EFFECT OF CHEMICAL MODIFICATION OF *LUFFA CYLINDRICA* FIBERS ON THE MECHANICAL AND HYGROTHERMAL BEHAVIOURS OF POLYESTER/LUFFA COMPOSITES

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Effects of surface chemical modifications of luffa fibers on mechanical and hygrothermal behaviors of polyester/luffa composites were studied. Unsaturated polyester resin was used as matrix. Untreated, alkali-treated, treated with combined process, and acetylated luffa fibers were used. Scanning electron microscopy and infrared spectroscopy were used to characterize the morphology and chemical structure of the luffa fibers. The mechanical properties of the composites were characterized using the three-point flexural bending test. Water absorption tests were conducted on both the fibers and the composite materials by immersion in distilled water at 25°C. The acetylation treatment improved the mechanical properties. As shown by infrared analysis, the process decreased the hydrophilic behaviour of the luffa fibers, improving their adhesion to the polyester matrix. The chemical modifications at the surface of the luffa fibers also decreased the diffusion coefficient and the maximum amount of water absorbed by these fibers. Moreover, it was observed that for the fibers tested in this study, the diffusion process was "Fickian" at earlier times of immersion, but became complicated towards the end of the immersion. Similar results have been obtained for composite materials at earlier stages of water immersion. The diffusion process was found to be affected by external loads applied on the exposed composite materials. As the load increased, the amount of absorbed water increased at a higher rate of diffusion.

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INTRODUCTION

A combination of properties of some natural fibers, such as low cost, low density, non-toxicity, high specific properties, lack of abrasion during processing of composite materials, and recyclability, has evoked interest in manufacturing using these fibers (Bledzki et al. 1999; Herrara-Franco et al.2004). These composite materials are used in different applications ranging from the automotive, aeronautic, and naval industries (Medina et al. 2008).

Recent literature (Bledzki et al. 1999) has indicated that due to the poor compatibility between these fibers and the polymer matrix, the surface of natural fibers should be treated to improve the adhesion at the interface of the composite materials. Many authors (Herrara-Franco et al.2004; Nabi et al. 1999; Arbelaiz et al. 2005) have

elucidated the contribution of different fiber surface treatments on both the hygrothermal and the mechanical properties of natural fibrous composite materials. For example, and according to Bessadok et al. (2007) and Paul et al. (1997), it has been shown that the treatment of the cellulose-based fibers with acetic anhydride decreases the hydrophilic behavior of these fibers by the substitution of hydroxyl groups with acetyl groups. Chemical modification takes place as the acetic anhydrides substitute the cell wall hydroxyl groups with acetyl groups, making the surface more hydrophobic and thus more compatible with the polymer matrix. Similar results were obtained by Tserki et al. (2005). Khalil et al. (2001) have indicated that the acetylation of oil palm fiber improves the interfacial shear strength (ISS) between the natural fibers and the various matrixes. Other coupling agents have also been used to modify cellulosic fibers. These include bifunctional anhydride, diisocyanate, and alkoxysilane (Ly et al. 2008). These methods increased the surface area of the fibers, inducing an increased contact surface area between the fibers and the matrix. Such an improved interface could explain the enhancement of the mechanical properties of composite materials. The alkali treatment is another treatment that has been widely used to improve the surface quality of natural fibers. It has been found that such treatment improves both the interfacial shear strength and the flexural properties of composite materials (Khanam et al. 2010; Shinoj et al. 2011; Boynard et al. 2003). This was attributed to the removal of the outer surface of the natural fibers, improving its surface quality. John et al. (2008) presented a detailed study on the effect of different chemical treatment modifications on the mechanical properties of natural fibrous composites.

Water absorption can also affect the mechanical proprieties of natural fibrous composite materials. Results published in the open literature indicated that the interfacial bonds between the natural fibers and the relatively hydrophobic polymer matrix could be easily damaged with high water uptake (Beg et al. 2008; Chow et al. 2007). This will in turn affect the mechanical properties of the composite materials. Changing the external morphology of the natural fibers seems to be the key factor that could change the fibers' hydrophilic behaviour and reduce the amount of water that could be absorbed by thefibers (Arbelaiz et al. 2005; Beg et al. 2008; Chow et al. 2007; Adhikarya et al. 2008; Espert et al. 2004).

In the literature the water absorption process in natural fibers was examined in order to better understand the diffusion in fibrous composite materials. According to Nosbi et al (2011) and Saikia (2010) the water absorption process of kenaf, okra, bowsting hemp, and betel nut fibers is non-Fickian, where the moisture uptake behavior is greatly altered by the degradation of the surface of the fibers. It has also been reported that alkali treatment of the fibers increases the moisture uptake (Modibbo et al. 2007) but grafting with poly-(MMA) chains reduces fibers welling (Shinga et al. 2010).

For composite materials, reinforced with natural fibers, the moisture uptake behavior was found to be Fickian. Such a result was confirmed for other natural fibers such as wood (Espert et al. 2004). Moreover, it has been noted that the tensile and flexural strengths of natural fibrous composites decrease after water absorption (Chow et al. 2007; Dhakal et al. 2007; Assarar et al. 2011; Akil et al. 2009; Rashdi et al. 2010; Mulinari et al. 2010).

Luffa cylindrica has a ligneous fibrous netting system in which the fibrous cords are disposed in a multidirectional array, forming a natural mat. Sequeira et al. (2010) reported that these fibers are composed of 65.5% cellulose, 17.5% hemicellulose, and 15.2% lignin. According to Boynord et al. (1999; 2003) the use of luffa fibers in composite materials may be very advantageous. They studied the effect of alkali treatment on the strength of luffa composite materials. The effects of coupling agents on the mechanical and water absorption properties of short luffa fibers/PP composites were studied by Demir et al. (2006). Results showed that silane and MAPP treatment reduce the water absorption and increase the tensile properties. Bal et al. (2004) have studied the water absorption of cell-fibers from luffa. They reported that the retention capacity of luffa fibers for aqueous solutions was improved with alkali treatment. They attributed this improvement to the changing of the fiber surface morphology after alkali treatment.

The aim of this work is to elucidate the effect of some chemical treatments applied on the surface of the luffa fiber on both the mechanical and hygrothermal proprieties of polyester/luffa fiber composites. The *Luffa ecorse* fibers were extracted from the raw luffa using two chemical methods. Untreated, extracted, and modified fibers were analyzed using infrared spectroscopy and scanning electron microscopy (SEM). The flexural proprieties of different composite materials using the luffa fibers were examined. The mechanical properties of the composite materials were determined using the three-point bending test.

MATERIALS AND METHODS

The luffa sponge, collected from Tunisia, was first washed three times in hot water, then with 0.2% of lauryl peroxide to ensure good cleaning (Bal et al. 2004).

Cell-fibers of *Luffa cylindrica* were extracted from fibrous strands using two methods. The first method included using a solution containing 4% sodium hydroxide to remove waxy and gummy substances. For alkali treatment, the temperature and the exposure time were fixed at 100°C and 2 hours. The second method consisted of treating cleaned luffa fibers with a specific solution containing 4% sodium hydroxide and 10% hydrogen peroxide, at 100°C for 2 hours. In the context of this report, this method will be called a "combined process". In both methods, the sodium hydroxide was used as a delignification agent for luffa fibers and the hydrogen peroxide as a bleaching agent. After treatment, the fibers were washed several times using distilled water and then dried at 80 °C for 2 hours.

Samples of the luffa fibers treated with the combined process were, in a second step, introduced in a round bottomed flask with a sufficient amount of acetic anhydride and brought to the desired reaction temperature of 100°C for 3 hours (Bessadok et al. 2007). The fibers were then washed with acetone at ambient temperature until the acetic anhydride was removed.

For convenience, the untreated luffa fibers, the alkali treated fibers, and those treated with the combined process and acetylated are called LB, LN, LC, and LA_c, respectively.

All fibers in this study were observed with a Philips scanning electron microscope (SEM), with an acceleration voltage of 20 kV to analyze the fibers' morphology. To identify the chemical modifications at the surface of these fibers, infrared spectroscopy analysis was conducted using an IR-840 SHIMADZU spectrometer. A mixture of 5 mg of dried fibers and 200 mg of KBr was pressed into a disk for FTIR measurements. 100 scans were collected for each measurement, in the spectral range of 400 to 4000 cm^{-1}. All the IR spectra presented in this work were obtained in an absorbance mode.

Both treated and untreated luffa fibers were used to reinforce an unsaturated polyester matrix. The composites were manufactured manually by placing one layer of these fibers inside a glass mould, pouring the pre-formulated resin, closing the mould, and then pressing. This procedure helps remove air bubbles that can be entrapped in the molded composite material and also enables the manufacturing of composites with uniform thickness. The obtained composite specimen was weighed and the percentage of fibers was estimated after composite fabrication by the formula below:

$$\text{Fiber weight ratio (\%)} = \frac{\text{fiber weight} \times 100}{\text{composite weight}} \tag{1}$$

From the obtained composites, specimens having dimensions of 80x15x4 mm^3 were cut for determination of the mechanical properties. A three-point bending flexure test was used in this study, according to the standard EN ISO 14125. The flexural properties of both the polyester resin and its composites were determined using a universal testing machine, LLOYD, with a test speed of 10 mm/min. The flexural strength, flexural modulus, and surface strain of the tested specimens were evaluated using the following equations, respectively,

$$\sigma_f = \frac{3FL}{2bh^2} \tag{2}$$

$$E_f = \frac{L^3 F}{4bh^3 w} \tag{3}$$

$$\varepsilon = \frac{6wh}{L^3} \tag{4}$$

Where F is the maximum load (N), L is the length (64 mm), h is the thickness (4 mm), b is the width (15 mm), and w is the defection (mm) of this specimen.

The ASTM D570-98 standard was used to characterize the hygrothermal behaviors of both luffa fibers and the fibrous composite materials. For these materials, dried specimens with dimensions of 80x15x4 mm^3 were submerged in distilled water at 25°C. These specimens were then removed from the water at predetermined times, weighed using a high precision balance to determine their instantaneous weight, and then resubmerged in the water. The weight gain was calculated according to,

$$H\% = \frac{M_t - M_0}{M_0} \times 100 \tag{5}$$

where M_t is the instantaneous weight of the immersed specimen and M_0 is its initial weight. $H\%$ can be predicted using the following equation (Boynard et al. 1999),

$$H\% = H_s \cdot \frac{4}{h}\left(\frac{D \cdot t}{\pi}\right)^{1/2} \tag{6}$$

where H_s is the percentage of weight gain at saturation, h is the thickness of the specimen, and D is the diffusion coefficient determined by:

$$D = \frac{\pi h^2 (H_2 - H_1)^2}{16 \cdot H_s^2 (t_2^{0.5} - t_1^{0.5})^2} \tag{7}$$

RESULTS AND DISCUSSIONS

Luffa Fibers Characterization

Analyses of the multiple varieties of luffa fibers were carried out using a scanning electron microscope. Figure 1a shows the presence of a thin layer of lignin covering the cellulosic fibrils. These fibrils are glued together by pectin substances and hemicelluloses. These results, when compared to those shown in Fig. 1b, indicate that the alkali treatment removes the waxy and gummy substances present at the surface of the untreated luffa fibers. Similar results have been obtained by the authors in an earlier work (Ghali et al. 2009). It has been noted that some chemical treatments of luffa fibers enhance the crystalline structure as they remove the amorphous compounds such as lignin and hemicelluloses. Figure 1c shows the presence of scratches at the surface of fibers treated by the combined process, which can be explained by the elimination of the gummy layers covering the luffa fibers. Such treatment seems to be the most effective treatment, since the topography of the surface of the fibers treated with this combined process appears to be much more ready to reinforce the polyester matrix during the eventual molding of composite materials. Figure 1d depicts a deposition on the fiber surface indicating the acetylating reaction. The surface shows the existence of many small attacked regions.

Figure 2 shows infrared spectra obtained at the surface of the different fibers shown in Fig. 1. The examination of these spectra reveals the presence of some characteristic peaks. The peaks located between 1050 cm^{-1} and 1100 cm^{-1} characterize the vibration of the $C-O-C$ groups. The $C-O-C$ symmetric stretch at 1100 cm^{-1} and the $C-OH$ stretching vibration of the cellulose backbone at around 1053 to 1060 cm^{-1} arose from the polysaccharide components. The peak at 1429 cm^{-1} is attributed to the CH_2 bending of cellulose. Peaks at 1370 cm^{-1} and 1330 cm^{-1} characterize the alcoholic groups of cellulose. The region located between 3500 and 2500 cm^{-1} is related to the OH and CH_2 groups, and the peak located at 1600 cm^{-1} is attributed to the stretching vibration of free hydroxyl groups (Tanobe et al. 2005).

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Fig. 1. SEM photos of untreated and treated luffa fibers: a) untreated, b) alkali treated, c) treated with combined process, d) acetylated

Examination of the spectra shown in Fig. 2a confirms that the luffa fibers preserved their cellulosic structure after treatment with the combined process. However, the peaks for the untreated (LB) and treated (LC) luffa fibers had different intensities. The decrease of the peak intensities after combined treatment indicates the removal of hemicellulose and lignin from the untreated fibers. Moreover, and as seen in Fig. 2b, the treatment of *Luffa ecorse* fibers with acetic anhydride led to the appearance of additional absorbance peaks between 1730 and 1740 cm^{-1} and at 1243 cm^{-1}. According to Bessadok et al. (2007) and Tserki et al. (2005) these peaks can be explained by the esterification of the hydroxyl groups, which result in an increased stretching vibration of the carboxyl ($>C=O$) groups present in ester bonds. The appearance of the peak at 1740 cm^{-1} for the acetylated fibers indicates that the acetyl groups are involved in an ester bond with the hydroxyl groups of the fibers. The esterification is also confirmed by the presence the peak at 1243 cm^{-1}. This result confirms the morphological observation shown in Fig.1 d. The infrared spectrum of LC fibers shows a more intense peak at 1600 cm^{-1}. Similar results were obtained by Tserki et al. (2005). Such a peak was attributed to the presence of a free hydroxyl bond at the surface of these fibers.
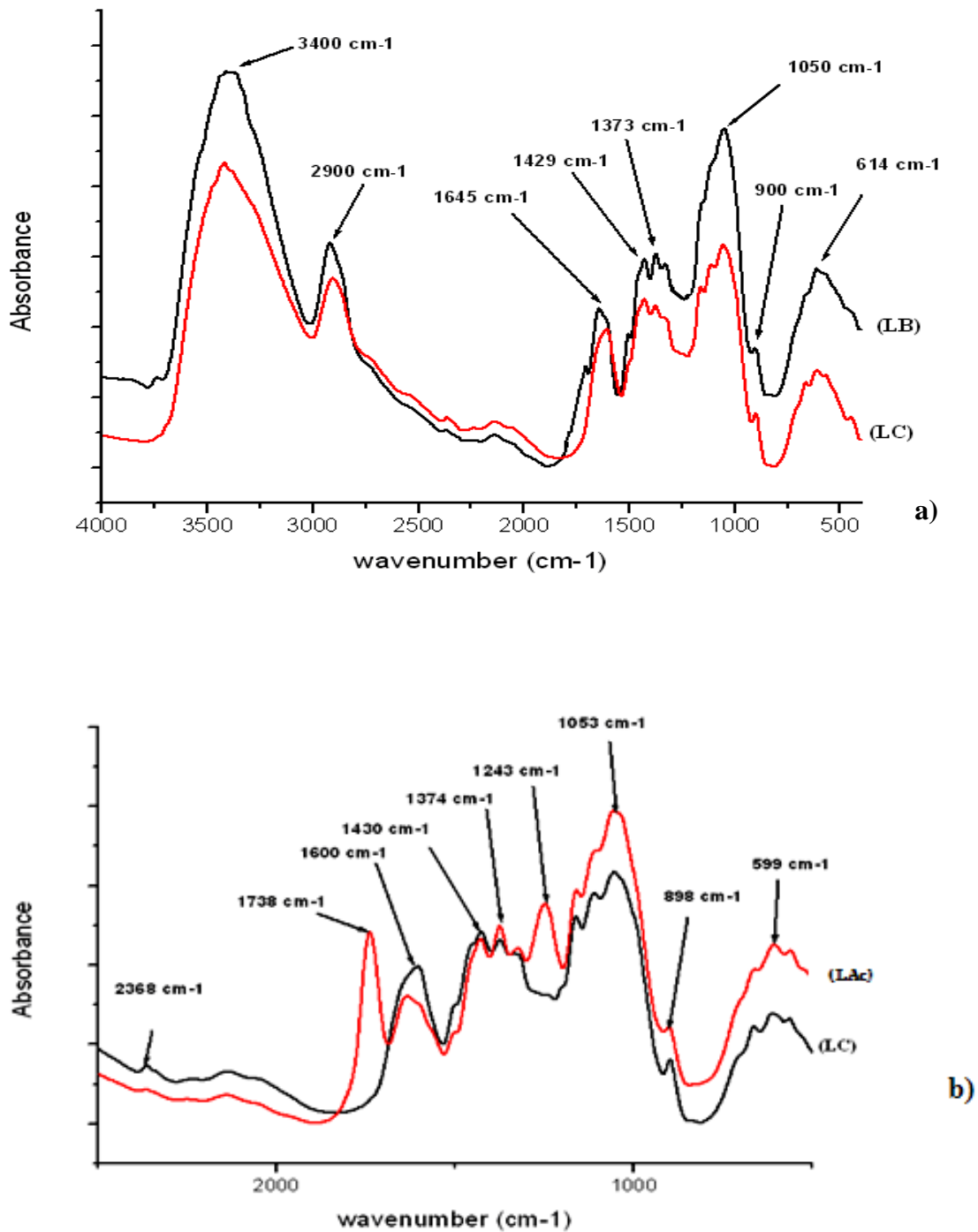
Fig. 2. Infra-red spectra of luffa fibers: a) LB and LC; b) LC and LA$_c$

Flexural Proprieties of Polyester / Luffa Fiber Composites

Three-point bending flexure tests were carried out on both the pure polyester specimens and the polyester composites reinforced with untreated and treated luffa fibers. Table 1 summarizes the obtained results.

Table 1. Flexural Properties of Polyester/Luffa Fibers Composite *

Specimen	Reinforcement	Fibers weight ratio (%)	$\sigma_f\,(MPa)$	$\varepsilon(\%)$	$E_f\,(GPa)$
Pure polyester (Pe)	--------	---	34.95(1.74)	1.51 (0.2)	2.30 (0.05)
Polyester/ Luffa untreated (PLB)	Untreated Luffa	5.3	39.35 (3.59)	1.48 (0.23)	2.67 (0.18)
Polyester/ Luffa NaOH (PLN)	Luffa fibers treated with alkali process	5,5	40.25(1.32)	1.56 (0.14)	2.69 (0.2)
Polyester/Luffa combined (PLC)	Luffa treated with combined process	6.4	37.13 (3.22)	1.42 (0.17)	2.60 (0.08)
Polyester/ Luffa acetylated (PLA$_c$)	Luffa acetylated	5.3	41.96 (3.35)	2.45 (0.27)	1.70 (0.05)

*The standard deviation for each value is indicated in parentheses

As shown in Table 1, the fibrous reinforcement improved the mechanical behavior of the composite material. The PLA$_c$ composite, containing a weight fiber fraction of 5.3%, had a greater flexural strength than the pure polyester; however, results alsoindicated that the combined treatment of luffa fibers seemed to decrease the mechanical properties of the composite material. This is likely due to the degradation of the fibers during such chemical treatment, which could greatly affect the quality of adhesion between the treated fiber and the matrix. According to the literature, the poor quality adhesion between natural fibers and the polyester matrix could be due to the difference in hydrophilic behaviors (Beg et al. 2008; Torres et al. 2005). The acetylating treatment of the luffa fibers was found, however, to improve slightly the flexural properties for the PLA$_c$ composite. This observation confirms those described above and clearly demonstrates that the reduction of the hydrophilic behavior of the luffa fibers improves the quality of the adhesion between the fibers and the matrix, which in turn should improve the mechanical properties of the composite material.

Hygrothermal Ageing of Luffa Fiber and Polyester / Luffa fiber Composites

Figure 3 shows the effect of the square root of the immersion time, $\sqrt{t}$, on the weight gain, $H\%$, for all luffa fibers used in the study. Also shown in Fig. 3 are the theoretical results obtained from Eqn. (6). For both treated and untreated fibers, the weight gains increased initially monotonically with $\sqrt{t}$ before reaching a maximum. Figure 3 also shows that the weight gain decreased with an increase in the immersion time. According to the literature (Saikia 2010), the initial and second absorption phases are governed respectively by the wetting of fibers and the capillary sorption process where the water diffuses into the fibers until reaching a maximum weight gain. When the immersion time becomes significant, a decrease in the weight gain is due to the hydrophilic property of cellulosic fibers as more water molecules penetrate into the micro cracks present in these fibers, making the fibers swell and eventually leading to failure (Nosbi et al. 2011).
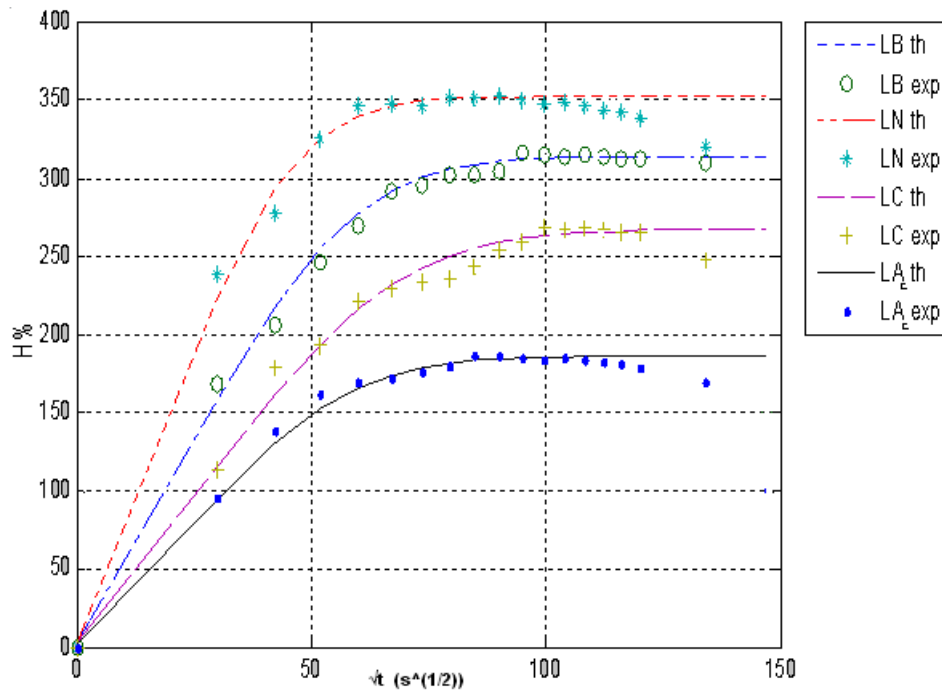
Fig. 3. Weight gain, $H\%$, against $\sqrt{t}$ for various luffa fibers

Figure 3 also shows that the alkali treatment improved the ability of the luffa fibers to absorb water. This improvement is likely due to the elimination of the gummy and waxy substances from the raw fibers. The improvement could also be explained by both the change in the quality of the surface of the luffa fibers after alkali treatment (Fig.1b) and the presence of several hydroxyl groups on these surfaces (Bal et al. 2004). Similar results were obtained for fibers treated with the combined process. The comparison between these experimental results and those predicted using Eqn. (6) clearly demonstrates that neither untreated nor treated fibers have Fickian water diffusion behaviors. As shown in Fig. 3, the acetylation treatment of luffa fibers considerably decreased the fibers' water absorption.

The water absorption behaviors of the different composites in this study are summarized in Fig. 4a. These materials exhibited Fickian water absorption behaviors, confirming theoretical predictions. Similar results were reported in the literature for other natural fiber-reinforced composite materials (Arbelaiz et al. 2005; Joseph et al. 2002). By comparing results shown in Figs. 3 and 4, it appears that the different chemical treatments used in this study similarly affect the hygrothermal behaviors of both the luffa fibers and their composite materials; however, the decrease in the water uptake observed during the immersion of these fibers in water was not observed in the case of the composites. The highest saturation weight gain was obtained by the PLN composite ($H_s = 3.35\%$), while the PLA$_c$ composite experienced the lowest saturation weight gain ($H_s = 2.25\%$). These observations confirm the FTIR results presented above and clearly indicate that the acetylation process decreased the water diffusion in composite materials compared to other chemical treatments. Because of this, the acetylation process appears to be the most efficient treatment for luffa fibers.

As shown in Table 2, the diffusivity decreased from 6.69×10^{-6} m^{2}s^{-1} down to $5.62 10^{-6}$ m^{2}s^{-1} after acetylating treatment of the luffa fibers. In the other hand, the highest diffusivity was obtained for the PLN composite ($7.41\ 10^{-6}$ m^{2}s^{-1}).

Table 2. Diffusivities Values of Polyester/ Luffa Composites

	D (10^{-6} m²s^{-1}) σ=0 MPa	D (10^{-4} m²s^{-1}) σ=8 MPa	D (10^{-4} m²s^{-1}) σ=16 MPa
Pur polyester (Pe)	3.13	------	-----
Polyester/ Luffa untreated (PLB)	7.045	------	-----
Polyester/Luffa NaOH treated (PLN)	7.41	3.75	5.57
Polyester/Luffa combined (PLC)	6.69	------	------
Polyester/ Luffa acetylated (PLA$_c$)	5.62	2.96	3.35

The hygrothermal behaviors of polyester/luffa fiber composites under various flexural loadings of 0 MPa, 2.4 MPa, 8 MPa, and 16 MPa are shown in Figs. 4a, 4b, 4c, and 4d, respectively. These figures show that the application of a low level of flexural stress did not change the hygrothermal behavior of the polyester/luffa fiber composites.

The saturation weight gain and the diffusion coefficient of all composite materials used in this study did not change under such ageing conditions. The hygrothermal behavior under a high level of applied stress is summarized in Figs. 4c and 4d and in Table 2.

As the applied load increased from 8 MPa to 16 MPa, the saturation weight gain increased from 4.1% to 5.2% for the PLN composite and from 2.5% to 2.92% for the PLA$_c$ composite. This increase is probably due to the increase in the number of microscopic cracks inside the loaded specimen. These cracks will in turn enable water molecules to easily diffuse into the loaded composites and both the saturation weight gain and the diffusion coefficient should increase (Table 2). With an increase of the applied stress from 0 MPa to 16 MPa, the diffusion coefficient increased dramatically, rising from 5.62×10^{-6} to 3.35×10^{-4} m²s^{-1}.

Results shown in Fig. 4 also indicate that the hygrothermal behavior of the pure polyester was not greatly affected by the change in the applied load. This result is rather surprising and could suggest that the presence of micro cracks in the stressed composite materials takes place mainly at the interface area, between the matrix and the fibers, and not on the matrix of these materials. As a result, composite materials should absorb higher amounts of water, as shown in Fig. 4. The validation of such results will be considered in a future study.
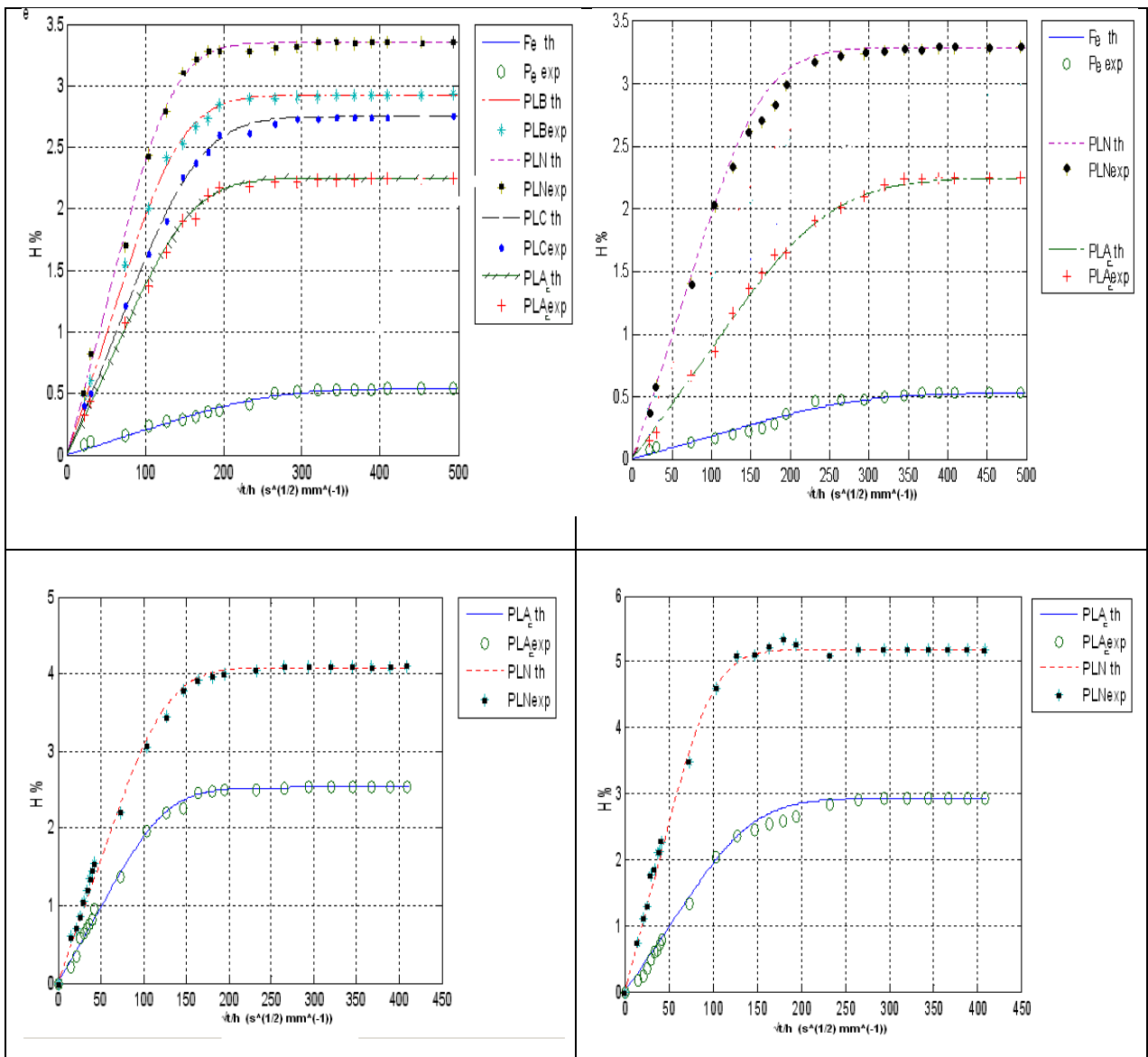
Fig. 4. Kinetic absorption curves of polyester/luffa fiber composites under a flexural loading of a) 0 MPa, b) 2.4 MPa, c) 8 MPa,and d) 16 MPa

CONCLUSIONS

Chemical modifications of *Luffa cylindrical* fibers were found to affect both the mechanical and hygrothermal behaviours of polyester/luffa fiber composites:

1. Acetylation treatment improved the mechanical properties of the composite materials, since such a treatment, as revealed by IR analysis, decreased the hydrophilic behavior of the luffa fibers, improving the fiber adhesion to the polyester matrix.

2. The alkali treatment removed the protecting layer of the cell-fibers, thus increasing the water absorption of the luffa fibers. In addition, the maximum absorption capacity of luffa fibers was observed when the fibers were treated with alkali.
3. The acetylation treatment decreased the moisture absorption of the luffa fibers. This was attributed to the substitution of hydroxyl groups by hydrophobic acetyl groups.
4. The water absorption behavior of luffa fibers follows Fick's law early in the immersion. A degradation process was observed later in the immersion. Such a degradation is characterized by a weight loss in the water absorption curves, which can be related to the hydrolysis phenomenon of waxy and gummy substances present in luffa fibers.
5. The saturation weight gain and the diffusion coefficient of composites are proportional to those obtained for the used fibers. The acetylation treatment was found to decrease the hygrothermal properties of the composite materials.
6. External loading and flexural loading accelerated the water diffusion process in composite materials since the loading caused microcracks that appeared in the composite materials.

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