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WATER ABSORPTION BEHAVIOR AND ITS EFFECT ON CHARPY IMPACT TEST OF JUTE YARN REINFORCED POLYESTER COMPOSITES

Plates of bidirectional jute/polyester composite material were manufactured by the contact molding method. These plates were cut to form notched test pieces 80x15x4 mm and immersed in water (1, 10, 30, 90, 180 and 270 days) in order to study the impact behavior of this material. The studied composite exhibited a water saturation limit after an immersion period of approximately 30 days, with Fickian diffusion of water within the material. Williams' method based on linear elastic fracture mechanics was used to calculate impact toughness GIC, which is due to the intrinsic characteristics of the material.

Keywords: jute, polyester, absorption, Charpy impact test, immersion, diffusion

INTRODUCTION

The use of natural fibers in composite manufacturing technology is progressively increasing owing to their availability and better properties, being comparable with other synthetic organic or mineral fibers.

The advantage of these fibers is their light weight, and low impact on the environment. There have been several attempts to use composites based on natural fibers such as jute fiber in place of ceramic fibers like glass, particularly in non-structural applications. Jute fiber offers good compatibility with different types of thermosetting or thermoplastic, biodegradable, and less abrasive matrices. However, jute fibers are hydrophilic and exhibit high moisture absorption compared to artificial fibers as they contain a high percentage of cellulose (around 56-64%) [1]. Water absorption is a function of the heterogeneity of the material and the nature of the interface which will create differential swelling within the composite. This can lead to a decrease in some properties and should be considered when selecting applications [2]. According to Roudier [3], plant fibers are not entirely surrounded by the polymer matrix, which results in a poor fiber/matrix interface and subsequently, uncontrollable behavior with regard to water absorption. The moisture concentration initially increases over time until it approaches a saturation point. The time required to reach the saturation point depends on the thickness of the sample and the ambient temperature. Drying can reverse the process, but cannot give rise to full realization of the original properties. Fluid chemistry and molecular structure have a significant effect on the absorption process. This has been shown by observations that the degree of saturation of a composite submerged in distilled water, fresh water, and sea water decreases in that order, respectively [4]. In general, the absorption of water by composites follows Fick's generalized law of diffusion. The rate of moisture absorption depends on several factors such as the void content, type of fiber, type of resin, orientation of fibers, temperature, and the presence of microcracks. Thomas et al. [5] studied the relationship between the moisture absorption of polyethylene composites reinforced with fibers from pineapple leaves (10%, 20% and 30% by weight). They found that moisture absorption grows almost linearly with the percentage increase in fibers. The diffusion of moisture in a composite material can be governed by three main mechanisms [6, 7]: the first mechanism involves the diffusion of water molecules within the micro-spaces between the polymer chains. The second mechanism involves capillary transport in the interstices and interface faults between the fibers and the matrix. The third mechanism comprises mainly the transport of microcracks in the matrix resulting from the swelling of the fibers (in particular in the case of composites based on natural fibers) [8]. Several works have been published in this context, the aim of which is to deduce the process of water absorption and the effect of humidity on the mechanical and chemical properties of composites [9-12]. Jute fibers

can undergo plasticization, which is characterized by changes in the mechanical properties. Hossain et al. [13] analyzed the effect of water absorption on the mechanical properties of a composite made from jute fiber. They found a remarkable decrease in breaking stress, as well as flexural modulus, with an increasing water immersion time up to 60 days. Athijayamania et al. [14] performed tensile and impact tests after the water aging of sisal/polyester composite material. The formation of a hydrogen bond between the water molecules and the cellulose fiber caused a decline in the static and dynamic mechanical properties.

This work aims to introduce the mechanism of water diffusion in a jute reinforced polyester composite material in order to study the effect of water absorption on the Charpy impact test using Williams' method based on linear elastic fracture mechanics.

EXPERIMENTAL TECHNIQUES

Materials

The material used in this work was a thermosetting matrix composite consisting of a polyester resin reinforced by three layers of bidirectional jute yarn with a mass fraction of 40%.

The unsaturated polyester resin consisted of a monomer (Polylite 420-852). It was cross linked at room temperature by adding an organic peroxide type catalyst. It successively changed from the initial viscous liquid state to the gel state, and then to the infusible solid state. Flexural modulus E is approximately 2350 MPa, with a tensile strength of 45 MPa, breaking stress about 72 MPa, density at ambient temperature about 1.11 g·cm^{-3} and 20 dPa·s viscosity [15].

A bidirectional jute yarn reinforcement with an average density of 1300 kg/m^3, modulus of elasticity of 20-55 GPa, and elongation at break of approximately 1.6% was used [16, 17].

Jute/polyester composite (JP)

The bidirectional jute/polyester composite plates were manufactured by the contact pressure molding method, which consists in depositing a succession of layers of jute yarn 40% by weight on the shape of the plate to be molded.

Immersion process

The jute/polyester composite sheets were cut into several specimens of a rectangular shape of the dimensions 80x15x4 mm (Fig. 1). These samples were immersed in water for 1, 10, 30, 90, 180 and 270 days at room temperature in a tank large enough to allow direct contact and total immersion of all the surfaces of each test piece. After removing the excess water, they were weighed on a precision analytical scale.

Fig. 1. Jute/polyester (JP) composite test pieces immersed in water

The amount of water absorbed was calculated by comparing the initial and final weights. An average of five test pieces was adopted for each immersion time. The weight gain process continued for the entire immersion time. Weight gain M_t is calculated according to the following equation:

$$M_t = \frac{m_t - m_0}{m_0} X\ 100 \tag{1}$$

where m_t and m_0 represent the weight of the test piece immersed in water for time t, and the weight of the test piece in the dry state (before immersion in water), respectively. There are three different categories of diffusion behavior: Fickian scattering, non-Fickian scattering and anomalous scattering [18]. The three main cases of diffusion can be distinguished theoretically by the shape of the absorption curve, which is represented by the following empirical equation:

$$\frac{M_t}{M_m} = Kt^n \tag{2}$$

where M_m is the equilibrium moisture content, and K and t are constants. The value of n indicates the mechanism of absorption, and constant K is a characteristic of the sample, which indicates the interaction between the sample and water.

The values of n and K were determined by linear regression analysis. The value of diffusional coefficient n shows a different behavior between the cases for Fickian diffusion (Case I) n = 0.5, for non-Fickian diffusion (Case II) n = 1, and for abnormal non-Fickian diffusion, n represents an intermediate value ($0.5 < n < 1$), whilethe transport mechanism is super Case II (controlled relaxation) for diffusional coefficient $n > 1$ [19]. The mechanism of water absorption and the kinetic parameters n and K were analyzed by putting the experimental values into the following equation, which is derived from Equation (2):

$$log\left(\frac{M}{M_m}\right) = log(K) + nlog(t) \tag{3}$$

Fick's second law is commonly represented as:

$$\frac{dC}{dt} = D\frac{\partial^2 C}{\partial x^2} \tag{4}$$

where: c – local solvent concentration, x – abscissa in the thickness [m], D – diffusion coefficient (or diffusivity) [$m^2 \cdot s^{-1}$].

Several models have been developed to describe the moisture absorption behavior of materials [20]. For one dimension of moisture absorption, where each sample is exposed on both sides in the same environment, total moisture content G can be expressed as follows [21]:

$$G = \frac{m_t - m_0}{M_m - m_0} = 1 - \frac{8}{\pi^2} \sum_{j=0}^{\infty} \frac{1}{(2j+1)^2} exp\left[-\frac{(2j+1)^2 \pi^2 D_x t}{h^2}\right] \quad (5)$$

where M_m is the mass of the sample at equilibrium. This is an efficient diffusivity since all the heterogeneities of the composites have been neglected; h is the thickness of the sample, t is the time, and j is the summation index. The diffusion coefficient is an important parameter in Fick's law. Solving the diffusion equation for the moisture weight, and rearranging with respect to the moisture content percentage, the following relationship is obtained [8]:

$$M_t = \frac{4M_m}{h}\left(\frac{t}{\pi}\right)^{0.5} D_x^{0.5} \quad (6)$$

The diffusion properties of the composites described by Fick's laws were evaluated by measurements of weight gain of the pre-dried sample immersed in water by considering the slope of the first part of the weight gain curve with respect to the square root of time using Equation (7) [22]. Diffusion coefficient D is defined as the slope of the normalized mass gain as a function of $t^{1/2}$ and has the form:

$$D = \pi\left(\frac{kh}{4M_m}\right)^2 \quad (7)$$

where k is the initial slope of the linear regression line of $M(t)$ as a function of $t^{1/2}$.

Charpy impact test

The specimens used in the impact test are of a prismatic shape (80x15x4), with a lateral notch of the SEN type (single edge notch) as illustrated in Figure 2. These specimens were cut from 300x210 mm^2 plates, then notched in the middle at different depths. Pre-notching was first carried out using a hacksaw and then the notching was continued with a rigid blade in order to have a sharp shape of the crack tip. The notch lengths are included in the ratio $0.2 < a/D < 0.6$, where a and D are the notch length and the width of the specimen, respectively. The dimensions of the test pieces are shown in Table 1. L and B are the length of the test piece and the distance between the support and the thickness of the test piece, respectively.

The tests were carried out on a Zwick 5113-type Charpy pendulum impact machine in three-point bending (Fig. 3). The device's trigger angle was 160° and the impact speed was 3.85 m/s. The pendulum used for the study materials was 7.5 joules. Figure 3 shows the employed experimental device, as well as the device for acquiring and processing data by a microcomputer equipped with "expert test" software.

TABLE 1. **Dimensions of Charpy impact specimens**

Jute/polyester composite material				
l [mm]	D [mm]	B [mm]	a [mm]	L [mm]
80 ±0.1	15 ±0.1	4 ±0.2	3-9	64

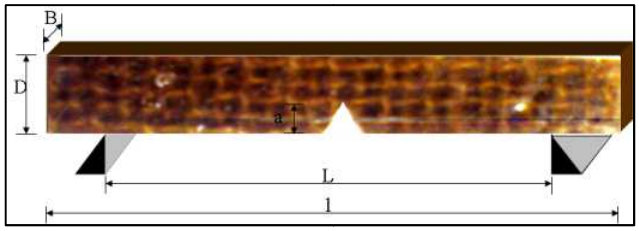


Fig. 2. Jute/polyester (JP) test pieces


Fig. 3. Charpy impact test setup

Williams' method based on the principles of linear elastic fracture mechanics was used to interpret the results of the impact tests on the notched specimens [23-25]. This method makes it possible to obtain an estimate of the energy or toughness G_{IC}, an intrinsic parameter of the material from the total energy U dissipated during the impact test according to the following equation:

$$U = G_{IC}.BD\phi \quad (8)$$

where ϕ is a calibration factor which depends on the geometry of the specimen and which was tabulated by Williams for different notch lengths and for different L/D ratios. Thus, recording the energy lost by the hammer at the time of impact for each notch plotted on a $U = f(BD\phi)$ diagram gives a straight line, whose slope measures the G_{IC}.

RESULTS AND DISCUSSION

Water absorption

Immersing the jute/polyester composite materials in water causes a change in the physical properties of the different samples. This change is a function of the chemical nature of the water as well as the characteristics of the materials: the nature of the fibers and the matrix, the geometry of the submerged specimens, the architecture, and the percentage of reinforcement. The jute/polyester (JP) composite material exhibited an increased weight gain with increasing immersion time (Table 2, Fig. 4).

TABLE 2. **Weight gain as a function of immersion time in water of jute/polyester composite**

Immersion time [days]	Weight gain [%]
0	0
1	0.952
10	3.201
30	4.336
90	4.338
180	4.381
270	4.391

The water absorption curve in Figure 4 shows that rapid penetration occurs at the initial stage as water mainly penetrates between voids and pre-existing microcracks. This curve becomes almost constant after an immersion time of about 30 days until the end of the immersion time (270 days). This implies significant reduction and stabilization as the material approaches saturation and is attributed to swelling of the components as the final equilibrium is achieved.

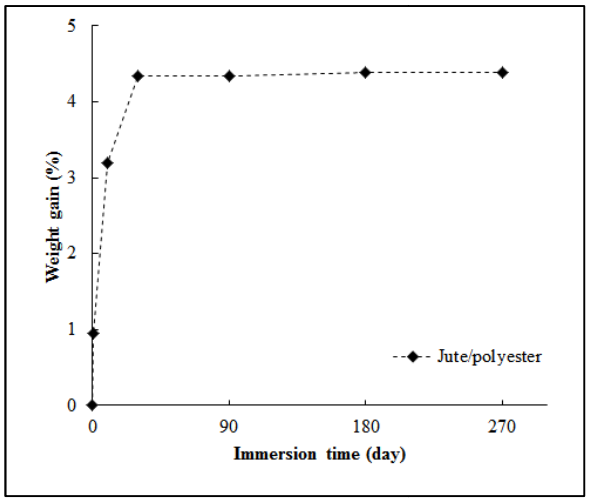


Fig. 4. Weight gain as a function of duration of immersion per day of JP composite in water

Equilibrium in the weight gain is reached after a certain time, and the absorption process depends on the swelling kinetics in the dispersed and continuous phases [26]. The hydrophilicity of jute yarn is responsible for the absorption of water due to hydroxyl groups [7]. Water can enter the cellulose network of the fiber and into the capillaries and spaces between the fibers and areas less bound by fibers, and it can attach itself by chemical bonds to hydroxyl groups in cellulose molecules [8]. Owing to this hydrophilic nature, swelling by the absorption of water can lead to microcracks within the material.

The mechanism of water diffusion within a JP composite material was studied in order to determine the values of n and K by fitting the experimental results of the diffusion equation (3).

Figure 5 presents the water diffusion curve of log (M_t/M_m) versus log (t) for the 40% jute/polyester composite material. The values of n and K were calculated from the slope of the linear regression line for a weight increase of 60% of the mass of each sample (M_t/M_m< 0.6; log (M_t/M_m) < –0.22) [18]. These values are summarized in Table 3.

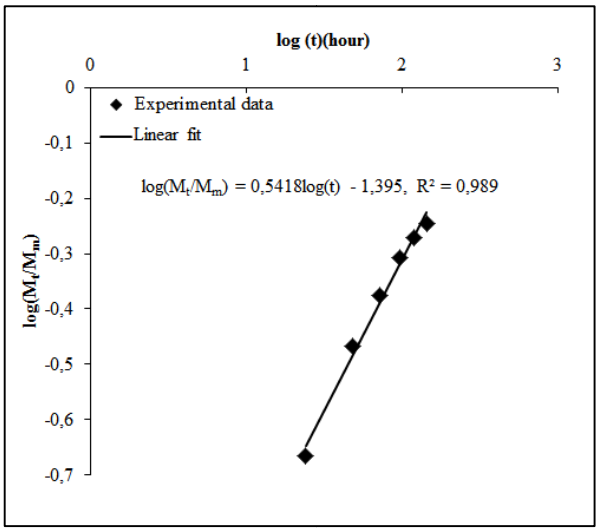


Fig. 5. Diffusion curve: graphical adjustment log(M_t/M_m) as a function of log(t) of JP composite

TABLE 3. **Values of n and K for jute/polyester composite immersed in water**

Jute/polyester composite material	n	K	R^2
	0.541	0.040	0.988

The n-value of the jute/polyester composite immersed in water is close to the n value = 0.5, which indicates that this type of composite approaches the Fickian diffusion behavior for water absorption. Hence, the rate of diffusion of water is slower than that of the relaxation of the material chains, and the mechanism is controlled by diffusion. Most studies on the water absorption behavior of natural composites follow Fickian behavior [5, 27]. The value of K is approximately 0.04, indicating that the presence of water within the jute/polyester composite causes strong interaction with the material itself. This strong interaction thus increases

the mobility of the polymer segments and creates additional free volume for the penetration of water [28].

Diffusion coefficient D is the most important parameter of Fick's model, which shows the ability of water molecules to penetrate inside composites [29]. Equation (7) is used to derive the diffusion coefficient by plotting the weight gain kinetics M_t/M_m curve as a function of ($t^{1/2}$) of the jute/polyester composite (Fig. 6). Coefficient D is a function of the slope of regression line k of the linear part which properly simulates the kinetics of water absorption. The symbols are the experimental points and the line shows the application of Fick's model. The diffusion coefficient and the slope of linear regression line k, as well as the jute/polyester composite confidence index, are shown in Table 4.

TABLE 4. **Diffusion coefficient for jute/polyester composites**

Jute/polyester composite material	k	M_m [%]	h [mm]	D [mm^2/s]	R^2
	$5.234\ 10^{-2}$	4.391	4.100	$2.510\ 10^{-6}$	0.995

It is quite clear that the diffusion coefficient for a composite immersed in fresh water is important. According to reports [7, 30-33], the diffusion coefficient values for composites reinforced with natural fibers are of the order of 10^{-6} and 10^{-7} (mm^2/s). While water can diffuse either by permeability through the polyester matrix or by percolation along the interface between the resin and the fibers, the associated rapid stress relaxation of the resin leads to disbonding generates a more porous matrix [20].

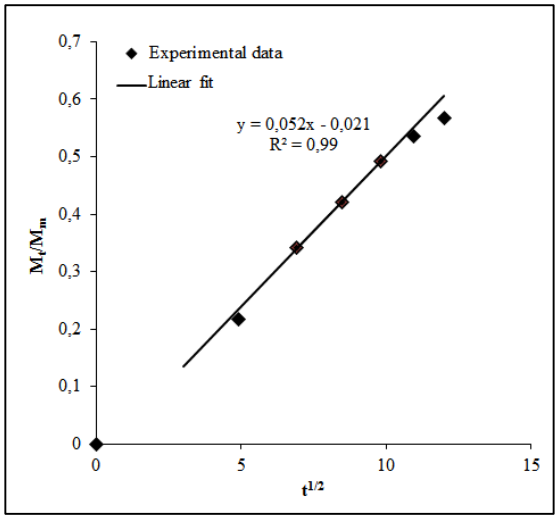


Fig. 6. Weight gain kinetics of jute/polyester composite immersed in water

Charpy impact test behavior

The principle of linear elastic fracture mechanics is used to interpret the Charpy impact test results by applying the compliance method, which makes it possible to deduce the critical energy release rate, or the dynamic toughness of the pre-cracked parts. Table 5 collates the results of the Charpy impact test of all the investigated materials.

TABLE 5. **Results of Charpy impact test of jute/polyester composite**

Immersion time	Number of specimens	U [J]	$BD\phi$ [mm^2]	G_{IC} [KJ/m^2]	U_{c} [J]	R^2
0 days	14	0.42 (0.03)	22.75 (2.94)	11	0.177	0.79
30 days	15	0.30 (0.03)	20.51 (3.47)	7.3	0.150	0.80
90 days	15	0.23 (0.11)	18.71 (2.42)	4,02	0.153	0.62

The immersion of the jute/polyester test pieces results in a weight gain of 4.34% for the two times of water immersion (30 and 90 days). Analysis of the Charpy impact test results based on the compliance method allows total fracture energy U to be plotted as a function of the $BD\phi$ fractured surfaces of the jute/polyester composite immersed in water (Fig. 7).

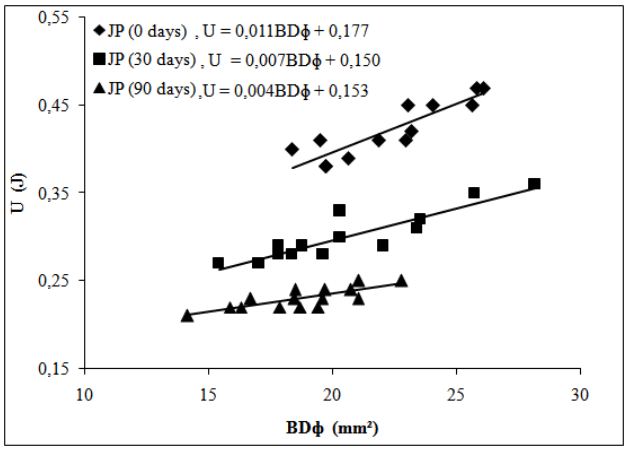


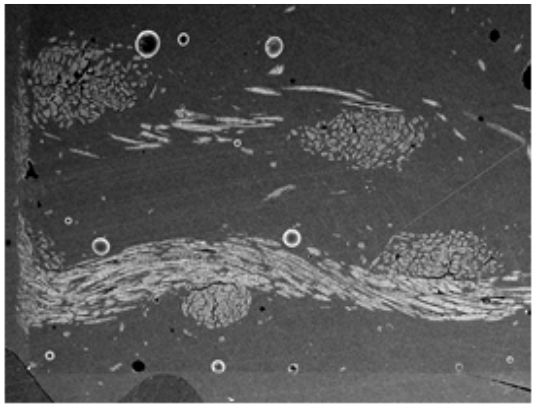
Fig. 7. Total fracture energy U as a function of $BD\phi$ fractured surfaces of JP composite immersed in water

It is clearly visible on the curve in Figure 8 that the weakest slope of the linear regression lines of the JP composite is that of the curve of the non-submerged composite. This is because the impact toughness of the JP composite decreases with the increasing duration of immersion in water. A drop of approximately 34% was recorded for the period of immersion of 30 days, followed by a drop of approximately 63% for the duration of water immersion of 90 days. This difference may be caused by the difference in the percentage of fiber added. The drop in the impact properties after immersion is due to the degradation of swollen fibers and damage to the fiber matrix interface, which lowers the charge transfer.

When immersed in water for a period of 90 days, the average impact energy required to rupture the test piece is almost half the impact energy for the non-submerged test piece that serves as the control test piece. It is cer-

tainly possible that the excessive absorption of water causes the penetration of a quantity of water to the cellulose network of jute fiber through the voids and the spaces between the fibrils and the less bound areas, which leads to a drop in the stiffness of the cellulose structure, and consequently, a decline in the impact strength of the composite [8]. In addition, the high cellulose content in jute yarns (approximately 64%) [34] also contributes to more water penetrating into the interface through the microcracks induced by swelling of the fibers [15, 35] (Fig. 8b). This creates swelling stresses between the fibrils as well as stresses between the fibers and the matrix leading to the damage of the fibers, in addition to the rupture of the interfacial bond [36].

a) Composite not immersed



b) Composite immesed in fresh water for a period of 90 days

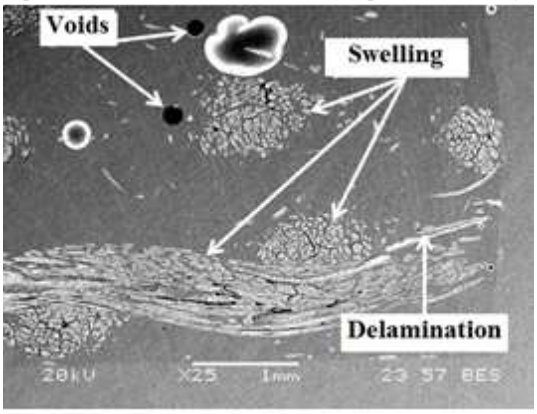


Fig. 8. Scanning electron microscope micrograph of fracture faces of jute/polyester composite

CONCLUSIONS

The jute/polyester composite material was immersed in water for several immersion times to study the effect of water absorption on the Charpy impact behavior. The following conclusions can be drawn:

- The water absorption of the bidirectional jute/ polyester composite increases with an increasing immersion time up to 30 days. After this immersion time, the material reaches the saturation limit until the end of the immersion period, a duration of 270 days.
- The mechanism of water transport in the composite presents Fickian diffusion, which is characterized by a diffusion rate that is slower than that of the relaxation of the material chains.
- The application of the Charpy impact test on the notched jute/polyester specimens causes brittle fracture of the composite material.
- The hydric immersion of the composites causes a significant drop in dynamic toughness, which is mainly due to the degradation of the fiber/matrix bond, as well as the presence of osmotic pressures within the material.
- The significant dispersion of the experimental results of the impact test is mainly due to the presence of manufacturing defects and the test conditions, as well as the method of sample preparation.

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