

# Ageing Studies of Pineapple Leaf Fiber–Reinforced Polyester Composites

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**ABSTRACT:** This experimental study evaluated the water absorption characteristics of pineapple leaf fiber (PALF)–polyester composites of different fiber content. The degree of water absorption was found to increase with fiber loading. The mechanism of diffusion was analyzed and the effect of fiber loading on the sorption kinetics was studied. The diffusion coefficient was calculated and found to increase with fiber content. Studies were also made to correlate water absorption with the cross-sectional areas of the specimens.

The effects of ageing on the tensile properties and dimensional stability of PALF polyester composites were studied under two different ageing conditions. Ageing studies showed a decrease in tensile strength of the composites. The composite specimens subjected to thermal ageing showed only a slight deterioration in strength. © 2004 Wiley Periodicals, Inc. *J Appl Polym Sci* 94: 503–510, 2004

**Key words:** diffusion; ageing; polyesters; fibers; composites

## INTRODUCTION

Composite materials involving fiber reinforcement are replacing traditional materials at a fast rate. Because natural fibers such as sisal, jute, banana, and pineapple are renewable and relatively less expensive than their synthetic counterparts, these promising fibers are used as a substitute for synthetic fibers in polymer composites. Natural fibers possess moderately high specific strength and stiffness and can be used to make useful structural composites.<sup>1–8</sup>

Synthetic fiber–reinforced composites impart good long-term behavior to various aggressive environments and an enhancement in strength and stiffness. However, it is found that natural fiber–reinforced polyester materials are more or less sensitive to humidity through absorption of water, leading to physical degradation such as plasticization of the matrix with water and the differential swelling between the fibers and the resin. The uptake of water can also lead to chemical degradation such as hydrolysis of the matrix. Consequently, the matrix structure and fiber matrix interface are affected, resulting in changes of bulk properties such as dimensional stability and both mechanical and electrical properties.

Pineapple leaf fibers (PALF) are easily available and they possess excellent mechanical properties.<sup>9</sup> These

fibers show high ultimate tensile strength and initial modulus because they have high cellulose content and comparatively low microfibrillar angle. Also its composites possess superior mechanical properties.<sup>10</sup> Because they are lignocellulosic, PALF fibers are hydrophilic in nature. It is well established that natural fibers that are hydrophilic in nature are susceptible to moisture absorption.<sup>11</sup> Water transport is strongly affected by the nature of fiber–matrix interface. If the interface is strong, it is difficult for the water molecules to diffuse into the composite system. Upon various treatments of the fiber and matrix resin, one can increase the interfacial interaction, thereby making the composite more resistant to water transport. To achieve the full potential of polymer composites, they must have good environmental conditions. Environmental conditions such as exposure to humid environment or change in temperature and exposure time cause dimensional instability and loss of mechanical properties. Absorbed moisture has many detrimental effects on material performance.<sup>12,13</sup> Sapiuha et al.<sup>14</sup> studied the kinetics and equilibrium of water sorption in polyethylene/cellulose composites. Several other studies have been reported concerning the effect of different ageing conditions on the physical and mechanical properties of natural fiber–reinforced polymer composites.<sup>15–17</sup>

PALF is obtained from the leaf of the plant *Ananas cosomos* belonging to *Bromeliaceae* family. PALF is used as potential fillers for thermosets, thermoplastics, and elastomers. George et al.<sup>18–20</sup> reported on the mechanical properties, rheological behavior, and viscoelastic

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**TABLE I**  
Physical and Mechanical Properties of Pineapple Leaf Fiber and Polyester Resin

Property	PALF	Polyester
Density, g cm <sup>-3</sup>	1.526	1.159
Tensile strength, MPa	170	22.9
Elongation at break, %	3	1.6
Young's modulus, MPa	6210	582

properties of PALF-reinforced polyethylene composites. Uma Devi et al.<sup>21</sup> reported the mechanical properties of PALF-polyester composites. The present study was aimed at evaluating the water absorption, ageing characteristics, and the effect of different ageing conditions on the mechanical properties of PALF-polyester composites.

## EXPERIMENTAL

### Materials

Pineapple leaf fiber (PALF) was obtained from South India Textile Research Association (Coimbatore). Washed and dried fibers were used for the study. General-purpose polyester resin (HSR, 8131, supplied by Bakelite Hylam Ltd., Hyderabad, India) was used for the study. The properties of PALF and polyester resin are given in Table I. Composites made from PALF and polyester resin of 30 wt % were used for this study.<sup>21</sup> The properties are detailed in Table II.

### Preparation of PALF-polyester composites

Randomly oriented pineapple fiber-polyester composites of 30 mm fiber length, with varying fiber loadings, were prepared by hand layup method. Composite sheets (size 150 × 150 × 3 mm<sup>3</sup>) were prepared using a closed mold. Polyester resin was mixed with 1 wt % cobalt naphthanate (accelerator) and 1 wt % methyl ethyl ketone peroxide (catalyst), degassed, and then poured onto the mat placed in the mold. When the mat was completely wet by the resin, the mold was closed and kept under pressure for 24 h. Samples were postcured and test specimens of the required size were cut from the sheets and used for testing.

### Ageing studies

Water absorption studies of the composites at room temperature were carried out according to ASTM D 570-81. The test specimens were 75 mm long, 25 mm wide, and 4 and 3 mm thick. The specimens were dried in an air oven for 24 h at 80°C, cooled in a desiccator, and immediately weighed. The conditioned specimens were placed in a container of distilled water maintained at room temperature and the samples were completely immersed. At the end of 2, 24, and 96 h, the specimens were removed from water one at a time, the surfaces were wiped off with a dry cloth, and samples were weighed to the nearest 0.0001 g. Weight measurements were repeated at the end of the first week and every 2 weeks thereafter until the increase in weight per 2-week period, by three consecutive measurements, remained more or less constant. The water absorbed by the sample was then determined.

### Immersion in boiling water

Test samples were immersed in boiling water for 7 h at atmospheric pressure. These samples were then dried between two sheets of filter paper and the weight and dimension of each specimen were measured. The samples were then subjected to tensile measurements.

### Thermal ageing

Test samples were heated at 100°C in an air oven for 48 h. After being conditioned at room temperature, the tensile properties of these samples were determined by an Instron (Canton, OH) UTM model 1121.

### Mechanical testing

Tensile testings were carried out using an Instron Universal testing machine model 1121, according to ASTM D 638, at a crosshead speed of 5 mm min<sup>-1</sup> and a gauge length of 60 mm. The specimens were clamped with screws.

## THEORY

The percentage increase in weight of the specimens after immersion in water for various time intervals was calculated to the nearest 0.01% as

**TABLE II**  
Properties of PALF-Polyester Composites of Different Fiber Loading

Fiber content (wt %)	Density (g cm <sup>-3</sup> )	Tensile strength (MPa)	Elongation at break (%)	Young's modulus (MPa)
10	1.03	17.1	1.3	1767
30	1.09	52.9	3.6	2290
40	1.09	63.3	5.0	2519

Increase in wt % =

$$\frac{\text{Net wt conditioned wt} \times 100}{\text{Conditioned wt}} \quad (1)$$

The mol % uptake  $Q_t$  is given by

$Q_t$  (mol %) =

$$\frac{\text{Weight of water taken up at time } t}{\text{Molecular wt of the solvent} \times \text{initial wt of the sample}} \times 100 \quad (2)$$

To study the mechanism of water sorption, the kinetic parameters  $n$  and  $k$  were analyzed from the following relationship:

$$\log Q_t/Q_\infty = \log k + n \log t \quad (3)$$

where  $Q_\infty$  is the mol % uptake at equilibrium,  $t$  is the time, and  $k$  is a constant characteristic of the polymer, which indicates interaction between polymer and water.

The values of diffusion coefficients were calculated from the relationship

$$D = \pi(h\theta/4Q_\infty)^2 \quad (4)$$

where  $\theta$  is the slope of the initial linear portion of the sorption curves,  $h$  is the initial thickness of the sample, and  $Q_\infty$  is the mol % uptake at equilibrium.

The sorption coefficient ( $S$ ) or solubility can be calculated from the equilibrium swelling using the relation

$$S = \frac{M_\infty}{M_p} \quad (5)$$

where  $M_\infty$  is the mass of the solvent taken up at equilibrium and  $M_p$  is the initial mass of the polymer sample. The permeability coefficient, which could be considered as the total effect of sorption and diffusion, can be estimated using the following equation:

$$P = DS \quad (6)$$

where  $D$  is the diffusion coefficient and  $S$  is the solubility.

The Jacobs–Jones model is used to describe the diffusion processes in two-phase materials consisting one dense and one less-dense phase. According to the model the diffusion proceeds by parallel Fickian processes in both phases.<sup>22,23</sup> The variation of normalized water content  $Q(t)$  with time can be described by the morphology-dependent equation:

$$Q(t) = V_d \left\{ 1 - \exp \left[ -7.3 \left( \frac{D_d t}{b^2} \right)^{0.75} \right] \right\} + (1 - V_d) \left\{ 1 - \exp \left[ -7.3 \left( \frac{D_l t}{b^2} \right)^{0.75} \right] \right\} \quad (7)$$

where  $Q(t) = Q_t/Q_\infty$ ;  $D_d$  and  $D_l$  are the diffusion coefficients of the dense and the less-dense phases, respectively;  $V_d$  is the volume fraction of the dense phase; and  $b$  is the thickness of the sample. In the case of PALF–polyester composites, the material can be considered as a two-phase system<sup>24</sup> with the nonpolar polyester matrix as one phase (phase 1) and the fiber as second phase (phase 2). In both phases, the diffusion takes place through the formation of hydrogen bonds between the hydroxyl groups of fiber and water molecules. In this case the Jacobs–Jones model can be modified to eq. (8), as follows:

$$Q(t) = Q_{(1)} \left\{ 1 - \exp \left[ -7.3 \left( \frac{D_1 t}{b^2} \right)^{0.75} \right] \right\} + Q_{(2)} \left\{ 1 - \exp \left[ -7.3 \left( \frac{D_2 t}{b^2} \right)^{0.75} \right] \right\} \quad (8)$$

where  $D_1$  and  $D_2$  and  $Q_{(1)}$  and  $Q_{(2)}$  are the diffusion coefficients and the normalized water content of absorbed water at saturation in the two phases, respectively.

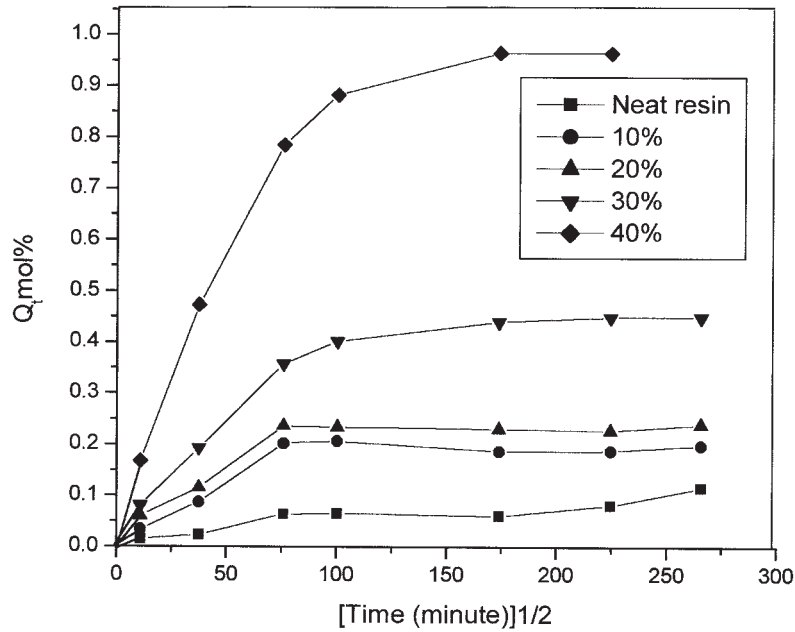
## RESULTS AND DISCUSSION

### Effect of water absorption

Water absorption in a fibrous composite depends on such factors as fiber loading, area of exposed surfaces, and diffusivity. The results of diffusion experiments were expressed as mol % uptake of water by 100 g of the polymer. The mol % uptake ( $Q_t$  %) was plotted against the square root of immersion time (in minutes) to obtain the sorption curves in Figure 1. At equilibrium,  $Q_t$  was taken as  $Q_\infty$ .

Water absorbed by the polyester sample was found to be very small in the initial stage. However, if crosslinked unsaturated polyester specimens are subjected to a lengthy immersion, the water absorption was found to be about 2% at equilibrium.

For PALF–polyester composites, the degree of water absorption increased with fiber loading. From the sorption curves it is seen that the uptake increased linearly at first, after which it leveled off, thus indicating the attainment of equilibrium. The percentage of water absorbed by 40% composites was 123% higher than that of composites containing 30% fiber at saturation point. For hydrophilic fibers such as PALF, an increase in water-sorption properties of the composite may be expected with the increasing concentration of

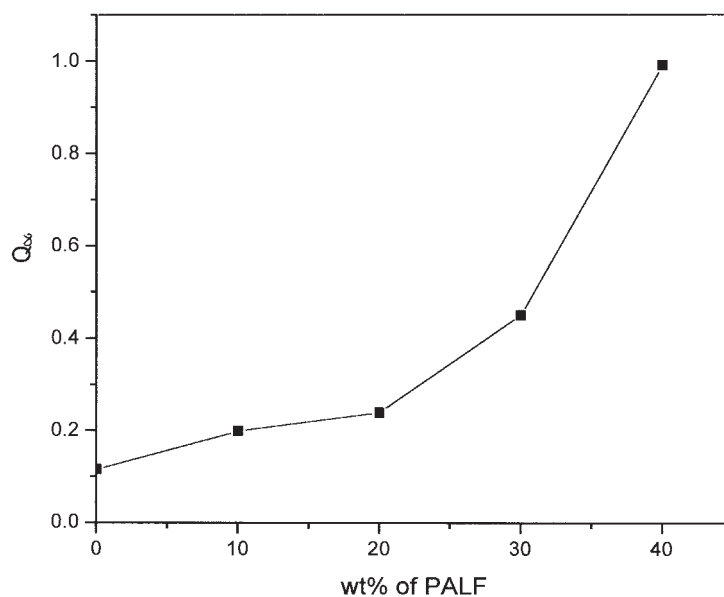


**Figure 1** Sorption curves showing the mol % uptake of PALF/polyester composites having different fiber loadings.

fiber. These fibers swell with absorption of water, which results in the development of shear stress along the matrix–fiber interface, leading to delamination and debonding. Also the absorption of water is attributed to the capillary phenomenon, although the presence of hydroxyl groups enhances the absorption of water by forming hydrogen bonding.

Figure 2 shows the variation of  $Q_{\infty}$  with fiber loading. From the figure, the rate of absorption is found to depend on cellulose content. This is because, with an increase in fiber loading, the rate of water absorption

increases as a result of the increase in total cellulose content. The sample containing lower fiber concentration (i.e., with 10 and 20% weight fraction of fiber) reached equilibrium more rapidly (i.e., after 1 week), whereas the samples with 30 and 40% fiber content reached the saturation point after 3 weeks. Furthermore, a very rapid initial sorption of water was observed in the case of composites with high fiber content (40%), which could be attributable to the uptake of water by the exposed cellulosic fibers located at the surface of the composite. The transport of moisture



**Figure 2** Variation of  $Q_{\infty}$  with fiber loading.

TABLE III  
Values of  $n$ ,  $k$ ,  $Q_{\infty}$ , Diffusion Coefficient ( $D$ ), Sorption Coefficient ( $S$ ), and Permeability Coefficient ( $P$ ), for PALF-Polyester Composites with Different Fiber Loading

Fiber loading (wt %)	$n$	$k$	$Q_{\infty}$	$D$ (cm <sup>2</sup> s <sup>-1</sup> )	$S$ (g/g)	$P \times 10^{-10}$ (cm <sup>2</sup> s <sup>-1</sup> )
0	0.320	-1.570	0.1156	$1.0 \times 10^{-9} \pm 1.3 \times 10^{-10}$	0.020	0.248
10	0.256	-1.180	0.1996	$1.3 \times 10^{-9} \pm 2.7 \times 10^{-10}$	0.03	0.42
20	0.243	-1.094	0.2386	$1.6 \times 10^{-9} \pm 4.4 \times 10^{-10}$	0.05	0.84
30	0.274	-1.264	0.4503	$2.3 \times 10^{-9} \pm 0.424 \times 10^{-10}$	0.08	1.79
40	0.268	-1.193	0.9909	$2.0 \times 10^{-9} \pm 1.3 \times 10^{-10}$	0.18	3.64

below the glass-transition temperature ( $T_g$ ) is a three-stage process in which the absorbed moisture first occupies the free volume present. In the second stage, water becomes bound to network sites causing swelling. Finally, water enters the densely crosslinked regions. According to the equilibrium theory, a polymer will absorb solvent until the solvent chemical potential in the polymer and that in the free solution are equal.<sup>25</sup>

#### Kinetics of water sorption

The equilibrium sorption values  $Q_{\infty}$  of the composites and the values of  $n$  and  $k$ , determined by linear regression analysis, are given in Table III. The value of  $n$  provides information about the mechanism of solvent transport under all experimental conditions. If  $n = 0.5$ , the mechanism is said to be Fickian.<sup>26</sup> Here at all fiber loadings,  $n$  is less than 0.5. Thus a deviation from Fickian behavior is observed in all cases.

#### Diffusion, sorption, and permeation

The values of diffusion coefficients obtained are also given in Table III. The  $D$  values increased with increase in fiber loading up to 30 wt % and thereafter decreased. The increase is attributed to the inherent hydrophilic nature of fibers. Thus the ability of water molecules to diffuse through the polymer increases.

Sorption describes the initial penetration and dispersal of penetrant molecules into the polymer matrix. Solubility is a thermodynamic parameter that depends on the strength of the interaction in the polymer/penetrant mixture. The values of  $S$  and  $P$  are given in Table III. The sorption coefficient values increased with increasing fiber loading and is maximum for 40 wt % composites. The permeation coefficient also increased for composites with 10 to 40 wt % loading. Compared to  $D$  and  $P$ , the value of  $S$  is higher, which implies an increased tendency of the penetrant to dissolve into the polymer.

#### Effect of water sorption on dimensional stability

Studies have been made to correlate water absorption with the cross-sectional area of the specimens. The

percentage increase in the cross-sectional area of the specimens after immersion in distilled water for 7 weeks is shown as a function of fiber loading in Figure 3.

The polyester samples showed no measurable area change, even after immersion in water for 7 weeks. The cross-sectional area of PALF-polyester composites increased after immersion in water for 7 weeks, reaching a maximum for 30 wt %, after which it leveled off.

#### Modeling of water sorption: Jacobs-Jones model

The experimental results of water diffusion in PALF-polyester composites are fitted by eq. (8) by a nonlinear least-square fit method and Figure 4 shows a typical experimental and fitted diffusion curve for 30% composite at 28°C. The fitted values of  $Q_{(1)}$ ,  $Q_{(2)}$ ,  $D_1$ , and  $D_2$  are listed in Table IV. The theoretical model shows good agreement with the experimental values. This confirms the existence of two different phases and mode of diffusion in PALF-polyester composites.

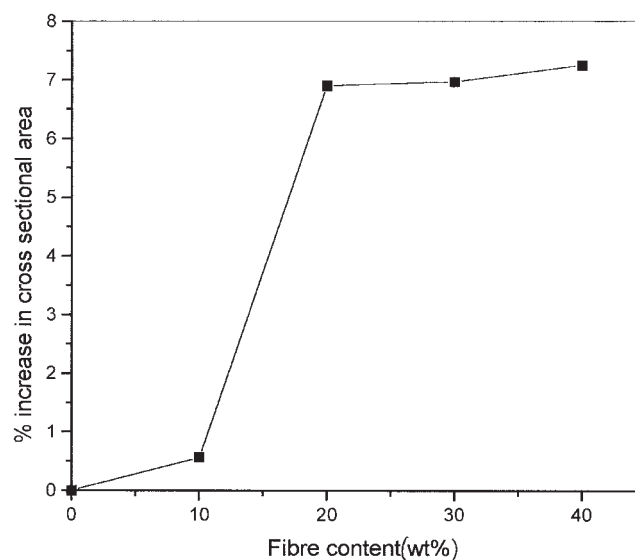
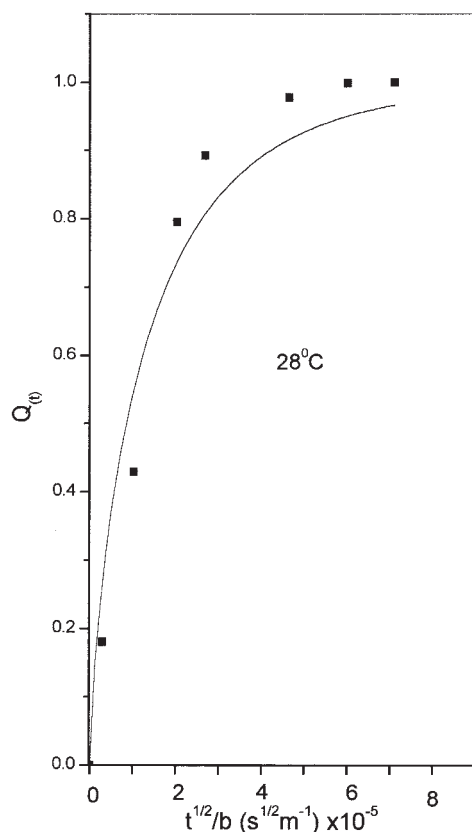


Figure 3 Effect of fiber content on the increase in area of PALF/polyester composites after immersion in water for 7 days.



**Figure 4** A typical experimental and fitted diffusion curve for 30% composites at 28°C.

The slight deviation from the experimental curve results from the deviation of diffusion processes from Fickian behavior. This was very clear from the  $n$  values.

### Immersion in boiling water

Table V shows that the tensile strength of pineapple fiber–polyester composites decreases on immersion in boiling water. The samples subjected to water ageing showed a decrease of 34%. The natural fibers absorb water and swelling of the fiber increases with exposure time in boiling water. As a result of the swelling of fibers, which are surrounded by the polymer matrix, cracks may be formed at the matrix. This may also

**TABLE V**  
Tensile Strength of PALF–Polyester Composites After Immersion in Boiling Water for 7 h<sup>a</sup>

Sample	Tensile strength (MPa)	Elongation at break (%)	Modulus (MPa)
Polyester			
Unaged	20.6	2	580
Aged	13.7	1	1600
Composite			
Unaged	52.9	4	2290
Aged	34.5	3	1900

<sup>a</sup> Fiber length: 30 mm; fiber loading: 30 wt %.

contribute to the penetration of more water into the composite during prolonged exposure in boiling water. The modulus of the composite was found to decrease with exposure in boiling water. The natural fibers absorb water because of its hydrophilic nature. Therefore as a consequence of the high water content after exposure in boiling water, the stiffness of the cellulose fiber decreased substantially.<sup>27</sup> At 30 wt % loading, because of the excessive absorption of water, the intramolecular hydrogen bonding in the cellulose fiber might be reduced by the formation of intermolecular hydrogen bonding between cellulose molecules and water molecules, thus leading to deterioration of the modulus.<sup>28</sup> The elongation at break values of neat polyester samples and PALF–polyester composites decreased slightly after exposure in boiling water, although the modulus of the neat polyester increases. The increased temperature leads to an increase in crosslinking, which thereby increases the modulus.

Dimensional stability of the composites was studied by measuring the changes in weight and cross-sectional area of the composites after exposure in boiling water for 7 h.

From Table VI it is clear that the absorption of water in the polyester samples was quite low (0.4%), although in the case of PALF–polyester composites, the change in weight was found to be 6%. An increase in cross-sectional area was also found as a result of exposure in boiling water. The polyester specimens showed no measurable area change after exposure;

**TABLE IV**  
Fitting Values as per Eq. (8) for Composites at 28°C

Fiber content (wt %)	$\log D_1$	$Q_{(1)}$	$\log D_2$	$Q_{(2)}$	$Q_\infty$
0	-12.1344	0.10467	-11.8742	0.89533	0.11559
10	-11.8464	0.01304	-11.3806	0.98696	0.19485
20	-11.2448	0.89905	-11.6818	0.10095	0.23599
30	-11.4730	0.54737	-11.3798	0.45265	0.4503
40	-11.4152	0.44619	-11.4168	0.5536	0.9906

however, in PALF-polyester composites, an increase in cross-sectional area was observed after exposure in boiling water. The increases in weight and cross-sectional area are attributed to absorption of water by the hydrophilic fibers in the composites. Because raw fibers were used in the fabrication of these composites, the water absorption was high, thus leading to deterioration in mechanical properties as well as dimensional stability.

### Thermal ageing

The mechanical properties of the composites after thermal ageing at 100°C are shown in Table VII. In the case of tensile strength values a decrease of 6% was observed upon thermal ageing; however, the modulus is decreased by 51%. Therefore the decrease of tensile strength is lower, whereas in the boiling water ageing, a decrease of 34% in tensile strength is observed. This may be a result of the loss of strength of fiber during prolonged exposure at elevated temperature (100°C) caused by the decomposition of volatile extractables present on the fiber surface.

Although the components of cellulose are supposed to remain stable up to 160°C the degradation reaction may be initiated slowly on prolonged heating at 100°C.<sup>16</sup> The degradation of fiber will develop voids at the interface and this leads to poor fiber-matrix adhesion. The modulus of the composite was also found to be less than that of the unaged specimens, although the elongation at break was not affected by ageing at elevated temperature. The loss in mechanical properties of PALF-polyester composites after water ageing and thermal ageing are comparable with the results of banana fiber polyester composites.<sup>29</sup>

## CONCLUSIONS

Results of this study indicate the environmental effects on the tensile properties of PALF-reinforced polyester composites. Ageing studies showed a decrease in tensile strength of about 6% for samples subjected to thermal ageing, whereas the composite specimens subjected to water ageing showed considerable loss in tensile strength, about 34%. The water uptake of the composites was found to increase with fiber loading

**TABLE VI**  
Variation in Weight and Cross-Sectional Area of the Polyester and PALF-Polyester Composite Specimens<sup>a</sup>

Sample	Increase in wt %	Increase in area %
Polyester	0.4	Nil
PALF-polyester composites	6	1.3

<sup>a</sup> Fiber length: 30 mm; fiber loading: 30 wt %.

**TABLE VII**  
Tensile Properties of PALF-Polyester Composites After Thermal Ageing 100°C, 48 h; Loading 30 wt %

Sample	Tensile strength (MPa)	Elongation at break (%)	Modulus (MPa)
Unaged	52.9	4	2290
Aged	49.8	4	1110

and leveled off at longer times. The kinetic parameters of water sorption of composites of different fiber loadings were calculated. The diffusion coefficient and sorption coefficient were found to increase with increasing fiber content of the composites. Water absorption leads to a slight deterioration in mechanical properties and dimensional stability. Models were used to fit experimental data, which showed good agreement with theoretical values initially but slight variation at equilibrium. The slight deviation from experimental values may be attributed to the deviation of diffusion from Fickian behavior.

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