Short Pineapple-Leaf-Fiber-Reinforced Low-Density Polyethylene Composites

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SYNOPSIS

Short pineapple-leaf-fiber- (PALF)-reinforced low-density polyethylene (LDPE) composites were prepared by melt-mixing and solution-mixing methods. In the melt-mixing technique, a mixing time of 6 min, rotor speed of 60 rpm, and mixing temperature of 130°C were found to be the optimum conditions. Tensile properties of melt-mixed and solution-mixed composites were compared. Solution-mixed composites showed better properties than melt-mixed composites. The influence of fiber length, fiber loading, and orientation on the mechanical properties has also been evaluated. Fiber breakage and damage during processing were analyzed from fiber distribution curve and optical and scanning electron micrographs. Considering the overall mechanical properties and processability characteristics, fiber length of 6 mm was found to be the optimum length of pineapple leaf fiber for the reinforcement in LDPE. The mechanical properties were found to be enhanced and elongation at break reduced with increasing fiber loading. Longitudinally oriented composites showed better properties than randomly and transversely oriented composites. Recyclability of the composite was found to be very good. A comparison of the properties of the PALF-reinforced LDPE composites with those of other cellulose-fiber-reinforced LDPE systems indicated superior performance of the PALF–LDPE composites. © 1995 John Wiley & Sons, Inc.

INTRODUCTION

The incorporation of stiff fibers in soft matrices can lead to new materials with outstanding mechanical properties encompassing the advantages of both the fiber and matrix.[1-7] These composites are generally characterized and evaluated by means of various standard tensile, flexural, and fatigue tests performed on composite specimens.[8,9] Fibrous fillers are widely employed in thermoplastics for reinforcement, and the stiffness and strength of fiber-reinforced plastics is a function of fiber properties and the quantity of fiber incorporated.[10-12]

Owing to the high price of composites, the user industries also demand a lower price for production of fiber components, at the same time an improvement in quality. It was found that these can be achieved by the use of natural fibers.[13-19] Compared to inorganic fillers, the main advantages of these fibers are their low cost, low density, high specific strength and modulus, renewable nature, and comparatively easy processability.[20] For better processability, these composite materials often incorporate short discontinuous fibers oriented in the direction of applied load in order to take full advantage of the reinforcing property of fiber.[21-24]

Properties of fiber-reinforced composites depend on many factors like fiber–matrix adhesion, volume fraction of fiber, fiber aspect ratio, fiber orientation, as well as stress transfer efficiency of interface.[25-27] Extensive research studies have been carried out over the last few years in the field of natural-fiber-reinforced thermoplastics. These include the interesting works of Kokta and co-workers.[28-34] Felix and Gatenholm[35] reported the effect of compatibilizing agent and nature of adhesion in composites of cellulose fibers and polypropylene (PP). Recently, Thomas and co-workers reported on the use of sisal fiber as a potential reinforcing agent in polyethylene, thermosets (epoxy resin, phenol-formaldehyde

polyester), and natural rubber.[36-44] Among various natural fibers, pineapple leaf fibers (PALF) exhibit excellent mechanical properties. These fibers are extracted from the leaf of the plant *Ananus cosomus* belonging to Bromeliaceae family. Physical properties, mechanical and dielectric properties, tensile behavior, surface, and fracture morphology of PALF were reported by many workers.[45-48] Bhattacharyya et al.[49] reported on the mechanical properties of PALF-reinforced elastomer composites. However, no serious attempts have been made to develop PALF-reinforced thermoplastic composites. In a recent communication, George et al.[50] reported on the viscoelastic properties of PALF low-density polyethylene (LDPE) composites.

In the present study we report the results of our investigations on the effect of short pineapple fiber reinforcement on low-density polyethylene. The effects of processing conditions, fiber loading, fiber orientation, and fiber length on the physico-mechanical properties of the composites are analyzed. The tensile failure surfaces are examined by scanning electron microscopy (SEM) in order to gain an insight into the fiber orientation, fiber damage, and fiber–matrix adhesion. Finally, the properties of pineapple leaf fiber composites were compared with those of other cellulose fiber (sisal and jute) reinforced LDPE composites.

MATERIALS

Low-density polyethylene granules (LDPE, 16 MA 400) were supplied by Indian Petrochemicals Corporation Ltd., Baroda. Pineapple leaf fiber (PALF) was obtained from South India Textile Research Association, Coimbatore. The properties of LDPE and PALF are given in Tables I and II, respectively. Fibers of different sizes were obtained by chopping them into 2, 6, and 10 mm, respectively, after removing the impurities. Pineapple fibers, a multicellular lignocellulosic material, contain 70–82% cellulose, 5–12% lignin, 1% ash, and other components.

Table I   Physical and Mechanical Properties of LDPE

Melt flow index (g/mm)	40
Vicat softening point (°C)	85
Density (g/cm^3)	0.918
Tensile strength (MPa)	8.6
Young's modulus (MPa)	130
Elongation at break (%)	110

Table II   Physical and Mechanical Properties of PALF

Density (g/cm^3)	1.526
Softening point (°C)	104
Tensile strength (MPa)	170
Young's modulus (MPa)	6260
Elongation at break (%)	3

EXPERIMENTAL

Fibers were separated from undesirable foreign matter and pith and manually chopped to different length of 2, 6, and 10 mm. The chopped fibers were washed with water and dried in an air oven at 70°C for 24 h before being mixed with polyethylene. Composites of PALF–LDPE were prepared by two different methods: melt mixing and solution mixing. Melt mixing was carried out in a Brabender Plasticorder (Model PLE 331). In order to optimize the mixing parameters, composites were prepared by varying the mixing time, rotor speed, and chamber temperature. In the solution-mixing method, a technique recently developed by our group, fibers were added to a viscous slurry of polyethylene in toluene, which was obtained by adding toluene to a melt of the polymer.[50] The solvent is then evaporated off. Both the melt-mixed and solution-mixed composites were then extruded at a temperature of 120°C through a hand-operated ram-type injection-molding machine. The extrudate was cut into 120 mm long rods. The oriented composites were prepared by closely aligning the cylindrical extrudates in a mold and then compression molding at a temperature of 120°C. Randomly oriented composites were prepared by direct extrusion of the mix into the mold.

Uniaxial tensile properties were measured using an INSTRON universal testing machine model 1121 at a crosshead speed of 50 mm/min and a gauge length of 50 mm. Specimens having a size of 120 × 12.5 × 2.5 mm^3 were used for testing. The tensile fracture surfaces were examined under a scanning electron microscope model JEOL JSM-35C. Tear strength of the sample was determined as per ASTM D 624 using unnotched 90° angle test pieces punched out from the molded sheets. Fiber breakage during mixing was analyzed by extracting the fibers from the composite using toluene as the solvent and observing under an optical microscope. Fiber size distribution curves were obtained based on measurements of 150 fibers using traveling microscope.
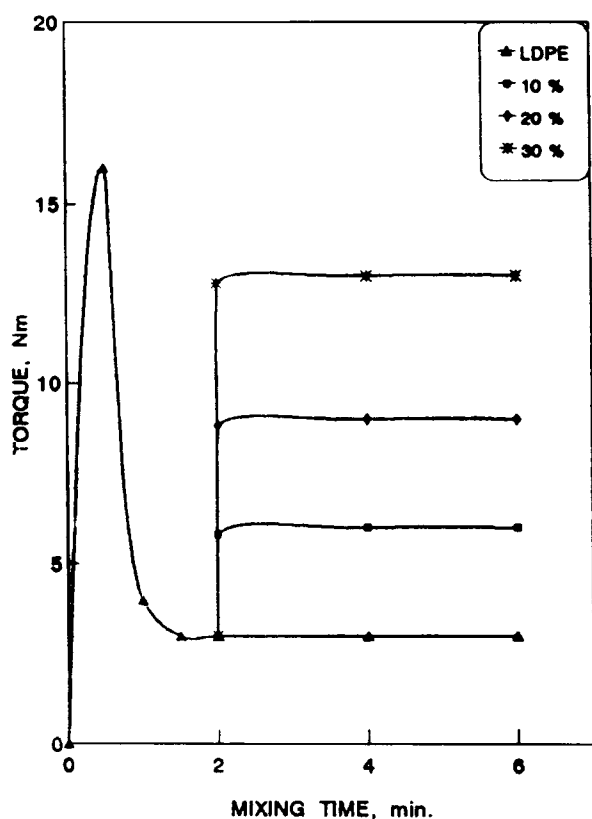
Figure 1 Plastographs for PALF–LDPE composites at different fiber loading. Rotor speed 60 rpm, mixing time 6 min, temperature 120°C.

RESULTS AND DISCUSSION

Melt-Mixed Composites

Mixing Characteristics

The mix characteristics of LDPE–PALF composites have been studied using the Brabender plastographs, which are plots of torque vs. mix time. As shown in Figure 1, the mixing torque initially increases rapidly when LDPE granules are charged into the mixer chamber. This is associated with the unmolten nature of LDPE granules. As the mixing time increases, LDPE undergoes melting, which results in a decrease of torque which levels off at longer times. Fiber was incorporated into molten PE after two minutes. Addition of fibers results in an increase of torque because of the increased viscosity of the system. The torque attains constant value at longer times when incorporation of PALF fibers in the LDPE matrix is complete. In melt-mixed composites, the properties of the system are dependent on mixing conditions such as mixing time, rotor speed, and temperature of mixing. In order to optimize

these parameters, a series of PALF–LDPE composites were made by changing rotor speed, mixing time, and temperature.

Mechanical Properties

The effect of mixing time on strength and Young's modulus of both oriented and random PALF–LDPE composites is depicted in Figure 2. These mixes were carried out at a temperature of 120°C and at a rotor speed of 60 rpm. When the mixing time is less, tensile strength and Young's modulus are low because of the ineffective mixing and poor dispersion of the fiber in LDPE matrix. However, as the mixing time increases, the tensile strength increases and attains maximum value at 6 min, which levels off at higher mixing time. The modulus exhibits maximum value in 6 min and decreases as mixing is continued. As expected, oriented composites show higher strength compared to randomly oriented composites, i.e., orientation provides better reinforcement to the matrix.

Figure 3 shows the effect of Brabender rotor speed on tensile strength and modulus of oriented and random composites mixed at 120°C for 6 min. Com-
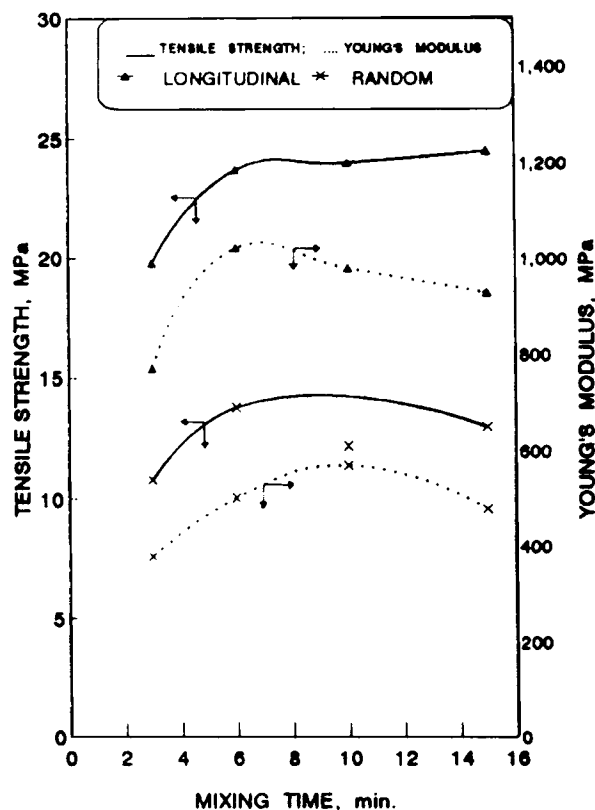

Figure 2 Variation of tensile strength and modulus with mixing time of melt-mixed composites. Fiber content 30%.
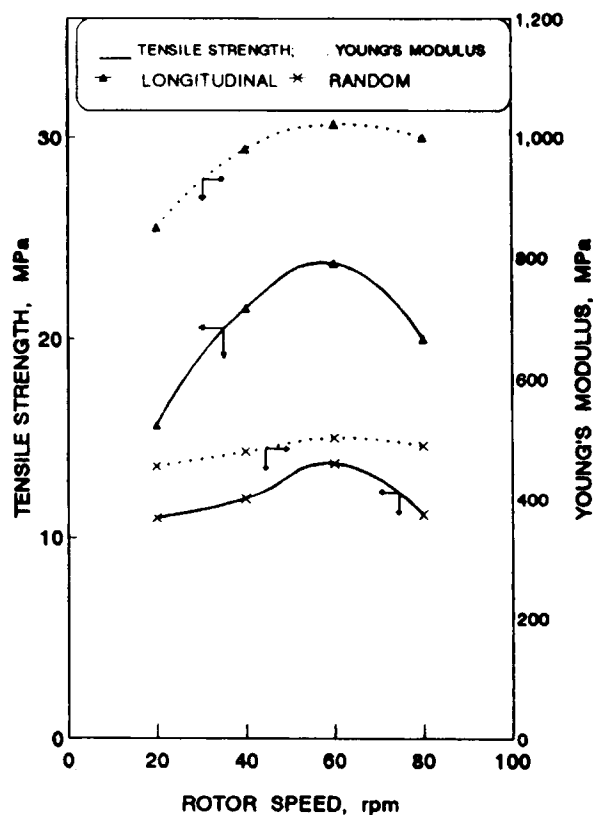
Figure 3   Tensile strength vs. rotor speed of melt-mixed composites. Fiber content 30%.

Figure 4   Fiber distribution curve showing fiber breakage at different rotor speed.

posites prepared by the mixing of fiber and LDPE at lower rotor speed show low tensile strength due to poor dispersion of fiber. But as the rotor speed is increased from 20 to 60 rpm, there is an increase of strength by 60% in the case of oriented composite. However, as the rotor speed is increased to 80 rpm, reduction in strength occurs due to the fiber breakage at higher rotor speed. A similar trend was observed in the case of Young's modulus also. Evidence for fiber breakage at higher rotor speed is provided in terms of fiber distribution curves given in Figure 4. It has been noticed that about 70% of fibers extracted from the composite prepared by mixing fiber and LDPE at 60 rpm are of 2.5–4 mm length. On the other hand, about 65% of fibers from composite that mixed at 80 rpm are in the range of 0.5–3 mm. This indicates that fiber breakage is extensive and severe at 80 rpm. Since the fiber length is extremely small, much shorter than the critical length, the stressed fiber debonds from the matrix and therefore the composite exhibits low strength.

Effect of processing temperature on the composite at a rotor speed of 60 rpm and a mixing time of 6 min on tensile properties is given in Table III. It
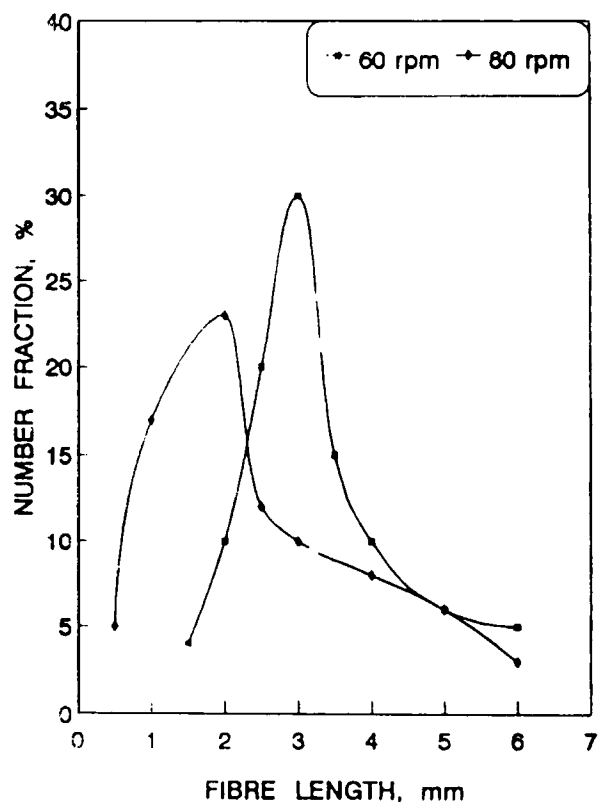
can be seen that the tensile strength and Young's modulus of both oriented and random composites increase with temperature and reach maxima at 130°C. However, above 130°C there is a reduction in strength and modulus. This may be due to the degradation of fiber at higher temperature. Moreover the dispersion of fiber in LDPE matrix will be poor due to the decrease in viscosity at high temperature.

Table III   Effect of Processing Temperature on the Tensile Properties of Melt-Mixed PALF–LDPE Composities[a]

Temperature (°C)	Tensile Strength (MPa)	Young's Modulus (MPa)	Elongation at Break (%)
120	23.7	1022	4
	(7)	(503)	(6)
130	25.3	1205	7
	(14)	(670)	(7)
140	22.8	1030	7
	(11.5)	(500)	(7)

Fiber length 6 mm, fiber content 30 wt %.
[a] Values in parentheses are the properties of random composites.

The optimization of melt-mixing parameters indicates that for the best balance of properties, LDPE–PALF composites should be melt blended in an internal mixer for a period of 6 min at a temperature of 130°C using a rotor speed of 60 rpm.

Melt-Mixed/Solution-Mixed Composites

Table IV shows the effect of fiber loading and mixing techniques on the tensile properties of randomly oriented PALF–LDPE composites. In the case of solution-mixed composite, by the addition of 10% fiber, the tensile strength and Young's modulus are increased by 20 and 68%, respectively. However, on further addition of fibers, the increase is gradual. This is associated with the fiber-to-fiber interactions at high fiber loading. The elongation at break of the composite undergoes a sharp fall upon the introduction of fibers in the mixes. But at higher loading, the decrease is not very sharp but gradual. On the introduction of fibers into LDPE matrix, the fibers inhibit the orientation of molecular chains and hence the elongation at break decreases substantially.

As compared to the solution-mixed composites, the melt-mixed composites show a lower mechanical properties at low fiber loading. For example, at 10% fiber loading, the Young's modulus and tensile strength of melt-mixed composites are lower than that of solution-mixed composites by 58 and 18%, respectively. The differences are less pronounced at high fiber loading. During melt mixing in a Brabender plasticorder the fibers undergo considerable damage like splitting and peeling due to the high shear forces. This can be seen from SEM photographs given in Figures 5(a) to 5(c). The extent of fiber breakage in melt-mixed composite is also evident from the optical photographs shown in Figures 6(a) and 6(b). Here fibers were extracted from the melt-mixed and solution-mixed composite using

toluene as solvent. The extracted fibers were examined under a microscope. It is seen that fiber breakage is severe in the melt-mixed composite than in solution-mixed composite. In solution-mixed composites, the fibers retain their original length. This can be further understood from Figure 7, which shows the fiber length distribution in melt-mixed and solution-mixed composites. It is interesting to note that, in melt-mixed composite, only 5% of fiber retains the 6 mm length. More than 70% of fibers are distributed in the range of 2–4 mm. But in solution-mixed composites maximum distribution occurs at 6 mm. Therefore the reduction in tensile properties in melt-mixed composites is due to severe fiber breakage and damage.

Solution-Mixed Composites

Effect of Fiber Length

The strength of fiber-reinforced composites depends on the degree to which an applied load is transmitted to fibers. The extent of load transmittance is a function of fiber length and magnitude of fiber–matrix interfacial bond. In short-fiber-reinforced composites there exists a critical fiber length that is required for the fiber to develop its fully stressed condition in the matrix. If the fiber is shorter than this critical length, the stressed fiber will debond from the matrix and the composite will fail at a low load. When the length is greater than the critical length, the stressed composites will lead to breaking of fibers and a high composite strength.

Tensile properties of longitudinally oriented solution-mixed composites at 30% loading as a function of fiber length are shown in Table V. When the fiber length is increased from 2 to 6 mm, there is an enhancement in strength by about 13%. Similarly there is an increase in modulus by 15% due to the

Table IV   Variation of Tensile Properties of Randomly Oriented Melt-Mixed and Solution-Mixed Composites[a]

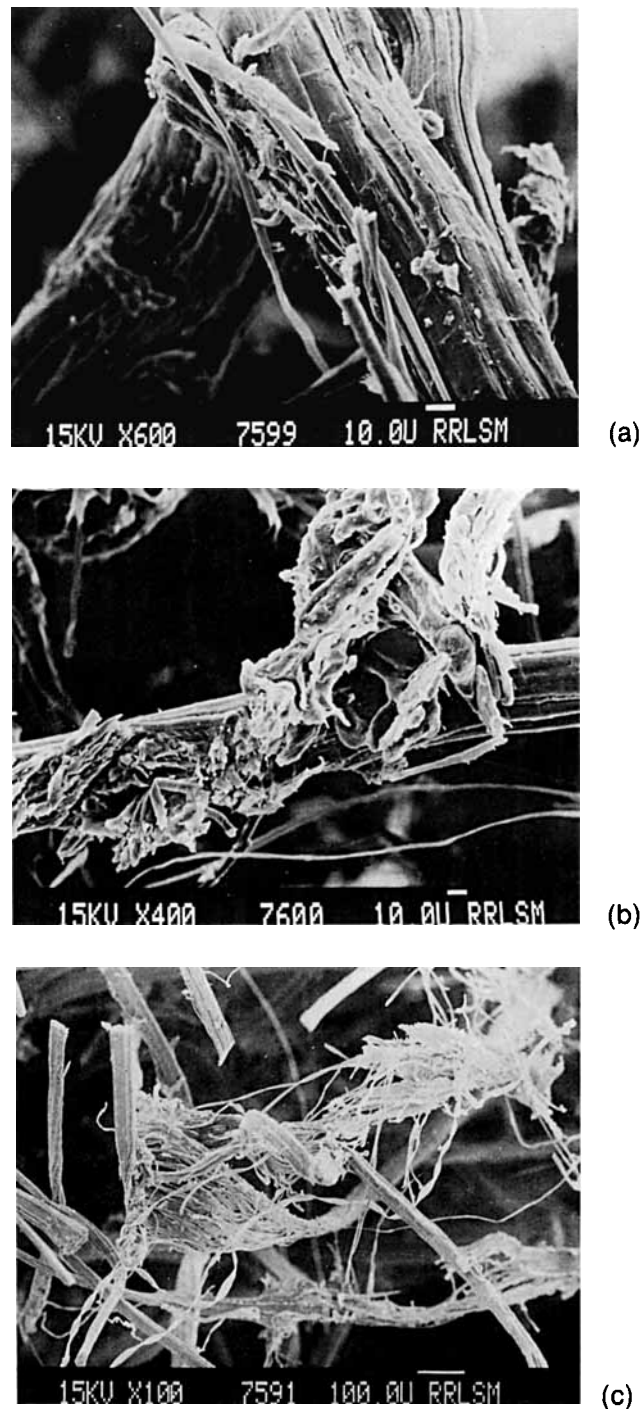| Melt-Mixed Composite				Solution-Mixed Composite			
Fiber Content (wt %)	Tensile Strength (MPa)	Young's modulus (MPa)	Elongation at Break (%)	Fiber Content (wt %)	Tensile Strength (MPa)	Young's Modulus (MPa)	Elongation at Break (%)
0	9.2	115	130	0	8.5	130	110
10	8.6	138	34	10	10.2	218	24
20	10.6	310	10	20	11.4	366	12
30	13.7	503	7	30	13.0	570	6

[a] Fiber length 6 mm.

Figure 5 Tensile fracture surfaces of melt-mixed composites showing fiber damage: (a) and (c) splitting, (b) peeling.

effective stress transfer between the matrix and fiber. When the fiber length is increased to 10 mm, there is only a 4% increase in tensile strength and modulus is almost unaffected. This clearly indicates that the mechanical properties level off beyond 6 mm fiber length. The plateauing off is associated with poor dispersion of fiber in the matrix and fiber-to-fiber entanglements at higher fiber length. The study was limited to 10-mm fiber length since the extrusion of composite was extremely difficult at higher fiber length. Even the extrusion of composite containing 10-mm fiber length was not easy. There-
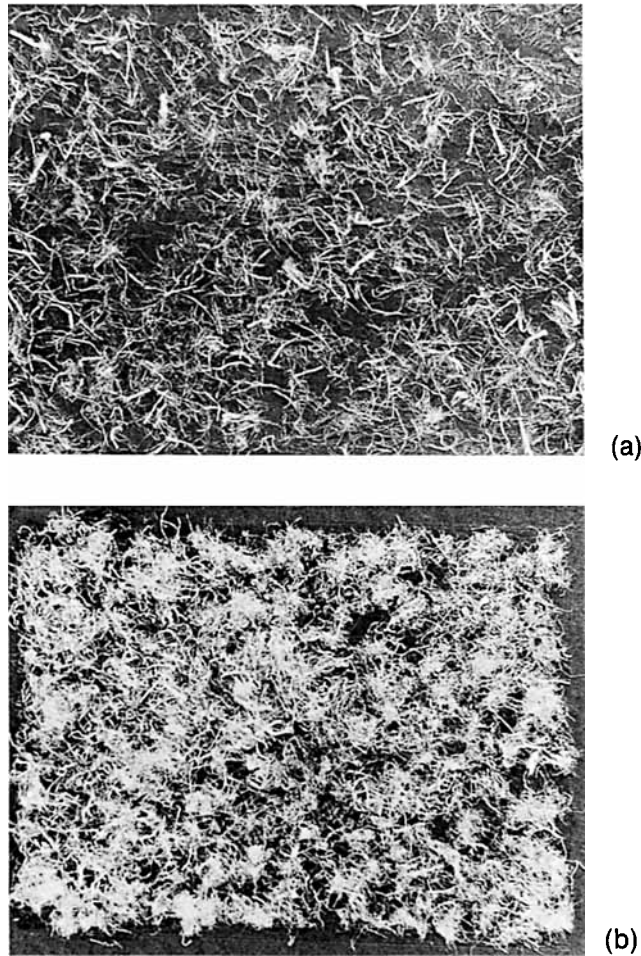
Figure 6 Optical photographs of fibers extracted from (a) solution-mixed composite and (b) melt-mixed composite showing extent of fiber breakage.

fore from the overall mechanical properties and processability, fiber length of 6 mm was found to be the optimum critical value for effective reinforcement in LDPE.

Effect of Fiber Loading

Figure 8 shows the stress–strain curve of longitudinally oriented solution-mixed PALF–LDPE composites. Stress–strain behavior of the composite is controlled by the characteristics of the fiber and the matrix. The deformation behavior of the composite under an applied load can be understood from the stress–strain curve. In the case of pure LDPE, elongation is quite high, i.e., the material shows low initial elastic modulus followed by yielding at larger strains and the failure is essentially ductile. Addition of 10% fiber results in an increase in the initial elastic modulus and reduction in elongation at break by about 90%. Further addition of fibers rapidly in-

creases the modulus of the composite and the system becomes more and more brittle.

Effects of fiber loading on the longitudinally oriented composites are shown in Table VI. It is observed that by adding 10% fibers there is an increase of 90% in tensile strength and by the further addition there is only an increase of 20%. The Young's modulus also increases with fiber loading. Addition of 10% of fiber, increases the modulus by 300%. It can be noticed that above 10% fiber loading, the increase in tensile strength and modulus is less pronounced. This is possibly due to high fiber-to-fiber interactions. The elongation at break of the composite exhibits a sharp fall by the introduction of fibers in these mixes. At higher fiber loading the decrease is gradual.

Tension set after failure was found to be lowered with fiber content as shown in Table VI. A tension set of 24% was found for LDPE, but it was 1% for the composite. This is due to the fact that the ad-
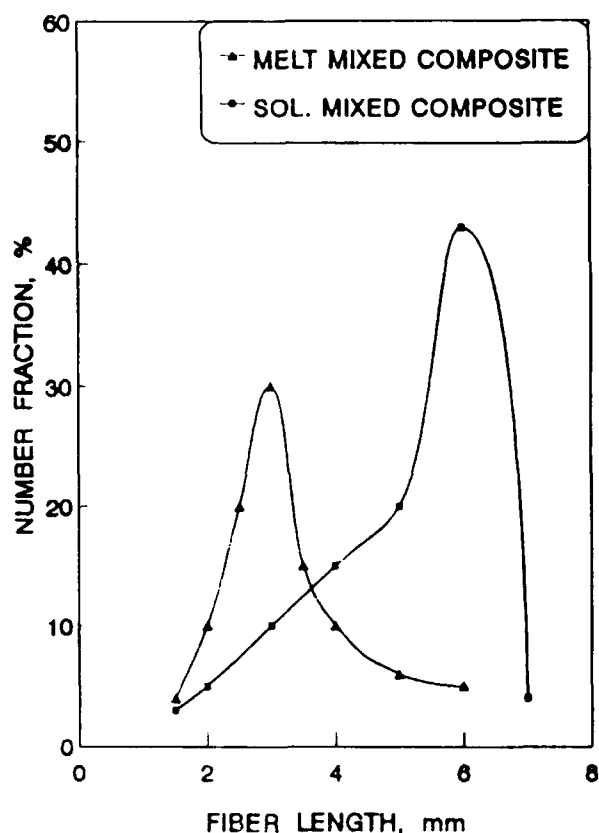
Figure 7 Fiber distribution curve showing fiber breakage in solution-mixed composite and melt-mixed composite.

Table V   Variation of Mechanical Properties of Unidirectionally Oriented Solution-Mixed PALF–LDPE Composites with Fiber Length[a]

Fiber Length (mm)	Tensile Strength (MPa)	Young's Modulus (MPa)	Elongation at Break (%)
0	8.5	130	110
2	19.7	932	4
6	22.5	1095	4
10	23.4	1100	7

[a] Fiber content 30 wt %.

random alignment. Longitudinally oriented composites are inherently anisotropic in that maximum strength and reinforcement are achieved along the direction of fiber alignment. In the transverse direction, reinforcement is virtually nonexistent and therefore fracture usually occurs at very low tensile stress. In randomly oriented composites, strength lies between these two extremes.

The SEM photographs given in Figures 9(a) and 9(b) show longitudinal and transverse orientation of fibers in composite. The figures indicate that the
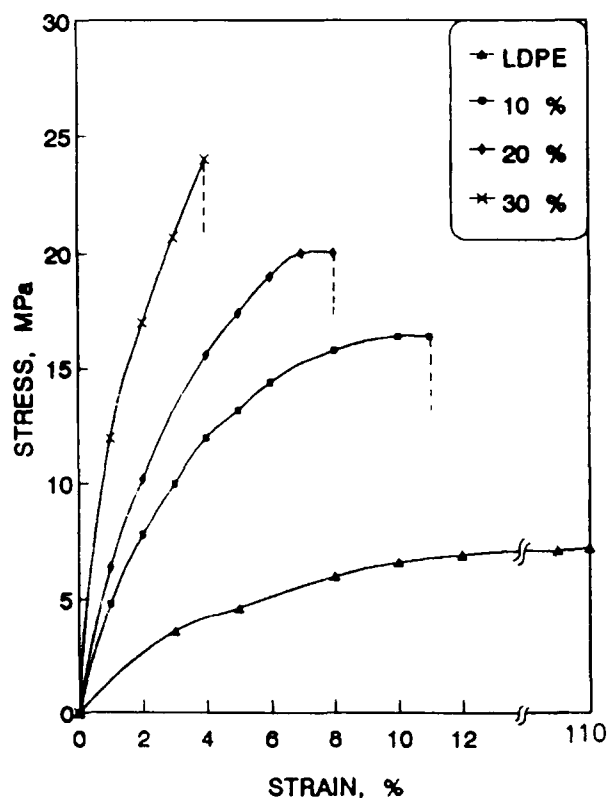
dition of fibers to LDPE makes the system highly brittle and the ductility of LDPE is completely lost.

It can be seen from the table that tear strength increases with fiber content. This indicates that incorporation of fibers makes the crack propagation difficult. When the fibers are arranged longitudinally, tearing takes place normal to the fiber orientation and tear strength depends largely on the obstruction offered by the fibers to the advancing tear.

As expected, the incorporation of fibers increases markedly the composite hardness. Hardness is related with toughness and strength of composite. The density of the composite increases with fiber content due to the close packing of fibers.

Effect of Fiber Orientation

Orientation of fibers related to one another plays a vital role in the performance of composite. With respect to orientation two extremes are possible. These include the parallel alignment of the longitudinal axis of the fibers in a single direction and the totally

Figure 8 Stress–strain curve of PALF–LDPE composites.

Table VI   Mechanical Properties of Solution-Mixed PALF–LDPE Composites (Longitudinally Oriented)[a]

| Fiber Content (wt %)	Tensile Strength (MPa)	Young's Modulus (MPa)	Elongation at Break (%)	Tear Strength (kN/m)	Hardness (shore-D)	Tension Set (%)	Density (g/cm^3)
LDPE	8.5	130	110	63	45	166	0.90
10	16.3	610	11	72	55	2	0.95
20	19.8	720-900	9	81	60	2	0.99
30	22.5	1095-1100	4	97	65	1	1.03

[a] Fiber length 6 mm.

fibers are well oriented during the processing of the composite. It can be seen from Figure 10 that longitudinally oriented composites show better tensile strength properties than transversely and randomly oriented composites. When the fibers are aligned perpendicular to the direction of force (transverse) fibers are not in conjunction with the matrix in in-

creasing the strength of the composite. As expected, randomly oriented composites show intermediate values. The same trend can be seen in the modulus curve as shown in Figure 11. It is clear that a slight misalignment of fiber from the direction of force may lead to drastic decrease in the modulus.

Effect of orientation on tear strength can be seen in Figure 12. Here also it is observed that longitudinally oriented composites exhibit higher properties than random and transverse speciemens. As explained earlier when fibers are oriented longitudinally, the failure takes place normal to the fiber orientation. But in the case of composites with transverse fiber orientation, the resistance offered by the fiber to the propagating tear is even less than that of matrix. As expected random composites show intermediate values.
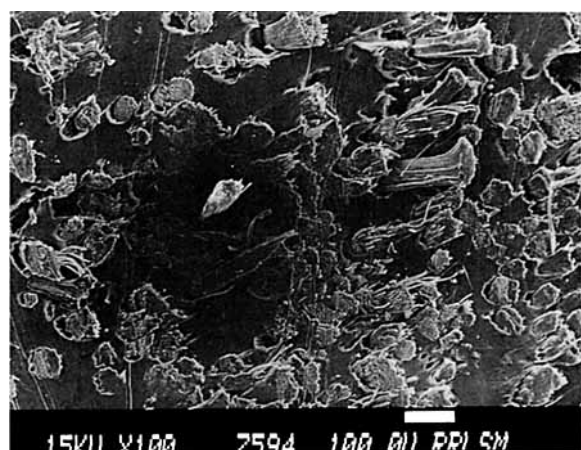
(a)
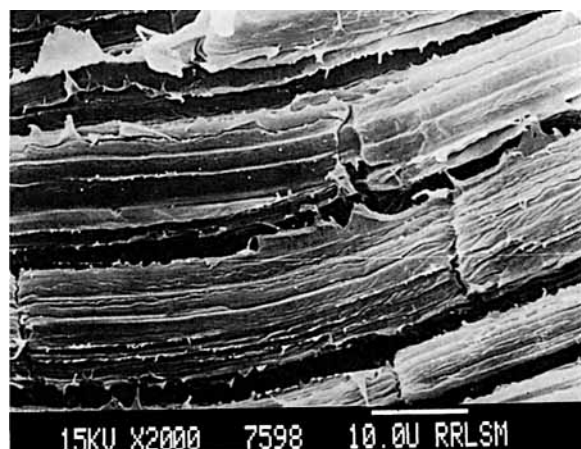
(b)

Figure 9   SEM photographs showing (a) longitudinal and (b) transverse fiber orientation. Samples were cut perpendicular to the direction of applied force.
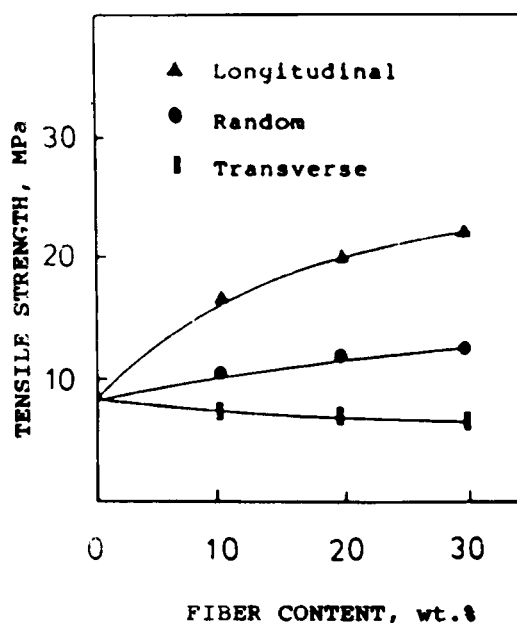
FIBER CONTENT, wt.%

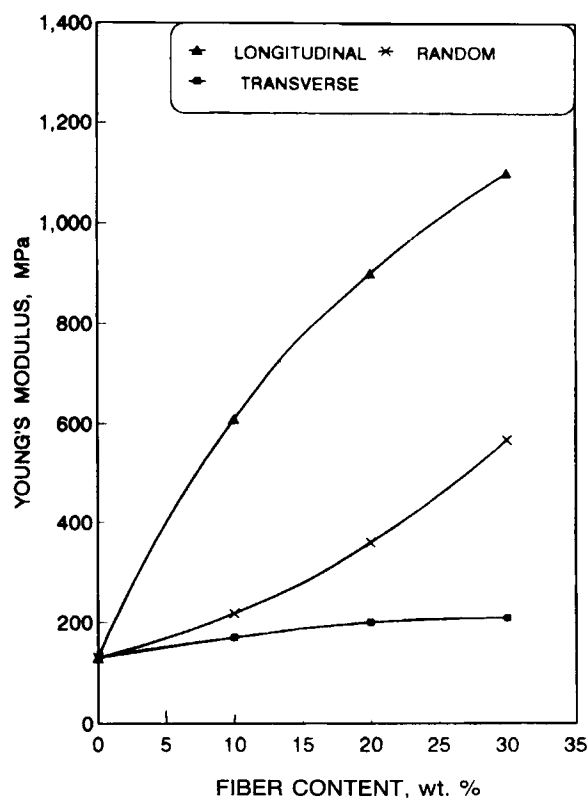**Figure 10**   Effect of fiber orientation on tensile strength of PALF–LDPE composites.

Figure 11 Effect of fiber orientation on Young's modulus of PALF–LDPE composites.

Recyclability of Solution-Mixed Composites

One of the important advantages of thermoplastic composites is their recyclability and reprocessability. The recyclability of the solution-blended PALF–LDPE composites containing 20% fiber has been analyzed by repeated extrusion at 120°C. The properties of the recycled composites have been given in Table VII. Composite properties remain constant, up to third extrusion. Beyond that the property decreases marginally due to thermal effect and degradation of the fiber.

Comparison of Pineapple-Fiber-Reinforced PE Composites with Other Natural Fibers

Tensile properties of LDPE filled with pineapple fiber, sisal,[37] and jute fiber[51] are given in Table VIII. The results show that pineapple- and sisal-fiber-filled composites show comparable mechanical properties. In the case of longitudinally oriented pineapple-fiber-filled composites, the addition of 10% fiber causes an increase of about 92% in tensile strength whereas in sisal composites the corresponding value is 83%. However, the Young's modulus of sisal–LDPE composites are superior to
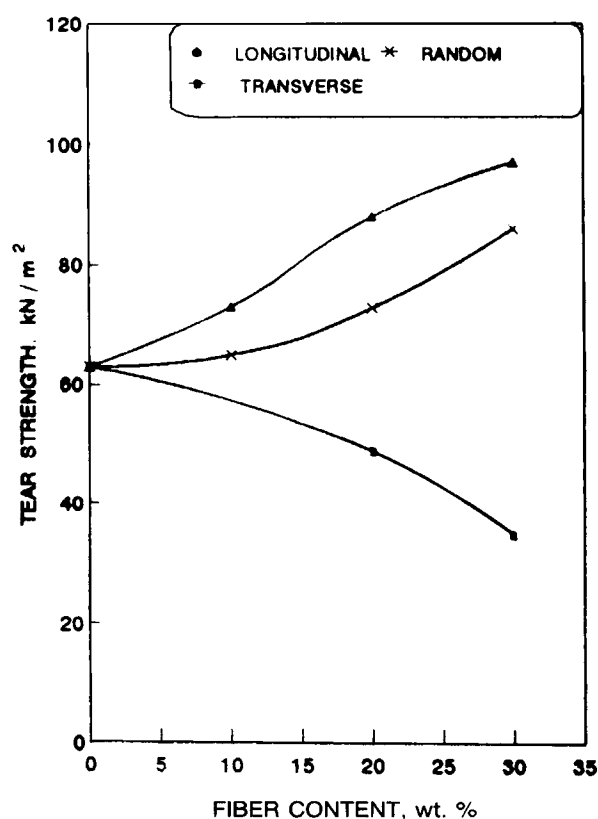

Figure 12 Effect of fiber orientation on tear strength of PALF–LDPE composites.

PALF–LDPE composites. But in the case of jute fiber–LDPE composites there is no improvement at low fiber loading (≤20%). It is seen that the tensile strength of LDPE is decreased by 30% by the addition of 20% jute fiber. However, at high jute fiber loading, the tensile properties of LDPE are improved. Among the three composites, the PALF–LDPE system shows highest elongation at break values. The superior mechanical properties of pineapple-reinforced composites are due to the high cel-

Table VII   Effect of Repeated Extrusion on the Tensile Properties of Solution-Mixed Composites[a]

No. of Extrusion	Tensile Strength (MPa)	Young's Modulus (MPa)	Elongation at Break (%)
1	19.8	720	9
2	19.9	775	10
3	19.1	710	11
4	17.9	625	12
5	16.7	530	14

[a] Fiber content 20%. Fiber length 6 mm.

Table VIII Comparison of Tensile Properties of Randomly Oriented PALF–LDPE, Sisal Fiber–LDPE and Jute Fiber–LDPE Composites[a]

Fiber	Tensile Strength (MPa) Fiber Loading			Young's Modulus (MPa) Fiber Loading			Elongation at Break (%) Fiber Loading		
	10	20	30	10	20	30	10	20	30
Pineapple	10.2	11.4	13.0	218	366	570	24	12	6
	(16.3)	(19.8)	(22.5)	(610)	(900)	(1100)	(11)	(9)	(4)
Sisal	10.8	12.5	14.7	324	453	781	2	1	Ñ1
	(15.6)	(21.0)	(31.0)	(1429)	(2008)	(3086)	(4)	(3)	(1.8)
Jute	—	4.99	8.03	—	—	—	—	11.8	4.9

[a] Values in parentheses correspond to longitudinally oriented composites.

lulose content of pineapple fibers as compared to the other natural fibers.[45] The high crystallinity of pineapple fibers as evidenced from the X-ray studies also contributes to the strength of pineapple-fiber-reinforced composites.[45]

CONCLUSION

PALF-reinforced LDPE composites were prepared by melt-mixing and solution-mixing methods. Processing characteristics of these composites were studied. Mixing time of 6 min, rotor speed of 60 rpm, and a mixing temperature of 130°C were found to be the optimum melt-mixing conditions for better fiber dispersion and mechanical properties. Tensile properties of melt-mixed and solution-mixed composites were compared. Melt-mixed composites showed lower properties than solution-mixed composites due to the extensive fiber damage and breakage during melt mixing. The fiber damage and fiber breakage were analyzed by optical and electron microscopies. Considering the overall mechanical properties and processability characteristics, fiber length of 6 mm was found to be the optimum length of pineapple leaf fiber for the reinforcement in LDPE. In solution-mixed composites, tensile properties, tear strength, hardness, and density were found to be enhanced and elongation at break reduced with increasing fiber concentration. Again, longitudinally oriented composites showed better properties than that of transversely and randomly oriented composites. SEM studies indicated that fibers are well oriented during composite fabrication. Recyclability of the composites was found to be very good. Comparison of tensile properties of LDPE–PALF composites with those of sisal and jute re-

inforcements indicated that pineapple fiber acts as an efficient reinforcement in LDPE.

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