Short Coir Fiber-Reinforced Natural Rubber Composites: Effects of Fiber Length, Orientation, and Alkali Treatment

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SYNOPSIS

Natural rubber is reinforced with untreated coir fiber chopped to different lengths, viz., 6, 10, and 14 mm. Mixes were also prepared using 10 mm-long coir fibers treated with 5% sodium hydroxide solution for different time intervals, viz., 4, 24, 48, and 72 h. These composites were vulcanized at 150°C. The vulcanization parameters, processability characteristics, and stress-strain properties of these composites were analyzed. The rubber-coir interface bonding was improved by the addition of a resorcinol-hexamethylenetetramine dry-bonding system. The reinforcing property of the alkali-treated fiber was compared with that of the untreated one. The extent of fiber orientation in the composite was determined from green strength measurements. From anisotropic swelling studies, the extent of fiber alignment and the strength of fiber-rubber interface adhesion were analyzed. Scanning electron microscopic studies were carried out to investigate the fiber surface morphology, fiber pullout, and fiber-rubber interface. © 1995 John Wiley & Sons, Inc.

INTRODUCTION

Composites in which short fibers are oriented uniaxially in an elastomer have a combination of good strength and stiffness from the fibers and elasticity from the matrix. These materials are used for the fabrication of a wide variety of products such as Vbelts,¹ hoses,² and complex-shaped articles.³ Studies on composites containing natural fibers are important because of their renewable nature, low cost, and amenability to chemical modification.

Many researchers have used short glass fibers for reinforcing rubbers because of their high modulus, high strength, and low creep.^{4,5} Coran et al.⁶ studied the morphological and mechanical properties of oriented cellulose fiber-reinforced elastomeric composites. Murty and De⁷ conducted studies on short jute fiber-reinforced natural rubber composites. Setua and De⁸ reported on the effect of bonding agents in short silk fiber-reinforced natural rubber composites. Recently, in this laboratory, short pineapple⁹ and sisal fiber have been successfully used for the reinforcement of natural rubber¹⁰⁻¹³ and low-density polyethylene¹⁴⁻¹⁸ and thermosets.¹⁹

Coir is a lignocellulosic fiber obtained from the fibrous mesocarp of coconuts, the fruit of coconut tree (cocos nucifera) cultivated extensively in the tropics. India and Srilanka account for nearly 90% of the world's coir production. Coir is a hard fiber renowned for durability because of its high lignin content (41–45%) compared to other natural fibers. The high wet strength values of coir is advantageous when the product is used in humid conditions. Also, the cost of coir fiber is comparatively less among the other natural fibers. Apart from the conventional uses of coir fiber as cordage and floor-furnishing materials, research has been progressing for the last few years to determine the efficiency of coir as a reinforcement in clay, cement, and polymers.

Owolabi et al.²⁰ used coir fiber as a reinforcing filler for plastics. The tensile strength, flexural strength, and water absorption of coir-polyester composites were improved by the addition of glass fibers.²¹ Very few studies have been reported using coir fiber as reinforcement for elastomers. Arumugam et al.²² investigated the effect of different bonding agents on the physical properties of coir fiberreinforced natural rubber composites. However, they

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did not investigate the effect of fiber length, fiber distribution, orientation, and fiber breakage.

Design of a short fiber-reinforced rubber composite depends on several factors such as the aspect ratio of the fiber, control of fiber orientation and dispersion, and existence of a strong interface between fiber and rubber.

This study deals with the optimization of the coir fiber length in natural rubber composites. To modify the fiber surface, the coir fiber was subjected to alkali treatment. The influence of alkali treatment, bonding agent, and fiber orientation on the mechanical properties was analyzed. An attempt was made to determine the extent of fiber orientation from the green strength measurements. Also, studies of equilibrium swelling in hydrocarbon solvent were carried out to obtain additional information on the fiber orientation and anisotropy of the composites.

EXPERIMENTAL

Materials

Coir fiber was supplied by the local processing unit in Kollam. It is reported to contain cellulose 36-43%, lignin 41-45%, hemicellulose 0.15-0.25%, and pectins 3-4% together with some water soluble materials.²³ It has an average diameter of 100-400 μ m and a specific gravity of 1.15. Natural rubber used for the study was ISNR 5 (light color) grade obtained from Rubber Research Institute of India, Kottayam. All other ingredients were of commercial grade.

Ingredients	Gum	А	В	с	D	\mathbf{E}	F	G	н
Natural rubber	100	100	100	100	100	100	100	100	100
Stearic acid	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
Zinc oxide	5	5	5	5	5	5	5	5	5
T.D.Q.ª	1	1	1	1	1	1	1	1	1
Resorcinol		_		_	8.75	8.75	8.75	8.75	8.75
Hexa ^b					5.6	5.6	5.6	5.6	5.6
C.B.S. ^c	0.6	0.6	0.6	0.6	0.6	0.6	0.6	0.6	0.6
Sulfur	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
Coir fiber	_	35	35	35	35				
(untreated)		(6 mm) ^d	(10 mm) ^d	(14 mm) ^d	(10 mm) ^d				
Coir fiber						35	35	35	35
(5% NaOH treated)						4 h	24 h	48 h	72 h
10 mm long							(treatme	ent time)	

Table I Formulation of Mixes

Fiber Preparation

Coir fiber was first separated from undesirable foreign matter and pith material. Then, it was chopped to different lengths, viz., 6, 10, and 14 mm. The coir fibers of length 10 mm were treated with a 5% sodium hydroxide solution for different time intervals, viz., 4, 24, 48, and 72 h. Finally, the fibers were repeatedly washed with water and air-dried.

Preparation of Composites

The formulations of mixes are given in Table I. The composite materials were prepared in a laboratory two-roll mill (150×300 mm). The nip-gap, mill roll speed ratio, and the number of passes were kept the same in all mixes. The samples were milled for sufficient time to disperse the fibers in the matrix at a mill opening of 1.25 mm. The bonding system, consisting of resorcinol and hexamethylenetetramine, was incorporated along with other ingredients. The fibers were incorporated at the end of the mixing process, taking care to maintain the direction of compound flow, so that the majority of fibers followed the direction of the flow.

Measurement of Properties

The fiber breakage analysis was carried out by dissolving 1 g of the uncured composite in toluene, followed by the separation of fibers from the solution. Then, the distribution of fiber length was determined using a traveling microscope.

^a 2,2,4-Trimethyl-1,2-dihydroxy guinoline polymerized.

^b Hexamethylenetetramine.

 $^{\circ}N$ -Cyclohexyl-2-benzothiazyl sulfenamide.

^d Values in parentheses show the fiber length.

Green strength values were determined, using dumbbell-shaped samples obtained from unvulcanized composites, on a Zwick Universal Testing machine at a stretching rate of 500% per minute.

Curing properties were measured in a Monsanto R-100 rheometer, at a temperature of 150°C. The composites were cured at their respective cure times as given in Table II. Dumbbell-shaped tensile specimens with longitudinal and transverse fiber orientations were punched out from the vulcanized sheets.

Stress-strain measurements were carried out at a crosshead speed of 500 mm per minute. Tensile strength and tear strength were measured according to ASTM methods D412-68 and D624-54, respectively. The tests were carried out both along and across the grain direction.

The anisotropic swelling studies were carried out after an immersion period of 48 h in toluene. The scanning electron microscopic (SEM) studies of fibers and composites were carried out using a JEOL scanning electron microscope.

RESULTS AND DISCUSSION

Fiber Breakage Analysis

The fiber-length distribution curve of composite B, which contains untreated coir fibers of length 10 mm, is given in Figure 1. It is clear that the majority of fibers have length in the range of 6-10 mm. This shows that the breakage of coir fibers is comparatively low when it is used as a reinforcing fiber in natural rubber composites. Synthetic fibers like glass and carbon undergo severe breakage during milling. O'Connor²⁴ studied the processing and properties of rubber composites containing different fibers and

Table II Vulcanization Parameters

Mix	$T_{ m min}$ (dNm)	$T_{ m max}$ (dNm)	ΔT (dNm)	t ₉₀ (min)
Gum	7	56	49	15.0
A (6 mm)	4	61	57	11
B (10 mm)	5	58.7	53.7	9.5
C (14 mm)	3.5	56	52.5	10.5
D	6.5	81	80.9	16
E	7	98.5	91.5	9.5
F	8	86.5	78.5	10.5
G	6	84	78	9.0
Н	6	99.8	93.8	9.4

showed that glass and carbon are poor in improving mechanical properties compared to cellulosic fibers. This has been explained in terms of the severe breakage incurred by the synthetic fibers during milling. Due to the intrinsically flexible nature of cellulosic fibers, they can bend and curl during milling and thus avoid severe breakage.

Effect of Fiber Length

Effect of Fiber Length on Vulcanization Characteristics

The rheographs of the mixes: gum, A, B, and C, are given in Figure 2. The initial decrease in torque to a minimum value is due to the softening of the rubber matrix. The drastic increase in torque after the minimum value is due to the cross-linking of the rubber phase. The curves cross over each other initially because of the differences in their cure rates. From Table II we can see that the maximum torque value, $T_{\rm max}$, decreases gradually with increase in fiber length. The highest viscosity of the mix containing coir fibers of length 6 mm can be attributed to the increased number of fiber ends, which enhance the overall friction of the compound.

From the rheographs, it is clear that after about 13 min the torque shows a constant value with time. At longer times, viz., after about 25 min, the torque values show a slightly decreasing trend with time because of the degradation of the rubber molecules and/or coir fibers.

Effect of Fiber Length on Mechanical Properties

The modulus and strength properties of short fiberreinforced rubber composites depend on the degree to which an applied load is transmitted to the fibers. The extent of load transmittance from matrix to fiber is a function of fiber length and the magnitude of the fiber-matrix interfacial bond. In fiber-reinforced composites, there exists a critical fiber length at which the load transmittance from the matrix to fiber is maximum. The critical fiber length is that fiber length which is required for the fiber to develop its fully stressed condition in the matrix. Thomas and co-workers^{10,14} determined the critical fiber length for different fiber/polymer systems. In the case of sisal fiber-reinforced natural rubber composites, the critical length was 10 mm. For sisal/ LDPE system, the critical fiber length was 6 mm.

The effect of fiber length on tensile moduli at different elongations, viz., 10 and 20%, is shown in Table III. The tensile moduli in the longitudinal direction increased rapidly and linearly as the length



Figure 1 Fiber distribution curve.

of the fibers was increased up to 10 mm length. The modulus at 20% elongation was almost constant after 10 mm length. But the modulus at 10% elongation reached a maximum for the composite containing coir fibers of length 10 mm and then decreased with increasing fiber length. However, the tensile modulus in the direction perpendicular to the orientation of fibers decreased considerably and more or less linearly as the length of the coir fibers increased.

As compared to that of the gum compound, the tensile strength decreased drastically up to the composite containing coir fiber of length 6 mm and then increased gradually reaching a maximum for the composite containing 10 mm-long fibers, as shown in Table III. Further increase in fiber length reduces the tensile strength due to fiber entanglement. This suggests that the critical fiber length is 10 mm for effective tensile stress transfer between fiber and matrix. The same trend can be seen in the case of tearstrength properties where the values show maxima at 10 mm fiber length in transverse and longitudinal directions. Addition of coir fiber decreases the elongation at break values of natural rubber composites. This is associated with the brittle nature of the fiber. Among the filled systems, the elongation at break value shows a maximum for 10 mm fiber length. The overall mechanical performance of the composite indicates that the critical coir fiber length is 10 mm in natural rubber composites.

Effect of Alkali Treatment/Bonding Agent

Surface Morphology of Coir Fiber

Good interfacial strength between fiber and rubber is the essential factor to achieve good fiber reinforcement. The interfacial strength depends on the surface topology of the fiber. Although coir fiber



Figure 2 Rheographs of the mixes: gum, A, B, and C.

possesses hydroxyl groups on its surface, its brittle nature and low cellulose content makes it a less effective reinforcement. Hence, to improve adhesion between coir fiber and natural rubber, it should be subjected to some sort of pretreatment. So, the mixes, E, F, G, and H, are prepared using coir fibers treated with sodium hydroxide having a concentration of 5%. To improve the fiber-rubber adhesion, a resorcinol-hexamethylenetetramine bonding system is added in mixes D, E, F, G, and H.

The surface topology of coir fiber was studied by SEM, which indicates an uneven surface as shown in Figure 3(a) and (b). It can be observed that certain globular protrusions are embedded on the fiber sur-

			Α	В	С	
Properties	Orientation	Gum	(6 mm)	(10 mm)	(14 mm)	
Modulus						
at 10% strain (MPa)	L	0.31	1.61	1.88	1.70	
	T	0.31	0.60	0.55	0.42	
at 20% strain (MPa)	L	0.42	1.57	1.69	1.60	
	T	0.44	0.79	0.70	0.53	
	L	17.95	5.74	6.69	5.46	
Tensile strength (MPa)	T	20.06	3.71	4.35	3.62	
	L	35.05	25.70	30.07	29.46	
Tear strength (kN/m)	T	37.93	21.39	25.20	23.37	
	L	1119	726	777	731	
Elongation at break (%)	T	1141	665	708	677	

Table III Effect of Fiber Length on Mechanical Properties of Mixes



а



b

Figure 3 (a) SEM of the surface of raw coir fiber: globular protrusions can be seen. (b) Magnified view of untreated coir fiber.

face. These have been identified as silicified stigmata, which are minute thickenings. Similar surface morphology has also been reported by other workers.²⁵

When the coir fiber was treated with 5% sodium hydroxide, the fiber changed color from pale yellow to brown. Fibers became thinner upon alkali treatment. This may be because of dissolution and leaching out of fatty acids, phenolic compounds, and their condensation products by alkali, which form the waxy cuticle layer or even the lignin component of the fiber. The changes in surface morphology of alkali-treated coir fiber for 48 h can be seen in Figure 4(a) and (b). Globular protrusions present in the untreated fiber surface disappeared, leading to the formation of a large number of voids. These voids promote better mechanical anchorage between fiber and rubber matrix. Loss of cuticle by the rupture of alkali-sensitive bonds leads to a rough surface.

SEM of the fractured end of the alkali-treated fiber in Figure 5 shows that the fibrillar structure



а



b

Figure 4 (a) SEM of alkali-treated coir fiber: Voids are produced by the removal of globular protrusions. (b) Magnified view of alkali-treated coir fiber.



Figure 5 SEM of fractured end of alkali-treated coir fiber: shows the fibrillar structure of the fibers.

of the fibers remains unchanged even after the alkali treatment. This indicates that a substantial amount of binding material is still present after the alkali treatment.

Vulcanization Characteristics

In Figure 6, the rheographs of mixes B, D, and G are compared. The higher $T_{\rm max}$ of composites D and G than that of B is due to the presence of the bonding system in the first two mixes. But it is interesting to observe that the $T_{\rm max}$ value of mix G is higher than that of mix D, which implies a higher cross-link density of the former, which may be caused by the alkali treatment given to the coir fibers in mix G.

Processability Parameters

Mooney scorch time values are given in Table IV. The large difference in scorch time of gum (29 min 48 s) and compounds D, E, F, G, and H may be due to the alkaline nature of the methylene donor, hexamethylenetetramine, present in the bonding system. There is, however, no remarkable change in scorch time of compounds D, E, F, G, and H. This indicates that the duration of alkali treatment has a negligible effect on scorch time.



Figure 6 Rheographs of the mixes B, D, and G.

Properties	Orientation	D	E (4 h)	F (24 h)	G (48 h)	H (72 h)
Mooney scorch at 120°C	-	5 min 20 s	5 min	5 min 3 s	4 min 7 s	5 min 12 s
Modulus at 20% strain (MPa)	$L \ T$	4.31 1.66	$\begin{array}{c} 4.47 \\ 1.60 \end{array}$	5.9 2.59	$6.28 \\ 1.97$	$5.75 \\ 1.83$
Tensile strength (MPa)	$L \ T$	4.50 2.81	$\begin{array}{c} 4.65\\ 3.11\end{array}$	$\begin{array}{c} 6.47\\ 4.18\end{array}$	6.98 3.98	$6.08 \\ 3.68$
Tear strength (kN/m)	$L \ T$	37.00 35.35	$\begin{array}{c} 48.58\\ 41.02 \end{array}$	$56.08 \\ 47.02$	54.38 42.43	49.83 45.01
Elongation at break (%)	$L \ T$	201 142	107 193	43 99	44 128	73 191

Mechanical Properties

The tensile moduli of the composites containing 35 phr coir fiber as a function of soaking time in 5%sodium hydroxide is shown in Table IV. It is seen that the tensile moduli of longitudinally oriented composites at 20% elongation and tensile strength increase with soaking time and reach a maximum at 48 h followed by a decrease. The tear strengths in both directions show maxima at 24 h NaOH treatment. This is associated with the better interaction between the treated fiber and rubber. However, prolonged treatment up to 72 h decreases the properties marginally. This may be due to the excessive removal of binding material such as lignin, hemicellulose, etc., on prolonged immersion in the alkali solution. This decreases the fibrous properties of coir fibers, and as a consequence of this, the properties are decreased. This trend is true in both transverse and longitudinal directions.

The stress-strain curves of mixes B and G are shown in Figure 7. Here, we can see that mix G has higher tensile strength and lower elongation at break than those of mix B. This confirms the better performance of composite G due to the combined effect of bonding system and alkali treatment given to coir fibers in mix G.

Fiber-Rubber Adhesion

Figure 8 shows the SEM of the fractured end of the tear specimen of the mix B containing untreated coir fibers without a bonding agent, and Figure 9, that of mix G containing alkali-treated coir fibers along with the bonding agent. In Figure 8, one can see a large number of holes that are produced as a result of the pullout of fibers from the rubber matrix.

This pullout of fibers is evidence of poor fiber-rubber adhesion. In Figure 9, broken fiber ends can be seen, instead of the pullout phenomenon. The cracks on the fiber ends support the fact that the fibers have undergone more breakage rather than pullout, which affirms a better interfacial strength. Also, the higher tear strength of mix G (54.38 kNm⁻¹), compared to that of mix B (30.7 kNm⁻¹), as given in Tables III and IV, support the above-mentioned observation.

The increased wettability and the enhanced fiberto-rubber adhesion in composites containing alkalitreated coir fiber could be observed from the gradation in roughness of tear-fractured specimens of the three types of composites, i.e., mixes B, D, and G. The rough surface of tear-fractured specimen of mix B, which contains untreated coir fiber without any bonding agent, shows poor adhesion between coir fiber and matrix. In this sample, the fibers were protruding out of the matrix as a result of the tear deformation. The pullout of fibers and surface roughness were comparatively low in mix D, which contains untreated fiber along with the bonding agent. The tear sample of mix G, which contains alkali-treated coir fibers along with the bonding agent, showed a very smooth surface. Here, pullout of fibers was completely absent. This supports the enhanced interfacial strength in mix G.

Fiber Orientation

Extent of Fiber Orientation

From the green strength measurements, we can obtain an idea about the anisotropic nature of these composites. The alignment of fibers in the flow direction increases the green strength in the longi-



Figure 7 Stress-strain curves of mixes B and G.

tudinal direction. The percentage orientation of coir fibers in the composite can be calculated using the following equation²⁶:

% Orientation =
$$\frac{S_L/S_{G,L}}{S_L/S_{G,L} + S_T/S_{G,T}}$$
 (1)

where S denotes the green strength of the composite and subscripts L, T, and G denote longitudinal, transverse, and gum compounds, respectively.

The extent of orientation of coir fibers in composites E, F, G, and H is given in Table V. It is observed that the orientation of coir fiber is at a



Figure 8 SEM of fractured end of tear specimen of mix B: shows holes produced by fiber pullout.



Figure 9 SEM of fractured end of tear specimen of mix G: shows no fiber pullout, which indicates better fiber-rubber adhesion.

	Green Stren			
Mix	Longitudinal	Transverse	Orientation (%)	
Gum	0.232	0.2475	_	
E	0.826	0.482	64.64	
F	1.64	0.462	79.10	
G	1.45	0.362	81.03	
н	1.15	0.34	78.30	

Table V Green Strength and Orientation

maximum in composite G, which contains coir fiber treated with sodium hydroxide for 48 h.

Effect of Fiber Orientation on Mechanical Properties

Tables III and IV show the effect of coir fiber orientation on the mechanical properties such as tensile moduli, tensile strength, tear strength, and elongation at break.

The higher mechanical properties in the longitudinal direction show the anisotropic behavior of coir fiber-reinforced natural rubber composites. When the fibers are oriented in the longitudinal direction, the mechanical property values are much higher than those in the transverse direction. Greater hindrance to the fracture front is experienced, when the fibers are oriented longitudinally (i.e., perpendicular to the fracture surface). Breakage and pullout of fibers take place mainly when the fibers are oriented in the longitudinal direction, whereas for transversely oriented fibers, the crack progresses in the direction of fiber alignment. The transversely oriented fibers act as barriers that prevent the distribution of stress throughout the matrix. This causes a higher concentration of localized stress and results in both poor and variable mechanical property values.

Anisotropic Swelling

The swelling behavior of fiber-reinforced elastomeric composites has been studied by Das.²⁷ Recently, swelling studies have been conducted using natural rubber composites with acetylated sisal fiber by Varghese et al.²⁸ The swelling studies in a solvent provide information on the resistance of an interface, the degree of dispersion of the fibers, and their alignment in the elastomeric matrix.²⁹

Table VI shows the swelling ratios of gum and composites B, D, and G. In the direction of the fiber orientation, a_L (see Fig. 10), the longitudinal swelling ratios of D and G are close to unity. This indicates the existence of a strong interface.

The a_T values (the transverse swelling ratios) of the gum and composite B show that perpendicular to the preferred orientation the swelling is independent of the presence of fibers and the value is equal to that of matrix. These values are, however, lower for composites D and G. The very close a_L values and the same a_T values of mix B and gum indicate that the fiber-matrix adhesion is practically nil in mix B, which contains untreated fiber without the bonding agent. The a_Z values indicate the swelling ratio in the Z direction. The difference between the a_T and a_Z values implies that the orientation of the fibers is not perfectly unidirectional, which may arise due to the flow produced during molding.

According to Coran et al.³⁰ for short fiber-reinforced rubber composites, the swelling ratio a_{θ} in any direction forming an angle θ with the fiber orientation is given by the expression

$$a_{\theta}^2 = (a_T^2 - a_L^2) \operatorname{Sin}^2 \theta + a_L^2$$
 (2)

where a_L and a_T are the dimensional swelling ratios in the longitudinal and transverse directions, respectively. Figure 11 shows dimensional swelling variation with angle θ in accordance with eq. (2) where various values of θ were assumed. The swelling increases with θ for all compounds, which confirms a preferential fiber orientation in the grain direction.



Figure 10 Schematic diagram of fiber-reinforced composite.



Figure 11 Dimensional swelling variation with angle θ between measurement direction and fiber orientation.

It can also be observed that the curve for composite B is only slightly lower than that of gum and it is even overlapping with the latter at higher θ values. This indicates that the swelling behavior of gum and B are roughly equal.

The anisotropic swelling studies provide an idea about the extent of fiber alignment. Table VI shows the slopes of gum and composites B, D, and G. It is reported that the steeper the line the higher the degree of fiber alignment.³¹ The line corresponding to composite D has the highest slope. This indicates that the fiber orientation is highest in composite D. However, the line corresponding to mix D is above that of mix G. This shows that the restriction to swelling is higher for mix G, which contains alkalitreated coir fibers, than that of mix D, which contains untreated coir fibers. This indicates that alkali treatment improves the interfacial bond strength.

CONCLUSION

The processability characteristics and mechanical properties of coir fiber-reinforced natural rubber

composites have been investigated as a function of fiber length, orientation, and surface treatment. The mechanical properties of the composites in the longitudinal direction are superior to those in the transverse direction. The optimum length for coir fiber was found to be 10 mm in order to achieve good reinforcement in natural rubber composites.

The surface morphology of coir fiber is modified by sodium hydroxide treatment. From the mechanical properties, it was observed that coir fibers should be immersed in 5% sodium hydroxide solution for 48 h for maximum tensile properties. The extent of fiber orientation was estimated from the green strength measurements. The fiber orientation was maximum for composites containing coir fiber treated in NaOH for 48 h. Scanning electron microscopic studies revealed better adhesion between alkali-treated coir fibers and natural rubber.

Anisotropic swelling studies indicated poor adhesion between untreated coir fiber and natural rubber. The swelling is smaller in composites containing alkali-treated coir fiber along with the resorcinolhexamethylene-tetramine bonding agent. An idea about the extent of fiber orientation in these composites was also obtained from the anisotropic swelling studies.

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