, earthen construction arouses a renewed interest in the current context of sustainable development. Indeed, to reduce the environmental impact of the construction sector which remains one of the most polluting industrial sectors, the earth represents the ideal material. The main advantages of earth building are the availability, abundance and shaping of this material which requires low energy. As well, this fully recyclable material possesses very interesting thermal, hygrometric and phonic properties.

Despite the many advantages related to the earth utilization in construction, this material nevertheless has some weaknesses limiting its dissemination. The issues encountered with such material are among others the low mechanical resistance, the high drying shrinkage and the high water sensitivity [2,3]. In order to address this, several stabilization techniques are used, leading to a large variety of earthen products of which the most recent is the compressed earth brick (CEB). In order to improve the mechanical performance and the durability of CEB, hydraulic binders such as cement or lime are sometimes added [4]. However, the binder contents generally between 8% and 12% remains high. Vegetable matter or fibers are more and more used for partial or total substitution of mineral binders. Their use has several advantages related to their abundance, low cost, low energy consumption which allows the reduction of environmental impacts [5,6]. Furthermore, the presence of these organic matters in the CEB improves properties such as thermal insulation, prevents cracking on drying, lightens the material and increases the tensile strength [7]. Today, several studies are therefore oriented towards the use of earth with the addition of plant materials which are renewable and locally available [8,9].

Côte d'Ivoire, a sub-Saharan country with an agricultural vocation, produces a wide variety of raw materials from food crops and exports whose shells and/or residues are not enough recycled. The national shea production estimated at around 250 thousand tonnes per year in the northern region of the country generates a significant amount of wastes which constitutes a real unhealthiness source causing a major environmental problem. It has been shown that the sticky black residue, resulting from the shea butter extraction, can be used to fill cracks in walls and as a waterproofing material [10]. In this context, such wastes could be recycled in earth bricks. The main purpose of this work is to study the mechanical and the microstructural properties of CEB incorporating shea wastes stabilized with cement, made from three local clay raw materials.

2. Materials and Methods

2.1. Materials

- There are three clay raw materials used in this study: The clays noted F and K collected respectively from Fronan (F) and Katiola (K) in the centeral region of Côte d'Ivoire. The extraction sites are located around the following geographic coordinates: 08°12'327" North, 005°07'078" West for F and 08°09'030" North, 005°05'850" West for K. The clay labeled Y comes from Yaou in the southern region of Côte d'Ivoire. The geographic coordinates of the sampling site are: 08°08'423" North and 005°06'125" West.
- The shea wastes (TK) was collected from the shea butter preparation sites in Korhogo in the northern region of Côte d'Ivoire.
- The cement used, class CEM I 42.5 R, is provided by Lafarge Holcim Côte d'Ivoire. Its chemical composition is presented in Table 1.

2.2. Methods

The chemical analysis was performed by means of plasma emission spectrometry ICP-AES after a chemical dissolution of elements using a microwave.

The X-ray diffractograms were obtained using a Bruker D8 ADVANCE type device. Measurements were carried out on non-oriented powders with average particle size inferior to 100µm using continuous scanning mode in 2θ range of 2° - 60° with a step size of 0.01° (2θ) and a counting time of 0.25 second.

The specific surface area was measured by the Brunauer Emmett and Teller method (BET) using the Micromeritics TriStar ii surface area analyzer device. Measurements were carried out after a 16 h degassing step at 200°C on samples crushed and sieved at 100 µm.

For shea wastes, the Blaine method was used. The Blaine is an apparatus that determines the fineness of products by air permeability. It measures the passage time of an amount of air through a powder bed. The test was carried out in accordance with the standard EN 196-6.

The limits of both the liquidity limit (W_L) and the plasticity (Wp) were determined using the Casagrande disc and the roller method respectively. The plasticity index (Ip) is given following equation 1. Tests were performed according to standard NF P 94-051.

$$I_P = W_L - W_P \quad (1)$$

The loss on ignition of clays and shea wastes was obtained by calcination. For the measurement, a sample of mass m_1 was calcined at 1000°C then cooled and the mass m_2 was determined. The loss on ignition was calculated using equation 2.

$$PF = \frac{m_1 - m_2}{m_1}. \quad (2)$$

The soil clayiness was determined by the methylene blue test in line with standard NF P 94-068.

The Mastersizer 2000 particle size analyzer was used to determine the particle size of the samples.

The biochemical composition of shea butter wastes was obtained by Van Soest method. This method is based on the difference in solubility of the constituents present within two types of detergents, a neutral or NDF and an acid detergent or ADF, which enables to determine the cellulose, lignin and hemicellulose contents in the sample.

The open porosity of the CEB samples was determined by hydrostatic weighing according to the standard NF P 18-459. The method principle consists in saturating the interconnected pores of the material with water and determining by weighing the apparent mass of the immersed material (M_{wet}), the mass in the air of the water-soaked material (M_{air}) and the mass in air of the dried material (M_{dry}). The opened porosity was deduced using equation 3.

$$\varepsilon = \frac{M_{air} - M_{dry}}{M_{wet} - M_{dry}} \times 100. \quad (3)$$

The total water absorption of CEB was determined by immersion. The principle of this method is the total immersion of the sample in water at (22 ± 3) °C for 24 hours until constant mass. Then, the saturated sample is dried to constant mass in a ventilated oven at (110 ± 5) °C. The weight increase, P_h, with respect to the weight of the dried brick is measured and the total water absorption is determined by equation 4.

$$A(\%) = \frac{P_h - P_s}{P_s}. \quad (4)$$

The mechanical bending and compression tests were carried out according to the methodology described by standard EN 196-1. For the flexural strength, the concentrated load method at mid-range using an automatic press on 4*4*16 cm^3 prismatic samples was used. For compression, the half-prisms obtained from the bending test were used in compression on the lateral molding faces.

The microstructure of CEB was carried out by means of scanning electron microscopy (SEM) coupled with energy dispersive spectroscopy (EDS).

Table 1. Chemical composition of cement (%mass of oxides)

SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	TiO_2	MnO	P_2O_5	SrO	Na_2O	K_2O	SO_3	PF
19.62	5.49	3.08	64.27	0.97	0.54	0.05	0.14	0.12	0.26	0.94	3.97	0.42

2.3. Compressed Earth Brick Mix Design

Seven different formulations in percentage by mass of the used materials (clay, shea wastes) stabilized with 5% cement were tested for the elaboration of the compressed earth bricks. These CEB are identified as $A_{95-X}TK_XC_5$, with A the clay, TK the shea wastes and C for the cement. X corresponds to the quantity of shea wastes used as a partial replacement for clay. The clays and the shea wastes were first ground to a granulometry less than 1mm. Then, a dry mixing of the various constituents was carried out with a mixer. Thereafter, the homogeneous mixture obtained was moistened with water followed by two (02) minute-mixing. Finally, the mixture was introduced into 4*4 *16 cm^3 prismatic molds and compacted using a hydraulic press under a pressure of 40 MPa. The demolding was carried out 24 hours later and the bricks were kept dry at 20°C for a period of 28 days before the various tests.

3. Results and Discussion

3.1. Physico-chemical Characterization of Clays

Table 2 shows the results of the chemical analysis of the clay samples. The silica (SiO_2) and alumina (Al_2O_3) are the two main oxides present in clays. The SiO_2/Al_2O_3 mass ratios of 3.37 for F; 2.48 for K and 3.24 for Y are high compared to that of pure kaolinite (1.18) [11], suggesting the presence of a significant amount of free silica and/or of type 2:1 clay minerals. The iron oxide content is also significant in the various samples, mainly in sample K (15.67%). Based on the classification of laterites proposed by Lacroix [12], which takes into account only the iron oxide contents, sample K with an iron oxide content of 15.67% can be qualified as lateritic clay. All studied samples also contain minor elements such as: calcium, potassium, sodium and titanium.

Figure 1 shows the XRD patterns of the samples. The major crystalline phases present in the three clay samples are kaolinite ($Si_2Al_2O_5(OH)_4$), quartz (SiO_2) and a micaceous phase. The micaceous phase corresponds to muscovite in the sample F and to illite in samples K and Y. In addition to these main phases, F contains rutile (TiO_2), K contains hematite and goethite while Y contains rutile and goethite.

Combining results of chemical analysis and those of X-ray diffraction, enables to evaluate the content of the crystalline mineral phases present in the different clays according to equation 5. The results are reported in Table 3.

$$T(a) = \sum M_i \times P_i(a). \quad (5)$$

Where T(a): oxide content (%) for the chemical element "a"; Mi: mineral content (%) "i" within the studied material and containing the element "i"; Pi(a): proportion of element "a" in mineral "i".

Table 2. Chemical composition of clay raw materials (%mass of oxides)

Sample	SiO_2	Al_2O_3	Fe_2O_3	CaO	K_2O	MgO	Na_2O	TiO_2	SiO_2 / Al_2O_3
F	69.92	20.76	4.65	0.33	2.11	0.53	1.45	0.26	3.37
K	57.51	23.12	15.67	-	2.35	-	0.73	0.83	2.49
Y	66.61	20.57	9.27	0.05	1.23	0.18	0.57	1.52	3.24

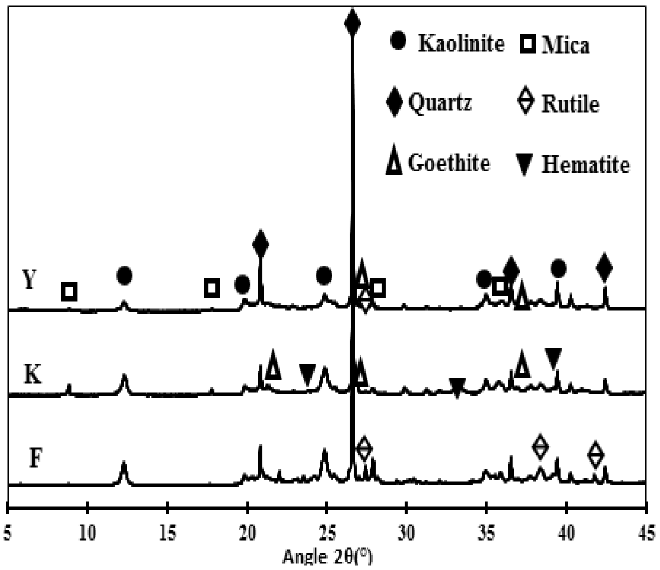

Figure 1. X-ray diffraction patterns of clay raw materials

Table 3. Mineralogical compositions of the samples (% mass)

Samples	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
Y	41.9	42.41	10.65	-	1.52	-	4.25

Quartz, kaolinite, illite and muscovite are the major crystalline phases in the three clay raw materials. The sample K also contains a significant amount of goethite.

Table 4 shows some physical parameters of the clay raw materials. The plasticity indices and the blue value of the soils show that the samples F and K are class A2 soils and Y a class A3 soil. The samples F and K are therefore suitable for use in the raw state for making of CEB. However, the use Y in the raw state could cause cracking due to shrinkage thus weakening the blocks. The loss on ignition results show a relatively low value for sample F (5.71%) and high values for samples K (10.16%) and Y (11.44%). The lower value of loss on ignition obtained for sample F could indicate a low amount of clay mineral phases. The higher values obtained for Y and K suggest a significant amount of clay minerals.

These loss on ignition could be due to the dehydroxylation of the clay minerals present in the different samples and to the decarboxylation or to the decomposition of organic matter. The densities of the three clay materials are similar and of the same order as those generally observed in kaolins and illite [13]. The slightly higher density for K is in line with that measured for the laterites which varies from 2.5 to 3.7 [14]. The specific surface area values of 18.32 m^2g^{-1} and 25.62 m^2g^{-1} respectively for F and K show that they are kaolinitic clays. Indeed, clays of this type have a specific surface area which generally varies between 10 and 30.2 m^2g^{-1} [15]. The higher specific surface area value for Y, i.e. 41.61 m^2g^{-1} in addition to the significant amount of iron oxide, suggests the presence in high quantity of fine particles (ϕ<80μm). The sample Y contains more fine elements with an average diameter d_{50} (6.36 μm) which is in accordance with the high value of the specific surface area.

Table 4. Some physical parameters of clay raw materials

Samples	F	K	Y
Plasticity index I_P (%)	23	17	29
Methylene blue value (g/100g)	1.35	0.5	1.56
Loss on ignition (%)	5.71	10.16	11.44
Density	2.64	2.78	2.63
Specific surface area (m^2g^{-1})	18.32	25.62	41.61
Average diameter d_{50} (μm)	35.15	17.47	6.36

3.2. Physico-chemical Characterization of Shea Wastes

Figure 2 shows the XRD pattern of shea waste powder. The result shows that the shea wastes are semi-crystalline containing cellulose with the main peaks 2θ at 17.67 °; 18.8 ° and 22.6 °. The peaks at 17.67 ° and 18.8 ° are quite marked and distinct one from another, indicating that the cellulose present in the shea wastes is the high crystallinity type I cellulose. The crystallinity of the shea waste was evaluated by the method proposed by Segal [16], the crystallinity index Ic is given by equation 6 using the intensity of the peaks 002 (I_{002}, 22 ° <2θ <23 °) and 110 (I_{AM}, 18 ° <2θ <19 °).

$$Ic(\%) = \frac{I_{002} - I_{AM}}{I_{002}} \times 100. \quad (6)$$

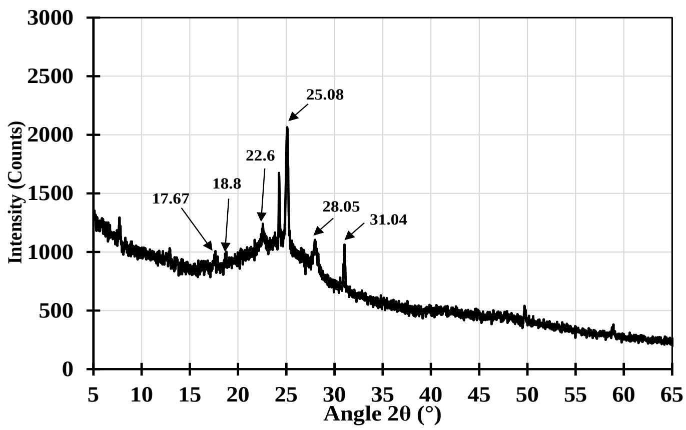

Figure 2. XRD pattern of shea waste

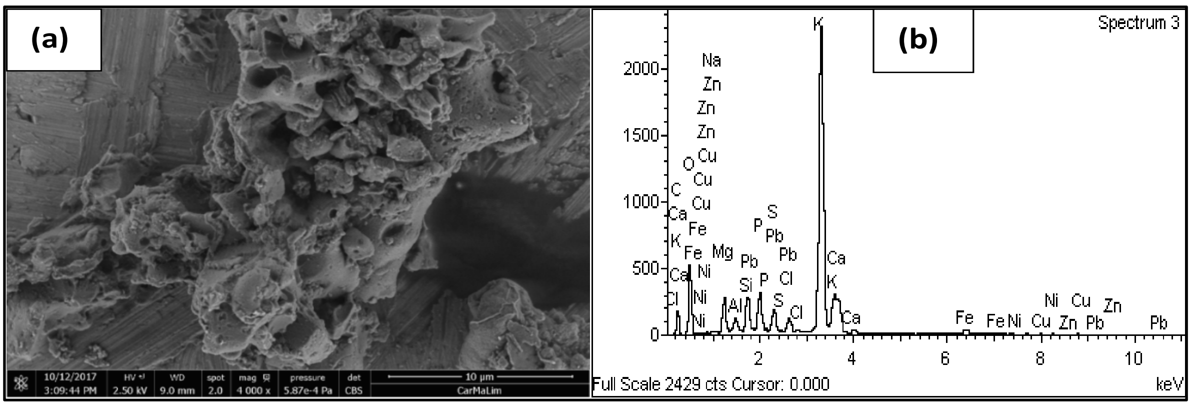

Figure 3. (a) SEM image; (b) energy dispersion spectrum of shea wastes

The value obtained is 20%, this low value would be linked to the presence of amorphous compounds that are lignins and hemicelluloses which contribute to the reduction of the index.

The morphology of shea waste was observed by means of scanning electron microscopy coupled with elementary chemical analysis by energy dispersion spectrometry. SEM observation (Figure 3a) shows an amorphous structure of shea wastes which confirms their semi-crystalline structures. EDS spectrum (Figure 3b) reveals the presence of carbon linked to the organic matter contained in the shea wastes. The potassium, which is always abundant in the dry matter of plants is also present. It is absorbed by the roots in the form of the K^+ cation and circulates in this form throughout the plant.

Table 5 shows some physical parameters and biochemical composition of the shea wastes. These results show a low apparent density (0.41). This value that is far from the measured specific gravity (1.33) suggests a significant proportion of pores in the shea wastes. The high value of loss on ignition (94.51%) confirms that shea butter wastes are very rich in organic matter. The combustion of this organic matter during the calcination of the shea wastes therefore explains the significant loss on ignition. The Blaine specific surface (3233 cm^2/g) shows the fineness of the shea waste particles, this value close to that of cement (3565 cm^2/g) could promote good adhesion between the cement particles and those of the shea wastes in the clay matrix of CEB. The biochemical composition of the shea wastes shows that they are composed of 28% cellulose; 19% hemicellulose and 32% lignin. These high lignin and hemicellulose contents are responsible for their amorphous nature. The high lignin content does not allow livestock to consume this waste [10], hence the need to find ways to recycle this agro-industrial wastes.

3.3. Mechanical and Microstructural Properties of Compressed Earth Bricks

Table 6 gathers the different studied mix design in dry mass percentage of each constituent. Before characterizations, the samples were kept dry at 20 °C for 28 days.

The open porosity of CEB increases with the addition of shea wastes in the different samples. There is therefore creation of pores with the shea waste addition. During the mixing phases, the shea waste particles absorb water due to their hydrophilic nature and expand and then repel the particles of the clay-cement matrix. After drying, the reduction in the volume of the particles due to the departure of the absorbed water, creates voids around them [17]. The shea waste particles would be therefore found as individualized particles in the clay-cement matrix.

Figure 4 illustrates the total water absorption of CEB after 24 hours of immersion. In general, a slight increase is observed with the addition of shea wastes. This slight increase can be attributed to the water absorption capacity of the organic material. In fact, due to their hydrophilic nature linked to the presence in large quantity of OH hydroxyl groups contained in cellulose and hemicellulose, plant material is generally very absorbent [18]. Also, this increase in absorption can be explained by the creation of pores in the matrix which will be filled with water during the CEB immersion.

Figure 5 shows the compressive (a) and the bending (b) strength of CEB**.** The results show an increase in both the compressive and the bending strengths with the addition of 5% of cement except for the formulation with Y where a decrease is observed. This improvement of the mechanical resistance when adding the cement can be explained by a more homogeneous microstructure linked to a smaller pore size and also to the absence of microcracks due to the formation of calcium silicate hydrates (CSH) which bind the isolated particles. The decrease in the compressive strength observed for the formulation with Y could be linked to the small sand content in this sample as the cement will preferably act on the sand for the formation of CSH. Thereafter, with the addition of shea wastes as a partial substitution for the clay material, a drop in mechanical strength was observed. This drop in mechanical resistance with the addition of organic matter has been widely commented in the literature and results mainly from the increase in the porosity of the materials [8,19]. It could also be linked to the decrease in the density of CEB of around 8% [20].

Table 5. Physical parameters and biochemical composition of shea butter wastes.

	Apparent density	Specific weight	Blaine specific surface (cm^2g^{-1})	Loss on ignition (%)
Physical Parameters	0.41	1.33	3233	94.51
	Cellulose (%)	Hemicellulose (%)	Lignin (%)	
Biochemical Composition	28	19	32	

Table 6. Formulations and open porosity of CEB

Formulations	Clay A(%)	Shea butter wastes TK(%)	Cement C(%)	Open porosity		
				F	K	Y
A_{100}	100	0	0	19.8	21.8	24.6
$A_{95}TK_0C_5$	95	0	5	20.5	22.8	24.8
$A_{93}TK_2C_5$	93	2	5	20.8	23.3	25.1
$A_{91}TK_4C_5$	91	4	5	20.9	24	25.4
$A_{89}TK_6C_5$	89	6	5	21	24.1	25.6
$A_{87}TK_8C_5$	87	8	5	21.3	24.2	25.7
$A_{85}TK_{10}C_5$	85	10	5	22.2	26.4	25.8

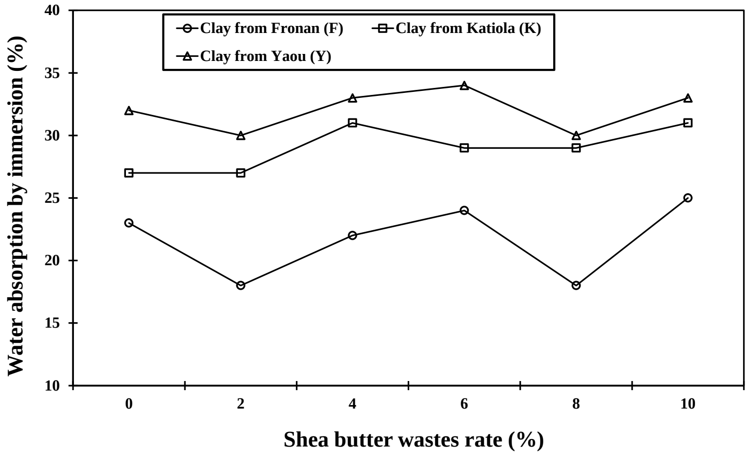

Figure 4. Total water absorption of CEB

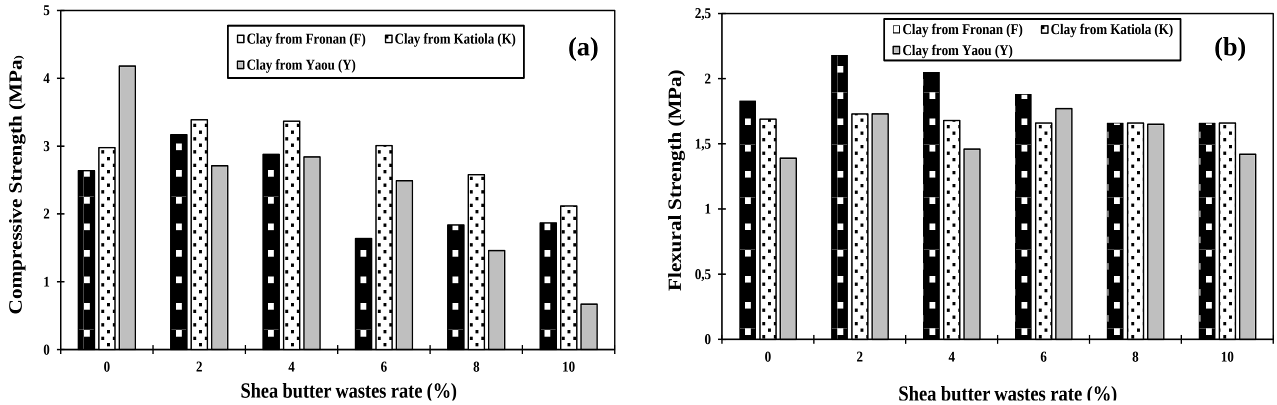

Figure 5. Mechanical resistance in compression (a) and in flexural (b) of CEB

Furthermore, by referring to the African standard relating to construction materials which require a compressive strength ranges from 2 to 4 MPa for non-load-bearing walls [21], the substitution of the clay material cannot be done beyond 4% with sample F. For an amount of 4% of shea waste the compressive strength is 2.88 MPa. For formulations with lateritic clay K, a substitution up to 10% remains possible. This can be explained by its good cohesion and the high content of goethite (12.28%) which is the variety of iron which seems to be the most active during the consolidation of lateritic soils [22]. But above 6%, the compressive strength (2.58 MPa) is lower than the one of the raw sample (2.98 MPa), which enables to set the substitution rate of shea waste for this sample at 6% leading to a compressive strength of 3.01 MPa in the case of clay Y-based formulations, a 6% shea wastes substitution is optimal because it leads to a compressive strength of 2.49 MPa.

Figure 6 presents the microstructure along with the EDS elementary analysis of stabilized CEB. The formations considered are those providing the best mechanical performance i.e. $F_{91}TK_4C_5$; $K_{89}TK_6C_5$ and $Y_{89}TK_6C_5$ with results depicted on Figure 6a; Figure 6b and Figure 6c respectively. The $K_{89}TK_6C_5$ formulation has a more homogeneous microstructure (Figure 6b) compared to the ones of $F_{91}TK_4C_5$ and $Y_{89}TK_6C_5$ which show a less homogeneous microstructure (Figure 6a and Figure 6c). This less homogeneous microstructure for these two formulations is characterized by an agglomeration of particles of different sizes and shapes and also by the presence of more pores in the matrix. This phenomenon can be explained by a weak adhesion between the organic material and the clay-cement matrix, linked to the non-rough character of shea wastes. The more homogeneous microstructure for the $K_{89}TK_6C_5$ formulation would be linked to its cohesion in the natural state which is a factor which influences the adhesion between plant particles or fibers and the clay-cement matrix [17,23]. Also, it can be related to the small number of pores in the matrix. On SEM images we also observe bright and clear zones due to the presence of calcium compound [24]. EDS analyzes carried out on the different zones show that they are rich in alumina, silica and iron oxide because of the presence of kaolinite, quartz and goethite in clay raw materials. There is also a significant amount of calcium oxide in these different areas, probably due to of the formation of portlandite (CH), calcite ($CaCO_3$) and/or calcium silicate hydrate (CSH). The latter help in improving the physical and mechanical properties of CEB. The portlandite is formed following the hydration of two calcium silicates, alite (C_3S) and belite (C_2S) according to equations 7 and 8.

$$C_3S + 3H \rightarrow C\text{-}S\text{-}H + 2CH \quad (7)$$

$$C_2S + 2H \rightarrow C\text{-}S\text{-}H + CH \quad (8)$$

The calcite is obtained following the carbonation reaction involving portlandite formed during the hydration of cement and carbon dioxide from the atmosphere according to equation 9.

$$Ca(OH)_2 + CO_2 \rightarrow CaCO_3 + H_2O \quad (9)$$

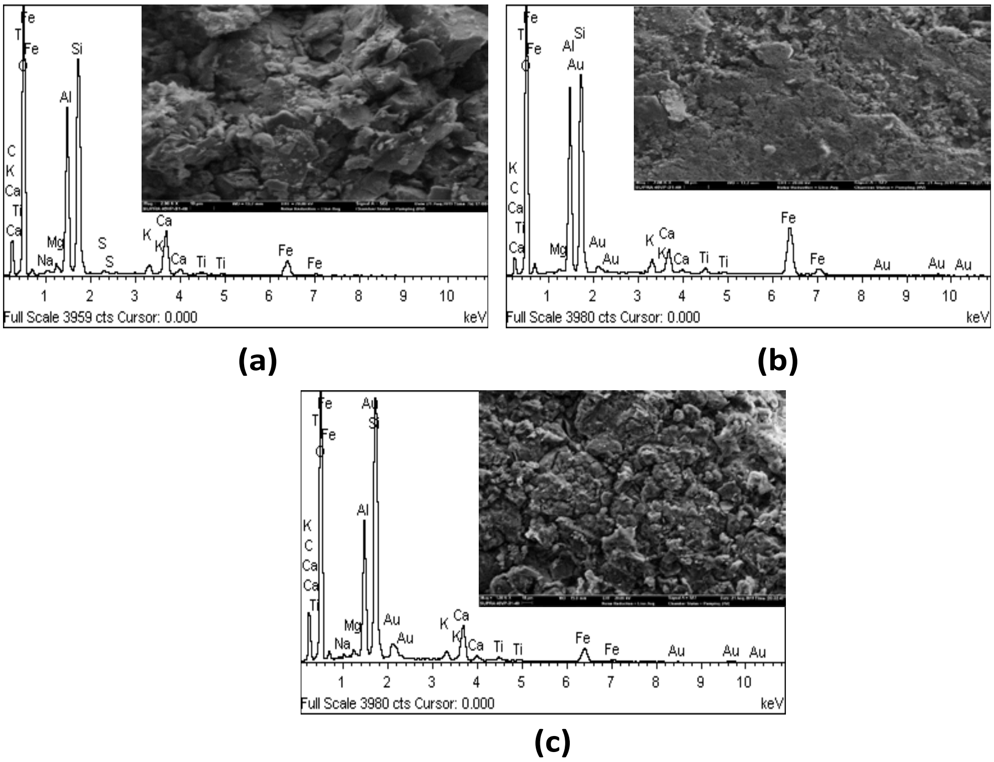

Figure 6. Microstructure and EDS analysis (a) formulation F91TK4C5; (b) formulation K89TK6C5; (c) formulation Y89TK6C5

Calcium silicate hydrates (CSH) result on the one hand from the hydration of the C_3S and C_2S of the cement and on the other hand from the pozzolanic reaction between the portlandite released during the cement hydration and fine quartz or silica from kaolinite silicates. The mechanism of this last reaction is expressed as follows:

- Dissolution of portlandite according to equation 10.

$$Ca(OH)_2 \leftrightarrow Ca^{2+} + 2OH^- \quad (10)$$

- Dissolution in basic medium of silica according to equation 11.

$$SiO_2 + 2OH^- \rightarrow H_2SiO_4^{2-} \quad (11)$$

- Reaction in an aqueous medium of Ca^{2+} ions released with the soluble silicates resulting in equation 12.

$$Ca^{2+} + H_2SiO_4^{2-} \rightarrow CaO.SiO_2.H_2O\ (CSH) \quad (12)$$

The presence of potassium oxide can come from the micaceous phases (illite and muscovite) present in clay raw materials and also from potassium present in shea wastes. The presence of sulfur oxide on the EDS spectrum of the formulation $F_{91}TK_4C_5$ could be justified by the formation of ettringite ($C_6A\bar{S}_3H_{32}$) which results from the reaction between gypsum and tricalcite aluminate in aqueous medium, according to the equation 13.

$$C_3A + 3C\bar{S}H_2 + 26H \rightarrow C_6A\bar{S}_3H_{32} \quad (13)$$

4. Conclusion

The aim of this work was to study the mechanical and microstructural properties of compressed earth bricks incorporating shea wastes and stabilized with cement. Thus, three clayey raw materials mainly consisting of kaolinite, quartz and micaceous phases; shea wastes rich in lignin, cellulose and hemicelluloses and cement were used. Different compressed earth bricks containing 5% cement and different quantities of shea waste varying from 0 to 10% in substitution for the clay material were developed. The various results obtained have highlighted the appearance of pores in CEB, favored by the addition of shea wastes. This led to a slight increase in the total water absorption. The addition of 5% cement induced an increase in the mechanical strengths which decline with adding shea wastes. Accordingly, to form good quality of CEB, the contents of shea wastes cannot exceed 4% for clay F-based formulations, against 6% for formulations with K and Y. Adding shea wastes to the clay-cement matrix led to a less homogeneous microstructure highlighting a poor adhesion between the shea wastes and the clay-cement matrix. Ultimately, the incorporation of shea wastes in bricks is a recycle avenue of these wastes source of environmental pollution.

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