

Influence of Treatments on the Date Palm Fiber and Cement Matrix Behavior: Tensile and Pull-Out Tests

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Abstract As part of our research on the adhesive bond between the vegetable date palm fibers and the cement matrix, we found that this phenomenon degrades progressively with the age of the matrix. It is well on durability that menaces this operation. To improve the behavior of the vegetable date palm fiber in cement matrix (mortar) vis-a-vis the bond stress, we conducted two types of treatments, the first directly addresses the fiber (alkaline and autoclave treatment), the second is done by replacing a portion of a cement by pozzolanic materials (pozzolana or métakaolin). Direct tests (pull-out test) were performed to find the bond stress fiber-cement matrix whose the mortar maturity being varied from 3, 7, 14, 28, and 90 days.

Keywords: fiber reinforced Concrete, date palm fiber, bond stress, durability, treatment, alkalinity, pozzolanic materials, autoclave

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1. Introduction

In our days, the use of fibers as reinforcement for cement matrices is a major importance, both to enhance local materials or for improving the characteristics of the cement matrix, i.e. their tensile and flexural strength, in added the increase of ductility and crack resistance [1,2]. The most widely fibers used are synthetic fibers such as (polypropylene, acrylic, aramid, nylon fibers ..). However the vegetable fibers are used recently, among these fibers we found the Bagasse, coconut, jute, banana, bamboo and date palm fibers. These fibers have several advantages such as low cost, biodegradability, renewable, and their availability everywhere. These properties favor the use of these fibers as reinforcement on the matrix [3,4].

The performance of the vegetable fibers in the concrete primarily depends on the phenomenon of adhesion between the latter and the matrix, Bessell & Mutuli found that bond stress between the sisal fiber and the concrete matrix is on the order of 0.6 MPa [5].

The main disadvantage of vegetable fibers is there low durability in alkaline environment and therefore in cementitious environments ($\text{pH} > 12$), this low durability appears by the degradation of the mechanical properties of the fiber and even the composite, Cesar et al found that sisal fibers have acceptable stress in the initial state, but a degradation about 78% of resistance was observed when these fibers were immersed in an alkaline solution [6,7]. A. Kriker et al result that the date palm fiber loses about 84% of its tensile strength after six months of immersion in a sodium hydroxide solution NaOH ($\text{pH} = 12.5$) [8].

For this purpose, several studies are started, to improve the durability of the vegetable fiber in alkaline environment

(concrete), whether by treatment of the fiber or by modification the matrix.

Several studies have shown the influence of various treatments of the vegetable fiber on their performances and however on the composite. Among existing chemical treatments found mercerizing, acrylation, permanganate, in added the Silane treatment, these treatments are intended to reduce the hydroxyl group in the fiber. The Alkaline treatment (treatment with sodium hydroxide solution NaOH) also called mercerizing improves the fiber-matrix adhesion by removing natural and artificial impurities that have accumulated in the surface of the fiber (Mishra et al. 2001a) [9,10]. Moreover, physical treatments have their large part in this procedure, such as the autoclave treatment. It cites as an example, a study based on flax fibers treated by autoclave consume 20% less moisture absorbing and retain about 20% more of their mechanical properties compared to the untreated fibers. [11].

On the other parte, literatures turn toward the modification of the cement matrix. This modification is made by replacing a part of the cement by pozzolanic additions in order to reduce the alkalinity of the matrix. Silva et al replace a portion of cement by calcined clay and the métakaolin [12], this study expresses a conservation of the composite toughness (fiber-matrix) after 100 cycles of (wet/dry).

2. Experimental Method

2.1. Materials Used

2.1.1. Date Palm Fiber

The fibers that we used in this work are the fiber of date palm (DPF), exactly from the parte so-called "leaflet"

(Figure 1). These fibers are from the region of Saoura (Bechar, Algeria), negligible cost and renewable source. They have a bright green or green color and a cross section that we can be considered as a rectangular section, the physical properties are regrouped on the Table 1.

2.1.2. Cementitious Matrix

The cement matrix used in this study is composed of one part of CPJ cement CEM II/B 42.5, and three parts of sand with a W / C equal to 0.55. (Table 2)

2.2. Treatments Carried out

2.2.1. Treatment of the Fiber

In our case, we performed a chemical treatment of the fiber (alkaline treatment) and other physical treatment (autoclave). The Chemical treatment is done by immersing the fibers in solution of Sodium hydroxide (NaOH) with a concentration of (0.5%, 1%, 1.5%, 5% and 10%) for one hour under a source of temperature at 100 ° C, they are then washed with water to remove the excess of (NaOH), the last wash is made with distilled water, after that the fibers are dried in the open air. The physical treatment (autoclave) is to put the fiber for 30 minutes in the presence of saturated water vapor at a temperature slightly greater than 130°C and wherein the vapor pressure is 2 bars, this step is followed by a further drying at ambient temperature [13].

2.2.2. The matrix Modification

For the modification of the matrix, we replaced 30% of the mass of cement by one of the two pozzolanic materials which are natural pozzolana or métakaolin, Natural pozzolana used is from the career of BENI SAF (West of Algeria), it was crushed and sieved through a sieve of 80 microns. The métakaolin is an artificial pozzolana obtained by calcined and grinding kaolin clay (ARGICAL M-100)' [14], the characteristics of these materials are summarized in Table 3.

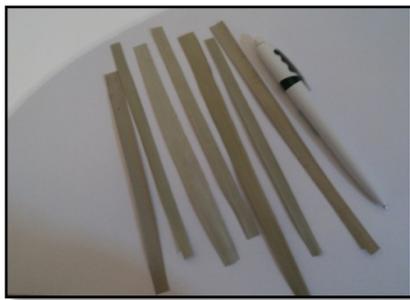


Figure 1. Date palm fibers (Leaflet)

Table 1. Physical Properties of date palm fibers

Date Palm fiber	
length (mm)	150
width (mm)	7
Thickness (mm)	0.55
Water content (%)	53.6
density (Kg/m ³)	720
Absorption coefficient (%)	132

Table 2. Composition of mortars used

		Type I	Type II
Sand (g)		1350	1350
Binder	Cement (g)	450	315
	Pozzolana (g)	0	135
	Métakaolin (g)	0	0
Super plasticizer (g)		0	6.75
Water/binder		0.55	0.55

Table 3. Chemical composition of materials used [14].

	Cement (%)	Pozzolana (%)	Métakaolin (%)
SiO ₂ (%)	23,7	46,86	55
Al ₂ O ₃ (%)	6,58	16,62	40
Fe ₂ O (%)	4	9,37	1,4
CaO (%)	64,95	9,38	0,3
MgO (%)	0,32	2,84	
CaO(free) (%)	0,33	/	/
SO ₃ (%)	/	0,36	/

3. Mechanical Behavior of the Fiber after Treatment

3.1. Behavior vis-a-vis the Direct Tensile

At the aim of studying the different treatments influence of different treatments of the fiber (DPF) on its own tensile strength and deformation, we conducted a direct tensile test on the fibers treated with two different ways:

- Chemical way: immersed the fiber in NaOH with a concentration of (0.5, 1, 1.5, 5 and 10%)
- Physical way: treated the fiber with autoclave.

In addition the untreated fibers values were kept as a datum.

The press used is a universal testing machine with a maximum capacity equal to 5 KN (accuracy of 0.01N), and which has to be used for two different tests (direct tensile and pullout test) of the fibers, linked to a digital data acquisition chain.

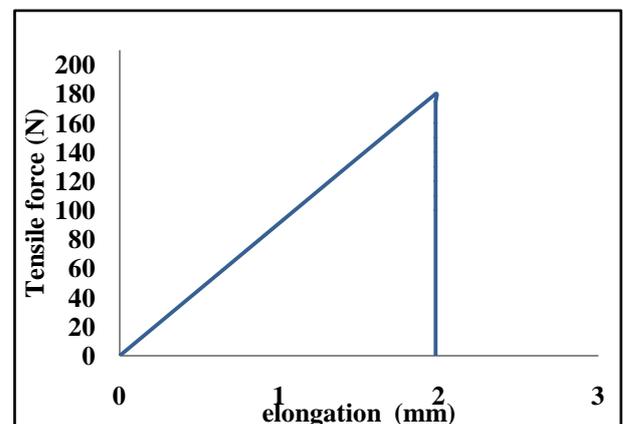


Figure 2. Force-elongation curve types of fiber (DPF) untreated subjected to tensile force

The Figure 2 is a force-elongation curve obtained from a tensile test of a date palm fiber. Note that the maximum force and elongation at break of the latter are: 180N and 1.85mm respectively, and the fiber breakage takes place immediately after exceeding the maximum force, the Young's modulus of the fiber is equal to ($E = 2670\text{MPa}$).

In order to find the efficiency of the different treatments on the fiber's property, we regrouped the results of these treatments that we carried out on the tensile stress, deformation, and elasticity modulus.

After the treatments performed on the fiber, it is clear that the tensile stress of the fibers increases with increasing the concentration of the sodium hydroxide solution (NaOH) till the value of 5% of this solution, while this stress is equal to 93.35 MPa for a concentration of 0% (untreated), then it reaches a value of 244.02 MPa for a concentration of 5%. This improvement of the stress is primarily due to the effect of sodium hydroxide solution which removes the impurities accumulated in the surface of the fiber which affects the properties of the latter, by against beyond this concentration a resistance drop occurs, this decrease is due to the high concentration of sodium hydroxide which enters inside the fiber and effects its structure, and therefore their mechanical properties.

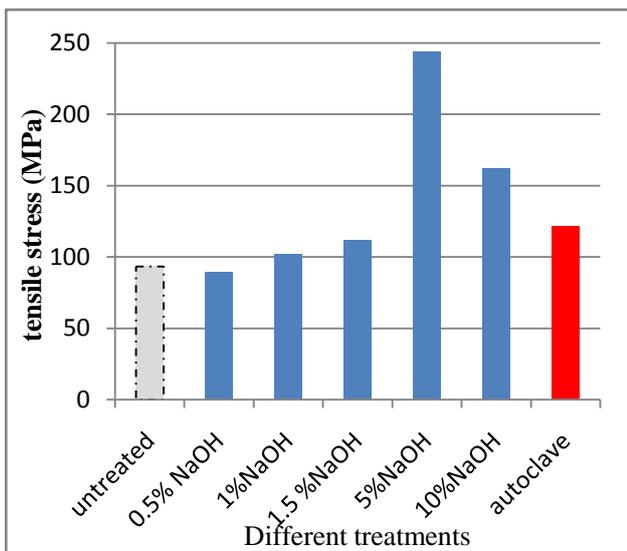


Figure 3. The tensile stress of treated and untreated fiber

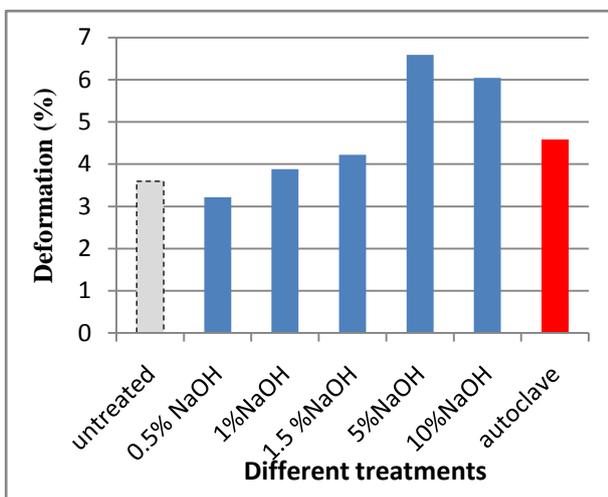


Figure 4. The deformation of treated and untreated fiber

However the autoclave treatment gives lower tensile strength compared to those found by the sodium hydroxide treatment, but the two are still higher than those of untreated fiber, it's equal to 121 MPa and 93 MPa for the autoclave and the untreated fibers respectively.

From the Figure 4 we show that the results of deformation have the same gait like that the tensile stress, where all treatments give an increase on the deformation values of the fiber. In added the immersion of the fiber in 5% of NaOH give the higher value (6.59%), also the treatment by autoclave give a good result but it's lower than those of 5% NaOH, and as we said these treatments remove the impurities of the fiber, which conduct the latter to give more elongation.

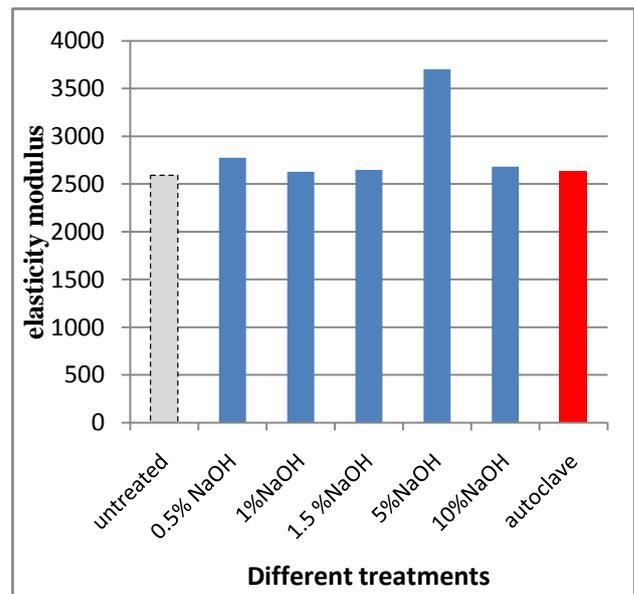


Figure 5. The elasticity modulus of the treated and untreated fiber

In the Figure 5 which is the values of elasticity modulus of treated and untreated fibers, we noted that the elasticity modulus of the fiber is around 2500 MPa for the most types of treatments, in the treatment by 5% of NaOH the elasticity modulus reached 3700 Mpa, this value is due to the high tensile stress with a little increase of the deformation value.

For all these results, we can say that the treatment by sodium hydroxide especially by (5% of NaOH) improve the tensile behavior of the date palm fiber.

3.2. Adhesion Fiber – matrix

To find the bond stress between the fibers (DPF) and the cement matrix (the mortar), the pull-out tests were conducted on the blocks of mortar, the dimensions of the block are (4x4x3 cm³) and the fiber length is 15 cm with 4 cm anchored length. The latter will be simply subjected to an axial tensile force, the mortar block is held fixed to allow the slip value of the fiber. The experimental device designed to ensure that handling is identical to that of the direct tensile test (Universal Press of 5 KN). The data acquisition is done simultaneously by a chain of acquisition integrated to the press, it allows the recording of the slip and pullout force of the fiber. The maximum force (Fmax) is acquired automatically and allows the calculation of the bond stress [15].

$$\tau = F/S \tag{1}$$

τ : bond stress.

F: maximum force of adhesion.

S: the contact area between the fiber and the matrix.

The protocol considered in this study for pull out test was devised into two parts ; the first is conducted for the treated fibers (physically treated by autoclave, and chemically by sodium hydroxide 5% and 10%). The second is conducted for the dosage of the cement which varied between 100% and 70%, where the supplement percentage (30%) was replaced by the pozzolana or the métakaolin. Noted that the composite used with untreated fibers or no modification matrix (100% cement) is taken as a control composite.

In added the age of the mortar was varied between (3 ; 7; 14; 28; and 90 days).

To study better the behavior of date palm fiber in adhesion with cement matrix, we carried out a typical curve (adhesion force-elongation) obtained from a pull out test of embedded palm fiber in a block of mortar.

From the [Figure 6](#), we show that the adhesion curve has the same behavior like the others materials i.e. started with a linear ascending branch of pull out force until a peak is reached (300N), in this branch the transfer of the force from the fiber to the matrix was occurred by the totality of the contact surface, after that we found a decreasing branch which mean the start of the partial debonding, thus this branch is followed by a more important step which is the phase of friction, in this phase called also a totality debonding, the bonding was occurred by the friction between the two materials (fiber and matrix).

[Figure 7](#) and [Figure 8](#) show the effect of various chemical and physical treatments of the fiber on the fiber-matrix bond stress, it is clear that the stress [τ] in the case of untreated fibers has a decreasing behavior at the time, because its own value starts with 300 KPa at the age of 3 days, however at days 90 days it is 60 KPa, this decrease is due to the specific behavior of vegetable fiber which declares a low durability in cementitious environments, this decrease is not a first, as others have mentioned this attitude in their research report on the vegetable fibers and special fibers such as date palm (Kriker et al).

A chemical treatment with sodium hydroxide gives an increase in bond stress till the age of 28 days, at this age it's equal to 210 KPa (10% NaOH), however it's equal just 140 KPa for untreated fibers. This increase on the bond stress is due to the effect of NaOH on the fiber which improve its contact surface state, moreover the sodium hydroxide reduce the hydrophilic character of the fiber which lead to reduce its quantity of the water absorption.

In addition, the adhesion of the treated fibers by autoclave is significantly lower than those of untreated fibers, but they maintain a durable behavior till the age of 28 days (with a value of [τ] equal to 210 KPa). This can be explained by the effect of autoclave treatment which changes the chemical composition of the fiber by reducing the quantity of the lignin and therefore the limitation of the reaction between the compounds of the fiber and those of the matrix. In addition, this treatment reduces also the surface roughness of the fiber, which decreases the bond stress from 300KPa to 225KPa for untreated and treated with autoclave respectively.

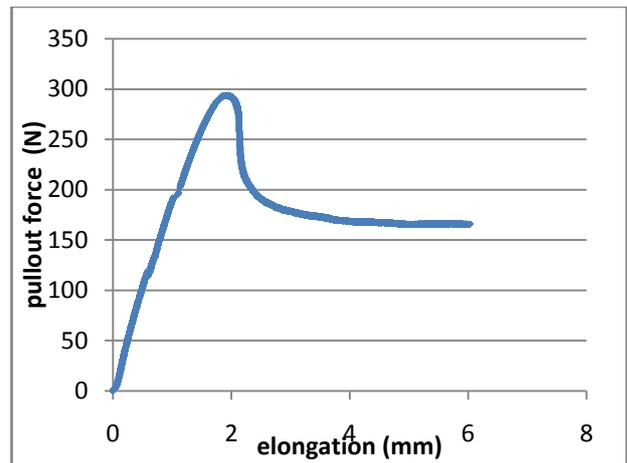


Figure 6. Typical pullout curve of fiber (DPF) anchored in a mortar block

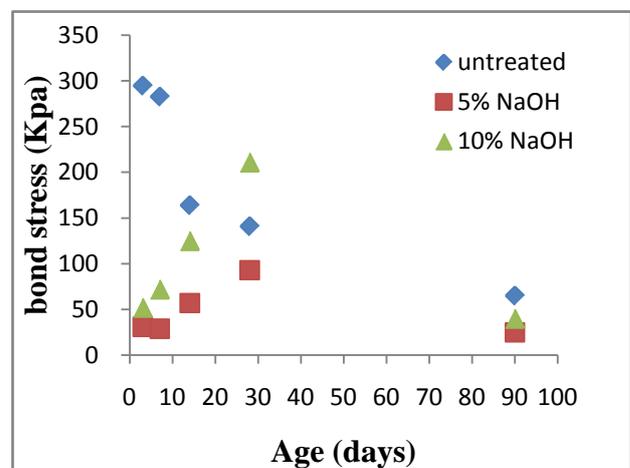


Figure 7. Bond stress of chemical treated fiber

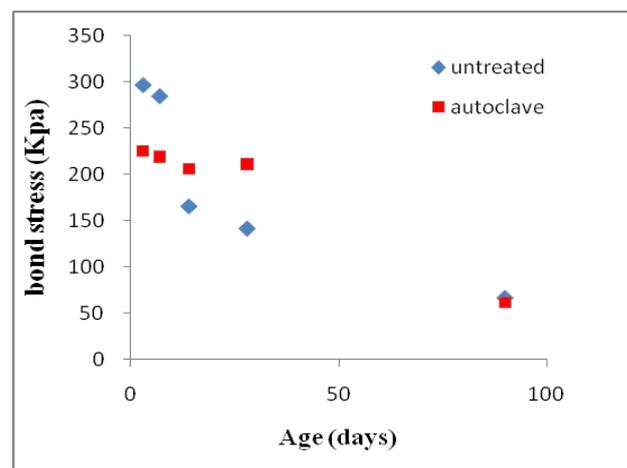


Figure 8. Bond stress of physical treated fiber

In the other hand of treatment which is the replacement of a part of binder by a pozzolanic materials, and as we show on the [Figure 9](#) these materials have a favorable effect on the durability of the composite vis a vis the bond stress, for example the métakaolin retains the value of the bond stress at 300KPa till the age of 28 days, this mean that is no reaction between the two materials (fiber, matrix), we can explain that by the low alkalinity environment of the matrix caused by the added of the métakaolin, and as we have mentioned on the table III the

percentage of the (CaO) is equal 64.95 % for the cement, however it's equal just 0.3% for the métakaolin.

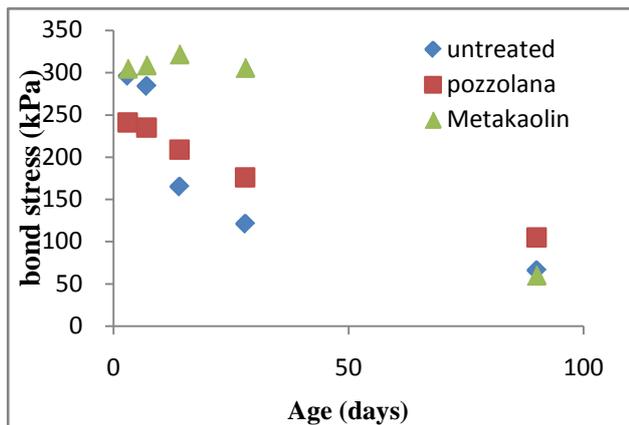


Figure 9. Bond stress before and after modification of the matrix

For the pozzolana treatment, the bond stress is less than that of the unmodified matrix till the age the 7 days, after this age the bond stress become higher even at the age of 90 days, its equal to 105 KPa for this treatment, however its equal to 66 and 60 KPa for untreated and métakaolin treated respectively, this caused by the effect of the pozzolana which start its activity at the late age.

4. Conclusion

From the tests that we performed and the results obtained, we can give the characteristics of date palm fiber, such as the tensile strength and the effect of some treatments of the fiber on its bond stress with the matrix. From the result obtained the fiber has an acceptable tensile strength (100 MPa) even without treatment, moreover the treatment let them to give a high tensile strength especially the treatment by 5% of sodium hydroxide, this strength is significantly higher than that of concrete which cannot reached 5MPa (ordinary concrete), but for the mechanical and environmental point of view the autoclave treatment gives the best result, even for the adhesion matrix fiber, this treatment gives the chemical stability of the fiber inside the cement matrix.

Concerning the bond stress of the fiber with the matrix, these fibers have a decrease behavior of this stress on the time. The treatments carried out even for the fiber or the replacement of a part of the cement give an improvement on the performance of the composite (fiber, matrix) till the age of 28 days, after this age a failing of the bond stress was recorded with all treatments. The treatment by pozzolana rests the effective treatment for the date palm fiber in the cement matrix.

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