

Study of Mechanical and Thermomechanical Properties of Jute/Epoxy Composite Laminate

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ABSTRACT: This article presents the development and the characterization of composite material (laminate) containing natural jute fiber reinforcement. Thermal characterization of jute fiber reinforcement shows the influence of the temperature on the mechanical behavior of fiber. At 180°C the jute fabric loses 50% of its mechanical characteristics. The laminate obtained by a process known as infusion is polymerized at a temperature lower than that which affects the mechanical properties of dry fabric. The digital image correlation carried out on laminated jute/epoxy (warp and weft direction) under tensile test shows the presence of a considerable gradient of deformation. This gradient is explained by the variability related to the local voluminal change of jute fibers of one place to the other and the nature of the weaving of the jute fiber. The three-point bending tests show a significant dispersion of rupture stress. The thermomechanical tests carried out on samples in the two principal directions, show that the thermal coefficient of expansion in warp direction is 48% larger compared to the weft direction. The thermogravimetric test shows that this laminate absorbs up to 4% water mass after 8 h in a climatic chamber with 70% moisture content.

KEY WORDS: jute, natural fibers, mechanical proprieties, thermomechanical analysis, thermogravimetric analysis.

INTRODUCTION

THE COMPOSITE TECHNOLOGY of a polymeric matrix, reinforced with man-made fibers such as glass, aramid, carbon, etc., has come of age especially with the advances in aerospace applications since the 1950s. The developments in composite material after meeting the challenges of aerospace sector have cascaded down for catering from domestic to industrial applications. Composites, the wonder material with light-weight, high strength-to-weight ratio, and stiffness properties have come a long way in replacing the conventional materials such as metals and woods. Material scientists all over the world

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focused their attention on natural composites reinforced with fibers made of jute, sisal, coir, pineapple, etc., primarily to cut down the cost of raw materials [1].

The work undertaken by Baley [2], shows that the properties of natural fibers change considerably and this variability begins from their harvests [3]. Khöler and Spatz [4] show that the genetic variability of jute fiber is related to the nature of jute. One of the principal problems of manufacture of jute fabric resides in the optimization of its use. To get the better mechanical properties the optimization of fiber use is significant [5,6].

The behavior of jute fiber is controlled by two parameters, one is the reorientation according to the axis of fibril and the slip compared to the other [7–9]. Hearle [10] shows that the angle of microfibrillar of the jute is influenced by the percentage of cellulose in the jute (61–71%). Work of Broutman and Sahu [5] show that this angle of microfibrillar is generally about 8° and this influences the mechanical behavior of the jute. For the improvement of the mechanical properties of the jute, especially its behavior in wet medium, several works are initiated to treat the surface of the jute fiber [11–20]. These treatments, which modify the interphase of the surface of fiber, produce morphological changes [12,21]. Treatments are carried out using alkaline, silane or alkaline, and silane [12–18] which decrease the absorption of moisture from 4.2% to 3.8%. The breaking stress of a fabric treated with silane is better than that of fabric untreated, on the other hand the breaking stress of a jute fabric untreated is better than a jute treated with alkaline [13,15–17]. Other treatments also done under UV radiation present an increase of 58% of its flexural strength [19,20,22].

The uses of jute-reinforced plastic are the current interest of many researchers. The most used thermoplastic resins are polyethylene (EP) [23,24], polypropylene (PP) [25,26], polystyrene (PS) [27,28], vinyl polychloride (PVC) [29,30], and the polyester [31,32,36] for economic reasons. The choice of a structural polymeric resin does not pose a problem of provisioning but constitutes a barrier to the recyclability of the unit. However, the ecological or natural resins do not answer the schedule of conditions of the end product because of the weak mechanical properties. These resins are rigid and breakable like the polylactone (PLA) with the jute [33], dissolve in water like the natural polysaccharose (TPS) [34]. Moreover, the migration of water into the resin can lead to disturbance of the fiber/matrix interface [35].

The influence of the stacking sequences on the mechanical properties (tensile, bending, and interlaminar shearing) of the untreated woven hybrid composites was studied by Sabeel and Vijayarangan [31]. Their results show that the mechanical properties of the laminate can be improved by incorporation of glass fiber in the jute/polyester composites. The moisture absorption of composite material containing jute, glass fiber reinforcement on an isothalic polyester resin [32] shows that the absorption of water decreases with the increase in the mass rate of glass fibers in the composite material. This is because the impermeable glass fibers act as barriers and prevent the direct contact between the jute and water [32,28]. Work of Alvarez et al. [36] show that the studies relating to the thermal degradation of the jute/vinylester composite are still limited for these materials, in spite of their use in the automobile.

For developing jute fiber reinforced plastic laminate, epoxy resin (LY 5052) was used by infusion process, which presents many advantages. Infusion process is easy and economical to fabricate, it also makes it possible to manufacture laminates of large size.

The mechanical behavior of the dry jute is significant and also the polymerization temperature of the resins, which can deteriorate the mechanical properties of the jute. Within the frame of this work, tensile tests are carried out on samples of dry jute fabric

at various levels of temperatures. Our objective is to develop a relation between the influences of temperature on ruptures stress of dry jute fabric. For that, we evaluated the mechanical and thermomechanical properties of the jute/epoxy laminate. Tensile tests, shearing, and three-point bending tests are carried out on standardized test specimens. For the tensile tests, the test specimens are instrumented on the surface with extensometric gages and randomly speckle pattern used for the measurement of the field of deformation by stereophony image correlation. Comparison between these two methods of measurements is carried out. The dilatometric and thermogravimetric analysis are carried out on jute/epoxy. The dilatometric analysis allows the identification of the thermal dilation coefficients in the warp and weft directions; the thermogravimetric analysis makes it possible to see the capacity for absorption of the jute/epoxy in wet place. The originality of this work consists initially in using the measurement technique of field by stereophony correlation of digital image for the identification of the mechanical characteristics of the jute/epoxy and the dilatometric and thermogravimetric analysis on jute/epoxy.

EXPERIMENTAL PROCEDURE

Mechanical Testing

The woven jute fibers and the jute/epoxy laminates are tested with an Instron tensile testing machine (equipped with a sensor of 10 kN) at a speed of 2 mm/min. In order to see the influence of the temperature on the behavior of dry fabric, tensile tests are carried on specimens at various levels of temperatures (25°C, 50°C, 80°C, 100°C, 150°C, 180°C). In order to maintain uniformity of specimen, they are cut from the same fabric (Figure 1). The specimens are clamped in mechanical jaws of Instron tensile testing machine

The jute/epoxy laminate is manufactured by the process known as 'infusion,' The fabric is prepared and cut out with dimensions of 300 × 300 mm². The lay-up sequence is [0]_S for the laminate, [90]_S for warp, and [+45/−45]_S for weft. The jute/epoxy laminates are cut out with a diamond saw following standard NF IN ISO 527-1. For identifying the mechanical properties (Young's moduli, Poisson's ratios, and tensile stresses in the

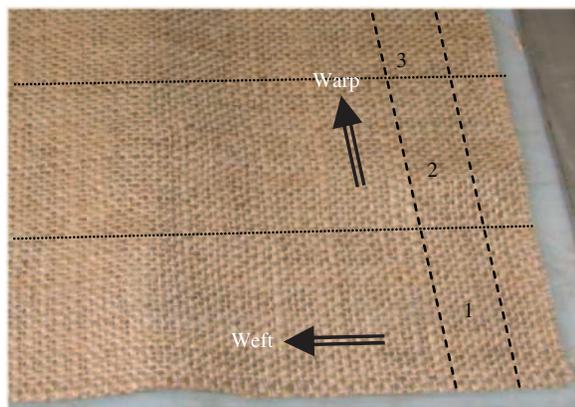


Figure 1. Cut of jute in the warp direction.

directions of warp and weft), tensile tests, and shear tests are carried out on specimens ($250 \times 50 \times 2.5 \text{ mm}^3$) in accordance with the standard IN ISO 527-5. These tests are carried out using hydraulic jaws with a pressure of 10 bar and at a speed of test 2 mm/min. For measuring deformation, the specimens are instrumented on the surface by strain gages of 25 mm length. The gages are connected on a Strainsmart de Vishay bridge. While conducting tensile tests in warp and weft direction, in addition to strain gages, digital image correlation technique is also used. Two numerical cameras (Figure 2(a)) are used for acquiring the images of the specimens. The images are stored in software (VIC3D) and analyzed. Stress rupture in shearing γ_{lt} and the modulus of rigidity (G_{lt}) are calculated respectively by Equations (1) and (2).

$$\gamma_{lt} = -\varepsilon_{ox} + \varepsilon_{oy} \quad (1)$$

$$G_{lt} = \frac{\sigma_{ox}}{2(\varepsilon_{ox} - \varepsilon_{oy})} \quad (2)$$

Three-point bending tests are carried out according to standard NF IN ISO 14125 on laminates ($50 \times 18 \times 2.5 \text{ mm}^3$), Figure 2(b). The maximum stress in three-point bending tests is given by Equation (3) with F_{\max} (N) the maximum force of rupture, L the distance between supports (mm), b and h are respectively the width and the thickness of the sample. Five samples are tested in the two directions (warp and weft). The bending equation is given in 4, where λ is the displacement (mm).

$$\sigma_{\max} = \frac{3 \times F_{\max} \times L}{bh^2} \quad (3)$$

$$E = \frac{FL^3}{4bh^3\lambda} \quad (4)$$

Thermomechanical analysis

The thermomechanical analyses (TMA) are carried out on two specimens with dimensions $4 \times 2.5 \times 8 \text{ mm}^3$. These tests are carried out in a thermal apparatus TMA7. The imposed thermal cycle starts at 20°C until it reaches 100°C with a 10-min hold, and then the same was reversed until the specimen reaches ambient temperature. The rate of rise and decrease is fixed at $5^\circ\text{C}/\text{min}$.

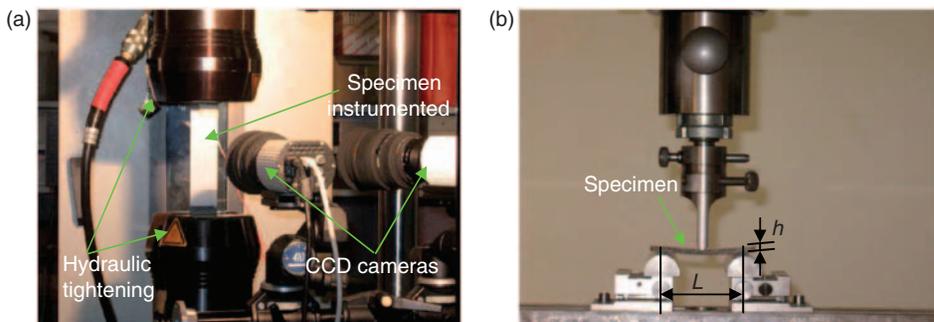


Figure 2. Experimental device for mechanical tests: (a) tensile test, (b) three-point bending test.

Thermogravimetric Analysis

The thermogravimetric analysis (TGA) is carried out in a controlled atmosphere on three specimens with a dimension of $3 \times 2.5 \times 6 \text{ mm}^3$. Before TGA analysis, the specimens are placed in a climatic chamber with the water content controlled at 70%. The first specimen is kept 2 h in the climatic chamber, the second one is for 8 h, and the third one is for about 24 h. The thermal cycle imposed on the specimens after their removal from the climatic chamber is as follows: the temperature of the specimen is increased from 20°C to 100°C , and then the specimen is held at 100°C for 100 min, then the same was descent to the ambient temperature with the rate of speed of $5^\circ\text{C}/\text{min}$.

RESULTS AND DISCUSSION

Jute

The tensile tests were carried out on jute fabric at various temperatures. At the start of the loading, upto 3 MPa, we note a non-linear behavior; of beyond 3 MPa and until the rupture all the specimens tested present a linear behavior. The non-linear behavior can be explained by the influence of the weaving of the fiber (warp and weft). Under tension, the fiber tends to becoming right, which modifies the undulation in the direction of tension (warp and also in the transverse direction; this biaxial phenomenon is amplified by the crushing of the wicks taking weaving into account [6]. This behavior in tensile is non-linear because of the connections and the undulations between the two networks, which lead to no local geometrical linearities [26].

Figure 3 shows the influence of failure stress according to the rise in temperature. It is noted that for temperatures raising from 25°C to 150°C the rupture stresses are not influenced by the temperature, the rupture stress reaches 11.8 MPa. The specimens

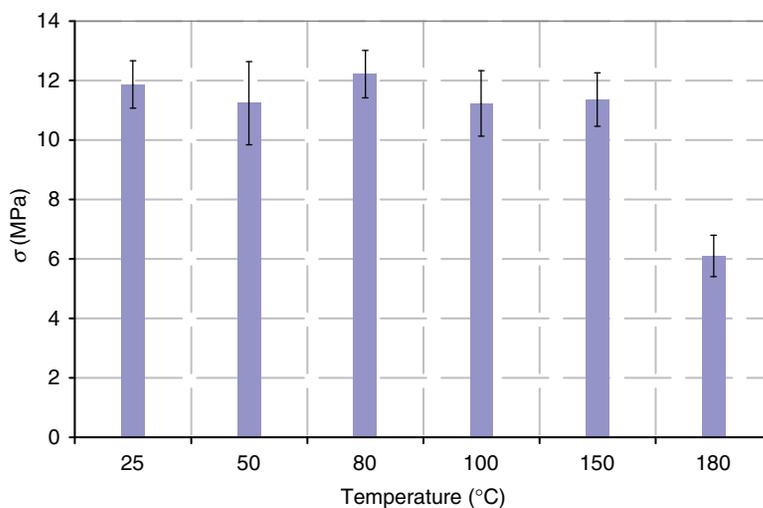


Figure 3. Failure stress of the tensile test on dry fabric.

heated at 180°C change its color to clear brown. The stress at which the fiber fails is 57% compared to the breaking stress of the samples at ambient temperature. This thermal limit is used to determine the choice of the resin for the manufacture of the laminates. Our choice is related to an epoxy resin of type LY 5052 with hardener DY 5052. The unit is polymerized at a temperature of 80°C during 8 h.

Laminate

MECHANICAL TESTING

Figure 4 shows the evolution of the constraint according to the axial deformation on two jute/epoxy specimens subjected to tension. The first specimen cut according to warp direction shows a linear elastic behavior up to 30 MPa, and then we observed a non-linear behavior until the breaking stress (43 MPa). The second specimen tested in the direction of weft presents a linear elastic behavior up to 47 MPa and finally non-linearity until rupture at 61 MPa. The difference in values of the breaking stress recorded in the two directions is primarily due to two significant factors: the variation of the diameter of the wire constituting fabric and the mode of weaving of fabric; we have more wire in the weft direction than warp direction.

Figure 5 represents the evolution of shear stress according to the deformation of a jute/epoxy laminate [+45/−45]_s. We observed a non-linear behavior during its testing. The modulus of rigidity was calculated on the rectilinear part at the beginning of curve.

The potential of measurements of field by stereophony digital image correlation (DIC) in the analysis of the structural behavior of composite materials is proven [27]. Figure 6 shows a comparison between the axial and transverse deformations obtained by digital image correlation and by strain gages. Lesser than 40 MPa, the difference between both the methods is lower than 5%, while approaching the rupture stress the variation reaches to 8%. This maximum variation corresponds to the axial deformation.

With the mesoscopic scale, the cartography of the longitudinal deflections reveals the presence of a strong gradient primarily dependent on the geometrical variability of fabric and that of the voluminal rate of the reinforcement at any point. Deformations shown in the warp direction vary from 6781 microdef to 12,427 microdef (6000 microdef). In the

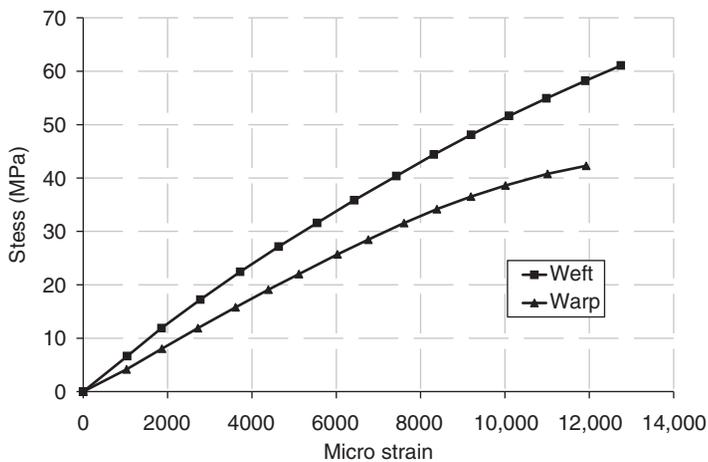


Figure 4. Stress vs. axial strain for the tensile test on jute/epoxy in weft and warp direction.

weaving direction, one can note that deformation gradient is less pronounced at about 200 microdef. Scanning electron micrograph of the fracture topographies after tensile tests on specimens showed a clear rupture of fiber in the warp direction and the presence of some fibrillates shredded in constituent fiber in the weft, this mode of rupture is identical to that of a jute/polyester presented by Sabeel et al. [31]. The same mode of rupture is observed after tensile test on specimens made in weft direction, on the other hand in the place of the shredded fibrillates, we note the presence of print of wire of warp. Fracture topographies in shearing of a jute/epoxy laminate $[+45^{\circ}/-45^{\circ}]_S$ showed a good cohesion between the various layers of reinforcement (no slip between layers). The modes of rupture are identical to the work of Park et al. [17].

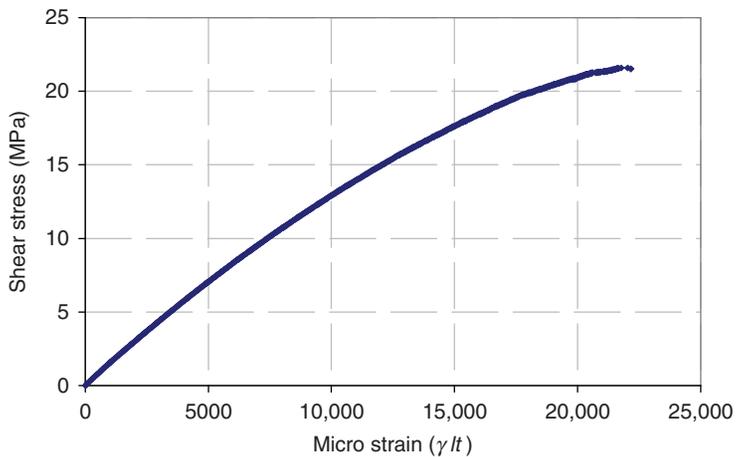


Figure 5. Shear stress vs. axial strain for the tensile test on jute/epoxy $[+45^{\circ}/-45^{\circ}]_S$.

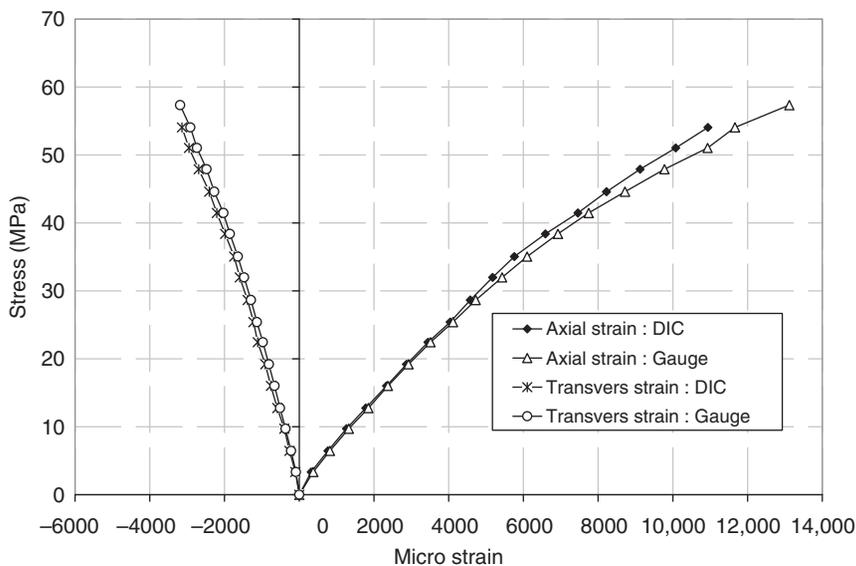


Figure 6. Stress vs. strain during tensile test on specimen made in weft direction: comparison between DIC and strain gage.

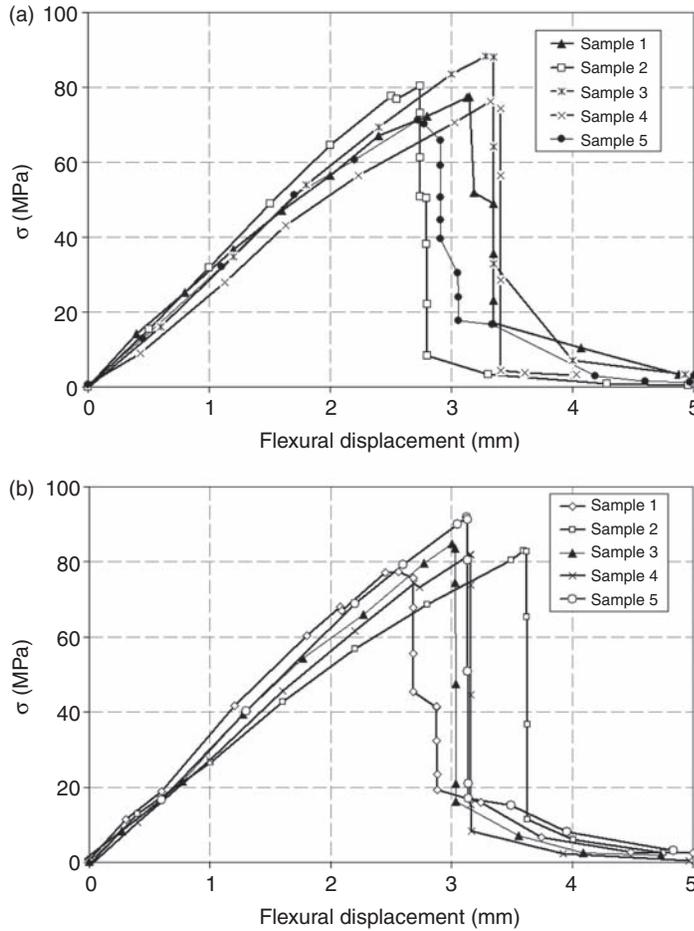


Figure 7. Curves of three-point bending test on jute/epoxy: (a) warp, (b) weft.

Figure 7 represents the evolution of the load according to displacement during the three-point bending test on jute/epoxy laminates in warp and weft. It is noted that all the specimens tested present a non-linear mechanical behavior. The average value of stress rupture of the specimen is 80.5 ± 8.78 MPa in warp direction, and 83.9 ± 5.25 MPa weft direction. The calculation of the modulus of inflection in warp and weft shows similar results: 3.24 ± 0.2 GPa.

Table 1 presents the values of the modulus of elasticity, modulus of rigidity, rupture stresses, and the Poisson's ratios obtained by strain gages. The jute/epoxy laminate after rupture by three-point bending were analyzed using SEM. The part of the specimen observed in tension shows the presence of a package of fibers (constituting warp) stretched. This can be explained by the following: under the effect of loading the fiber of warp elongated by exceeding their yield stress; after rupture, the specimen remains stretched. This phenomenon is not observed in a warp thread tested in compression. The analysis with the SEM on the zone of rupture of the specimen in warp and weft direction presented the same observations.

Table 1. Mechanical characteristics obtained by gages.

	Tensile modulus Warp	E (GPa) Weft	G (GPa)	Poisson's ratio		σ rupture (MPa)	
				ν_{lt}	ν_{tl}	Warp	Weft
Tensile test	4.5 ± 0.6	5 ± 0.8	—	0.24	0.27	38 ± 6	51 ± 4
Bending test	3.2 ± 0.2	3.2 ± 0.3	—	—	—	80 ± 8	83 ± 5
Shearing test	—	—	1.45	—	—	23	

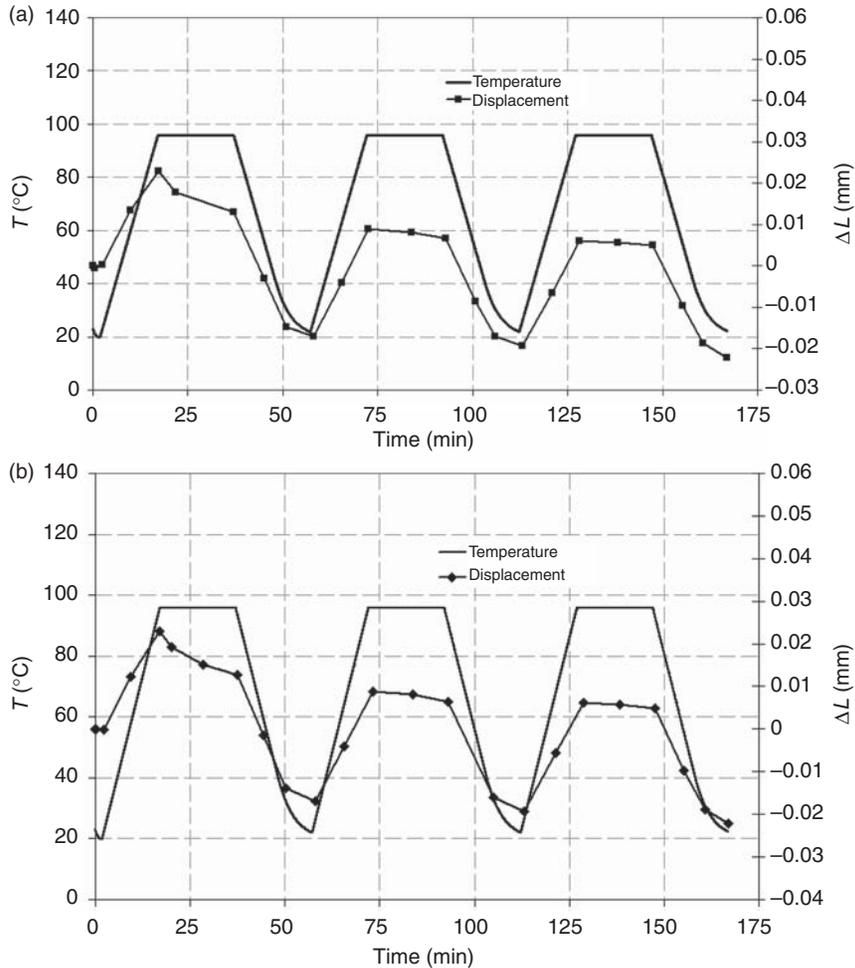


Figure 8. Evolution of the temperature and length of the specimen with time: (a) warp direction, (b) weft direction.

THERMOMECHANICAL ANALYSIS

Figure 8(a) and (b) respectively shows the TMA and dilation coefficients of the jute/epoxy laminate in the warp and weft directions after three successive thermal cycles. In the isothermal stage of the first cycle, we note a variation in the length of the specimen;

Table 2. Thermal dilation coefficients (α).

Dilatation coefficient α (Warp)	Dilatation coefficient α (Weft)
$40 \cdot 10^{-6}/^{\circ}\text{C} \pm 1$	$27 \cdot 10^{-6}/^{\circ}\text{C} \pm 0.4$

this variation is significant which is not true for the other two isothermal stages. This length variation can be explained by the presence of moisture in the specimen. At 100°C (which characterizes the isothermal stage) we have the phenomenon of water evaporation in material. The same phenomenon is also noticed in the specimen made in the weft direction. To confirm this analysis, thermogravimetry tests are carried out. For calculating the thermal dilation coefficients we excluded the first cycle because of the presence of moisture in material. The thermal dilation coefficients obtained in the warp and weft direction show a variation of 66% of the thermomechanical behavior of the laminate compared to weft direction. Table 2 shows the average value of the thermal coefficient of expansion in both the directions (warp and weft) and its standard deviation.

THERMOGRAVIMETRIC ANALYSIS

The TGAs carried out in samples at an atmosphere controlled climatic chamber with a water content of 70% for different durations show a significant variation of mass (Δm), this variation is caused due to the absorption capacity of the jute [28]. During the analysis, the specimens studied undergo a thermal cycle characterized by a stage with 100°C. The loss of mass of the specimen having spent 2 and 8 h respectively in climatic chamber showed the variation of mass, which is more significant than that of the sample spent 2 h in climatic chamber with a difference of about 1.5%. This can be explained owing to the fact that the first sample has lost less mass because it has absorbed less moisture, while the second sample having absorbed much more moisture lost a more significant mass. Tests were carried out over 8 h of duration and the variation of mass is 4%. It is observed that the most significant variation is during the first 70 min.

CONCLUSION

On the basis of eco-design and development of composite using the local resources, the study of the natural jute reinforcement initially enabled us to identify the influence of the temperature and its behavior in tension. The rupture stress under tension of dry fabric heated at a temperature higher than 150°C fails at a stress of 54% compared with that of a dry fabric at ambient temperature. In the second time, thanks to the mechanical tests (traction, inflection, and shearing) carried out on laminates of the jute/epoxy show certain variability mainly dependent on nature of fabric and its mode of weaving. The dimensional checks of fields by stereophony correlation of digital images highlight this variability. On the cartographies of deformations in warp and weft direction, we observed a micro-gradient of deformation of about 6000 micro-deformation.

The tests of TMA showed that the dilation coefficient in weft is 48% larger than that in warp direction. We also note a variation of the mass of the laminate in the presence of moisture due to the capacity of absorption of jute fiber. This absorption is more significant

in the first hour of test (TGA). The laminate jute/epoxy thus obtained will be used like skin in the realization of a sandwich with natural core.

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