Mechanical Properties of Pineapple Leaf Fiber-Reinforced Polyester Composites

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ABSTRACT: Pineapple leaf fiber (PALF) which is rich in cellulose, relatively inexpensive, and abundantly available has the potential for polymer reinforcement. The present study investigated the tensile, flexural, and impact behavior of PALF-reinforced polyester composites as a function of fiber loading, fiber length, and fiber surface modification. The tensile strength and Young's modulus of the composites were found to increase with fiber content in accordance with the rule of mixtures. The elongation at break of the composites exhibits an increase by the introduction of fiber. The mechanical properties are optimum at a fiber length of 30 mm. The flexural stiffness and flexural strength of the composites with a 30% fiber weight fraction are 2.76 GPa and 80.2 MPa, respectively. The specific flexural stiffness of the composite is about 2.3 times greater than that of neat polyester resin. The work of fracture (impact strength) of the composite with 30% fiber content was found to be 24 kJ m^{-2}. Significant improvement in the tensile strength was observed for composites with silane A172-treated fibers. Scanning electron microscopic studies were carried out to understand the fiber–matrix adhesion, fiber breakage, and failure topography. The PALF polyester composites possess superior mechanical properties compared to other cellulose-based natural fiber composites. © 1997 John Wiley & Sons, Inc. J Appl Polym Sci **64:** 1739–1748, 1997

INTRODUCTION

As fiber-reinforced structural plastics are taking the central stage in almost every sphere of material science, lignocellulosic natural fibers like the pineapple leaf fiber come as a viable and abundant substitute for the expensive and nonrenewable synthetic fiber. These fibers with high specific strength improve the mechanical properties of the polymer matrix.

In tropical countries, fibrous plants are available in abundance and at least some of them are agricultural crops. Pineapple is among them. Pineapple leaf fibers at present are a waste product of pineapple cultivation. Hence, without any additional cost input, pineapple fiber can be obtained for industrial purposes.

Among various natural fibers, pineapple leaf fibers (PALFs) exhibit excellent mechanical properties. These fibers are multicellular and lignocellulosic. They are extracted from the leaves of the plant *Ananus cosomus* belonging to the Bromeliaceae family by retting. The main chemical constituents of pineapple fiber are cellulose (70–82%), lignin (5–12%), and ash (1.1%).[1] The superior mechanical properties of pineapple fiber are associated with its high cellulose content and comparatively low microfibrillar angle. Among matrix resins, unsaturated polyesters have been commonly used for making thermoset composites, especially with glass fibers.[2]

Natural fibers like jute, coir, sisal, sunhemp, straw, etc.,[3–7] are incorporated into the polyester

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matrix to make composites. Thomas and co-workers[8–10] reported on the use of sisal fiber as a potential reinforcing agent in polyethylene, polystyrene, natural rubber, and various thermosets such as epoxy, phenol–formaldehyde, and polyester. Although composites based on polyester matrix resin are well known, there have been only limited studies on the properties of PALF-reinforced polyester composite. Mukherjee and Satyanarayana[11] reported on the mechanical properties of PALF. Pavithran et al.[12] studied unidirectionally aligned PALF–polyester composites and the toughness of these composites were found to increase with the microfibrillar angle of the fibers. Recently, George et al.[13,14] reported on the processing characteristics, viscoelastic properties, and mechanical behavior of PALF–LDPE composites. The present investigation was aimed at analyzing the mechanical properties of a PALF-reinforced polyester composite. The influence of fiber length, fiber loading, and coupling agents on the tensile, flexural, and impact properties of the composites was analyzed. The properties of the composites were compared with other natural fiber-reinforced polyester composites.

EXPERIMENTAL

Pineapple leaf fiber (PALF) was obtained from South India Textile Research Association, Coimbatore. The fiber was chopped to the desired length, washed with water, dried in air, and used for composite fabrication. Room-temperature cure general-purpose polyester resin (HSR 8131, supplied by Bakelite Hylam Ltd., Hyderabad, India) was used for the study. Vinyltri(2-ethoxy methoxy)silane (Silane A172) and γ-aminopropyltrimethoxysilane (Silane A1100) were supplied by Union Carbide Co., Montreal, Canada. The physical and mechanical properties of the pineapple fiber and polyester are given in Table I.

Table I Physical and Mechanical Properties of Pineapple Fiber and Polyester Resin

Property	PALF	Polyester
Density (g/cm^3)	1.526	1.159
Tensile strength (MPa)	170	22.9
Elongation at break (%)	3	1.6
Young's modulus (MPa)	6210	580
Specific strength (MPa)	110	19.7
Specific modulus (MPa)	4070	502

For NaOH treatment, the chopped fiber was dipped in 2% NaOH solution for 1 h, washed with water, and dried in air. For acetylation, 10 g of pineapple fiber was soaked in glacial acetic acid for 1 h at room temperature, decanted, and then soaked in acetic anhydride (50 mL) containing 2 drops of concentrated sulfuric acid for 5 min. The fiber was filtered and washed with water until it was free from acid and then dried in air for 24 h.

For silane treatment, the fibers were treated with 0.3% solution of silane in a water–ethanol mixture (40 : 60) for 1.5 h, dried in air for 30 min, followed by drying at 50°C for 1 h.

Randomly oriented pineapple fiber polyester composites with varying fiber length and fiber volume fraction were prepared by a hand layup method. Composite sheets of size 150 $\times$ 150 $\times$ 3 mm^3 were prepared using a closed mold. First, the mold was polished and then a mold-releasing agent was applied on the surface. General-purpose polyester resin was mixed with 1 wt % cobalt naphthenate (accelerator) and 1 wt % methyl ethyl ketone peroxide (catalyst). The resin mixture was degassed in a vacuum desiccator and then poured onto the mat placed in the mold. When the mat was completely wet by the resin, the mold was closed, pressed, and cured at room temperature for 24 h. Sheets were prepared with fibers of varying length (5, 10, 20, 30, and 40 mm) and of varying fiber loading. The specimens of required dimensions were cut from the sheets, smoothed by sanding, and used for testing.

Tensile testing of pineapple fibers was carried out in an Instron universal testing machine Model 1121 at a crosshead speed of 1 mm per min. Specimens were prepared by mounting single fibers on a stiff cardboard piece with a 50 mm window. The ends of the fibers were fixed on the cardboard. The diameters of the fiber specimens were measured using an optical stereomicroscope. Since the diameter of the fiber is found to be different in different places, an average of about six readings were taken for diameter determination.

Tensile testing of the composite specimens was carried out using an Instron universal testing machine Model 1121 at a crosshead speed of 5 mm min^{-1} and a gauge length of 60 mm. Rectangular specimens of size 150 $\times$ 10 $\times$ 3 mm^3 were used for testing. Flexural tests were performed according to ASTM D 790. Rectangular strips of size 75 mm long, 15 mm wide, and 3 mm thick were used for these tests. A crosshead speed of 5 mm min^{-1} was used. The flexural strength and flex-

ural modulus were calculated using the following relationships:

$$\text{Flexural strength} = \frac{3PL}{2bd^2} \quad (1)$$

$$\text{Flexural modulus} = \frac{L^3m}{4bd^3} \quad (2)$$

where L is the support span; b, the width of the specimen; d, the thickness; P, the maximum load; and m, the slope of the initial straight line portion of the load-deflection curve.

Charpy-type impact tests on unnotched specimens were performed using a pendulum impact testing machine PSW 0, 4. The test specimen was 50 mm long, and the cross-sectional area, 24 mm^2. For evaluation of tensile, flexural, and impact properties, five specimens were tested and average values are reported.

The surfaces of the fractured specimens under tensile, flexural, and impact tests were examined using a scanning electron microscope (SEM) JEOL Model JSM-35C and a Cambridge 250 MK_3 stereoscan.

RESULTS AND DISCUSSION

Mechanical Properties in Tension

The uniaxial stress–strain diagrams of PALF–polyester composites with varying fiber length and fiber loading are given in Figures 1 and 2. The deformation behavior of the composites under an applied load can be understood from the stress–strain curve (Fig. 1). The stress increases linearly with strain at low elongation. However, at higher elongation, nonlinear behavior is observed. This is true for all lengths of fibers used. It is evident from the figure that for a given strain level stress increases with fiber length up to 30 mm and then decreases, indicating an optimum fiber length.

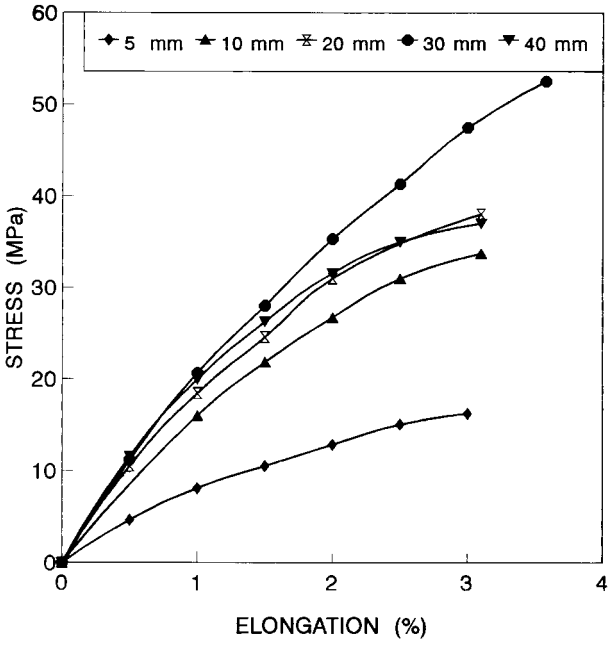


Figure 1 Dependence of stress–strain behavior of PALF–polyester composites on fiber length (fiber loading 30 wt %).

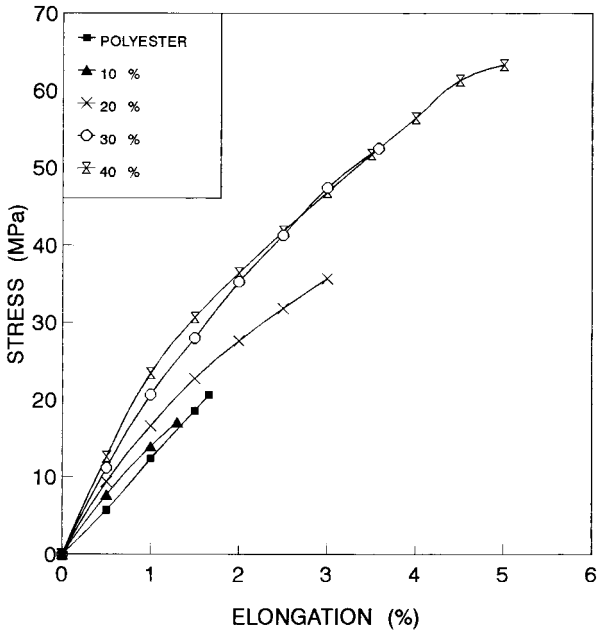


Figure 2 Stress–strain curve of PALF–polyester composites at various fiber loadings (fiber length 30 mm).

From the stress–strain curve of the composites with varying fiber loading (Fig. 2), it is found that the stress–strain curve of pure polyester is similar to that of brittle materials. The behavior is perfectly elastic, i.e., the stress increases linearly with strain. However, addition of fibers makes the matrix more ductile. This is evident from the high elongation-at-break values of the composites.

Effect of Fiber Length

The effect of fiber length on the tensile properties of PALF–polyester composites (containing 30% fiber by weight) can be readily assessed from the data shown in Table II. In general, the polyester composites showed an increasing trend in the mechanical properties up to a fiber length of 30 mm. At a fiber length of 30 mm, the Young's modulus

Table II Effect of Fiber Length on Tensile Properties of PALF-reinforced Polyester Composites (Fiber Content 30 Wt %)

Fiber Length (mm)	Young's Modulus (MPa)	Tensile Strength (MPa)	Elongation at Break (%)
5	815	15.6	3.0
10	1870	35.0	3.0
20	1990	39.2	3.0
30	2290	52.9	3.6
40	1970	38.4	3.0

of the composites is 180% higher than that of a 5 mm length due to effective stress transfer between the fiber and the matrix. When the fiber length is increased to 40 mm, a decrease in moduli is observed.

The change in tensile strength of the composites with fiber length followed the same pattern as that of the modulus. The tensile strength value of the composites with 30 mm-long fibers is about 240% higher than that with a fiber length of 5 mm. When the length of the fiber is increased from 30 to 40 mm, the tensile strength is decreased by 38%. The decrease in strength above a fiber length of 30 mm is due to fiber entanglements that occur above an optimum size of the fibers. However, no significant changes in the elongation at break of the composites are noticed with respect to variation in fiber length.

Observation of the composite specimens have shown that long fibers tend to bend or curl during molding and cause a reduction in the effective length of the fiber below the optimum length (30 mm), which results in a decrease of properties. Thus, there exists a critical fiber length that is required for the fiber to develop its fully stressed condition in the matrix in these composites. From the overall mechanical properties, a fiber length of 30 mm was found to be the optimum length for effective reinforcement in a polyester. In the case of fibers shorter than the critical length (<30 mm), the stressed fiber will debond from the matrix and the composite will fail at low strength.

Table III Variation of Tensile Properties of PALF–Polyester Composites as a Function of Fiber Loading (Fiber Length 30 mm)

Fiber Content (Wt %)	Young's Modulus (MPa)	Tensile Strength (MPa)	Elongation at Break (%)
0	580	20.6	1.6
10	1770	17.1	1.3
20	1830	40.0	3.0
30	2290	52.9	3.6
40	2520	63.3	5.0

Effect of Fiber Loading

The effect of fiber loading on the randomly oriented composites is shown in Table III. It is observed that the Young's modulus of the composites showed an increase with increase in fiber content. Addition of 40% fiber increased the modulus by 340%. The high stiffness of these materials makes them suitable for structural applications.

The tensile strength of the composite is slightly decreased with the addition of 10% fiber. This is because at low fiber loadings the fibers act as flaws. However, further increase in fiber content up to 40% increases the tensile strength. Addition of 40% fiber increases the tensile strength of the composite by about 210% than the neat polyester.

The percentage elongation at break is quite low in the case of pure polyester resin. The elongation at break decreases for 10% fiber composites and then increases with increasing concentration of fiber loading as observed from Table III. The brittle nature of the polyester resin decreases with the addition of pineapple fiber and, therefore, the elongation at break increases. It is interesting to note that the failure elongation of the composite is much higher than that of the individual components (polyester and PALF) at higher fiber loadings, indicating a synergistic effect.

The tensile fractographs of PALF–polyester composites containing 10 and 40 wt % of PALF examined with SEM are presented in Figures 3 and 4. Figure 3 shows fiber pullout, debonding, and fibrillation. At high fiber loading, fiber-to-fiber contact is greater, which is evident from the SEM photograph (Fig. 4). The presence of polyester particles on the fiber surface indicates better interaction between the fiber and the matrix.

Mechanical Properties in Flexure

The stress–strain behavior of PALF–polyester composites under flexure is shown in Figure 5. In the case of pure polyester, the flexural stress increases linearly with strain. But the flexural behavior of the composites is nonlinear. These curves show an initial linear portion followed by a nonlinear portion from the middle of the curve

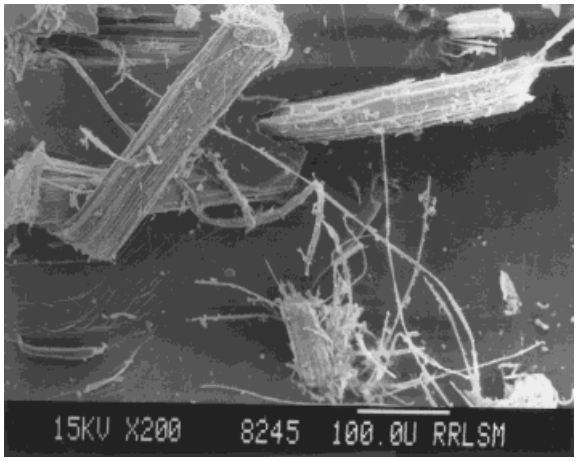


Figure 3 SEM of tensile failure surface of PALF–polyester composites (fiber content 10 wt %).

up to the fracture point. The addition of fibers makes the composite more ductile.

The dependence of flexural properties of the composites on fiber length is shown in Figure 6. The flexural strength of the composites containing 30 mm-long fibers was 23% higher than that of composites containing fibers of 5 mm length. The flexural stiffness of the composites containing 30 mm-long fibers was also found to be higher than that of 5 mm-long fibers by 57%. However, the flexural properties showed a decrease for composites having a fiber length of 40 mm. The best flexural properties of the composite are obtained at a fiber length of 30 mm.

Figure 7 shows the variation of flexural strength and flexural modulus with fiber loading. From the figure, it is seen that the flexural

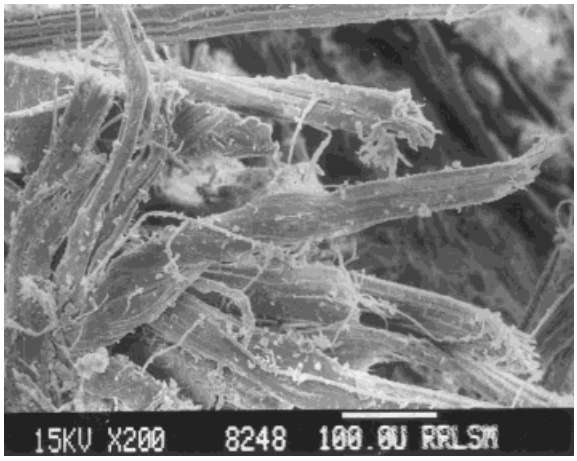


Figure 4 SEM of tensile fracture surface of PALF–polyester composites (fiber content 40 wt %).

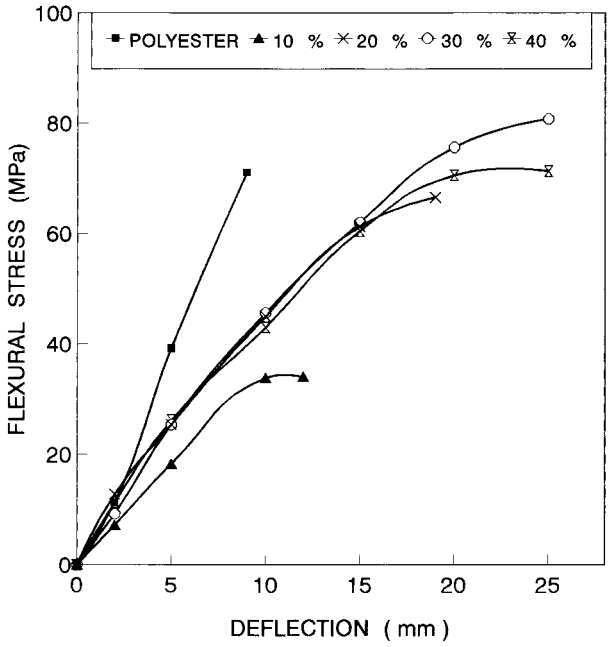


Figure 5 Variation of flexural stress with deflection of PALF–polyester composites.

strength values of the PALF–polyester composites are found to be less than the neat resin at low weight fractions of the fiber. But increase in fiber content from 10 to 30% increases the flexural strength by 120% for composites containing 30

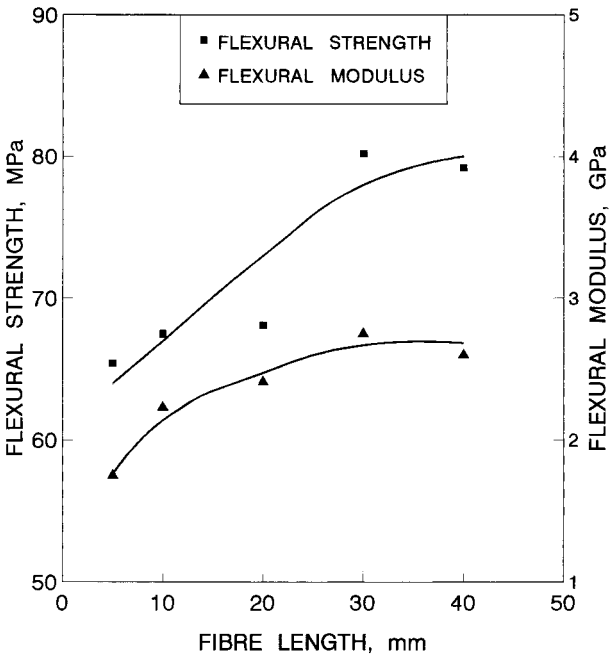


Figure 6 Variation of flexural strength and flexural modulus with fiber length (fiber loading 30 wt %).

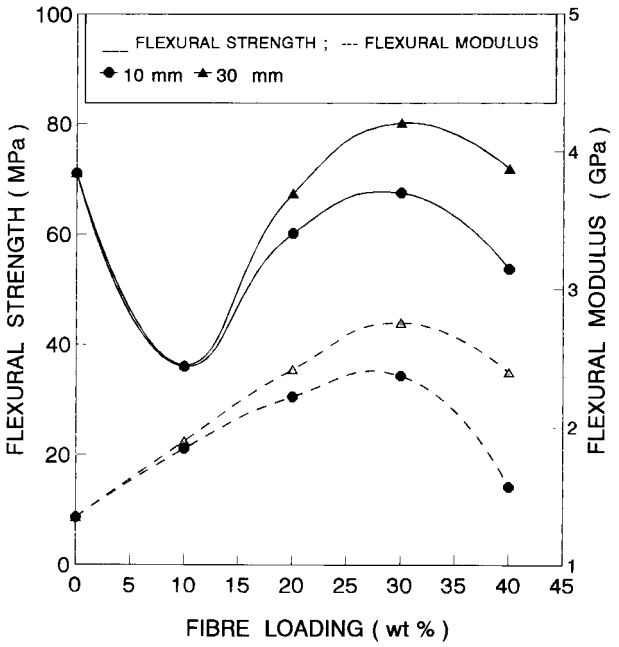


Figure 7 Variation of flexural strength and flexural modulus with fiber loading of PALF–polyester composites.

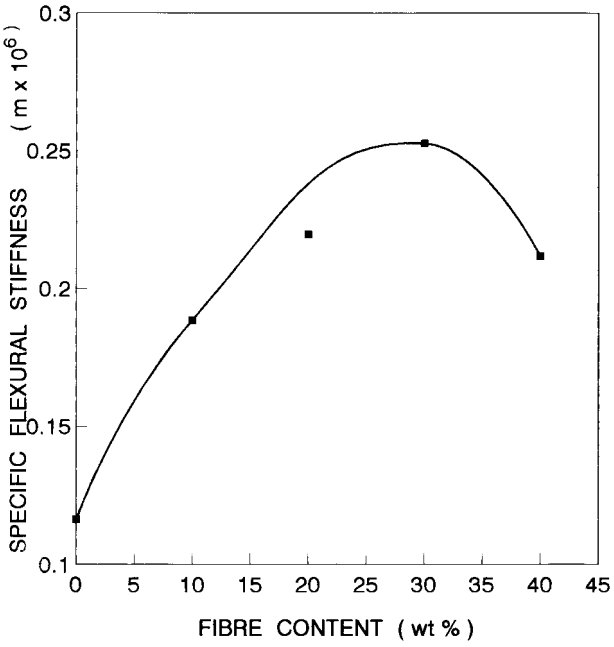


Figure 8 Variation of specific flexural stiffness of PALF–polyester composites with fiber content (fiber length 30 mm).

mm-long fibers. Further increase to 40 wt % results in a slight lowering in flexural strength by about 12%. The flexural strength of the composites is significantly higher than the corresponding tensile strength. The flexural modulus increases with fiber content up to 30 wt % and the value for 30 wt % is 105% greater than that of the neat polyester resin. However, further increase of fiber loading to 40% decreased the value by 13%. The flexural properties of the 10 mm fiber composite vary in the same manner. The decrease of the flexural modulus at 40 wt % fiber loading is clearer in the system with short fiber lengths (i.e., 10 mm). The best flexural properties are observed in composites with 30% fiber content. The decrease in the flexural properties at higher wt % (40%) of fiber loading is due to the increased fiber-to-fiber interactions and dispersion problems.

The specific flexural stiffness is plotted against fiber wt % in Figure 8. The specific flexural stiffness increases with the addition of fiber and is maximum for 30 wt % composites. Its value decreases for 40 wt % composites. The 30 wt % composite has a specific flexural stiffness of 0.25 (m $\times 10^6$), which is 2.3 times greater than that of polyester.

Scanning electron micrographs of the fractured surfaces of the composites after the flexural test are given in Figures 9 and 10. Large fiber breakage and delamination are seen in Figure 9. Figure 10(a) and (b) shows a high extent of fiber pullout and fibrillation.

Impact Behavior of PALF–Polyester Composites

The work of fracture (impact strength) of PALF–polyester composites at 30% loading as a function of fiber length is shown in Table IV. It is seen that a comparatively higher impact strength is

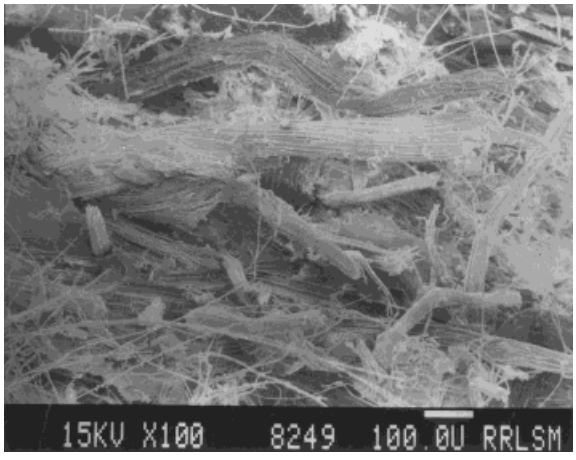
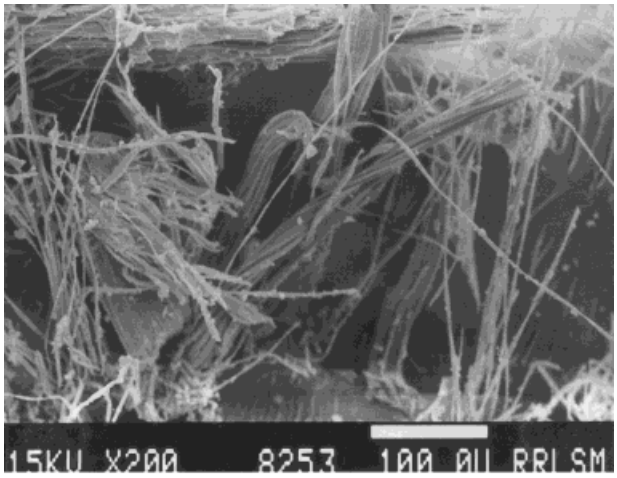
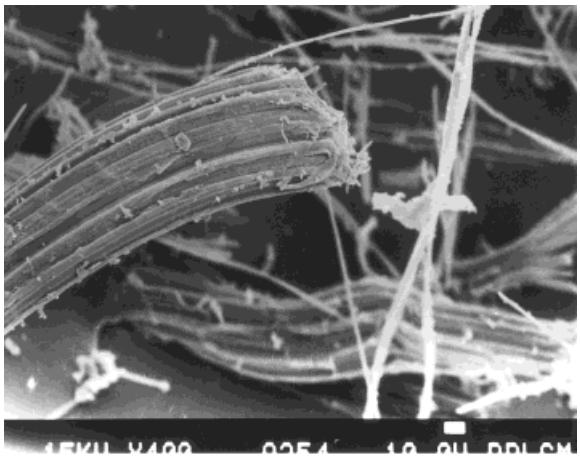


Figure 9 SEM of fracture surface of PALF–polyester composite under flexural failure (fiber content 30 wt %).



(a)



(b)

Figure 10 (a) SEM of fracture surface of PALF–polyester composites under flexural failure (fiber content 10 wt %). (b) SEM of magnified view of a pulled-out fiber during flexural failure (fiber content 10 wt %).

observed for composites of fiber length 10 and 30 mm (critical fiber length). In fact, there is a decrease in impact strength for composites of higher fiber length (40 mm).

The energy-absorbing mechanisms built in the composite include

1. Utilization of the energy required to debond the fibers and pull them completely out of the matrix.
2. Use of a weak interface between the fiber and the matrix.[15]

It is clear that the conditions leading to (1) and (2) are at odds with those required for good stiffness and strength of the composite.

In most fiber-filled composites, a significant part of the energy absorption during impact takes place through the fiber pullout process. The energy involved and, hence, toughness are greatest when the length of the fibers is equal to the critical length (l_c) in accordance with (1). Again, it is apparent that maximum strength and maximum toughness cannot be achieved simultaneously and that composites must be designed for an optimum combination of the desired mechanical properties. The variation of the work of fracture with fiber length was calculated by Copper[16] using a model based on (1) given above. Fibers shorter than l_c (30 mm) will be pulled out from the matrix rather than broken when a crack passes through the composite. The fracture energy will then largely be a combination of the work needed to debond the fibers out of the matrix and the work done against friction in pulling the fibers out of the matrix. The fracture energy (U) arising from fiber pullout is given by the following expressions due to Cottrell[17]:

For $l < l_c$,

$$U_1 = \frac{v\tau l^2}{12d} \tag{3}$$

where d = the fiber diameter, τ = the interfacial frictional stress, and v = the volume fraction of the fiber. Hence, $U_1 \propto l^2$ for $l < l_c$.

The energy reaches a maximum when $l = l_c$ and will have a value

$$U_{\max} = \frac{v\tau l_c^2}{12d} \tag{4}$$

Strictly speaking, the work of fracture arising from the fibers and matrix and the energy to debond the fibers should be added to this energy.

Table IV Variation of Work of Fracture (Impact Strength) with Fiber Length for PALF–Polyester Composites (Fiber Content 30 Wt %)

Fiber Length (mm)	Work of Fracture (kJ m^{-2})
10	24.4
20	19.9
30	24.2
40	22.0

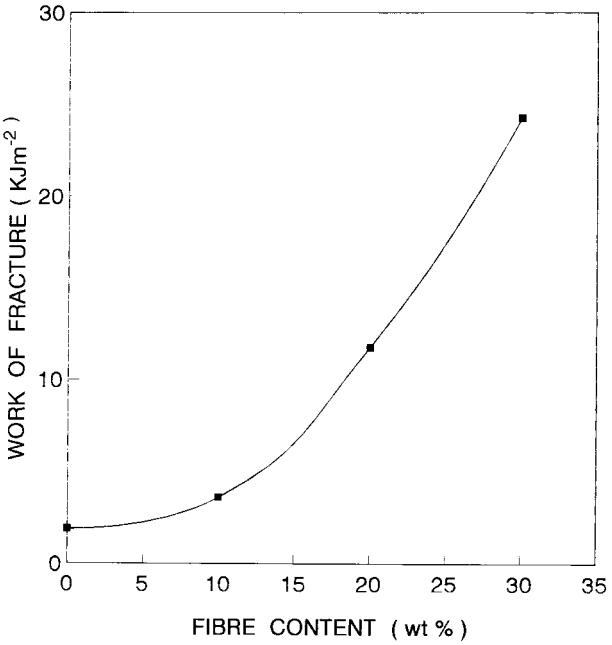


Figure 11 Variation of work of fracture of PALF–polyester composites with fiber content (fiber length 30 mm).

However, in many cases of practical interest, the energy of pullout considerably exceeds the other contributions. Thus, for maximum toughness, it is desirable to use fibers having a length $l < l_c$ based on eqs. (3) and (4) since $U_{max} \propto l(l_c)$ when $l \leq l_c$. The observed high impact strength of the composites with fiber length 10 and 30 mm (critical fiber length) (Table IV) can thus be explained. Also, progressive delamination occurs in these cases, resulting in high energy absorption. However, for fibers having length greater than the critical length, only a proportion of the fibers will pullout and then the energy is given by

$$U_2 = \frac{v\tau l_c^3}{12dl} \qquad (5)$$

The impact energy or strength decreases as

$$U_2 \propto \frac{1}{l}$$

The slight decrease in impact strength for composites with fibers of length 40 mm can thus be explained.

The work of fracture (impact strength) of the composites is found to increase linearly with the weight fraction of the fiber as shown in Figure 11

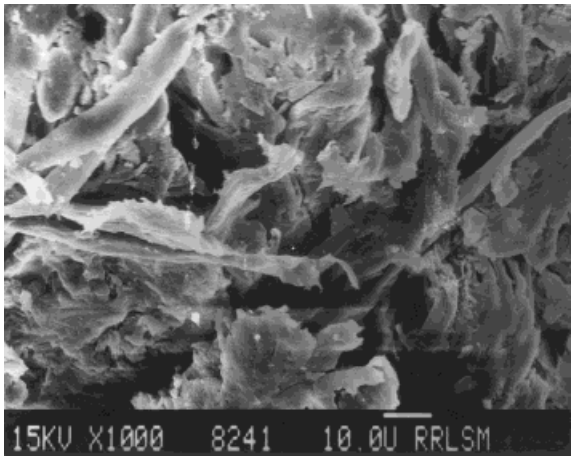


Figure 12 SEM of fractograph of the composite during impact test (fiber content 30 wt %).

and the work of fracture for a 30 wt % composite is found to be 24 kJ m^{-2}. It is about 1200% greater than that of pure polyester resin.

It is generally accepted that the toughness of a fiber composite is dependent mainly on the fiber stress–strain behavior.[18] Pineapple fibers are comparatively strong fibers with high failure strain and therefore impart a high work of fracture to the composites. The fiber pullout, interface fracture, and delamination are the major contributions toward the high toughness of these composites.

SEM of the impact fracture surfaces of PALF–polyester composites containing 30 and 10 wt % fibers are shown in Figures 12 and 13. Figure 12 shows mainly the corrugated surface of the fiber and Figure 13 shows debonding between PALF and polyester.

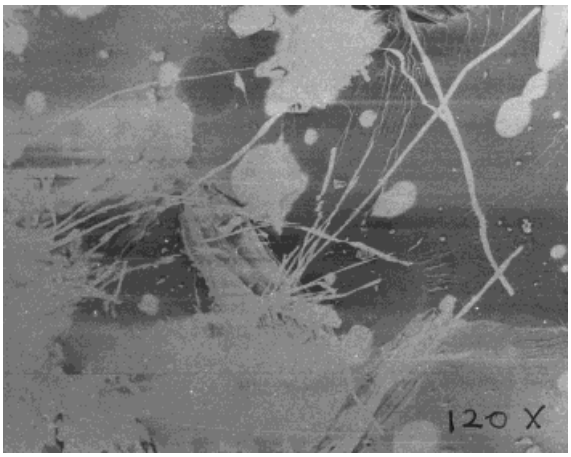


Figure 13 SEM of fracture surface (impact) of PALF–polyester composites (fiber content 10 wt %).

Table V Effect of Chemical Treatment on Mechanical Properties (Fiber Length 30 mm, Fiber Content 30 Wt %)

Type/Nature of Treatment	Tensile Strength (MPa)	Elongation at Break (%)	Young's Modulus (MPa)	Flexural Strength (MPa)	Flexural Modulus (GPa)
Untreated	52.9	3.6	2290	80.2	2.76
2% NaOH (treated for 1 h)	55.4	4.7	1460	77.4	2.93
Silane A172	73.5	4.3	—	85.6	—
Silane A1100	52.7	3.5	2690	79.6	2.84
Glacial acetic acid and acetic anhydride	42.7	3.6	2260	71.5	2.55

Effect of Chemical Treatment on Mechanical Properties

The performance of composites is to a greater extent influenced by the interfacial interaction between the fiber and the matrix. The interfacial bond may be improved by surface treatment.

Table V shows the mechanical properties of composites obtained with chemically treated PALF. Reagents such as NaOH, silane coupling agents (silane A172 and A1100), and glacial acetic acid were used for treatment.

NaOH treatment showed marginal improvement in tensile strength. However, flexural strength and the Young's modulus decreased. The slight enhancement in tensile strength in these composites is attributed to the improved wetting of alkali-treated PALF with polyester. NaOH treatment is mainly a process of surface activation. It leads to the formation of a rough surface which would increase the mechanical interlocking between the fiber and the polyester.

A comparison of the effect of two silane coupling agents on the mechanical properties of the composites was carried out. A 40% increase in the tensile strength was observed when the fibers were treated with silane A172 [vinyltri(2-ethoxy methoxy)silane]. The flexural strength of these composites was also increased by about 7%. In silane A172-treated composites, the alkoxy groups of silane hydrolyze to form silanols (—OH). This —OH group interacts with the —OH groups of lignocellulosic PALF, forming hydrogen bonds, and the vinyl group reacts with the polyester. This would cause the resin to be less interconnected, resulting in a higher elongation of the silane-treated composites. Addition of the coupling agent silane A1100 has improved the Young's modulus of the composites marginally. However, other properties were unaffected. Thus, the treatment of the fibers with silane A1100 has a negligible effect on the properties of the composite as it is a nonreactive type on the basis of its reactivity with the polyester matrix. The mechanical properties of the composites with acetylated fibers were found to be less than those of the untreated fiber composites.

Physical Properties

The density and hardness of the composites with varying fiber content is given in Table VI. The density of the composites slightly decreased with fiber content for a low weight fraction (10 wt %), but remained steady for a high weight fraction of the fiber. Incorporation of the fiber reduces hardness at a low weight fraction of the fiber. However, at high fiber concentration, the hardness values of the composites are found to increase.

Comparison with Other Short Natural Fiber Polyester Composites

A comparison of the mechanical properties of the PALF–polyester composite with other natural fiber–polyester composites at 30 wt % fiber content

Table VI Variation of Density and Hardness of PALF–Polyester Composites with Fiber Content (Fiber Length 30 mm)

Fiber Content (Wt %)	Density (g/cm^3)	Hardness (Shore D)
0	1.15	82
10	1.03	73
20	1.10	75
30	1.09	83
40	1.09	84

Table VII Properties of Natural Fiber Polyester Composites Prepared by Hand Layup Method (Fiber Content 30 Wt %)

Property	Straw-reinforced Polyester	Sisal Polyester	PALF–Polyester
Tensile strength (MPa)	—	28	52.7
Flexural strength (MPa)	47	53	80.2
Impact strength (unnotched) (kJ/m^2)	2.6	11	24.2

Source from Refs. 5 and 7.

is given in Table VII. The mechanical properties (tensile, flexural, and impact) of short PALF–polyester composites are significantly higher than those of straw and sisal fiber polyester composites. This is due mainly to the high cellulose content of PALFs.

CONCLUSION

The results of the present study showed that a useful composite with good strength could be successfully developed using pineapple fibers as a reinforcing agent for the polyester matrix. The optimum length of the fiber required to obtain PALF–polyester composites of maximum properties was found to be 30 mm. The stress–strain behavior in tension reveals that neat polyester is brittle and the addition of fibers makes the matrix more ductile. The tensile strength and Young's modulus of these PALF polyester composites increased linearly with the fiber weight fraction. But in the case of flexural strength, there is a leveling off beyond 30%. The impact strength also increased linearly with the weight fraction of the fiber. The composite with 30 wt % fiber content exhibits an impact strength of 24 kJ/m^2. The high toughness of this natural fiber polymer composite places it in the category of tough engineering materials. A significant increase in the strength of the composites was observed after treatment of the fibers. The best improvement was observed in the case of silane A-172-treated fiber composites. The PALF–polyester composites exhibit superior mechanical properties when compared to other natural-fiber polyester composites and can be used as structural composites.

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