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Short Polyester Fiber Reinforced Natural Rubber Composites

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Short Polyester Fiber Reinforced Natural Rubber Composites†

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The effect of fiber orientation, concentration and L/D ratio on the mechanical properties of short polyester fiber-natural rubber composites have been studied. High anisotropy in the mechanical properties were observed with respect to fiber orientation. Tensile strength, tear resistance and compression set were higher in the longitudinal direction than in the transverse direction. Hardness and abrasion resistance increased and resilience decreased on increasing fiber concentration. However increasing fiber length to diameter ratio (L/D), decreased the anisotropy in tensile and tear properties at 30 phr fiber loading.

INTRODUCTION

Short fibers are known as reinforcing agents in polymers for more than three decades. It has much advantages over continuous fibers. Short fibers can be incorporated into elastomer matrix easily using conventional two-roll mixing mill and this avoids dipping and

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calendering operations as in the case of continuous fibers. Short fiber reinforced composites can be processed easily by compression molding. This saves much energy and time. High degree of anisotropy in properties is obtained by orienting these fibers.

Much work has been done on short fiber reinforcement of elastomers. Mechanism of reinforcement by short fibers have been studied extensively.¹⁻⁹ Effect of short Nylon¹⁰ fibers and silk fibers¹¹ on the mechanical properties of elastomers have been reported. Not much work has been done on the effect of short polyester fibers on the natural rubber compounds. The aim of the present study is to find the effect of fiber concentration, orientation and *L/D* ratio on the mechanical properties of short Polyester fiber-Natural rubber composites.

EXPERIMENTAL

Materials

Crumb natural rubber (ISNR-5) was obtained from Rubber Research Institute of India, Kerala. Chemically treated Polyester cords as per specifications given in Table I, were supplied by M/s Dunlop India Ltd., Sahaganj and antioxidants and accelerators were supplied by M/s Bayer India Limited, Thane.

TABLE I
Specification of polyester cord

Type of fabric	Dipped polyester cable cord
1. Construction in raw stage	1100 Denier \times 3 \times 3
2. Breaking strength (N)	627 min
3. Elongation at break (%)	13
4. Dip pick-up	8
5. Chemical treatment for adhesion	Not supplied

Note: Chemically treated cords were supplied by M/s. Dunlop India Limited for carrying out the above study.

Processing

Formulations of the mixes are given in Table II. The cords were first chopped to lengths of 6.5 mm and 12.5 mm approximately by a semiautomatic chopping machine. The mixing was carried out as per ASTM D 3182-80 on a two-roll laboratory size mixing mill (152 mm × 330 mm). The stock was then divided into four parts and individual parts were subjected to different number of passes, folding and passing each time in the same direction through tight nip. 2–3 passes gave optimum orientation as is evident from Table V and in all subsequent mixing the orientation was effected by passing twice through the tight nip. Optimum cure time was determined by a Monsanto Rheometer R-100. Vulcanization was carried out at 150°C on an electrically heated single daylight hydraulic press at 4.5 N/mm² pressure. For thicker samples sufficient extra cure was given so as to get the same extent of cure. The vulcanizates were tested for different physical properties according to respective ASTM standards. Tensile and tear properties were studied using an Instron Universal Tester (model 1195)

TABLE II
Composition of the mixes

Ingredients	Mix No.					
	A	B	C	D	E	F
Natural rubber	100	100	100	100	100	100
Zinc oxide	5	5	5	5	5	5
Stearic acid	2	2	2	2	2	2
IPPD ¹	1	1	1	1	1	1
Vulkanox HS ²	1.5	1.5	1.5	1.5	1.5	1.5
TMTD ³	1.0	1.0	1.0	1.0	1.0	1.0
MOZ ⁴	2.0	2.0	2.0	2.0	2.0	2.0
Sulphur	0.3	0.3	0.3	0.3	0.3	0.3
Polyester cords (L = 6.5 mm)	0	10	20	30	40	0
Polyester cords (L = 12.5 mm)	0	0	0	0	0	30

Note: 1. N-isopropyl N'-phenyl p-phenylene diamine. 2. 1,2-dihydro-2,2,4 trimethyl quinoline. 3. Tetramethyl thiuram disulfide. 4. Benzothiazyl 2-sulphene morpholide.

at a cross head speed of 500 mm/min. For tensile properties, samples were punched out using die E from moulded sheets and were tested according to ASTM D412 method A. For tear strength measurements, standard test pieces (ASTM D624—Type C) were used. Hardness measurements were made according to ASTM D2240 using Shore A durometer. Resilience was tested using a Dunlop Tripsometer at 35°C. For heat build up measurements, Goodrich Flexometer was used according to ASTM D623-28. Abrasion loss was determined using a Dupont abrader and the values were expressed as volume loss per hour. Compression set was determined according to ASTM D395-78. In the stress-strain tests, modulus at 300% elongation could not be taken because in all cases the elongation at break values were much less than 300%.

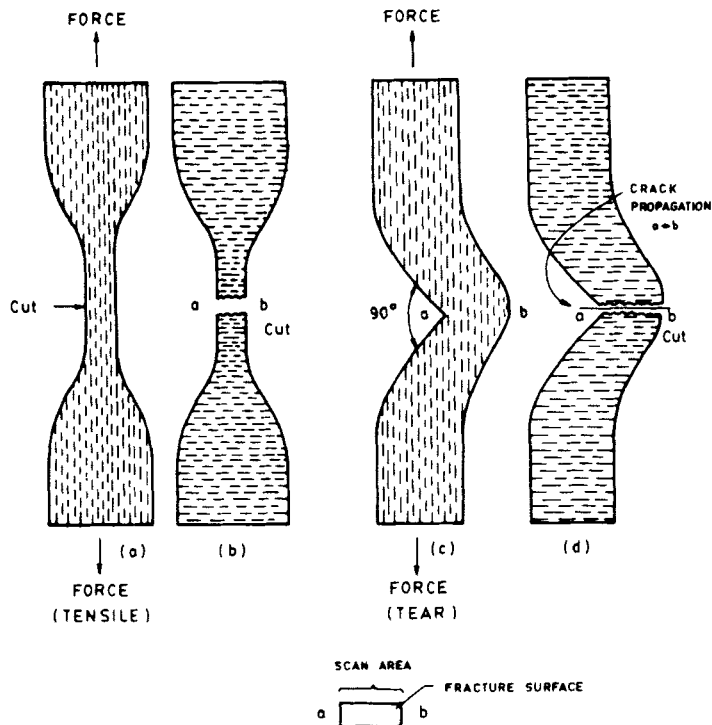


FIGURE 1 Fractured surfaces and scan areas of specimens.

SEM studies of the tensile and tear fracture surfaces were carried out on a scanning electron microscope (model ISI-60). The specimens were sputter coated with gold and then examined under SEM within 24 hours of testing. The angle of tilt of the specimens was 0° to the beam and the orientation of the photographs were kept constant. The scan areas and fractured surfaces of specimens are shown in Figure 1. The lengths of the fibers were found out by means of a travelling microscope and the diameter by an optical microscope to determine the length to diameter (L/D) ratio. L/D of the fibers after mixing was found out by extracing the fibers from the elastomer matrix after dissolving in benzene and drying at 70°C for 1 hour in air oven. The length of the fibers after extraction was measured for a representative sample and the average was taken. Some of the properties of the composites such as tensile strength, tear resistance, and Goodrich heat build up were determined in both longitudinal (along the grain) and in transverse (across the grain) orientations of the fibers.

RESULTS AND DISCUSSION

1. Effect of orientation and concentration of short fibers

1.1. Processing characteristics Table III gives the curing characteristics of the mixes A–F. In the case of the mixes A to E minimum torque and maximum torque values, as expected, increase with fiber concentration. Scorch time decreases with fiber loading. This is attributed to the higher time the stock remains on mill during mixing. As fiber concentration increases, the time of incorporation also increases. So it is quite obvious that the scorch time will decrease. Optimum cure time also decreases with fiber concentration. In the case of mix F, the scorch time is higher than mix D which has the same phr of fiber. This is because of low mixing time. Since the fiber length is higher, the number of fibers to be incorporated is less for a given fiber loading and hence the low mixing time.

1.2. Fiber breakage Table IV gives the lengths of the fibers before and after mixing. It shows that the fibers do not undergo much

TABLE III
Curing characteristics of the mixes

Parameters	Mixes					
	A	B	C	D	E	F
Initial viscosity = L_0 (N.m)	0.8	1.6	1.8	2.5	2.9	2.7
Minimum viscosity = L_1 (N.m)	0.3	0.7	0.7	0.9	1.1	0.7
Thermoplasticity = $(L_0 - L_1)$, (N.m)	0.5	0.9	1.1	1.6	1.8	2.0
Induction time = t_1 , (min.)	6.50	4.85	4.25	4.00	3.60	4.50
Scorch time = t_2 , (min.)	7.25	5.25	4.70	4.50	4.00	5.00
Maximum torque = L_f (N.m)	4.6	6.1	6.5	6.8	7.4	6.7
Optimum cure, (N.m)	4.13	5.56	5.92	6.21	6.77	6.10
Optimum cure time, t_{90} , (min.)	14.00	11.50	9.75	9.00	8.00	12.00
Cure rate, $\frac{100}{t_{90} - t_2}$	14.8	16.0	19.8	22.2	25.0	14.3

TABLE IV
Aspect ratio of polyester fibers

Mix No.	Length of the fibers before mixing (mm)	Length of the fibers after mixing (mm)	Diameter of the fibers		Aspect ratio before mixing (L/D)	Aspect ratio after mixing (L/D)
			before mixing (mm)	after mixing (mm)		
A-E	6.57	5.65	0.0262	0.0249	251	227
F	12.50	11.66	0.0262	0.0222	477	525



FIGURE 2 SEM photomicrograph of a chemically treated polyester cord (40 \times).

breakage during mixing. Figures 2 and 3 give the SEM micrographs of the cord and the fibers after extraction respectively.

1.3. Orientation during milling Table V gives the tensile and elongation at break values for different number of passes of mix D at tight nip in the mill. In the longitudinal direction the tensile strength increases with number passes, the increment being higher

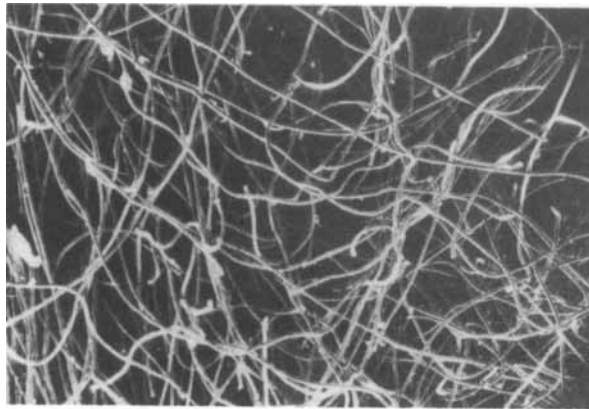


FIGURE 3 SEM photograph of extracted fibers showing the reduced diameter (30 \times).

TABLE V

Variation of stress strain properties with number of passes through tight nip (mix D)

Direction of orientation of fibers	No. of passes through the tight nip	T.S. MPa	E.B. %
L	1	27.2	20
T		6.0	102
L	2	29.6	20
T		8.0	125
L	3	30.1	39.7
T		5.7	128
L	4	30.2	42.8
T		5.7	163

in the second pass. This is because optimum orientation of fibers in the mill direction is effected by passing twice and there is not much improvement in the orientation on further passing. At this stage 60–70% of the fibers will be oriented along the mill direction.^{8,9} In the transverse direction, the tensile strength remains almost constant. Elongation is more in the transverse direction.

1.4. Mechanical properties Table VI gives the mechanical properties of the mixes A–F.

1.4.1. Stress-strain properties Stress-strain curves for the mixes A–F in the longitudinal and transverse directions are given in Figures 4 and 5 respectively. The tensile strength first decreases and then increases with fiber loading in the longitudinal direction, the minimum being at 10 phr (mix B) for a given L/D . At 10 phr loading, the reinforcing effect brought about is insufficient to compensate for the dilution effect of the fibers in the elastomer matrix and hence the decrease in tensile strength. The number of fibers being very small, (mix B), they are unable to deviate the fracture path appreciably and effectively.^{12,13} On the other hand it serves to dilute the rubber matrix. SEM micrograph of the tensile fracture surface (Figure 6) shows scattered fibers on the surface. It is quite insufficient for uniform transmission of stress in the matrix resulting in localised stresses at the fiber matrix interface leading to poor reinforcement.

TABLE VI
Mechanical properties of the composites

Property	Mixes						
	A	B	C	D	E	F	
Tensile strength, MPa	L 22 T 21	13 6	23 7	30 8	22 8	18 7	
Tear strength, N/mm	L 23 T 25	54 62	85 80	116 68	107 97	101 92	
Elongation at break, %	L 650 T 670	30 300	26 150	20 125	32 100	25 125	
Hardness, Shore A	41 55	70 53	85 50	91 43	92 40	91 43	
Resilience, %	1.2	0.72	0.54	0.42	0.30	0.24	
Abrasion loss (cc/hour)	L 9 T 9	7 6	6 5	5 4	5 5	5 4	
Compression set, %							
Goodrich heat build-up T°C, 50°C (longitudinal)	11	34	—	—	—	—	
(a) Time to blowout, min.	—	—	16	10	5	8	
(b) Temp. at blowout, °C	—	—	100	104	102	93	
Goodrich heat build-up °C, 50°C (transverse)	11	—	—	—	—	—	
(a) Time to blowout, min.	—	4	3	2	1.5	4	
(b) Temp. at blowout, °C	—	79	82	83	79	90	
Permanent set, %	L 0.9 T 0.9	4 —	— —	— —	— —	— —	

Note: L = longitudinal orientation, T = transverse orientation.

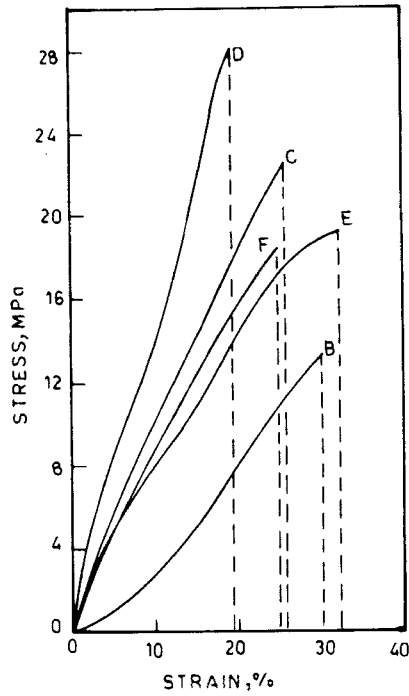


FIGURE 4 Stress-strain relation of polyester fiber natural rubber composites with fibers in longitudinal orientation.

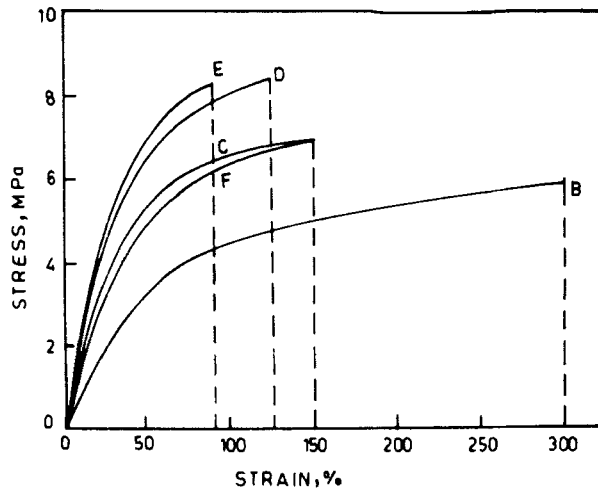


FIGURE 5 Stress-strain relation of polyester fiber natural rubber composites with fibers in transverse orientation.

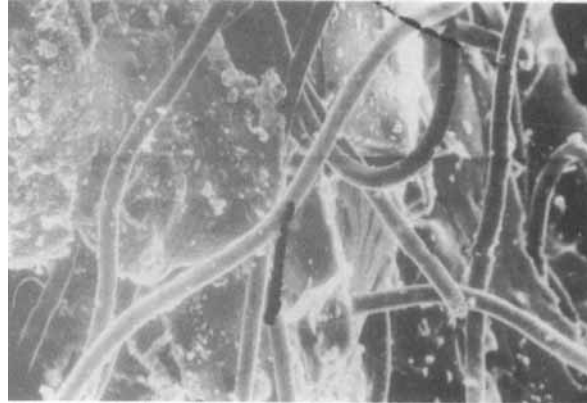


FIGURE 6 SEM photomicrograph of tensile fracture surface of mix B with longitudinally oriented fibers (100 \times).

In the transverse direction, the tensile strength remains almost constant with fiber loading, indicating an insignificant contribution from fibers in carrying the load. Tensile strength in this direction is very small because the fibers, being in a direction parallel to the propagation of the fracture, are unable to give effective hindrance to advancing fracture front. During failure, fiber-matrix debonding occurs when the fiber loading is low as seen in Fig. 7, showing



FIGURE 7 SEM photomicrograph of tensile fracture surface of 10 phr loaded fibers in transverse orientation (100 \times).

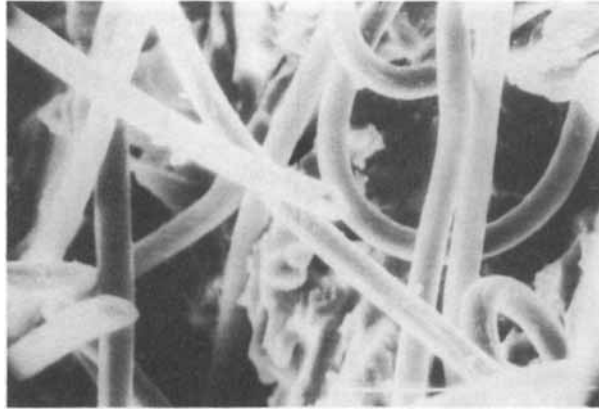


FIGURE 8 SEM fractograph of tension failure surface of mix D with longitudinal oriented fibers (240 \times).

peeled fibers as well as pulled out fibers. At higher fiber concentration in the longitudinal direction, there are more fibers to hinder the fracture front, and the stress is more evenly distributed. Thus tensile strength increases with fiber loading up to 30 phr. With higher fiber loadings the matrix is more restrained as is evident from the fractograph of tensile fracture surface of mix D (Fig. 8). In the transverse direction the tensile strength, which remains almost

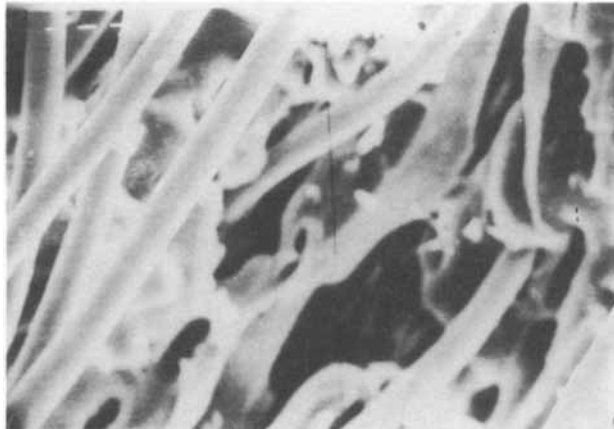


FIGURE 9 SEM fractograph of tension failure surface of mix D with transversely oriented fibers (260 \times).

constant, is very low compared to that in the longitudinal direction. Fiber matrix separation, due to the crack propagation at the fiber-matrix interface, resulting in very low tensile strength is evident from the tensile fractograph of mix D (Figure 9).

At 40 phr fiber loading in the longitudinal direction (Mix E) tensile strength decreases in the longitudinal direction. This may be due to higher stress concentration in the closely spaced fibers. Similar observation have also been made by earlier workers using short jute and silk fibers as reinforcing materials.^{6,7}

Elongation at break in the longitudinal direction drops drastically on addition of 10 phr of fiber (mix B) and drops only marginally by further addition of fiber. In the transverse direction, the fall is not so sharp as in the other direction.

1.4.2. *Tear resistance* Tear resistance is also found to increase with fiber concentration upto 30 phr in the longitudinal direction. As fiber concentration increases, there is more and more hindrance to the propagating tear by the fibers and hence the improved tear strength. In the case of transversely oriented fibers, most of them being parallel to the direction of crack propagation, offers less resistance to the propagating tear. This fact has been further established by the SEM study of the torn surfaces of the tear test specimens. SEM photomicrograph (Fig. 10) shows the tear fracture

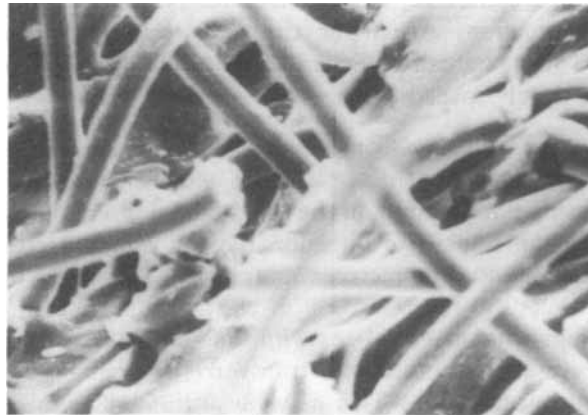


FIGURE 10 SEM photomicrograph of tear fracture surface of mix D with fibers in longitudinal orientation (260 \times).

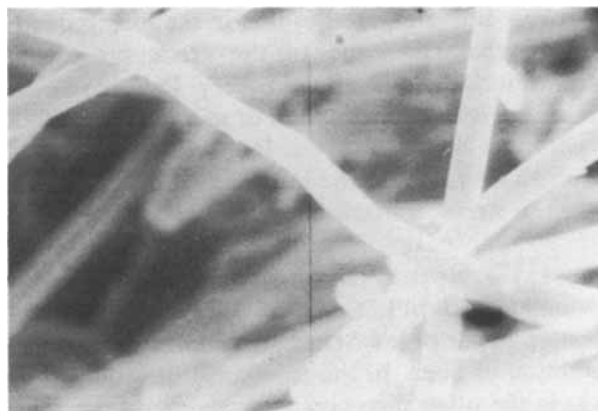


FIGURE 11 SEM photomicrograph of tear fracture surface of mix D with transversely oriented fibers (260 \times).

surface of mix D with the fibers oriented in the longitudinal direction. The dispersed and oriented fibers obviates the tearing process and the tear path terminates in the middle. Because of strain at 90° to the fibers, some of fibers are pulled out of the matrix leaving holes behind and some of the fibers are bent forming loops because of the relaxed strain. Figure 11 shows the SEM photograph of tear fracture surface of mix D with fibers oriented in the transverse direction. At 40 phr the tear resistance is found to decrease. This again can be explained in terms of stress concentration in the closely spaced fibers.

1.4.3. *Compression set* Compression set steadily increases at lower loadings of fibers (Mixes B & C) and then remains constant with increasing fiber loading in the longitudinal direction (Mixes D, E & F). In the transverse direction the set values decrease upto 30 phr (Mix D) and then increase marginally. This slight increment can be attributed to the reduction of elastic property of the rubber matrix at higher loadings. Mixes with longitudinally oriented fibers exhibit more set than those with transversely oriented fibers at the same fiber concentration. This may be attributed to the fact that stress acting along the fiber axis for a long period of time may cause bending of the fibers when they are oriented longitudinally whereas,

in the transverse direction the fibers do not undergo bending since the fiber axis is perpendicular to the stress. Hence set is low.

1.4.4. *Hardness and resilience* Shore hardness of the composites increase as the fibers are introduced and the increase is very rapid upto 30 phr loading of fibers beyond which the change in degree of hardness is very low. However the rebound resilience of the short polyester reinforced natural rubber composites decrease continuously with increase in fiber concentration.

1.4.5. *Heat build up* Figure 12 shows that the hysteresis loss of the composites increase as short fiber loading increases. The heat build up is so high that the test could be completed only for 10 phr loading of fibers in the longitudinal orientation, where as in the transverse orientation of fibers none could be conducted to completion. The samples generated cracks a few minutes after the start and blew out immediately developing large amounts of heat in the system. Table VI shows the temperature rise above 50°C and the

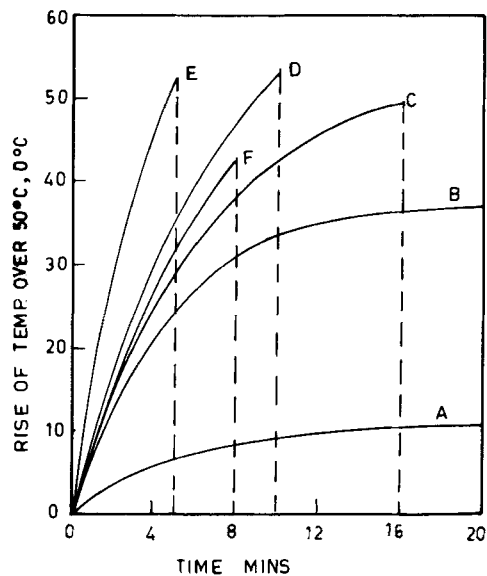


FIGURE 12 Plot of temperature rise above 50°C vs. time for mixes A to F with fibers in longitudinal orientation.

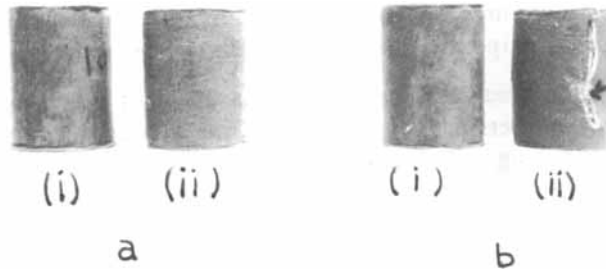


FIGURE 13 Samples before and after heat build up testing of mix B, showing the crack developments and nature of cracks in longitudinal (a) as well as transverse (b) orientations.

time and temperature of blow out of the composites. In the longitudinal orientation of fibers, buckling of the fibers in the matrix prevents fiber breakage or pulling out leading to absorption of greater part of the applied stress in stress-strain cycle.⁷ There is, thus, less residual energy to dissipate as heat, whereas, in the case of transversely oriented fibers, buckling of fibers does not occur and therefore less amount of the applied stress is absorbed leading to generation of more heat. In addition to it, the friction among the fibers leads to further temperature rise. As a consequence of the stiffness of the polyester fibers, flexing is poor and more heat is generated resulting in development of cracks on the surface of the specimen at an early stage of the stress strain cycle. Figures 13 and 14.

After the Goodrich flexometer test, permanent set could be

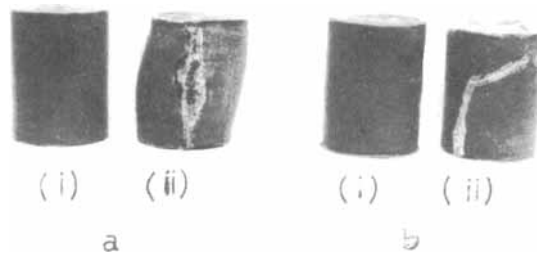


FIGURE 14 Heat build up samples before and after testing for mix D (a) longitudinal orientation (b) transverse orientation.

determined only for one sample, containing 10 phr fibers, in the longitudinal orientation. It is observed that the set property increased by 340 percent on incorporation of 10 phr of short fibers. This is because, the temperature developed with fibers is higher and at higher temperatures, the matrix gets softened and the molecules slip past each other, under stress, irreversibly, resulting in high set values.

1.4.6. *Abrasion loss* Abrasion loss decreases as the fiber concentration increases. Since abrasion loss is a combination of mechanical loss due to repeated shearing and chipping action and the hysteresis loss, the abrasion resistance is directly proportional to the product of specific mechanical loss in an elementary stressing cycle and the fatigue resistance of rubber. This is agreed upon from the results obtained for abrasion loss from compounds A to E. As the fiber concentration increases and hysteresis property deteriorates, the resistance to matrix wear increases, because of very good mechanical strength property of the fibers. However, the mechanical loss is low because of good reinforcement of matrix by the short fibers. Thus, the overall effect i.e. the sum of mechanical loss and hysteresis loss, known as abrasion loss is decreased as fiber concentration is increased.

SEM study of the abraded surface of Mix B (Figure 15) shows the



FIGURE 15 SEM photomicrograph of abraded surface of mix B with ploughing marks and disintegrated fibers (40 \times).

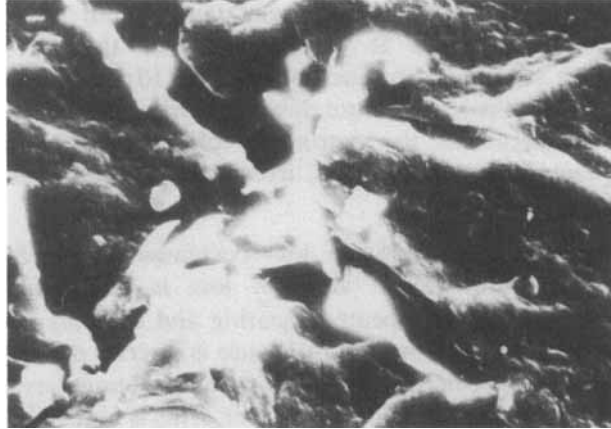


FIGURE 16 Magnified view of the abraded surface of mix B showing the matrix flow and small dimples (340 \times).

disgruntled and mutilated fibers causing ploughing marks on the surface. Minor cracks propagate longitudinally on the surface and fibers are dismantled, supporting the phenomenon of debonding of fibers at lower concentrations (Mix B). On examining the surface closely (Figure 16) it shows matrix flow in the direction of abrasive force and small dimple structures characteristic of natural rubber vulcanizates. The zig zag ridges in the middle simulate the

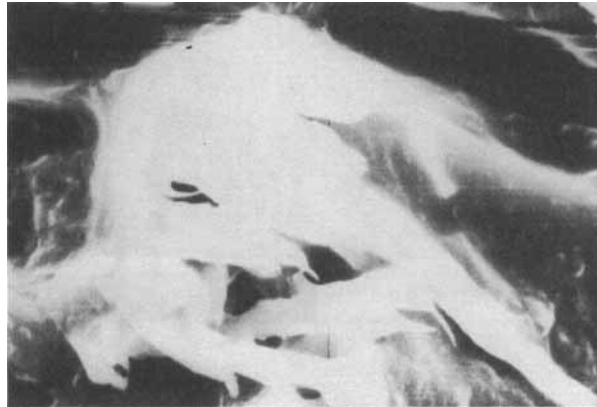


FIGURE 17 Magnified view of a ridge shown in Fig. 16 with tapered and worn out fibers (690 \times).

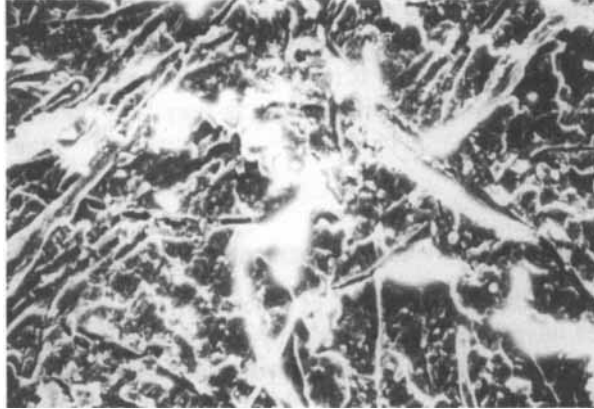


FIGURE 18 SEM photomicrograph of abraded surface of mix D with deep cuts on the surface (70 \times).

ribbed structure of filled natural rubber vulcanizates.¹⁴ A magnified view of one such ridge (Figure 17) shows the worn out fibers pointing away from the direction of abrasive force. Sometimes this causes overlapping of the worn out fiber ends. This shows that the fibers actually obstruct the wearing process and undergo strenuous cycles restricting the wear of fiber rubber composites. This type of wear may be characterised as that in between abrasive wear and frictional wear.

SEM photomicrograph (Figure 18) of the abraded surface of mix



FIGURE 19 Magnified view of abraded surface of mix D (300 \times).

D shows random fissures and deep cuts on the surface. There is an indication of flow of matrix in the direction of the abrasive force. The long furrows on the surface may be caused by the sharp abrasive surface and fiber dismantling is very less. There are small dimple structures which become evident on magnification (Figure 19).

2. Effect of change in aspect ratio of the fibers

The effect of the aspect ratio of fibers on the properties of the composites have been investigated by many researchers.¹⁵⁻¹⁷ Mixes D and F, contains 30 phr of fibers having an aspect ratio of 251 and 477 respectively before mixing. After mixing the corresponding values are 227 and 525.

2.1. Processing characteristics Table III shows that the minimum torque, maximum torque and optimum cure time are reduced slightly and scorch time is increased by increasing L/D ratio. The increment in the scorch time is due to the reduced mixing time. Since the number of fibers, to be incorporated is less, at high fiber length, for a given amount of fiber, the mixing time is reduced.

2.2. Mechanical properties

2.2.1. Stress-strain properties Figures 4 and 5 show the stress strain curves of the Mixes D and F in the longitudinal and transverse directions respectively. Tensile strength is lowered and elongation at break values are increased in the longitudinal direction by increasing L/D . This is due to more entanglements, as the fiber length is more, which will result in less orientation during milling and hence poor transfer of stress between the rigid fibers and the soft matrix.¹⁸ When the fibers are not in the same direction as the applied stress and form an angle with it, failure of the rubber-fiber interface is facilitated. This causes reduction in the stress value.

2.2.2. Tear resistance Tear resistance decreases in the longitudinal direction and increases in the transverse direction. This is due to more entanglements caused by higher length of fibers. In the longitudinal direction, due to the entanglements, the hindrance to

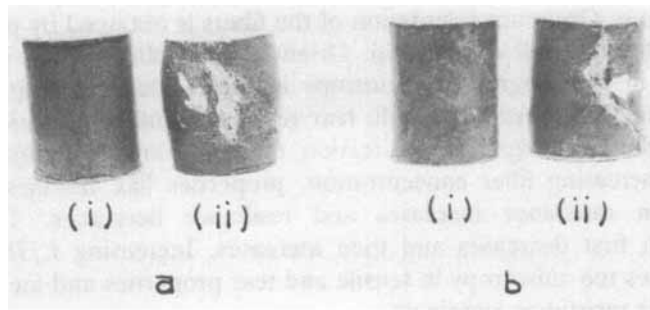


FIGURE 20 Heat build up samples of mix F before and after testing (a) longitudinal and (b) transverse orientation of fibers.

the propagation of tear lines, by the longitudinal fibers becomes less since the orientation is less, as is evident from the tensile value. In the transverse direction, due to entanglements there will be more hindrance to tear lines propagation. Thus, an increase in the aspect ratio of fibers categorically decreases the anisotropic character of the composite.

2.2.3. Other mechanical properties The properties like hardness, compression set, and resilience remained unaffected by the change in the aspect ratio of fibers. Abrasion resistance of the composites increases marginally as the aspect ratio increases. In the heat build up test, the samples cracked and were blown out before the test was complete. The development of cracks after 4 minutes of testing is shown in Figure 20.a(ii) showing the samples with longitudinal orientation of fibers and in b(ii) showing the samples with transverse orientation of fibers. In sample a(ii) the specimen bulges in the middle and a deep vertical crack is developed along the direction of fiber orientation on application of the cyclic stress. But in sample b(ii) the crack deviates at an angle of 90° from the direction of application of stress because of transverse orientation of fibers and more than one crack is generated (not visible in the photograph).

CONCLUSIONS

Mechanical properties of short polyester fiber reinforced natural rubber composites vary with fiber orientation, concentration and

L/D ratio. Optimum orientation of the fibers is obtained by passing twice through mill at tight nip. Orientation of fibers in the matrix results in high degree of anisotropy in the mechanical properties. Properties like tensile strength, tear resistance and compression set are higher in longitudinal direction than in transverse direction. With increasing fiber concentration, properties like hardness and abrasion resistance increases and resilience decreases. Tensile strength first decreases and then increases. Increasing *L/D* ratio decreases the anisotropy in tensile and tear properties and increases abrasion resistance marginally.

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