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Performance of lightweight mortar reinforced with doum palm fiber

Fatma Naiiri^{1,2} , Allègue Lamis¹, Salem Mehdi², Zitoune Redouane² and Zidi Mondher¹

Abstract

Natural fibers are increasingly used in composites because of their low cost and good mechanical properties. Cement reinforced with natural fibers is contemplated as a new generation of construction materials with superior mechanical and thermal performance.

This study of three sizes' effect of Doum palm fiber explores the mortar's behavior reinforced with different fiber ratio. The aim is to determine the optimal addition to improve mechanical and thermal properties of natural fiber reinforced cements. Physical, mechanical and thermal properties of composite are examined. Tensile properties of Doum fibers are verified to determine their potential as reinforced material. Findings prove that the use of alkali-treated Doum fiber as reinforcement in cement mortar composite leads to the upgrading of the mechanical properties including thermo-physical properties against composites reinforced with raw fibers and control cement mortars. While, the compression and flexural strength of the cement mortar reinforced with alkali-treated Doum fiber with diameter 0.3 mm (CT3) are metered to be 11.11 MPa, 5.22 MPa, respectively for fiber content 0.5%. Additionally, based on thermo-physical tests, it is assessed that the thermal conductivity and diffusivity decrease for cement mortar reinforced with Doum fiber with diameter 0.2 mm (CT2).

Keywords

Cement mortar, doum palm fiber, treatment, mechanical properties, thermal properties

Introduction

Owing to rapid urbanization, buildings are designed without taking into account their environment. Standard mortar is deemed as an important construction material, it is gradually spent for civil infrastructure all over the world, which has poor thermal insulation properties. Contrary to vernacular and traditional buildings where people used to build their houses using natural materials.

Frequently used construction materials, including synthetic fibers, increase pollution, regarding their disposal environmental issues and CO₂ manufacturing emission.¹

Industries have recently been focusing on the development of lightweight and eco-friendly construction materials.² Therefore, they have marked an awareness to limit the use of cement reinforced with synthetic fibers. For this reason, researchers have records to innovate sustainable materials to replace synthetic

fibers by natural fibers as reinforcement of structural materials.³

Many researchers deal with the use of palm fibers as reinforcement in construction materials due to their important insulation properties. Mansour et al.⁴ investigated the effect of rachis and petiole of palm trees on compressive, flexural strength and thermal conductivity of cement mortar.

Agoudjil et al.⁵ highlighted that the date palm waste is a good product for the development of safe and efficient insulating materials. For this purpose, Chikhi et al.⁶ studied a new biocomposite material elaborated

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with date palm fibers and gypsum. The fibers were obtained by grinding the date palm waste wood. These authors publicized that this type of biocomposite exhibits good mechanical and thermal performances, which allows for being appropriate for thermal insulation in building. In a similar way, Benmansour et al.⁷ investigated the use of new material composed of mortar reinforced with date palm fibers. The authors underlined that using these fibers reduces the density and the thermal conductivity of this kind of bio composite. Relating to RILEM classification,⁸ these bio composite materials (according to the concentration and fibers size) can satisfy both mechanical and thermal requirements of lightweight concrete.

Another disadvantage of standard mortar is the fact that it has an elastic deformation followed by a sudden fracture. They do not have a post cracking ductility.¹

Natural fibers, such as hemp, palm, coconut, and cotton have been examined as potential alternative for synthetic fibers in accord with their low cost and availability in fibrous form.

By focusing our attention on the natural fibers, several studies in the similar field are those by Savastano et al.^{7,9-11} who determines elastic modulus, flexural strength and toughness of cement matrix, alternatively reinforced with banana, eucalyptus or sisal pulps fibers. They draw attention to the fact that by adding 8% of fibers to the mix, the properties mentioned above increase, compared to the reference mixture. They also show a decrease in the workability of cement reinforced with coir or eucalyptus pulp. This decrease in workability is the result of moisture absorption by natural fibers.

The result of Ramakrishna¹² reports that the impact resistance of mortar reinforced with plant-fiber are from 3 to 18 times higher than unreinforced mortar. However, the addition of microfibers also reveals a decrease in the compressive strength of the mix that is relative to plain mortar due to the increase in the air content of the mixture.

Compared to composite without fibers, Mansour and Azur¹³ show a decrease in the mechanical properties of composites reinforced with jute fibers as the content and length of fibers decrease.

However, in spite of the natural fiber advantage, their usefulness as reinforcement of a cement matrix, are restricted by their degradation in an alkaline environment which conducted to, a loss of the composite strength.^{9,11} This problem presents an important matter which needs to be solved before the commercialization of natural fiber as reinforcement for cement matrix in industries.

On the other hand, mechanical properties of composites depend on the adhesion between fibers and matrix. The higher water absorption of natural fibers

linked to their dimensional variations leads to forming to voids and micro-cracking at the interfacial transition zone, which results in the reduction of the interfacial adhesion between cementitious matrix and fiber.^{14,15} It is obvious that producing composite with higher mechanical properties from adding natural fiber is definitely linked to good adherence between the matrix and the fiber. Natural fiber has different chemical structures which induces to an inefficient transfer of stress to the interface. For all these findings, treatment of the fiber surface becomes a requirement.¹⁶

For all these reasons, in order to improve the interfacial adhesion between the matrix and fiber and to minimize the absorption of water, numerous researchers have used different chemical pretreatments of the fibers.¹⁷⁻²⁰

Among these treatments, using alkaline treatment with Sodium hydroxide solution (NaOH) removes the amorphous materials, such as lignin, hemicellulose, pectin and waxes from the outer surface of the fiber. This treatment increases the degree of the fiber's crystallinity and its surface roughness.²¹

Fantilli et al.²² experimentally investigates mortar reinforced with both raw and treated wood fibers, by evaluation flexural strength, fracture toughness, and elastic modulus. They find a remarkable increase of the mechanical properties by adding 1% of treated wood fibers by volume, with an increase of fracture toughness equal to 300% compared to plain mortar.

Doum palm trees are abundant in Tunisia. A mature palm tree has generally 64-128 leaves with fibers and spines on the petiole and renews 10-30 leaves every year.

In this context, this paper primarily provides new information about the Doum palm fibers and presents a variety of results on the effect of NaOH treatment of Doum palm fibers, like,

Fourier-transform infrared spectroscopy (FTIR), X-ray diffraction (XRD) and mechanical properties. The alkali treatment proves itself to be effective to upgrade the physical and mechanical properties of Doum palm fibers, and as such improving fibers matrix-interface interaction. Elsewhere, the effectiveness of alkali-treated Doum palm on the compressive strength and thermal properties has been discussed.

Materials

Presentation of doum palm fiber

The doum palm is composed of a long branch, a mesh surrounding the trunk, leaves (containing Petiole and Feliolo) and fruit branch.

The doum palm is commonly founded in North Africa and grows up to 17m. In Tunisia, this tree is

used as a decorative plant in urban and rural areas and the annual trimming operation generates tremendous quantities of agricultural waste, which are usually discarded.

These wastes can represent a plentiful source of low-cost material for industrial uses. Thus, the development of cement mortar reinforced with this abundant material depict a great solution for the economical utilization of the same wastes as a renewable resource.

The Doum palm fiber is a natural fiber extracted from the petiole of Doum palm which is harvested in Tunisia (Figure 1).

Firstly, petiole is crushed using grinder made in a mechanical engineering laboratory of the national engineering school of Monastir. Secondly, the fiber is screened using different meshes of the grid. From this method, a fiber with diameter varies from 90 μm to 500 μm is obtained (Figure 2).

Extracted fiber is washed to remove impurities and residues. The washing processes are important to enhance fiber durability because residues can induce the development of fungus on the fiber surface which decreases their utility.

Alkali treatment's objective is to remove lignin and hemicellulose from the fiber wall and enhance surface roughness which has a positive impact on the fiber-matrix bond.²³ The treatment decreases the degree of crystallinity and polymerization as well as the destruction of structural linkage between cellulose and lignin and decomposition of the lignin structure.²⁴

Collected fiber is screened using different normalized meshes of the grid. Three types of fiber sizes were obtained; they are listed and presented in Figure 3

- Reinforcement sieve1 with 0.1 mm mesh noted (RS1)

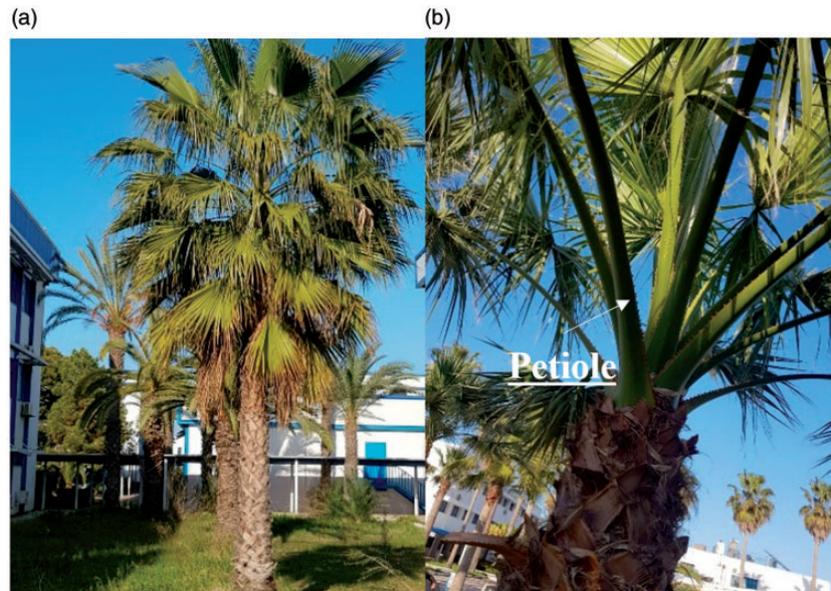


Figure 1. Doum palm: With (a) palm tree, (b) palm petiole.

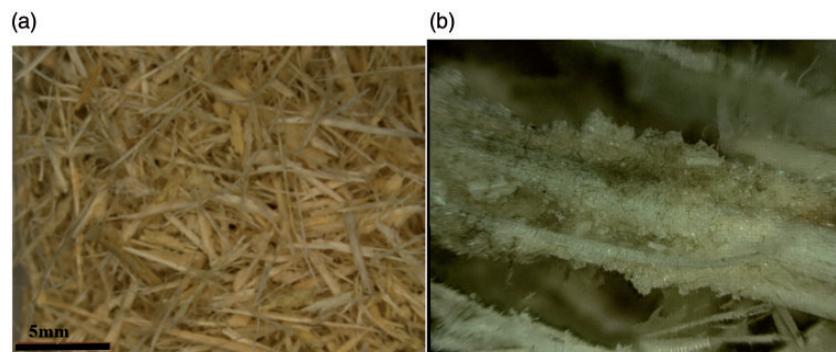


Figure 2. Digital microscope of raw Doum fiber.



Figure 3. Pictures showing the three kinds of Doum palm fibers with: (a) fibers referenced under RS1, (b): fibers referenced under RS2 and (c): fibers referenced under RS3.²⁵

Table 1. Cement characteristic.

CaO(%)	SiO ₂ (%)	Al ₂ O ₃ (%)	Fe ₂ O ₃ (%)	MgO(%)	K ₂ O(%)	Na ₂ O(%)	SO ₃ (%)
60	17	4	3	5	1	1	3.5

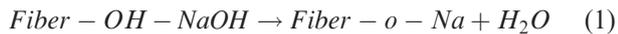
- Reinforcement sieve2 with 0.2 mm mesh (RS2)
- Reinforcement sieve3 with 0.3 mm mesh (RS3)

Fiber treatment

The main component of natural fiber is crystalline cellulose. It also contains lignin, hemicellulose and wax substance. Nevertheless, hemicellulose and lignin can degrade over time. Accordingly, to enhance the long-term durability of cement mortar reinforced with natural fiber and to raise interfacial bonding, an alkali fiber treatment with NaOH solution can be employed.

Hemicellulose which mainly consists of hexosan, xylan, and polyuronide, is very delicate to the action of caustic soda. Thus, alkali fiber treated with NaOH can enhance adhesion characteristics of natural fiber by striking out pectin, hemicellulose, and lignin consequently giving to the fiber a rough surface.

The major modification in this treatment is the removal of hydrogen bonding from the structure. The reaction, that takes place between fiber and alkali treatment, is presented in the following equation (1):



The Doum palm fiber was treated using 1 wt% hydroxide sodium in water. They are soaked in the alkaline solution for 1 hour at temperature 105 °C. Subsequently, the alkalinized fiber is washed using distilled water until it eliminates all the residues of the sodium hydroxide. Afterward, the washed fiber is bleached with sodium chloride and finally washed with distilled water.²⁵

Cement

A Portland cement CEMII 32.5 R is used for all mixture, produced by Carthage Company of Tunisia conforming to ASTM C1329 – 05 requirements.²⁶

The chemical compositions are shown in Table 1.

Portland cement presents an alkali environment because it contains important quantities of CaO (Table 1). When it reacts with water it produces a solution oversaturated by Ca(OH)₂, (CH) and small percentages of Na₂O and K₂O which forms NaOH and KOH solutions.

Sand

Rolled sand with granulomere (0/2) is used in the experiments according to ASTM C348 requirements.²⁷ It is purchased from the quarries of the National company of Granulats Jbel OUST (GJ) in Jabal OUEST Tunisia.

The cumulative percentage graph of sand is given in Figure 4.

After analysis, the values appeared are tabulated below Table 2.

$$\text{FM} = 2.315$$

The fineness modulus of fine aggregate is 2.315. It means that the average aggregate size is between 2.2–2.8. The used sand is preferential sand according to Standard NF 18-540.²⁸

Composite preparation

The operating procedure for the manufacture of mortar consists of manufacturing traditional mortar (a mixture of sand, cement and water) and after adding different length of Doum palm fibers. For the

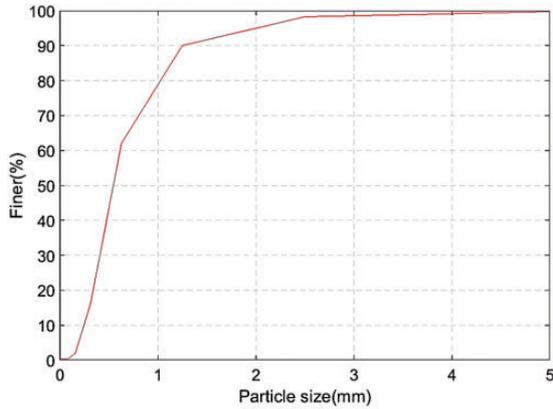


Figure 4. Grain size distribution.

preparation of composite, mixing is executed using an automatic mortar mixer with maximum capacity 5 l.

Figure 5 illustrates the used method to prepare the composite.

To minimize the absorption of water by fibers, fibers are soaked in 10% of water. In preparation of mixtures, firstly sand with 30% of water is added to the mixing container. The mixture is started to be mixed at low speed for 30 seconds. After those fibers with 30% of water are gradually added to the mixture to avoid the grouping of the fibers and also the abrupt absorption of large quantities of water. The mixer is stopped in 30 min. Finally, cement and water are continuously added and the mixer is adjusted to high speed.

Table 2. Values after analysis.

Sieve size	Weight retained (g)	Cumulative weight retained (g)	Cumulative percentage weight retained (%)
5	0.5	0.5	0.25
2.5	3	3.5	1.75
1.25	16.5	20	10
0.63	56	76.0	38
0.315	91.5	167.5	83.75
0.16	28.5	196.0	98
0.080	3.5	199.5	99.075
Bottom	0.5	200	100

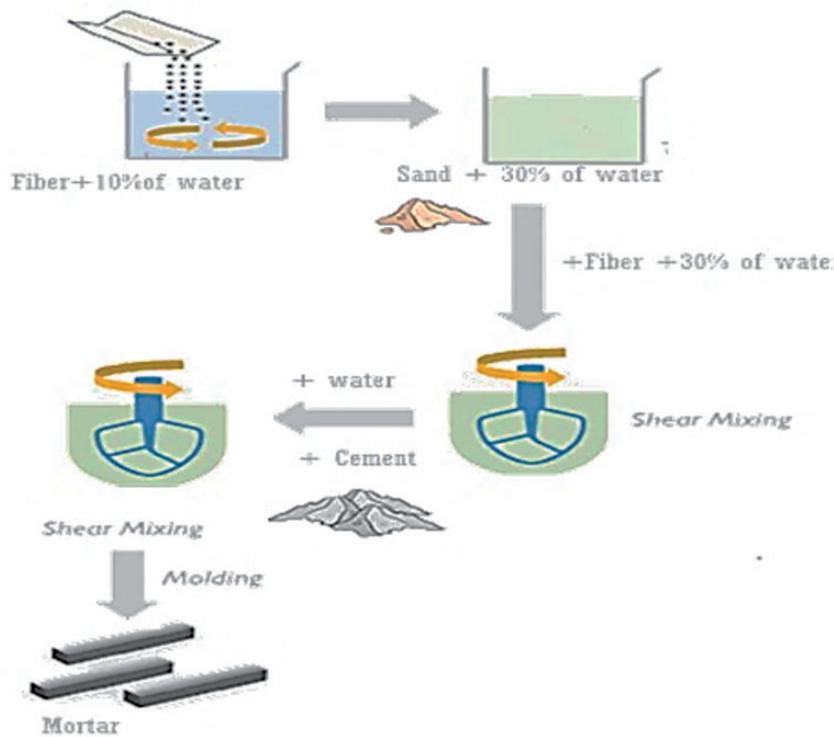


Figure 5. Schematic representation of the manufacturing process for cement mortars.

Table 3. Mix proportion of the cement mortar (Kg/m³).

Fiber ratio %	Cement (Kg)	Sand (Kg)	Water (Kg)	Fibers (Kg)
0	450	1350	225	0
0.5	450	1350	250	10.12
1	450	1350	265	20.25
1.5	450	1350	300	30.37
2	450	1350	300	40.49
2.5	450	1350	300	50.625

The mixer is stopped after 2 min and 30 s and the adhered mortar to walls is scratched by scraping. The obtained mortar is placed into 4 mm × 4 mm × 16 mm dimensions molds.

Firstly, the half volumes of molds are filled with mortar and are shaken using a shaking table for 30 s. After that, the molds are filled and shaken for another 30 seconds. The specimens are unmolded after 24 hours and they are cured under laboratory conditions for 28 days. Each experimental test is repeated 3 times.

The proportions of materials for the reference mortar was one part of cement to 3 parts of sand by weight with 0.5 water-cement ratio. For the reinforced specimens, the quantity of water is adjusted to have the same workability as the reference mortar

To study the effect of the quantity of fibers added to cement in the composite properties, a five-weight fraction of Doum palm fiber is used from 0.5% to 2% with a step of 0.5%. We notice that we used untreated and treated fibers for all the study.

Natural fiber presents a sensibility to the alkalinity of cement. To remedy this problem, doum fiber is treated with 1% NaOH solution for 1 hour at a temperature of 105 °C.²⁵

The details of the mix proportions per cubic meter for all composites are presented in Table 3.

The designations of the various cement mortars are presented in Table 4.

Experimental methods

Fiber chemical composition properties

To enhance and develop better evaluations of Doum fiber and their capacity, many factors are determined for their selection as reinforcement in the bio-based material. The considered evaluation criteria includes chemical, physical and mechanical tests are evaluated.

The cellulose, hemicellulose and lignin contents are calculated by the traditional Van-Soest method (Figure 6).²⁹ Doum powder of 0.50 g is properly weighed for the chemical measurement. The chemical measurements are repeated for three sample with a relative error lower than 5%.

Table 4. Composite designation.

Mix type	Matrix
RCM	Reference cement mortar
CNT1	Cement mortar + untreated fiber sieve 1
CNT2	Cement mortar + untreated fiber sieve 2
CNT3	Cement mortar + untreated fiber sieve 3
CT1	Cement mortar + treated fiber sieve 1
CT2	Cement mortar + treated fiber sieve 2
CT3	Cement mortar + treated fiber sieve 3

**Figure 6.** Van-Soest method.

Water capacity absorption

In order to measure the water absorption capacity of raw and treated fiber, a procedure inspired by the German codex's STN2: 117/87 standard is used.

In practice: a selected mass of each sample is dried in the oven until its mass is constant. The sample is then placed in a hydrophobic veil bag (the weight of which is well known in advance) and weighed (M_0). Then immersed in distilled water for 30 minutes at room temperature. The sample is then drained for 5 minutes and weighed again (M_1).

Water absorption capacity is determined by the following formula:

$$\frac{M_1 - M_0}{M_1} \text{ (g/g)}$$

X-ray diffraction

The crystallinity of fiber is studied by X-Ray diffraction (XRD). The XRD analysis of Doum palm fiber is carried out using a D8 advance diffractometer.

Diffraction diagrams are monitored by recording X-ray diffraction patterns between 0° and 70° (2θ angle range).

The crystallinity index (I_C) is calculated using the following expression used by Segal³⁰ (equation (2))

$$I_C = \frac{I_{002} - I_{am}}{I_{002}} \times 100 \quad (2)$$

I_{002} is the maximum intensity of diffraction of the (0 0 2) lattice peak 2θ between 22° and 23° , and I_{am} is the intensity of diffraction of the amorphous material, which is taken at 2θ angle between 18° and 19° where the intensity is at a minimum.³¹

Scanning electron microscopy image

The scanning electron microscopy (SEM) is used to examine the influence of chemical treatment on the surface morphology of Doum palm fiber and the interfacial properties of Doum mortar composites.

The microscopic examinations are performed using scanning electron microscopy type HITACHI-TM3030 which provides magnifications up to 60.000 times. The accelerating voltage is 15 kV and 5 kV. This test is realized in the Roberval laboratory, Compiègne University of Technology in France.

Density in hardened states

Bulk density is measured in hardened state, according to EN1015-10, using $40 \text{ mm} \times 40 \text{ mm} \times 160 \text{ mm}$ test specimens, following 28 days of curing at a temperature of $20 \pm 1^\circ \text{C}$, at a relative humidity of $65 \pm 5\%$.

Composite mechanical properties

Specimens are removed from molds after 24 hours and are left under normal conditions (Temperature $T = 20 \pm 2^\circ \text{C}$ and relative humidity $\text{RH} = 65 \pm 5\%$) during 28 days until testing.

Compressive strength and flexural strength were measured at 28 days, as per Standard EN 1015-11,³² using a LLYOD equipped with a 20 KN load cell. Three different test specimens are tested under flexion for each combination and six under compression. The test specimens measured $40 \text{ mm} \times 40 \text{ mm} \times 160 \text{ mm}$ and the bottom support rollers are separated at intervals of 100 mm.

The flexural strength is calculated through the following relationship (equation (3)) according to NF EN 12390-3 standard.³³

$$\sigma = \frac{3FL}{2wh^2} \quad (3)$$

Where L is span length, w is the width of the specimen, F is applied load and h is the thickness of the specimen.

The compressive strength is calculated with this formula (equation (4)):

$$\sigma_c = \frac{F_c}{S} \quad (4)$$

Where F is the load and S is the section of the applied force.

Thermal conductivity

All thermo-physical tests of composites are performed using samples with dimensions of $4 \times 2 \times 1 \text{ cm}^3$.

The thermal conductivity and diffusivity are determined using the Hot disk method TPS 2500 S with a precision of 5% under the following climatic conditions: $T = 21 \pm 2^\circ \text{C}$ and relative humidity $\text{RH} = 45\% \pm 10\%$.

The hot disk technique is used for a transient measurement of thermal conductivity and thermal diffusivity. It consists of fitting the sensor between two pieces sample, each one with a plane surface. The sensor used in this test method is made of nickel foil, which is wound in a double spiral pattern with a 6.403 radius.

For all specimens, each measurement is relative to three similar tests.

To estimate the potential of composite for building, the thermal diffusivity is calculated using the following (equation (5)):

$$a = \frac{\lambda}{\rho c} \quad (5)$$

Where λ is the conductivity (W/m K), ρ is the density (Kg/m^3) and c is the specific heat (J/kg K)

Results and discussions

Fiber chemical composition properties

To study the effect of NaOH treatment on the mechanical and physical properties of fiber, many tests are carried out on the raw and the treated fiber. The results were presented previously by Naiiri et al.²⁵

The NaOH treatment leads to a reduction of the fiber diameter with a decrease of the density by removing the lighter compounds from its surface.

An important amelioration of the mechanical properties and tensile strength is noted due to the higher homogeneity inside the fibers.²⁵

The chemical characterization of raw Doum fiber as shown in Table 5 prove that it contains cellulose (42.3%), hemicellulose (21.85%), lignin (18%). It also reveals that the pectin and protein content is 5.51% and moisture 17.61%.

Table 5. Various properties of the commonly used fibers compared with used fiber in this study.

Type of fiber	Doum palm fiber	Date palm fiber	Flax	Hemp
Cellulose (%)	42.3	46	62–72	68–74.4
Hemicellulose (%)	21.85	18	18.6–20.6	15–22.4
Lignin (%)	18	20	2.3	3.7–10
Pectin and protein (%)	5.51			
Moisture content (%)	17.61	5–21.1	88–12	6.2–12
Elongation at break (%)	7.89	2–19	1.2–3.3	1–3.5
Tensile strength (MPa)	155.88	97–275	343–2000	270–900
Reference	This study and Fatma et al. ²⁵	Hamza et al. ³⁴	Salit et al. ³⁵	Li et al. ³⁶

It can be concluded that fiber with higher cellulose contents has better mechanical properties. Hemp and flax have relatively higher values of tensile strength than the date and Doum palm fiber as they have higher cellulose content. On the other hand, the quantity of cellulose hurts the fiber elongation to break property. From this table, results reveal that Doum and date palm fiber have a better elongation to break characteristics due to their lower content of cellulose.

Absorption capacity

Absorption capacity being the amount of liquid remaining associated with the sample after impregnating and draining.

The absorption capacity of alkali-treated fiber increasing from 0.52 g/g to 0.71 g/g, a result that confirm that treated fibers have interesting hydrophilic traits due to the elimination of hydrophobic materials and the increase in hydroxides.

X-ray diffraction method

An alkaline treatment using a NaOH solution is frequently used to increase the crystallinity index of natural fiber.

This index is related to the arrangement of cellulosic chains. The removal of an amorphous constituent from the surface of the fiber provides an increase of the crystallinity index and accordingly of the cellulosic chains. The X-ray diffraction patterns of Doum palm fiber are shown in Figure 7.³⁷ The major crystalline peak occurs at around $2\theta = 22.4^\circ$, which represents the cellulose crystallographic plane (0 0 2). In our case, a pronounced peak at 22.5° is observed.

After the treatment, the main peak is increased which translates to an increase in the degree of crystallinity of the fiber during their immersing.³⁷

Alkali treatment leads to the elimination of amorphous or poorly crystallized constituent for the fibers. The estimated values for treated and untreated Doum palm fiber are presented in Table 6.

Thus, the presence of important quantities of amorphous elements inducing spaces and disoriented areas could negatively influence the crystallinity index of fiber.³⁸

When fibers contain large quantities of amorphous material, the two-diffraction peak around 14.8° and 16.4° correspond to the (101) and (101) crystallinity planes appears as one broke peak. From Figure 7 we can observe that these two peaks are more defined for the treated Doum palm fibers than for the untreated fibers. It is in accord with the increase of the crystallinity index presented in Table 6.

Morphological characterization of fibers

Knowledge of fiber morphology is important because it provides information on the interfacial adhesion between the fibers and the matrix. To characterize the morphology of Doum palm fibers, a HITACHI TM3030 scanning electron microscopy (SEM) and Keyence Digital Microscope VHX 2000 are used.

Figure 8 represents the longitudinal views of Doum palm fibers. They show that they have irregular shapes covered with a layer of gummy and waxy substances. Their structure is similar to natural composites.

The cellulose fibrils are held together by lignin and hemicellulose to form fibers. For plants, the fibers are linked together by lignin which has a high solubility in the alkali environment of the matrix.

A cross section of petiole fiber (Figure 9) shows the presence of many numbers of hollow single fibers bonded by a layer. Elsewhere, the shape and the structure of petiole Doum palm fiber are similar to the most natural fiber and particularly to the coir fiber.^{39,40}

Each fiber is cylindrical in shape and formed with multicellular fibers each containing a central void (lumen). According to morphological studies performed on palm fiber, Agoudjil et al.⁵ predict that the external layer can be lignin and the withdraw of this layer lead to a stable bond between the fibers and the matrix.^{41–43}

Based on the structure, cleaning the surface of the fibers from a large number of impurities seems to be

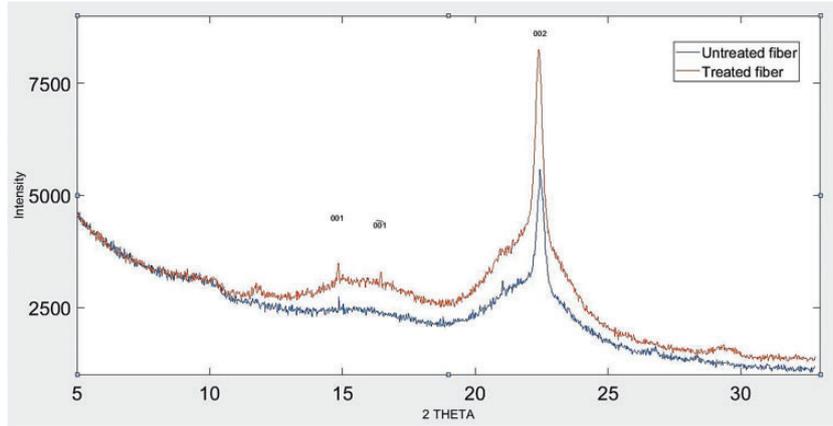


Figure 7. X-Ray diffractogram of Doum palm fiber.

Table 6. XRD diffraction of doum palm fiber.

	$I_{0,02}$ ($2\theta = 22.5^\circ$)	I_{am} ($2\theta = 18.1^\circ$)	The crystallinity index (%)
Untreated doum palm fiber	5066	2163	57.30
Treated doum palm fiber	8430	2719	67.74

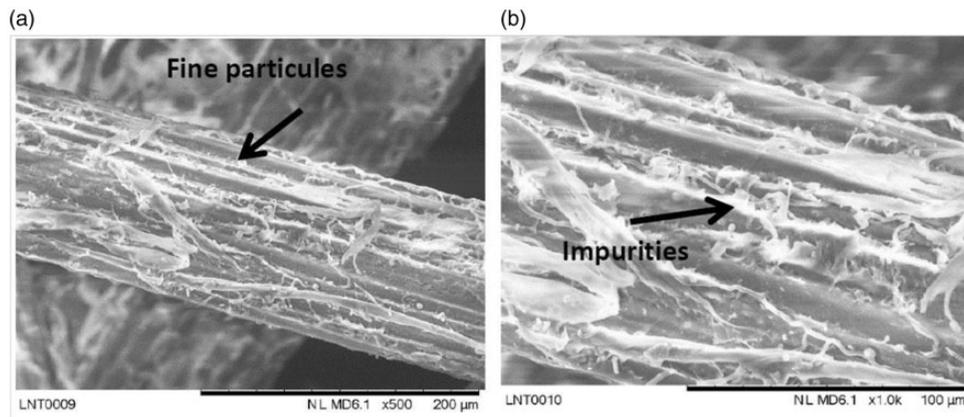


Figure 8. SEM image of untreated Doum palm fiber: (a) Magnificationx 500; (b) Magnificationx1000.

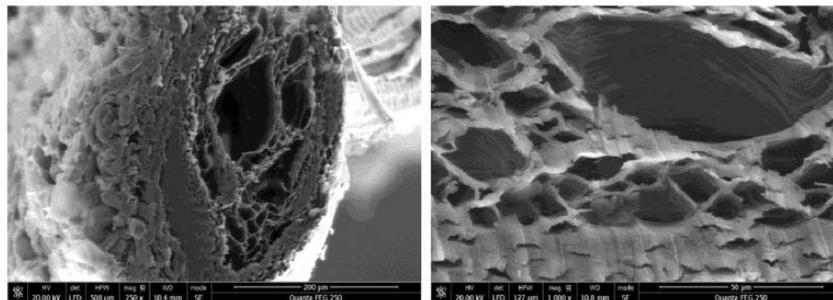


Figure 9. Cross-section of Doum palm fiber.

crucial for overcoming the poor adhesion between the fibers and the matrix. These sightings confirm that the fiber structure is very important to have a good adhesion between Doum palm fibers and cement mortar. Thus, the Doum palm fiber could be good competitor for the development of a natural composite.³⁹

The surface of raw Doum palm fibers is covered with a large number of amorphous materials predominantly composed of hemicellulose and lignin and other impurities (Figure 10). Raw Doum palm fibers are presented in Figure 10. Its surface is irregular and has many impurities and residual lignin. These impurities decrease the tensile properties of Doum palm fibers because they raise the diameter of the fiber without sustaining any tensile loads. Moreover, these impurities can cause an unstable adhesion between the fiber and the matrix and minimize the mechanical properties of the composites.

The use of alkali treatment removes the impurities and the fatty substance from the surface of the fiber and this decreases the fiber diameter as indicated in Figure 11.

The alkali treatment removes some lignin and hemicellulose and allows the fibrils to rearrange themselves, which leads to a better packing of cellulose chains.

Before treatment, the surface contains gummy and waxy substances as shown in Figure 10.

This component facilitates the extraction of fibers from the matrix but after treatment, the surface becomes much smoother without attacking cellulose microfibrils, which improves adhesion between the fibers and the cement matrix.³⁶

Effect of fiber length and alkali treatment on the composite mechanical properties

Flexural strength. The flexural properties of composite reinforced with treated and untreated fibers are investigated by using a three-point bending test. The addition of fibers to specimens changes the flexural strength behavior of composite. It does not only improve the flexural strength but also improves their stiffness. Figure 12 shows the variation of the flexural strength of raw and alkali-treated Doum palm fiber reinforced cement mortars with the increase of fiber ratio (mass %). As can be seen, the flexural strength of composite reinforced with treated Doum palm fibers is higher than that reinforced with untreated fibers. An important decrease of flexural strength by adding untreated Doum fiber compared to control mortar. This decrease is related to poor bonding between raw fiber and matrix due to the presence of amorphous materials on the fiber surface. The chemical treatment makes the fiber surface cleaner and rougher resulting in an

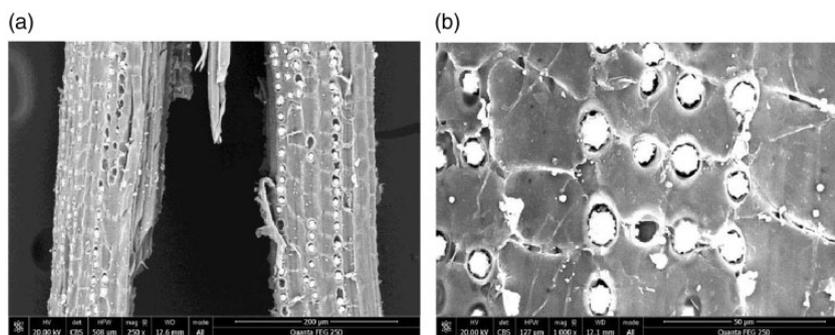


Figure 10. SEM micrographs of raw Doum palm fiber: (a) magnification $\times 250$; (b) magnification $\times 1000$.

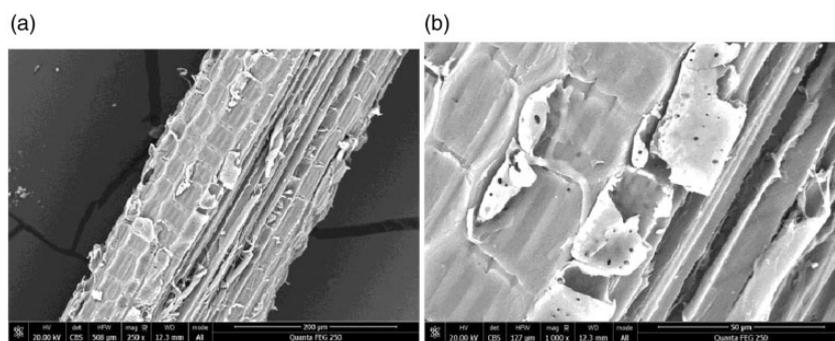


Figure 11. SEM micrographs of alkali treated Doum palm fiber: (a) magnification $\times 250$; (b) magnification $\times 1000$.

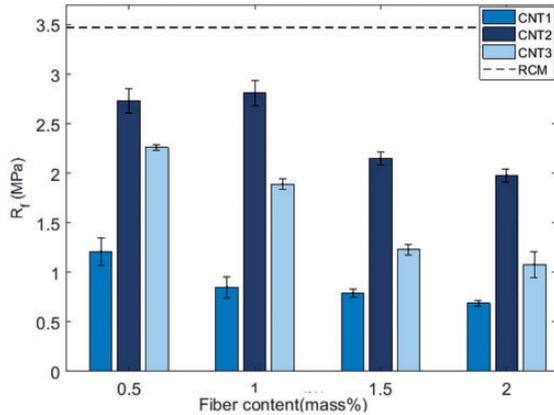


Figure 12. Flexural strength of raw Doum palm fiber reinforced cement mortar.

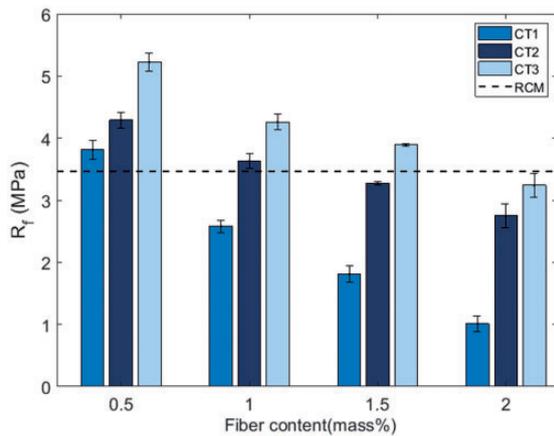


Figure 13. Flexural strength of treated Doum fiber reinforced cement mortar.

increase in the tensile strength of composite reinforced with treated fiber by improving the interfacial adhesion between the cementitious matrix and the fiber.^{39,40}

Besides, untreated fibers have an important capacity to absorb water than treated fibers and they liberate it in the mortar after mixing. This behavior makes the composite more porous and contains a higher quantity of water than mortar reinforced with treated fibers and it decreases their flexural strength.

The flexural strength of composite reinforced with treated fiber firstly show an increase until an optimum value of fiber content and then a shortage with increasing fiber content. The flexural strength of control cement mortars is measured to be 3.47 MPa, whilst they are measured to be 5.22 MPa, 4.29 MPa and 3.81 MPa for respectively CT1, CT2, and CT3 with adding 0.5% of treated Doum palm fibers (figure 13). Such behaviour is essentially linked to the fiber bridging mechanism, which consists of transmitting supplementary tensile stress caused by Doumfiber through the

crack surface and they limit the progression and the propagation of the crack, by a sewing effect, leading to increase the tensile strength of composite samples. The same results are obtained by previous research.^{44,45}

The maximum flexural strength for the composite CT3 reinforced with a 0.5% mass fraction was 5.22 MPa.

Figure 14 presents flexural deflection curves for control cement mortar and cement mortars reinforced with treated Doum palm fiber. For the reference cement mortar (RCM), the curves are a linear brittle behavior. On the other hand, the curve of composite reinforced with treated Doum palm fiber (CT3) can be divided into three parts separated by two different force values. The post-peak response is controlled by the performance of the fibers while the pre-peak response is dominated by the performance of the matrix.

Control mortar reached a maximum load value of approximately 1400 N and then the load value dropped suddenly. The load value of Doum palm fiber reinforced mortar (CT3) exceeded 2400 N, achieving a 70% strength increase.

The first region I present a linear evolution of the load versus, the first region presents a similar behavior that the control cement mortar, it is characterized by a linear evolution of the load versus deflection.

In region II, the first macroscopy damage in the material occurred with a notable change of the stiffness.

For the region, III a slow increase in load after the fibers took over because, after the peak, the response is controlled by the performance of the fibers. (Figure 14)

Compressive strength. The mean values obtained from the compression tests are presented in Figure 15 as a function of fibers content.

As expected, for all specimens reinforced with untreated fiber, the breakage decrease in proportion to the amount of fiber present in the mixture. We can observe that compressive strength is about 62% lower for samples CNT2 reinforced with 1.5% of fiber, in relation to the reference mortar. This behavior can be explained by the low surface protection in the fiber, which permits degradation due to the higher alkalinity of the matrix. This can adversely affect the adhesion between the cementitious matrix and fiber, producing spacing inside the mortar. These spaces reduce the compactness of the cement and consequently the compression strength of the reinforced mortars

A second source of the lower compressive strength of untreated fiber-reinforced mortar compared to control mortar was the bleeding which happens in a mortar with a higher water/cement ratio. Untreated fiber has high porosity, which means greater water absorption in its pores and they release it when mixing the mortar. The same result is obtained by Orge⁴⁶ and Orzekan.⁴⁷

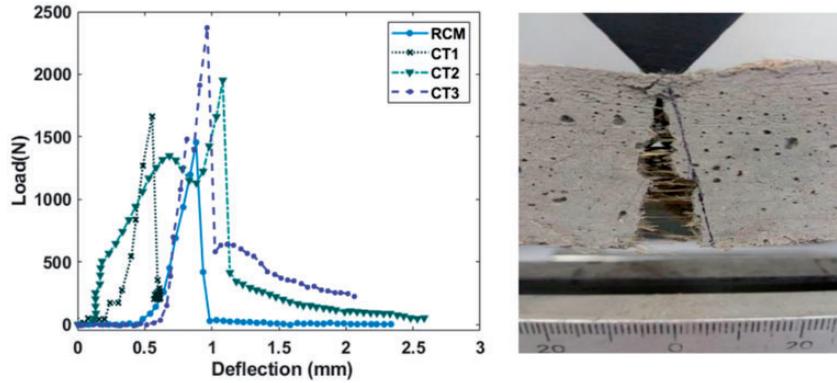


Figure 14. The load -deflection curve of control, treated Doum palm fiber reinforced cement mortar.

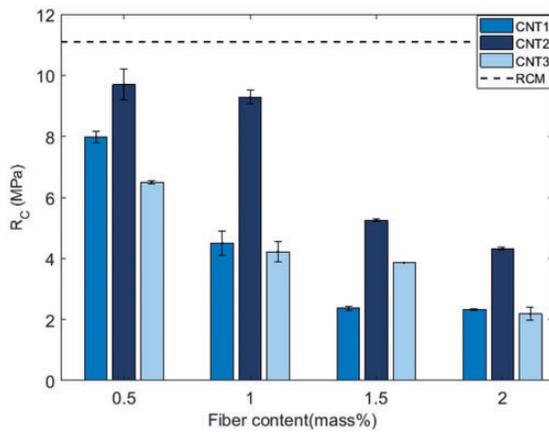


Figure 15. Compressive strength for raw Doum fiber reinforced cement mortar.

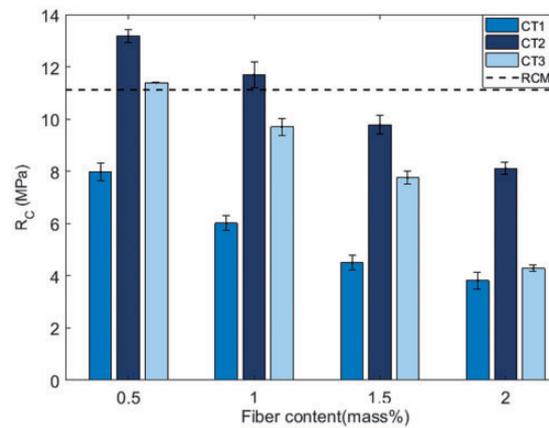


Figure 16. Compressive strength for treated Doum fiber reinforced cement mortar.

Contrarily, comparing the mechanical properties of control cement mortar and cement mortar reinforced with treated Doum palm fiber, a gain of compressive strength of about 18.63% for CT2 with 0.5% mass fraction can be observed (Figure 16). The maximum value is obtained for an optimum length and fiber content. Beyond this value, an incremental decrease of the material strength can be detected due to the non-homogenous mix and the higher quantities of the fiber-matrix interface. This decrease can be associated with an increase of porosity, the higher quantity of fiber make mixing difficult and aims to clump together, producing inadequate adhesion between the matrix and the fiber, which decreases the workability of fresh mortar and reduces their strength.

This result confirm what was found by Konin⁴⁸ and khedari.⁴⁹

Fiber–matrix interaction

The interface between the matrix and the fibers plays an important role in determining the mechanical properties of composites. Good interfacial bonding is

required to have a maximum reinforcement because the stress transferred between fiber and matrix passes through the interface.

Interfacial properties of Doum palm -cement mortar composites are investigated by digital microscope Leica DMS300 and HITACHI TM3030 scanning electron microscopy (SEM).

Figure 17 presents the broken surface of cement mortar reinforced with 0.5% of treated fiber (CT3). It demonstrates that the alkali-treated fiber is surrounded by the cement, proving a strong fiber-matrix adhesion. It also shows the existence of damage and cracks in the broken fiber ends due to the strong adhesion between the fibers and the matrix.

Composites prepared with treated fibers have a minimum pull out of the fiber, which reflects a better load transfer from the matrix to the fiber, and also an increase in the mechanical properties of the composites. The alkali treatment induced a better adhesion between fiber and matrix. The same results were obtained by Zukowski et al.²³

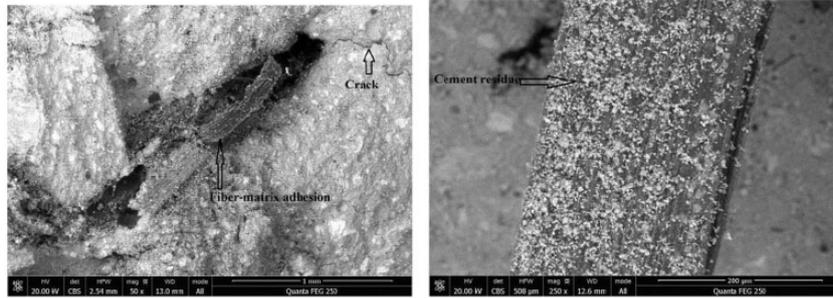


Figure 17. Break surface of cement mortar reinforced with treated fiber.

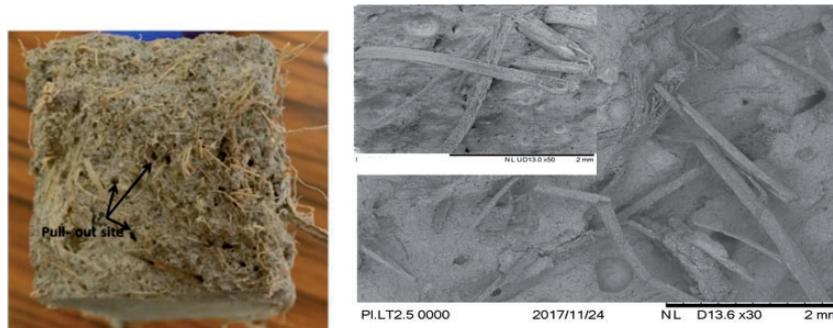


Figure 18. Break surface of cement mortar reinforced with untreated fiber.

While Figure 18 present cement mortar reinforced with 0.5% of untreated fiber (CT3). It shows that, the untreated fibers partly adhered to the matrix and pulled out from the matrix. After applying stress, several holes appears due to the debonding of the fiber from the matrix, which indicates a weak adhesion between the fiber and the matrix. This behavior has contributed to lower mechanical properties. However, for the composite reinforced with untreated Doum palm fibers, there is poor interaction between the fiber and the matrix, leading to a limited interfacial bonding and hence low mechanical properties.

Effect of the fiber length and alkali treatment on the composite thermos-physical properties

Thermal conductivity. The thermal conductivity is an important factor, which characterizes the effectiveness of the composite as a building material. Before testing, all specimens are dried at 105 °C obtained a constant mass. The thermal properties are measured using the hot-disk method.

Figure 19 shows the influence of Doum palm fiber content on the thermal conductivity of the composite. As expected the thermal conductivity decreases as the fibers content increases.

While thermal conductivity of the control mortar is (W/m K), it drops from 1 W/mK to 0.36 W/mK for CT2, which corresponds to a reduction of about

64%. This decrease is due to the increase of porosity of the dry mortars cause, by the high capacity of water absorption of Doum fiber. Furthermore, adding fiber into mortar increase the inner air of samples which make it more porous, the air has a lower thermal conductivity of about 0.026 W/mK and it is lower than that of the solid material bringing to reduce the heat transfer, accordingly, the increase of the inner air of samples conducting to a decrease in thermal conductivity. The results following the observations reported in the literature.^{50,51}

In general short fibers have too much difficult to align compared with the long fiber which creates a lot of voids leading to low thermal conductivity of specimen.³⁹ In our case for particular fiber content, the thermal conductivity is lower for specimen CT2. However, the reduction of thermal conductivity values is important indicating that the use of Doum palm fiber has a positive effect.

Thermal diffusivity. First of all thermal conductivity is the rate at which heat flows through any kind of material, such as mortar. Thermal diffusivity, on the other hand, is the rate at which temperature changes occur in the mortar and it presents a major factor to estimate their potential as a good insulation material. It measures the rate of heat propagation through a material.

Figure 20 presents the evolution of thermal diffusivity as a function of Doum palm fibers and for different size of fibers. It can be realized that all sizes of Doum palm fiber have a positive effect to amortize the heat diffusion in the composites. Indeed the incorporation of Doum palm fiber that the cement mortar reduces the thermal diffusivity of the composite. This is due to the opposition between the heat flow and the alveolar structure of fibers.^{52,53} According to these results, the optimal fibers content is 2% and CT2 fibers because it has lower thermal diffusivity.

Thermo-mechanical diagram. Mechanical strength, thermal conductivity, and density are important parameters, which should be considered to select the greater building thermal insulating material. Compressive strength depends on the specimen density.

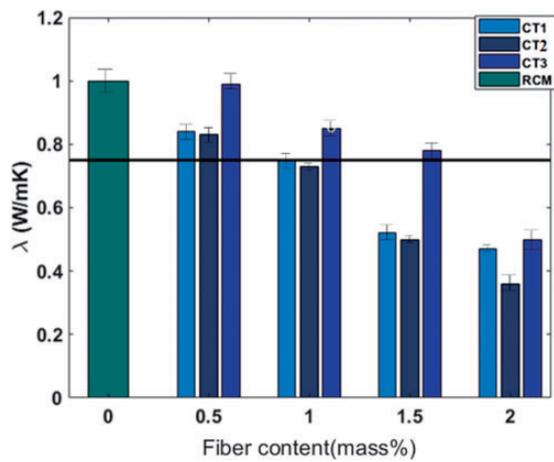


Figure 19. Thermal conductivity of composite as a function of fiber ratio.

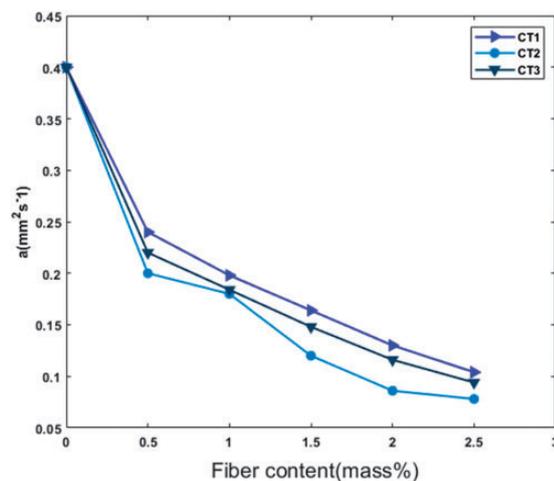


Figure 20. Thermal diffusivity as a function of fiber ratio.

Composite with lower density is considered as a light-weight material with a reduced thermal conductivity.

Thermo-mechanical diagram illustrates the relation between mechanical properties, thermal properties, and density. It permits the analysis of the thermomechanical properties of thermal insulation materials.

Figure 21 presents the thermomechanical diagram of cement mortar reinforced with treated Doum palm fiber (CT2).

From Figure 21, by increasing Doum palm fiber ratio cement mortar becomes lighter and the thermal conductivity is meaningfully reduce. Indeed to obtain a composite with interesting thermal insulation capacity induces a detriment in the compressive strength.

The compressive strength raises suddenly from 11.11 MPa for reference composite to 13.18 MPa for composites with a mass fraction of 0.5%. Beyond this fiber fraction, the compressive strength of the composite decrease slightly to 7.8 MPa, which corresponds to 2.5% fiber content.

According to the functional classification of light-weight concrete conducted by Rilem⁸ and Boumhaout et al.¹ are presented in Table 7, the compressive strength of our composite is permissible for structure and insulating lightweight material.

Therefore, cement mortar composite reinforced with Doum palm fiber of fiber content below 0.5% for CT1 and CT2, for CT3 with fiber content more than 1.5%, has a coherent thermal conductivity and mechanical properties with structural and insulating materials in buildings.

The test results of dry density, compressive strength, and thermal conductivity of mortar are summarized in Table 8. The values were ranged from 1507–1812 kg/m³ for dry density and 0.5–0.99 w/m.k for thermal conductivity. The density of CT1 and CT3 mortars is slightly higher than CT2 mortar. As can be seen, the values of

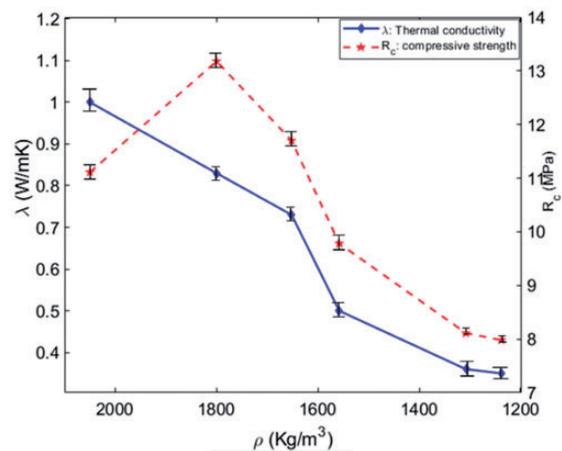


Figure 21. Compressive strength and thermal conductivity as a function of density.

Table 7. Functional classification of light weight concrete.⁸

	concrete of lightweight concrete		Autoclaved aerated concretes	
	Class II (insulating and construction)	Class III (insulating)	Class II (insulating and construction)	Class III (insulating)
R _C (MPa)	>3.5	>0.5	>2.5	>0.5
λ (W/m.k)	<0.75	<0.3	<0.75	<0.3

Table 8. Composites properties.

	CT1			CT2			CT3		
	0.5	1	1.5	0.5	1	1.5	0.5	1	1.5
φ (%)	0.5	1	1.5	0.5	1	1.5	0.5	1	1.5
R _C (MPa)	7.97	6.01	4.5	13.18	11.7	9.78	11.39	9.7	7.75
λ (W/m.k)	0.84	0.75	0.52	0.83	0.73	0.50	0.99	0.85	0.78
ρ (kg/m ³)	1812	1683	1558	1800	1652	1507	1829	1750	1740

thermal conductivity tends to decrease with a decrease in densities of mortars reinforced with Doum palm fiber. Moreover, the values of density of mortars reinforced with treated Doum fiber also decrease with the fiber content. These test results are in agreement with those obtained by other research work.⁴⁹ Khedari et al.⁴⁹ study the thermal conductivity of cement mortar reinforced with coconut fiber and explain that the density tended to decrease with the fiber mass fractions because fibers clumped together during mixing, capturing water-filled spaces that successively turn into porosities or air voids.

Conclusion

This paper highlights the potential behind using Doum palm fiber in structural and insulation cement mortar mixes.

The problem of biodegradability of natural fibres was investigated in this study. An alkaline treatment of fibers with a 1% concentration NaOH solution combined with sodium hypochlorite (NaOCl) was performed. The results obtained from X-ray diffraction (DRX), infrared spectroscopy (FTIR) and scanning electron microscope observation show that this treatment is effective in removing amorphous materials such as lignin and hemicellulose, increasing the crystallinity of the fiber leading to greater mechanical properties which leads to an improvement in adhesion between cement and fibers.

It is generally seen that the introduction of raw fiber into cement mixes causes a decrease in flexural and compressive strength due to the poor bond between fiber and matrix.

When failure propagates inside the cement mixes in tension, the poor bond around fibers leads to decrease flexural and compressive strength.

A notable improvement of the thermal properties of cement mortars reinforced with Doum palm fiber is observed as compared to reference cement mortar.

In addition, the density, and thermal conductivity decrease by increasing fiber ratio into the composite, while the obtained values reveal that adding 1% of CT2 fibers results composite with $\rho = 1652 \text{ kgm}^{-3}$ and $\lambda = 0.73 \text{ W/m.k}$. These gotten values following the Rilem recommendation for insulating material.

The most important conclusions are the following:

1. Taking into account the results obtained in 'Results and discussions' section, comparing treated and raw doum fiber composites, there is a significant improvement with the treated fibers. It is now possible to exclude the incorporation of raw fibres with a cement binder, because of the presence of amorphous materials that lead to poor hydration of the fibers with the cement binder.
2. Adding 1% of CT2 results in a considerable improvement in the mechanical properties of the cement mortar. The 28 days flexural and compressive strength of CT2 are 23.6% and 18.63% respectively superior to the control mortar. Reverse, the 28 days compressive strength of the other composite still complies with the strength requirement for lightweight concrete.
3. Adding, treated Doum palm fiber to cement mortar trained in changing the failure mode of the composite. The flexural test reveals that the reinforced specimens have a ductile mode with a minimizing of the failure propagation by fibers.

4. The thermal conductivity and the density are meaningfully (on average 26.22% and 27%, respectively) lower than control mortar.

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