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Physicochemical and mechanical characterization of Benin's Kenaf fibers and its effect on the building compressed Earth Blocks (CEB) mechanical properties

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Abstract

The physical, chemical and mineralogical characteristics of Kenaf fibers from the Republic of Benin were studied by using the X-ray diffraction (XRD), infrared spectroscopy (IR), thermal gravimetric analysis coupled withdifferential scanning calorimetry (TGA/DSC) and chemical analysis using the method of VAN Soest. It emerged from study that the fibers of Kenaf essentially consisted of cellulose (73wt %), hemicelluloses (18 wt%) and lignin (6 wt%). Kenaf fibers of 10, 20 and 30 mm lengthwere used at dose rate of 1.2% weight to reinforce the mechanical and thermal properties of Compressed Earth Blocks (CEB)developed from a clay soil;constituted of kaolinite (33, 46wt%); illite (14.90wt%%); microcline (2.11wt%); quartz (48.78wt%) and 0.86wt% of anatase. The incorporation of Kenaf fibers permitted to reduce the distribution of cracks in CEB. Analysis of the mechanical behavior of different formulations soil/fiber in terms of flexural strength and compression demonstrated the beneficial effect of the fibers. The best result of mechanical strength standpoint was obtained with the fibers length of 30 mm. Here we successfully proved that it is possible to improve the mechanical and thermal properties of CEBby using fibers for reinforcement; and the composite can be optimized by altering the fiber content and length.

Keywords: Kenaf fibers, Clay soil, Chemical and mineralogical Composition, Mechanical resistance.

Introduction

Kenaf is an herbaceous plant which annual height is between 1.5 and 3.5 m with a woody base. It belongs to the family of Malvaceae. The diameters of the rods varied from 1 to 2 cm and the leaves, which are of variable sharp, have lengths in the ranging between 100-150 mm. The leaves nearby the stems were strongly lobate (3-7 lobes), whereas the superior nearby stem leaves were slightly lobate. In the Republic of Benin and Burkina Faso, Kenaf leaves are often used to prepare Sauces[1]. White, yellow or purple flowers have diameter varying from 8 to 15 cm. When they are white or yellow, the center is still deeply purple. The fruit is a capsule of 2 cm of diameter containing several seeds. Furthermore, knowing and believing in the high mechanical strength of Kenaf fibers, the latters are often used to produce artisanal bags in West Africa. Benin northern breeders also use Kenaf fibers in the rope in orderto attach animals. In Nagoand Yoruba area in the republic of Benin, the earth blocks are traditionally stabilized or reinforced with organic materials available locally such as straws, herbal decoctions and especially cow dung. Many studies have focused on the physical and mechanical characteristics of compressed earth blocks stabilized or reinforced with natural fibers[2-11], but little attention was paid to how the physical and chemical characteristics of fibers incorporated affect physical and mechanical properties of the produced CEB. In addition, the chemical compositions of Kenaf fiber have been in some Asia, South America countries, and Burkina Faso in Africa[11-13]. Despite the fact that their chemical composition is climate, plant species and soil type dependent, any information is available in the literature regarding Benin's Kenaf fibers. Consequently, this work aims at first to study toinvestigate the properties physical, chemical and mechanical; of Kenaf fibers from the Republic of Benin. Secondly and finally, investigate its impact on the enhancement of the mechanical properties of compressed earth blocks. In short, the main objective of this work is to valorize Kenaffibers in building earth block fabricationas it is produced and easily available in Benin.

Materials and methods

Raw materials: Kenaf plants were collected from Kandi, a town in northern Benin (West Africa). They were cut at the age of 6 months and retted in rivers and manually defibrated. The fibers were cut with a massicot to obtain fibers of 10 mm, 20mm or 30 mm length. The soil used were collected from the region of Parakou in northern Benin (between latitudes 9°00' and 10°00' North and longitudes 2°20'and 3°40' East). Figure-1 shows the particle size distribution curve.

Table-1 presents the geotechnical characteristics of the used land; itwas a sandy loam.

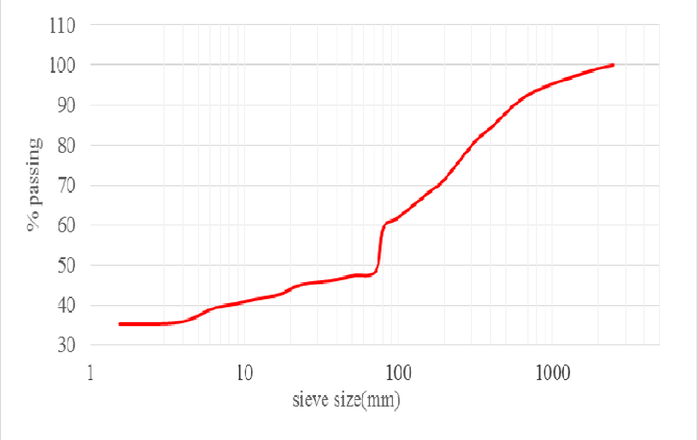

Figure-1: Grain size distribution of the used soil

Table-1: Geotechnical characteristics of the soil sampling area (Parakou town).

Composition			
Sand			61.7 %
Limon			23.5 %
Clay (particle size <2 µm)			14.8 %
Atterberg limits	Liquid limit LL	Norm NF P94-052-1	28 %
	plastic limit PL		14 %
	plasticity index PI		14 %
Other features	Blue value Norm NF P 94-068		1,1
	Sand equivalent SE Norm NF EN 933-8		13
	Optimum water content Norm NF P94-093		12 %

Physicochemical, mineralogical and mechanical characterization of fiber: To assess the mineralogical composition of the fibers, ground fibers (size <80 pm) was used; on which different analysis including X-ray diffraction, thermal gravimetric (TGA) and Fourier transform infrared (FTIR) were performed. Analysis regarding thermal gravimetric was carried out at temperature heating rate of 10°C/min. The diffractometer used was Bruker D 5000 brand apparatus with a graphite rear tube operating with Cu Kα radiation. Thermogravimetric curve up to 1100°C of the fibers was obtained with a SETARAM apparatus at heating rate of 10°C/min. The FTI Ranalysis was carried outby the mean of Nicolet 510FT-IR spectrometer operating at the wave length of 4000-400 cm^{-1}. The fibers for chemical analysis were pulverized in a mortar. The experimental technique used was the Van Soest procedure using four detergents: NDS (Neutral Detergent Soluble) NDF (Neutral Detergent Fiber), ADF (acid detergent fiber) and ADL (Acid Detergent Lignin) to quantify the amount of cellulose, hemicelluloses and lignin[15,16].

Cellulose is a linear polymer of β- (1-4)-D-glucopyranode, which could be classified into five different types. The first type I is the cellulose with the best physical and mechanical properties (150 GPa Young's modulus). It is a crystalline polysaccharide. The main components of hemicellulose is constituted include saccharides such as xylose, mannose and glucose. It forms a strong block with cellulose fibrils via the multitude of hydrogen bonds. According to Toledo Filho[17], lignins, as amorphous polymers formed by aromatic units such as guaiacycle the syringyl and phenylpropane; act as a cementing agent.

The mechanical characterization of Kenaf fibers follows the same methodology as cotton fibers. The fibers are glued on a paper frame of 3.5 cm high and 2 cm wide. The gauge lengths used are 5, 10, 20 and 30 mm. The frame is then placed in the manual clamping jaws of the testing machine (Instron 5566) with a 10 kN cell for monotone uniaxial tensile test. The stress is applied at aconstant displacement cross speed (10% l_0 per minute). During the test, the force applied to the sample and the displacement of its point of application (movement of the movable cross member) are recorded.

Compressed earth blocks (CEB) mechanical characterization: The flexural and compressive strength were determined accordance to European standard EN 196-1[18]. The measurements were performed on a Zwick mechanical press with a capacity of 50 kN. The 4x4x16cm^3 prism samples were first submitted to a bending test of three points until the rupture in order to assess the flexural stress. The two parts resulting from samples rupture were then recovered and submitted to a single compression test on a plan of 4x4cm^2 area. For Each formulation, we made: three trials of three-point bending tests and six replication of compression test.

Experimental design: The compressed earth blocks (CEB) were made using the conventional technic (kneading, compression and release). The dry soil was first coarsely crushed to obtain particles smaller than 5 mm. It is then mixed with sufficient water to achieve the optimum Proctor to obtain maximum compactness after compaction. Kenaf fibers were then added to the mixture. The weight of fiber was fixed at 1.2% of the dry mass of earth. The mixture is then placed in prismatic specimens molds of 4x4x16 cm^3 dimensions. It is then compacted based on the static mode with simple effect by a hydraulic press until a compaction stress of 5 MPa.The test pieces are demolded after compaction and kept in a conditioned chamber at a temperature of 20 ± 1°C and a relative humidity of 50 ± 5%. It were then dried untilmass stabilization (after 21 days) (Table-2).

Results and discussion

Characterization physical chemistry, mineralogy and mechanical of Kenaf fiber compared with flax fibers: The X-ray diffraction allow to observe the crystal structure of vegetable fibers. The crystalline fraction of vegetable fibers corresponds to one ofthe polymorphic states of cellulose. Diffractograms of Figure-2 are similar and allow to identify clearly 4 peaks. The peaks at 15.02 and 16.03° (2θ CuKα) correspond to the crystallographic planes (110) and (111) of the cellulose I. The main peak at 22.7° corresponds to the plane (002) and finally, the one identified at 34, 34 corresponds to the plane (023) or (004). For Kenaf, it is difficult to distinguish the 15.02 peak from the one at 16.03°. This reflects a high content of amorphous materials such as lignin, hemicellulose and amorphous cellulose. In contrast, when crystalline cellulose content is high, these two peaks are well separated as observedon flax diffractogram. The crystallinity index of the fibers, corresponds to the ratio of the mass of the crystal phase of cellulose versus the total mass of the sample (cellulosesemi crystalline + hemicellulose + pectin + lignin). This crystallinity index was determined for Kenaf and flax fibers based on the method described by Segal et *al*[19] according to thee quation-1 below:

$$Ic = \frac{(I_{002} - I_{AM})}{I_{002}} \quad (1)$$

I_{002} corresponding to the plane (002) of the crystalline phase (2θ = 22.7°) and represents the intensity of both amorphous and crystalline material. I_{AM} represents the diffraction intensity of the amorphous phase at $2\Theta = 18$ and corresponds only to the intensity of the amorphous portion of the material. In practice, I_{AM} is the minimum between the diffraction peaks of the planes {110} and {002} in the crystalline phase. Using the above-mentioned formula, the Ic values of 49.2 and 77.8 were found respectively for Kenaf and flax crystallinity.

In order to perfect the mineralogical characterizationof the CEB and particularly that of amorphous compounds within, milled samples were passed through TGA and FTIR analysis (Figure-3).

During their sintering, the thermal property of the fibers was determined as described by El-Shekeil et *al.*[20] and Morán et *al.*[21]. The profileof samples weight loss as a function of temperature revealed the presence of three characteristic zones:

The first zone (between 30 and 110°C) marks the elimination of the water initially present in the sample. This fraction is not important because the samples were dried before the test;

The second zone, covering the temperature range from 210 to 400°C, characterized by a significant loss of weight of the samples, corresponds to the active step of fibers pyrolysis. During this stage the main components of fibers (cellulose, hemicellulose and lignin) are degraded. This degradation is characterized by the presence of an exothermic peak around 335°C at the DSC attributed to the decomposition of the cellulose. This great mass loss indicates that cellulose is an important constituent of fibers. Finally the last area between 410 and 500°C corresponds to the passive step of the pyrolysis of lignin. This area is also marked by the presence of an exothermic peak around 450°C. The assignment of this peak to the decomposition of lignin is supported the very high thermic resistance of lignin macromolecule. Lignin has great thermal stability due to the possibility of forming hydrogen bonds, which would strengthens the stability of the molecules. Fourier Transform Infrared Spectroscopy is a widely used analytical technique for the identification of chemical bonds characteristics of a given material. The spectra obtainedby applying it to Kenaf and flax fibersare shown in Figure-4.

Table-2: Experimental design.

Treatments	Fibers contents	Fibers length [mm]	Mass content of soil water	water content of the mixture	Remark
A0	0	-	0%	12 %	not bundle reference mixture
A1	1.2 %	10	0%	11.4%	
A2	1.2 %	20	0%	11.6%	
A21	1.2 %	20	0%	12.5%	
A3	1.2 %	30	0%	11.8%	

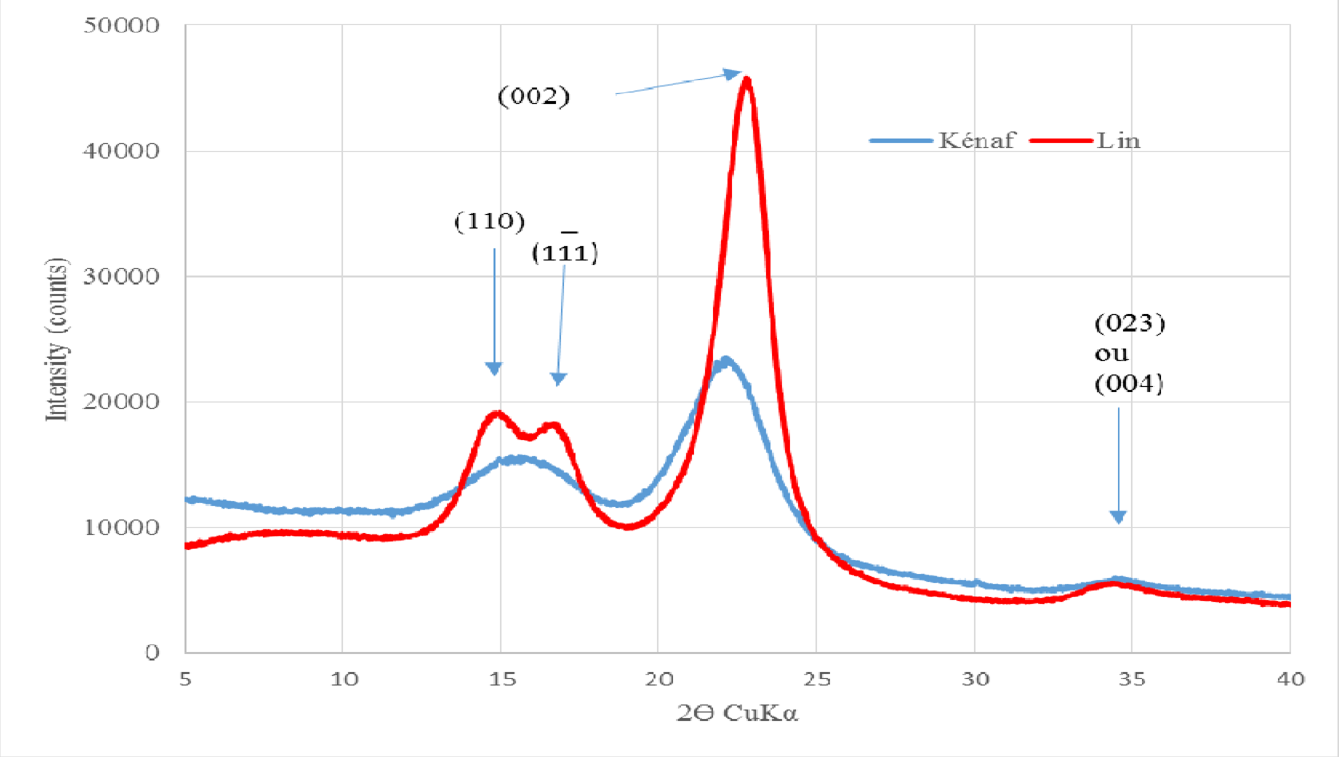

Figure-2: Kenaf fibers diffractogram compared to that of flax.

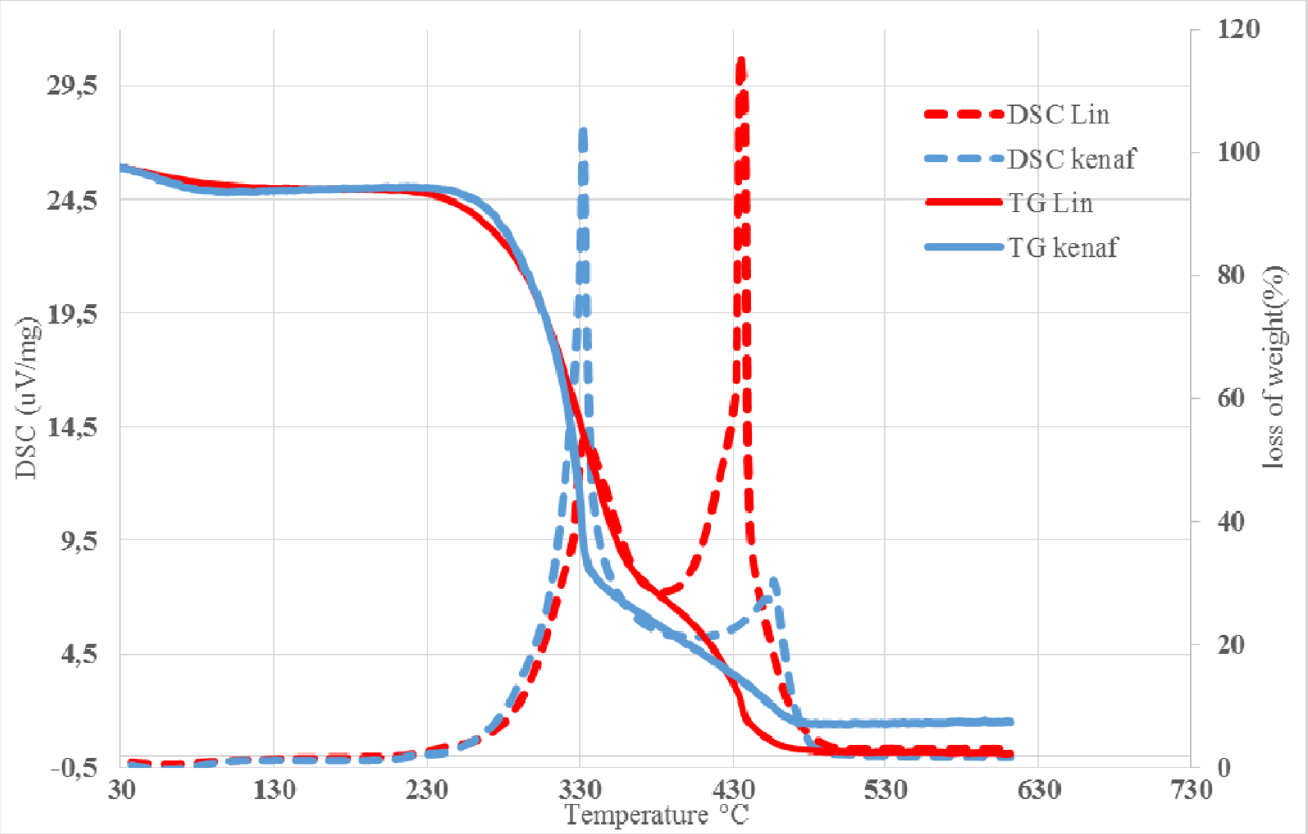

Figure-3: Fiber's TG and DTA curves.

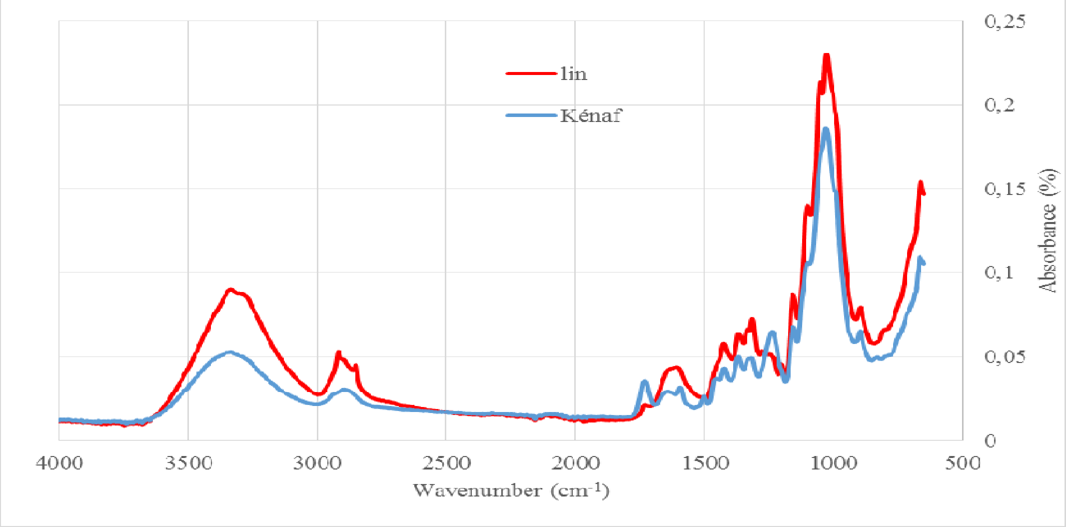

Figure-4: Infrared spectrum of Kenaf fibers and flax.

The wide absorption around 3400 cm^{-1}would correspond to O-H stretching. The same absorption would also characterizes absorbed water by the fibers' O-H stretching. The main peaks were identified based on the statement made by Millogo et al.[1], Ivanova et al.[22], Morán et al.[21] and Shin et al.[12] as reported in Table-3.

Table-3: Main infrared absorption bands Kenaf and flax[1,12,21,22.]

Wave number cm^{-1}	Designation
3400-3345	Characteristic stretching vibrations of O-H bonds of cellulose and hemicellulose
2915	Stretching bonds of C-H of cellulose and hemicellulose chains
2848	Stretching of $-CH_2$ bonds of cellulose and hemicellulose
1725	Stretching of C = O bond of carboxylic acids present in pectins and in acetyl groups present in the hemicelluloses
1625	Shear bond vibration-OH characteristic of free water
1503	Cycles aromatiques de la lignine (seulement pour la fibre de Kenaf)
1420	Vibration of the characteristic carbonyl bond pectins
1360	shear vibration of C-H bonds of cellulose and hemicellulose
1310	Stirring of the two H-C bonds of CH_2-groups of cellulose and hemicellulose
1230	Characteristic peak of lignin (Kenaf)
1162	Stretching of C-O bonds of acetyl groups (found in hemicellulose or pectins)
1108	skew distortion of the group C-O-C
1032	Stretching of the C-O bond
890	characteristic vibration of the β binding cellulose

Lignin, cellulose and hemicellulose's backbone bands, have been highlighted.

Chemical compositionof Kenaf and flax fibers have been compared to other types of fibers (Table-4). It could be seen that the fiber content in hemicelluloses and lignin depends on the origin of the Kenaf material used. Benin's Kenaf fibers cellulose content was in the same order of magnitude as that fund in Burkina which was previously studied by Millogo et al[1] (Table-4). This difference between Benin's fibers cellulose contents and others reported in the literature would be explained by the difference in the climate, soil type and plant species. The hemicellulose content was similar to that of Burkina; whereas lignin content is lower than the values availablein the literature. The discussion was mainly on cellulose contents, which playsa greatrole on the mechanical properties of the fibers due to its high tensile strength. The cellulose content of the fibers shown in Table-4 is almost the same for flax, hemp, jute and ramie. Studied Kenaf fibers contained cellulose than that sisal, ramie and jute fibers which emphasized Kenaf high mechanical strength. Figure-5 shows the variation of the average tensile strength of the Kenaf fibers as a function of the gauge length. One can note that the resistance decreases when the gauge length increases. This could be explained by the fact that the rupture occurs by the sudden spread of pre-existing defects and also by the fact that the density and the severity of these defects increase with the tested length. This was in agreement with Mouhoubie[23] and Anthony[24] findings reported on alfa, the diss, and flax fibers, and also synthetic fibers such as glass, carbon[25]. There is also a significant dispersion of the breaking stress. This dispersion may be explained by the fact that the section of those fibers varies along their length. Regardless of the gauge length considered, the average tensile strength of the Kenaf fibers is greater than that of sisal, coconut and palm oil fibers[17-19]. These values are in the same order of magnitude as those obtained by Millogo et al on Kenaf fiber harvested in the Bobo-Dioulasso region (Burkina Faso)[20].

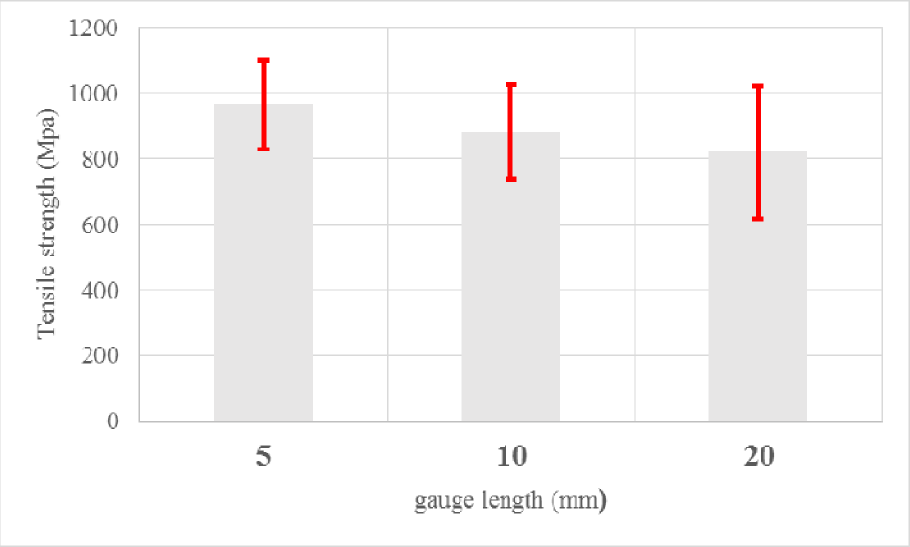

Figure-5: Tensile strength - Influence of the length of the Kenaf fibers.

Table-4: Composition of the fibers and comparison with other types of fibers.

Fiber	Cellulose (%)	Hemicellulose (%)	Lignin (%)	Other (compounds water-soluble, waxes, fats, etc) (%)	Reference
Studied Kenaf	**73**	**18**	**6**	**1.6**	
Kenaf	70	19	3	1.3	1
	53±4	18±1.4	8±1.2	-	30
	45-57	21.5	8-13	-	14
	58±1	22±1	17.5±1,3	2.4±0.4	13
	60.8	19.2	14.7	-	12
	31-39	21.5	15-19	-	31
Studied flax	**78.9**	**7.3**	**4.0**	**2.6**	
Flax	65-75	15	-	2-5	32
	71.2	18.6	2.2	6.0	33
	67	11	2	-	33
	80.1	14,9	3.1	-	34
	82± 3	7 ±3	-	5± 2	35
Chanvre	55	16	18	5	36
	74.4	17.9	3.7	0.8	37
	56.1	10.9	6	7.9	38
	76.12	12.28	5.65	3.29	39
Ramie	68.6	13.1	-	-	37
Jute	33.4	22.7	28	-	40
	64.4	12	-	-	37
Sisal	70	12	10	-	41
Coir	33.2	31.1	20.5		40

Mechanical properties of compressed earth blocks (CEB): Figure-6 presents the variation of the flexural strength function of the fiber length. The average values range from 1 to 2 MPa and an important dispersion of the flexural strength values for the treatment A3 it is noticed. This could be explained by the fact that during the preparation of the mixture, the use of low fiber length results in a more homogeneous mixture.

There was also a slight decrease in strength in the treatmentA1 compared to A0. This reflects the fact that the fiber length was not sufficient to prevent the propagation of cracks. Furthermore there is a positive effect of the fiber length in treatmentA3 showing that l = 30 mm is sufficient to prevent the propagation of cracks. The flexural strength of the reinforced BTC (1.2% Kenaf fiber lengths, l = 20mm and b = 30mm), is larger than that of the others values reported in the literature regarding the earth blocks stabilized with fibers[42-44]. This might be due to the strong tensile stress of Kenaf fibers relative to those used in others studies.

We distinguished three phases in the behavior to flexion of A2 and A3 (Figure-7): Phase I: quasi-linear behavior similar to that of A0, at this stage, efforts are mostly taken up by the matrix as evidenced by the bending energy. There was also a slight increase in the occurrence of the first crack stress compared to A0; Phase II: from **σ ***, there was appearance of the first crack of the matrix, followed by the charge resuming by the fibers, which limit the propagation of the crack by a bridging effect; Phase III: beyond σmax, there is a controlled stress reduction unlike A0 which suddenly breaks. This phase is associated with a progressive rupture of fiber/matrix interfaces followed by a loosening of fibers which causes a loss of adhesion between the fibers and the matrix.

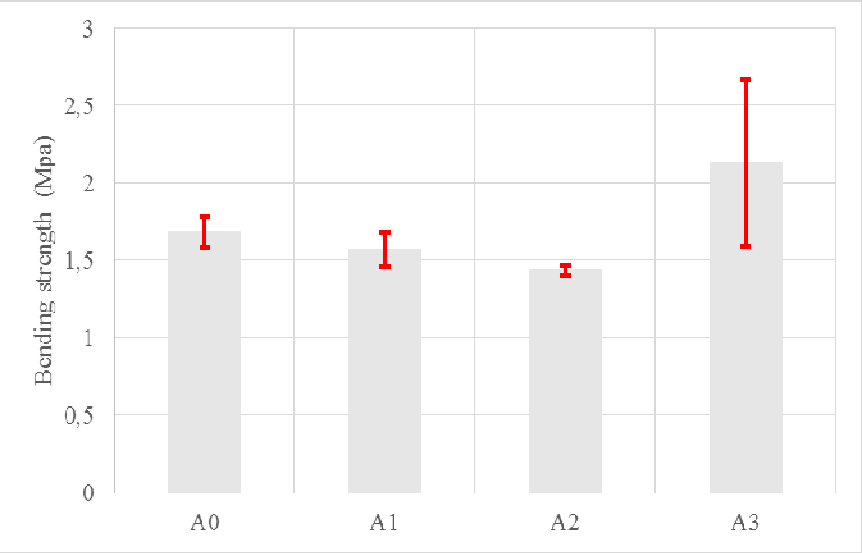

Figure-6: Evolution of the flexural strength in the treatments - Influence of fiber length.

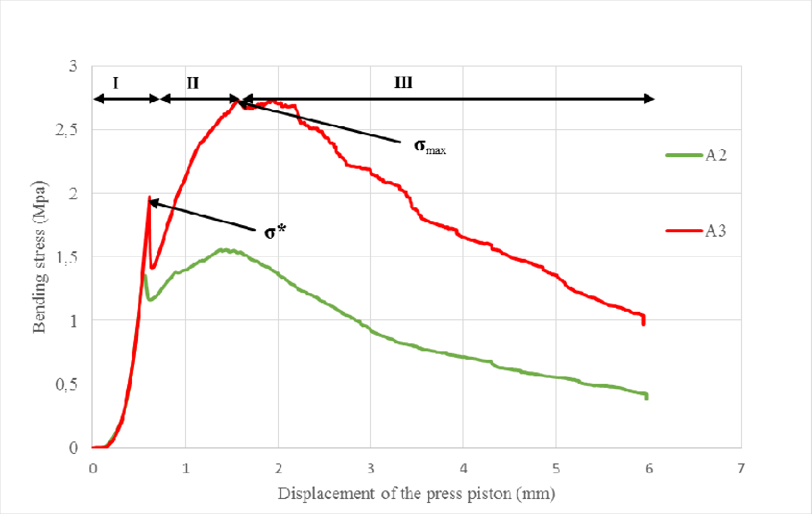

Figure-7: Bending behavior of CEB bundles.

Unreinforced BTC were fragile during bending tests, while those reinforced with fibers have a higher ductility, which is very important for masonry and usually show a brittle behavior. Blocks and mortar higher ductility makes masonry more stable to resist earthquakes and differential settlements, which limits the growth of cracks. In summary, the reinforced BTC ductility's increase erelied on the fibers which hold the cracks after the matrix crack. The resistance strength of the fibers through the cracks is mobilized by their integrated length, where a link is created primarily between cellulose molecules (negatively charged -OH bonds) and, cations such as Fe^{3+}, Ca^{2+} and Mg^{2+} flocculated within the soil. Figure-8 shows the variation of the compressive strength of BTC for different lengths of fibers. The addition of Kenaf fibers in the ratio of 1.2% dry weight increases the compressive strength of BTC. Furthermore there is an increase of the compressive strength in the treatments A2 compared to A1 and A3. In contract, when considering Figure-9, it appears that, for a deformation of 5%, the strain energy absorbed by the treatment A3 is greater than that absorbed by A1 and A2. This fact led us to conclude that in the mechanical point of view, the formulation A3 is the one having the higher resistance.

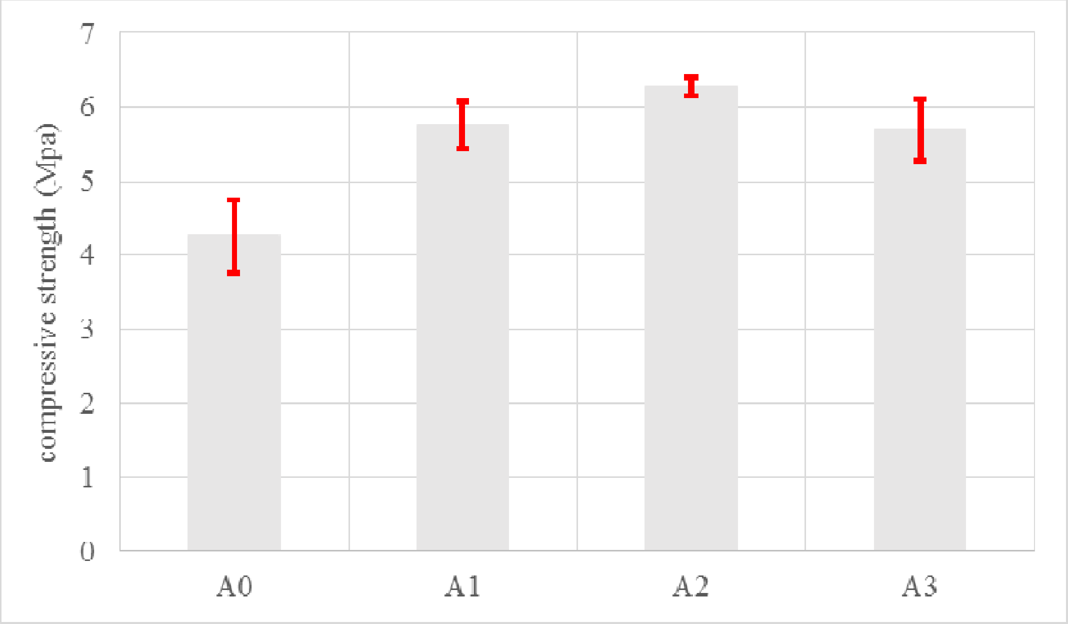

Figure-8: Compressive Strength - Influence of the length of the Kenaf fiber.

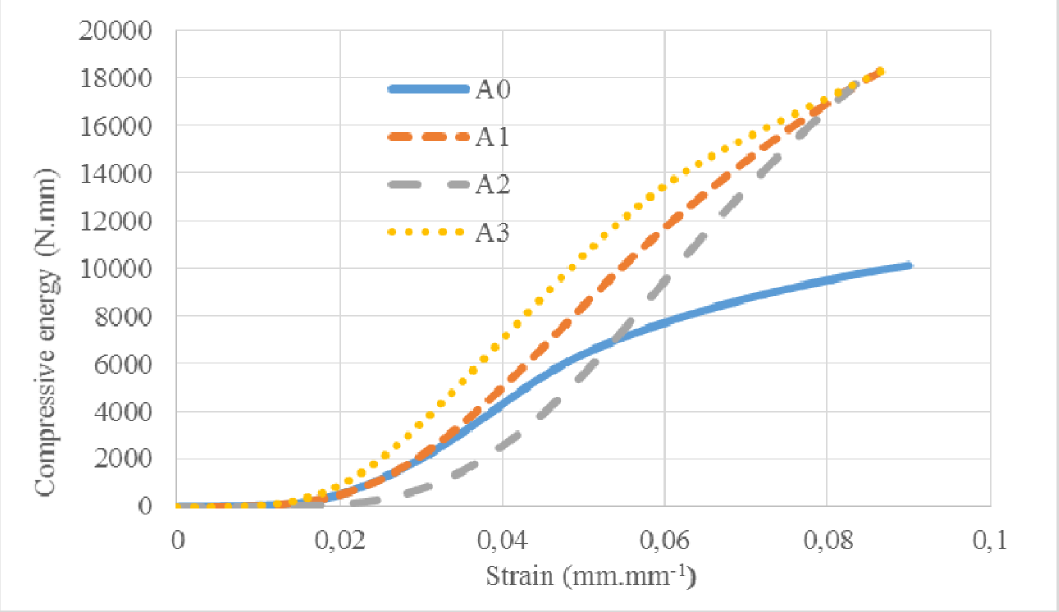

Figure-9: Influence of fiber length on the energy absorbed by the compression CEB.

Conclusion

Benin's Kenaf fiber physicochemical and mechanical characteristics were determined; and its impact of the mechanical and thermal properties of compressed earth blocks (CEB) were studied. The findings could be summarized as follows: i. The tensile strength of the Kenaf fibers decreases as the gauge length increases and regardless of the gauge length considered the average tensile strength of the Kenaf fibers is greater than the tensile strength of sisal, coconut and oil palm fibers. ii. The studied Kenaf fibers were mainly consisted ofcellulose I (73% wt; Ic = 49.2), hemicelluloses (18 %wt) and lignin (6%wt). iii. The Addition ofKenaf fibers in earth for compressed earth blocks (CEB) fabrication mainly enhanced the tensile ductility of the blocks; which could be attributed to the high mechanical strength of fibers. iv. The incorporation of Kenaf fibers 30cm is the best treatmentas far as mechanical behaviors are concerned.

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