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Phisycal and Thermomechanical Characteristics of Epoxy Biocomposites Laminates Reinforced by Mat of Date Palm Tree Fibers

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ABSTRACT

This paper considers the analysis of the physical and thermomechanical behavior of a laminate biocomposite constituted of three layers of vegetal fibers mat and epoxy resin. This material is made by contact molding process. We then characterized the physical, thermal and mechanical properties of this material. The results experimental obtained show this material had low density in the range (0.911-1.030) g/cm^3 and also exhibited low thermal conductivity, in the range (0.372-0.521) W/mK. That the incorporation of date palm tree fibers in an epoxy matrix reduces water uptake despite their hydrophilicity. The interesting mechanical properties of the material have been demonstrated by video tensile tests. These results make it possible to exploit this vital composite material for potential industrial applications.

1. INTRODUCTION

Composite laminates are made up of layers of fiber composite materials that can be stacked together to provide engineering properties like elastic moduli, Poison's ratio, in-plane stiffness, bending stiffness, strength, and coefficient of thermal expansion. The popularity of composite material shell panels is increasing day-by-day due to their light weight, high stiffness to weight ratio, and excellent structural tailoring capabilities [1].

During the past decades, increasing demand for a variety of industries, such as high-performance and lightweight structures, has stimulated a strong and growing development of fiber-reinforced polymer laminates [2]. Fiber-reinforced composite laminates commonly used in industries mainly include carbon fibers, glass fibers and metal fibers [3-5]. The use of synthetic fiber composite laminate during the twentieth century due to the industrial explosion generated enormous ecological and environmental problems. This is due to the non-degradability of these materials. Composite natural fiber laminates are an interesting alternative in cases where significant mechanical rigidity is not required, and because of the advantages of their properties such as biodegradability, low density, light weight, abrasion resistance, inexpensive, and environmental friendliness, they are very likely to replace synthetic fibers in the composite product [6]. Recent legislation on environmental protection has led to the emergence of several scientific works on the development of this type of material. Hemp, jute and flax are the natural fibers most used in the implementation of this type of composite material.

Abdallah Mir et al. [7] in their paper studied the development and characterization of composite materials (laminates) containing natural jute fiber reinforcement. laminate obtained by a process known as infusion is polymerized at a temperature lower than that which affects the mechanical properties of dry fabric.

Scarponi et al. [8] in their work experimentally determined some important mechanical characteristics of RTM/epoxy hemp plain weave fabric laminates.

Suneel Motru et al. [9] developed and evaluated the mechanical properties of biodegradable PLA/green flax fiber composite laminates in this research. The PLA/Lin composites were manufactured by manual hot plate compression molding using a locally developed test rig.

Another type of natural fiber that has received a lot of attention lately is the fiber sourced from date palm trees. There are over 120 million date palm trees all over the world which are widely spread in North Africa, the Middle East, Pakistan, India and the United States [10]. The total number of date palm trees planted in Algeria alone is about 18.4 million trees [11]. Date palm tree have four types of fibers, which are leaf fiber in the peduncle (leaflets), fiber in the rachis, fiber in the grappe and mesh fibers around the trunk (*Lif*) Figure 1 shows the different type of date palm tree fibers.

Once the date palms fruit are harvested, large quantities of date palm rachis, grappe and Fibrillium accumulated every year in agricultural lands of different countries. In Algeria alone, more than 80,000 tons of date palm tree waste accumulate annually. These amounts of important and valuable biomass wastes are of potential interest in different countries since they can be considered as new cellulosic fiber sources. Thus, innovative ways of valorizing this abundant renewable resource should be found [12, 13].

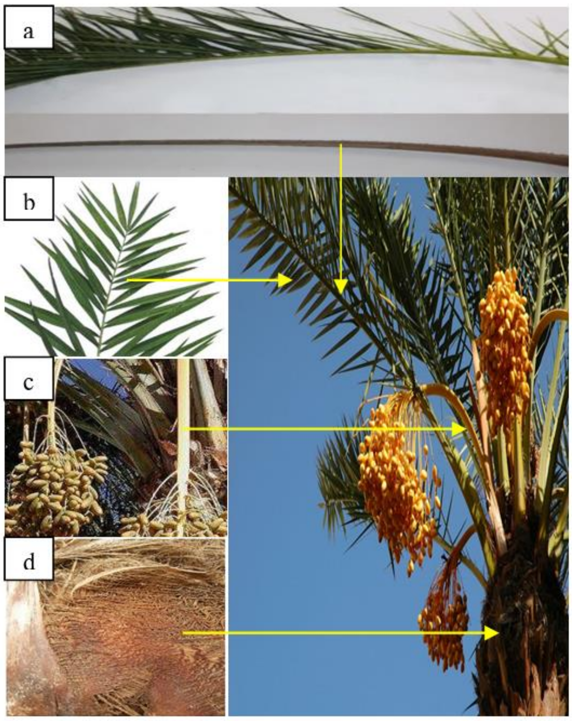

Figure 1. Different type of fibers: (a) Rachis, (b) Leaflets, (c) Grappe, (d) Mesh fibrers (*Lif*)

The Date palm tree fibers contains lignin from 20 to 30% and have a density from 0.45 to 0.90 g/cm^3, their tensile strength varies from 30 to 350 MPa, Young's modulus from 2 to 24 GPa and Poisson's coefficient from 0.100 to 0.176 [12, 14, 15]. Through these scientific results, date palm tree fibers have proven to have the potential to be effective filler in both thermoplastics and thermosetting materials for use in various industrial applications.

Several studies aimed to prepare and test bio-composites materials by using the different types of date palm tree fibers in the form of powder or short fibers to obtain new physical, thermal and mechanical properties at low cost [12, 16, 17].

This research aims to prepare a three-layer bio-composite material of the mats prepared from the fiber mesh surrounding the trunk of the date palm tree and immersed in epoxy resin. This composite material is prepared by contact molding. Then the physical, thermal and mechanical properties were examined. To the knowledge of the authors, there are no previous works involving the preparation and study of biobased nature materials fortified from date palm tree fiber mats.

2. MATERIALS AND METHODS

2.1 Matrix

The epoxy used in this study is of the MEDAPOXY STR type, which we got from Granitex Company Algeria. It is a two component with a Weight ratio (1kg resin and 0.666 kg hardener), polymerizable at room temperature, rigid and unfilled. The amber color mixture and the properties:

- Very good resistance to chemical products;
- Very good adhesion to concrete and steel supports;
- Good wettability of the support and the glass fiber.

Table 1 shows epoxy resin Characteristics.

Table 1. Epoxy resin characteristics (*)

Characteristic	
Density (ISO758)	1,1± 0,05 g.cm^{-3}
Viscosity (NF T76-102) at 25°C	11000 MPa.s
Curing time at 20°C	65% HR
Flexural strength (NA 324)	>25 MPa

(*) Granitex data sheet, edition February 2017.

2.2 Date palm tree fiber (*Lif*)

The vegetable fibers used in this work are date palm tree surface fibers from the region of Biskra (southern Algeria). Surface fibers are naturally meshed. They are formed by the overlay of three plates of lattices. These fibers were extracted manually in the form of a pararectangular plate (200x300) mm^2 during the harvest of dates and the period of maintenance of the trunk of the date palm El ghers. Figure 2 shows the location of *Lif* fibers in date palm tree.

The researcher [15] conducted mechanical experiments on this type of fiber by date palm El ghers, so that the mechanical properties listed in the following table were obtained. Table 2 shows the fibers characteristics.

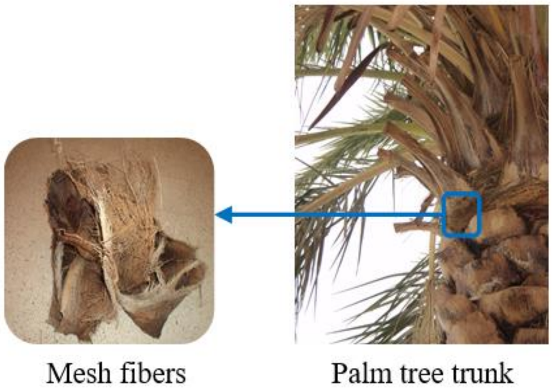

Figure 2. Location of *Lif* fibers in date palm tree

Table 2. Fibers characteristics [15]

Characteristic	
Density (g/cm^3)	0.47
Stress of rupture (MPa)	38.02 – 89.35
Strain of rupture (%)	1.62 – 7.95
Modulus of elasticity (GPa)	4.16 – 4.30
Poisson's ratio (/)	0.115- 0.136

2.3 Mat fibers preparation

In order to prepare date palm tree fiber mats, we brought the fibrous mesh surrounding the surface of the date palm tree trunk. Then we cleaned the fibers well from dust and impurities and filtered them by removing the thick fibers and keeping the fine fibers. Then we washed it well with water and left it for a whole day to dry. We collected the mesh fibers in the form of mats and then put them in a pressing machine, at a pressure of 20 Pa, a temperature of 100°C and a time of 10 minutes. Figure 3 shows the mat fiber preparation protocol. In the end, we obtained textile fiber mats with dimensions (180×180) mm^2, thickness 0.85 mm and surface density 0.034 g/cm^2 Shows in Figure 4.

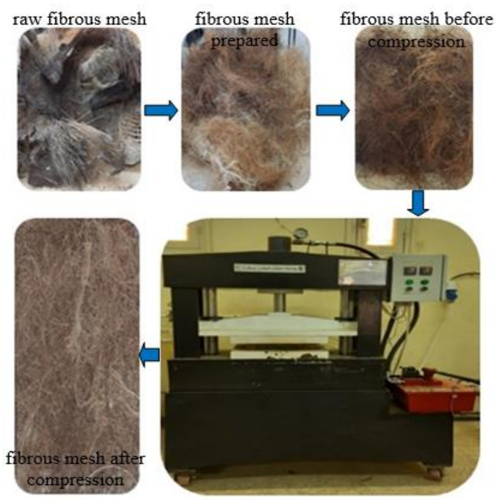

Figure 3. Mat fiber preparation protocol

Figure 4. Date palm tree fiber mat

2.4 Composite laminate preparation

Figure 5. Method of preparing biocomposite laminate

The composite material is made by contact molding. We used a plexiglass sheet Coated with Vaseline. The reinforcement is impregnated with the matrix using a paint brush then debubbling is carried out to eliminate air bubbles and ensure homogeneity in the distribution of the resin. After the composite has gelled and hardened (24 hours), the composite is removed from the mould. The polymerization process continues at ambient temperature (25°C) for several days. The elaborate composite contains three layers of date palm tree fiber mats, with a fiber volume fraction of 20%. Figure 5 shows the method of preparing biocomposite laminate.

3. EXPERIMENTAL PROCESS

3.1 Density test

The density of materials is defined as the ratio of the mass of samples taken from the material to their volume. The volume was determined from the variation in water volume caused by soaking the samples (water displacement technique), according to ISO 10119:2002.

3.2 Water absorption test

Water absorption and biodegradability, which are important properties for vegetal fiber materials, were also studied. In this study, the water absorption experiments are carried out according to the conditions of the literature [18, 19].

Water absorption test was conducted on the three samples having the dimensions of 3 mm × 20 mm × 20 mm, according to ASTM D570. The samples where dried in a vacuum oven at 70°C for 24 hours prior to the test. Then the samples were weighted immediately with a precision of 0.001g. In order to determine the water absorption ratio, the specimens were weighed every 2 hours for 1 day and afterward once daily for 1 week. Before weighing, the surfaces of the specimens were dried with a filter paper. Figure 6 shows the water absorption test instrumentation. The water absorption ratio of composites is calculated by the following equation:

$$Water\ absorption\ (\%)=\frac{(W_a - W_i)}{W_i}\times 100 \qquad (1)$$

where, W_i: mass of the dry sample, W_a: mass after absorption.

The water absorption rate of composites is calculated by the following equation:

$$Water\ absorption\ rate =\frac{\Delta(Water\ absorption\ ratio)}{\Delta t} \qquad (2)$$

where, Δt : time difference between two weighings.

Figure 6. Water absorption test instrumentation

3.3 Conductivity thermal test

The sample whose thermal resistance is to be determined is placed between two aluminum plates measuring (5x5) cm^2. The two aluminum plates ensure temperature homogeneity on each side of the sample by clamping. The upper plate is in contact with a cold source θ_2 and the lower plate is in contact with a hot source θ_1. The thermal power of which is adjustable. The thermal power is adjusted so that the lower plate is maintained at the temperature θ_1. Figure 7 shows the thermal conductivity device.

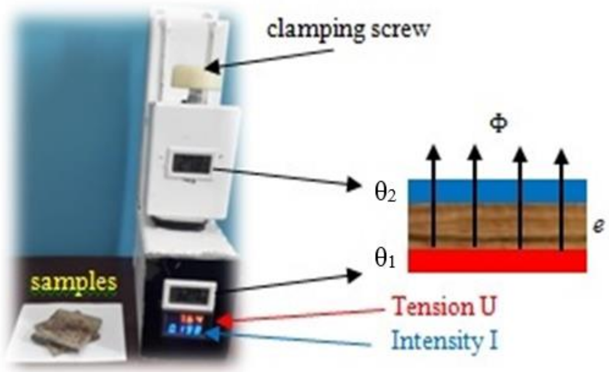

Figure 7. Thermal conductivity device

When steady state is reached, the thermal conductivity λ is determined by:

$$\lambda = - \frac{\Phi . e}{\theta_2 - \theta_1} \quad (3)$$

This thermal flux Φ corresponds to the electrical power of the energy transfer:

$$\Phi = P = U . I \quad (4)$$

where: λ: conductivity thermal, Φ: Thermal flux, θ_1 and θ_2: lower temperature and upper temperature, *e*: thickness, *P*: electrical power, *U*: tension, *I*: intensity.

3.4 Video-tensile test

This test was conducted to determine the elastic modulus E, stress of rupture σ_r and strain of rupture ε_r. Five samples of material composite were used to reproduce the test. The material was subjected to the tensile test on a universal Instron 5969 testing system connected to a computer equipped with Bluehill 3 testing software. Load-displacement curves were obtained by using a 5 KN static load cell at a jaw displacement rate of 2 mm/min.

To determine Poisson's ratio, an advanced extensometer video sensor device was used for longitudinal and transverse strains measurements. The five test samples are marked with four dots in the middle of each specimen using a specific marking guide. The advanced extensometer video sensor is placed in front of the specimen to detect the displacement of the four points. Figure 8 shows the Video-tensile test.

Figure 8. Video-tensile test

The Poisson's ratio ν_{LT} is calculated according to the following formula.

$$\nu_{LT} = - \frac{\varepsilon_T}{\varepsilon_L} \quad (5)$$

where, ε_T: transverse strain, ε_L: longitudinal strain.

4. RESULTS AND DISCUSSION

4.1 Density

The results of the material (epoxy reinforced of the *Lif* fibers mat) density is from (1.07±0.17) g/cm^3. It is about 30% lower than that of synthetic composites material and 6% to other composite reinforced by vegetal fiber [20-22]. This is due to the low density of the *Lif* fibers of date palm tree. Thus, *Lif* fibers offer an important advantage in the design of lightweight biocomposite materials.

4.2 Water absorption

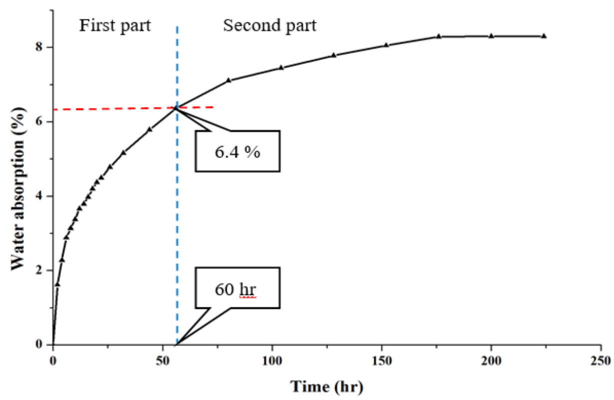

Figure 9. The water absorption of epoxy composite reinforced with *Lif* fiber mat

The curve of the evolution of the water absorption ratio as a function of the composite immersion time is shown in Figure 9. This curve is made up of two distinct parts. The first part (before 60 hours approximately where the water absorption rate is almost zero (Figure 10)) corresponds to the transient absorption regime, the water absorption rate is relatively high. In the second part of this curve, the water absorption rate decreases with time where saturation is reached.

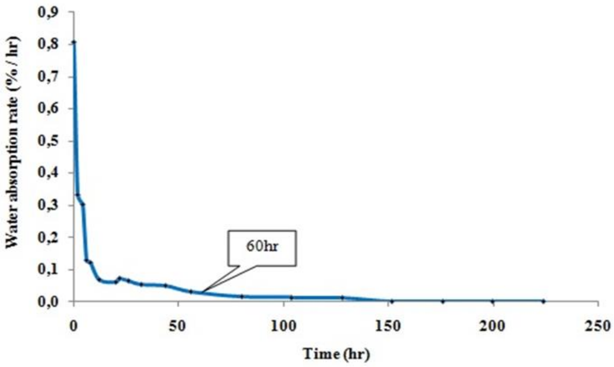

Figure 10. The water absorption rate of epoxy composite reinforced with *Lif* fiber mat

4.3 Conductivity thermal

The thermal properties of composite materials depend upon the constituent's nature, the volume fraction of their component, the size and shape of reinforcement fibers, method for making and the interface bonding between the matrix and fibers.

The results obtained by measuring the thermal properties of composite materials in the range (0.372-0.521) W/mK. This result is less high compared to the literature [23, 24]. This is because the composite material is made up of layers linked in series. Mathematically the conductivity thermal is determined by:

$$R_c = R_m + R_f \quad (6)$$

with:

$$R_i = \frac{e_i}{\lambda_i A_i}, \qquad i = c, m, f \quad (7)$$

where, R_c: thermal resistance composite, R_m: thermal resistance matrix, R_f: thermal resistance fibers, A: area.

Finally, according to the results obtained, the present material is classified in composite materials with low thermal conductivity.

4.4 Video-tensile

The results of the video-tensile test on specimens of virgin resin and composite materials are presented by typical stress-strain curves shown in Figure 11. They show a relatively large linear part characterized by a constant elastic modulus. Then, the curves lose their initial linearity, reflecting the onset of damage to the specimens. In the last phase, the specimens regain irreversible behavior until the occurrence of a sudden rupture.

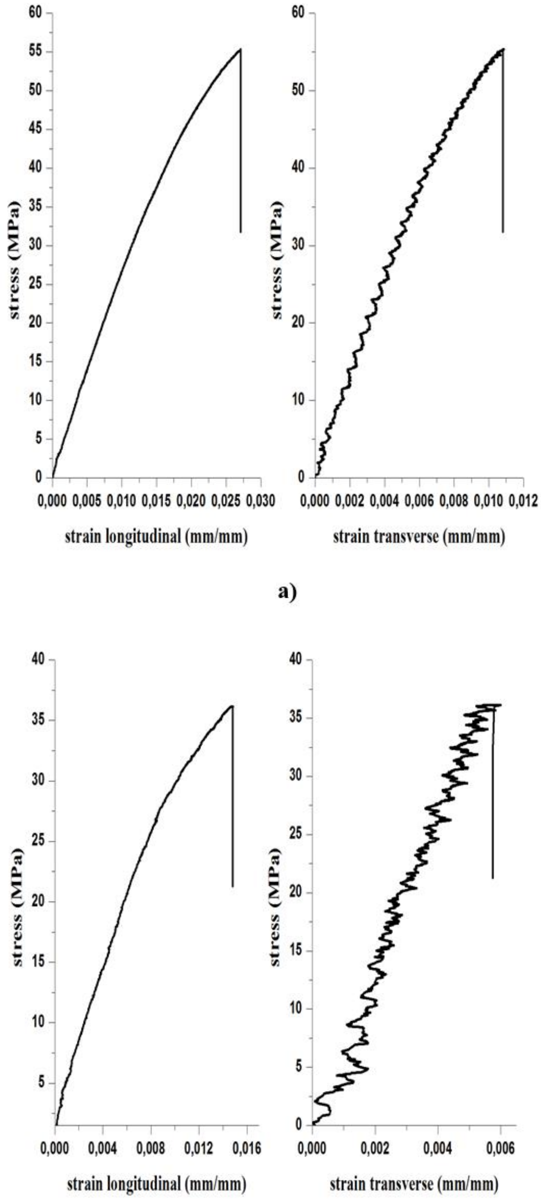

Figure 11. Video-tensile test on specimens of a) virgin resin b) composite materials

Table 3 illustrates the mechanical properties of virgin resin and composite materials obtained from several tests.

Table 3. Virgin resin and composite materials characteristics

Characteristic	virgin resin	composite materials
Stress of rupture (MPa)	56,37±1,230	30,40±3,980
Strain of rupture (mm/mm)	0,039±0,009	0,011±0,001
Modulus of elasticity (GPa)	2,679±0,082	3,669±0.560
Poisson's ratio (/)	0,404±0,008	0,384±0,053

The important results are observed in the mechanical test in this work. It is noted that the modulus of elasticity and the breaking stress vary according to this manufacturing process technique. There is an improvement 50% in the elastic modulus value of composite material reinforced with 20% date palm tree fiber mat compared to the virgin resin, while the stress of rupture value decrease by 20% compared to the virgin resin. The significant decreases observed in stress of rupture data could be ascribed to tow main factors: fiber-matrix adhesion problem represented by loosening of the fibers are shown in Figure 12, method of preparing biocomposite laminate by problem of creating defects like air bubbles.

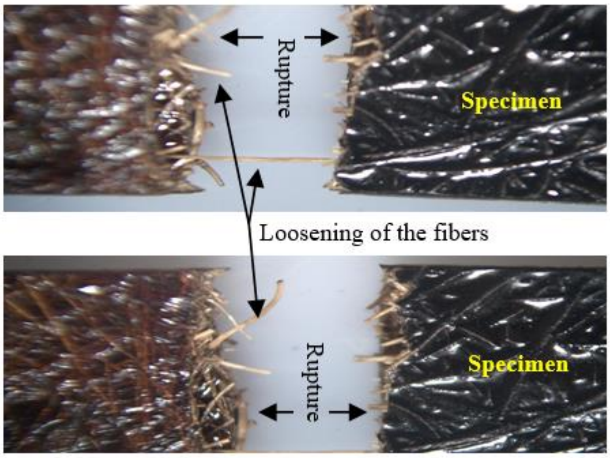

Figure 12. Loosening of the fibers

5. CONCLUSIONS

In this work, biocomposite laminates were prepared, using date palm tree fibers mat from the region of Biskra, Algeria, as reinforcing materials and epoxy as the matrix. The ratio of fibers in the composites laminates is 20%. In this experimental study examined the density, water absorption, conductivity thermal and tensile of materials.

The results of the study show that *Lif* fiber can be used like other vegetable fibers. The low density and the interesting mechanical properties of the *Lif* fiber indicate the possibility of using it as reinforcement in composite materials for various industrial uses. The water absorption leads to volume variations of the vegetal fibers in the composite and generates modifications to the fiber-matrix interface and can reduce the performance of the composite. Where the behavior of date palm tree *Lif* fibers with respect to water influences their long-term behavior due to low hydrophilic lignin content. The implementation of the epoxy matrix composite gives significant protection against humidity is harmful. The thermal conductivity measurements show that date palm tree fiber reinforcement gives low thermal conductivity, thus, we can benefit from it in the manufacture of insulating composite materials. The reinforcement in the form of a mat, which in our case is a mat made of date palm tree fibers, significantly modifies the mechanical behavior of the composite materials compared to the virgin resin. This case is observed in the video-tensile test response. Where our laminates have a higher elastic modulus than that of virgin resin. On the other hand, the breaking stress of the composite is lower than that of the unreinforced resin. This is related to the microstructure of the composite which is determined or influenced by the processing procedure and its constitution.

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NOMENCLATURE

W_i	mass of the dry sample, g
W_a	mass after absorption, g
e	thickness, m
U	tension, Volt
I	intensity, A
P	electrical power, W
R	thermal resistance, K .W^{-1}
A	area, m^2
t	time, s

Greek symbols

ε	Strain, mm/mm
σ	Stress, MPa
ν	Poisson's ratio
λ	Thermal conductivity, W.m^{-1}. K^{-1}
θ	Temperature, K
Φ	Thermal flux, W.m^{-2}

Subscripts

r	rupture
L	longitudinal
T	transverse
c	composite
m	matrix
f	fiber