

Cure Characteristics and Mechanical Properties of Short Nylon Fiber-Reinforced Nitrile Rubber Composites

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ABSTRACT: Acrylonitrile butadiene rubber (NBR)-based composites were prepared by incorporating short nylon fibers of different lengths and concentration into the matrix using a two-roll mixing mill according to a base formulation. The curing characteristics of the samples were studied. The influence of fiber length, loading, and rubber crosslinking systems on the properties of the composites was analyzed. Surface morphology of the composites has been studied using Scanning Electron Microscopy (SEM). Addition of nylon fiber to NBR offers good reinforcement, and causes improvement in mechanical properties. A fiber length of 6 mm was found to be optimum for the best balance of properties. It has been found that at higher fiber loadings, com-

posites show brittle-type behavior. Composites vulcanized by the dicumyl peroxide (DCP) system were found to have better mechanical properties than that by the sulfur system. The swelling behavior of the composites in *N,N*-dimethyl formamide has been analyzed for the swelling coefficient values. Composites vulcanized in the DCP system were found to have higher rubber volume fraction than that in the sulfur system, which indicates better rubber–fiber interaction in the former. The crosslink densities of various composites were also compared. © 2004 Wiley Periodicals, Inc. *J Appl Polym Sci* 92: 1023–1030, 2004

Key words: nylon; fibers; rubber; composites

INTRODUCTION

Fiber-reinforced rubber composites are of immense importance both in the industrial field and in the area of research and development.^{1–4} Reinforcement of rubber with short fiber combines the elasticity of rubber with strength and stiffness of the fiber. The additional benefit is that the fiber is incorporated into the compound as one of the ingredients of the recipe, and hence, they are amenable to the standard rubber processing steps of extrusion, calendaring, and various types of molding. Short fibers are also used to improve or modify certain thermodynamic properties of the matrix for specific applications or to reduce the cost of fabricated articles.⁵ The properties and performance of short fiber-reinforced rubber composites depend on several factors such as nature and concentration of the fiber, its aspect ratio, orientation, and the degree of adhesion of the fiber to the rubber matrix. Various synthetic fibers such as glass, rayon, nylon, asbestos, aramid, and cellulose have been studied as reinforcement in both natural and synthetic rubber matrices.⁶ Effect of aramid, glass, and cellulose short fibers on the processing behavior, crosslink density, and me-

chanical properties of natural rubber, styrene–butadiene rubber, and ethylene propylene diene terpolymer rubber has been investigated by Manchado and Arroyo.⁷ Tensile and tear properties of short fiber-reinforced chloroprene rubber have been studied by Ryu and Lee.⁸ The effect of filler loading and bonding agent on bamboo fiber-filled natural rubber composites has been studied by Ismail and coworkers.⁹ Use of sisal and coir fibers as reinforcing fillers for NR has been investigated by Varghese et al.¹⁰ and Geetamma et al.¹¹ Prasantha Kumar et al.¹² studied the use of sisal as reinforcing fillers in SBR matrix. Nune and Afonso¹³ studied the interaction of NBR with cellulose fiber. Ashida and coworkers^{14,15} investigated the dynamic properties of various fiber–rubber composites. The general mechanical properties of cellulose fiber in NR, CR, and SBR have been investigated by Subic et al.¹⁶ Setua and coworkers^{17–22} studied short silk fiber as a reinforcement for rubber. Murthy²³ studied the reinforcing effect of fiberglass on the physicomachanical properties of NR. Short glass fibers were used for reinforcing rubber due to their high modulus, high strength, and low creep.²⁴ Ibarra and Chamorro^{25,26} investigated the mechanical properties of carbon, polyester, glass, polyamide, and cellulose in ethylene propylene diene monomer (EPDM), natural rubber (NR), chloroprene rubber (CR), nitrile rubber (NBR), and styrene–butadiene rubber (SBR) matrices. Coran et al.²⁷ have investigated the effect of various fibers

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TABLE I
Physical Properties of Nylon Fiber

Physical property	Nylon fiber
Density (g cm^{-3})	1.14
Diameter (μm)	25.0
UTS ^a (GPa)	0.9
Modulus (GPa)	4.9

^a Ultimate tensile strength.

such as glass, nylon, rayon, and cellulose in both natural and synthetic rubber matrices.

Acrylonitrile butadiene rubber, commonly known as nitrile rubber (NBR), is a synthetic rubber that possesses good heat and oil resistance. It has a wide range of product applications. Reinforcement of NBR matrix with suitable fibrous fillers can considerably improve its physical properties. The present study deals with the curing characteristics, mechanical properties, and swelling behavior of short nylon fiber-reinforced NBR composites, with special attention to the effects of fiber length, fiber loading, and rubber crosslinking systems.

EXPERIMENTAL

NBR was obtained from Apar Industries, Mumbai, India. It is reported to contain 35% acrylonitrile. Nylon fiber was obtained from SRF Polymers Limited, Chennai, India, in yarn form. Physical properties of nylon fiber used are given in Table I.

Formulations of the mixes used in the present work are given in Table II. A, B, C, D, G, H, and J represent mixes vulcanized by sulfur. Mix A represents the gum vulcanizate. B, C, and D represent mixes at constant fiber loading but with different fiber lengths. G, H, and J represent mixes with a constant fiber length but with different fiber loadings. K, L, and N represent

mixes cured by the dicumyl peroxide (DCP) system. Composites were prepared using a laboratory two-roll mixing mill (150×300 mm). NBR was masticated on the mill for 2 min, followed by addition of the ingredients. The nip gap, mill roll speed ratio, and the number of passes were kept the same in all the mixes. The samples were milled for sufficient time to disperse the fibers in the matrix at a mill opening of 1.25 mm. The cure characteristics of the mixes were investigated by using a Monsanto Rheometer R-100 at a rotational frequency of 100 cycles/min. Vulcanization of the mixes were done at 153°C in a hydraulic press having steam-heated platens to their respective cure times as obtained from the Rheometer.

Stress-strain measurements were carried out using a Universal Testing Machine (ZWICK-1474) at a cross-head speed of 50 cm min^{-1} . Tensile measurements of the composites were carried out using samples cut along (longitudinally oriented fiber) the grain direction. Modulus, tensile strength, and elongation at break were determined according to ASTM test method D412-68. Hardness was measured at room temperature by using a shore-A hardness tester according to ASTM D-2240 test method. In the measurement of mechanical properties, the standard deviation was below 2%.

Swelling studies were carried out using circular samples of diameter 1.9 cm, which were punched out from the vulcanized composite sheets using a sharp steel die. Initially, samples were dried overnight in a vacuum desiccator and the original weights were taken. They were then swollen in *N,N*-dimethyl formamide at room temperature for 72 h.

Samples for scanning electron micrographs (SEM) were prepared by cryogenically fracturing the samples in liquid nitrogen. The samples were sputter coated with gold and the photographs were taken using a scanning electron microscope (S-2400, Hitachi).

TABLE II
Formulations of Mixes (phr^a)

	A	B	C	D	G	H	J	K	L	N
NBR ^b	100	100	100	100	100	100	100	100	100	100
Zinc oxide	5	5	5	5	5	5	5	—	—	—
Stearic acid	2	2	2	2	2	2	2	—	—	—
MBTS ^c	1.5	1.5	1.5	1.5	1.5	1.5	1.5	—	—	—
TMTD ^d	0.5	0.5	0.5	0.5	0.5	0.5	0.5	—	—	—
Sulfur	1.5	1.5	1.5	1.5	1.5	1.5	1.5	—	—	—
DCP ^e	—	—	—	—	—	—	—	5	5	5
Nylon fiber	—	6	6	6	9	12	15	—	6	15
Fiber length (mm)	—	2	6	10	6	6	6	—	6	6

^a Parts per hundred rubber.

^b Acrylonitrile butadiene rubber.

^c Mercapto benzo thiazyl disulphide.

^d Tetramethyl thiuram disulphide.

^e Dicumyl peroxide.

TABLE III
Fiber Length Distribution Index

	Fiber length (mm)	\overline{Ln} (mm)	\overline{Lw} (mm)	$\overline{Lw/Ln}$
Before mixing	2	1.94	1.87	0.963
	6	5.92	5.97	1.008
	10	9.78	9.62	0.983
After mixing	2	1.50	1.41	0.940
	6	4.60	5.07	1.102
	10	8.31	8.11	0.975

RESULTS AND DISCUSSION

Fiber breakage

Fibers usually undergo breakage due to the high shear force caused during mixing. The distribution of fiber lengths can be represented in terms of moments of distribution.^{28,29} The number and weight average fiber lengths can be defined as

$$\overline{Ln} = \frac{\sum Ni Li}{\sum Ni} \quad (1)$$

$$\overline{Lw} = \frac{\sum Ni Li^2}{\sum Ni Li} \quad (2)$$

where \overline{Ln} is the number average fiber length, \overline{Lw} , the weight average fiber length, and Ni the number of fibers having length Li .

The value of $\overline{Lw/Ln}$, the polydispersity index can be taken as a measure of fiber length distribution. The values of \overline{Ln} , \overline{Lw} , and $\overline{Lw/Ln}$ were calculated based on 100 fibers for the chopped nylon fibers extracted from the mix. Table III shows the fiber length distribution index of nylon fibers before and after mixing. The value of $\overline{Lw/Ln}$ remains almost the same before and after mixing, indicating that no considerable fiber breakage occurred during mixing.

Processing characteristics

The cure characteristics and thus processibility of the compounds have been studied from the rheographs. The maximum torque in the rheographs is a measure of crosslink density and the stiffness of the NBR matrix. In general, for all mixes, the torque initially decreases and then increases, and finally levels off. The initial decrease in torque to a minimum value is due to the softening of the rubber matrix, while the increase in torque is due to the crosslinking of the rubber. The leveling off is an indication of the completion of curing. It is found that, generally, the presence of fibers increases the maximum torque.

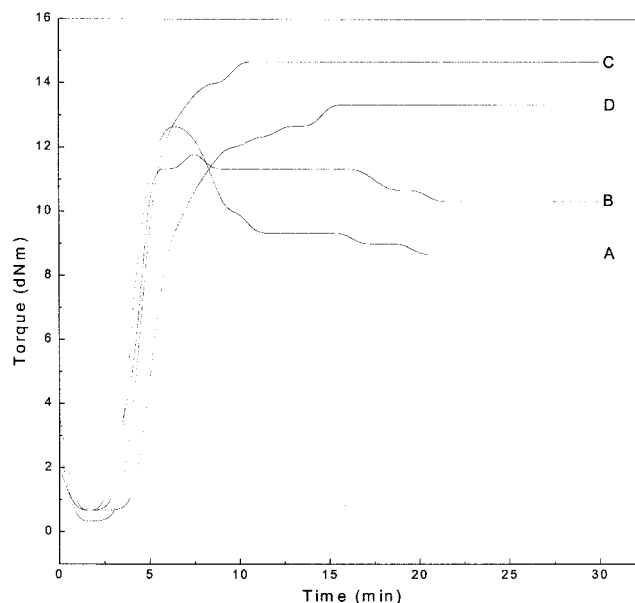


Figure 1 Rheographs of mixes A, B, C, and D.

Effect of fiber length

The curing behavior of mixes A, B, C, and D are given in Figure 1. It can be seen that the torque value increases with an increase in fiber length up to 6 mm and then decreases. The increase in torque is due to the increased restriction to deformation caused by the fiber reinforcement. The decrease in torque after 6 mm clearly indicates that the effective reinforcement decreases due to fiber entanglement, and 6 mm is the optimum length for short nylon reinforcement into NBR matrix. It is reported that the increase in torque is directly proportional to crosslink density.³⁰ Table IV shows the effect of fiber length on the cure time of mixes A, B, C, and D. It can be seen that as the fiber length decreases the cure time decreases. This is due to the fact that for the same loading, as the fiber length decreases; the number of fiber ends increases. The increased ends generate more heat due to friction,¹¹ and thus decreases the cure time.

TABLE IV
Cure Characteristics of Mixes

Mixes	Max. torque (dNm)	Min. torque (dNm)	ΔT (dNm)	Cure time (min)
A	11.79	0.50	11.29	4.60
B	12.31	0.64	11.67	4.88
C	15.20	0.48	14.72	7.22
D	13.45	0.42	13.03	9.32
G	15.68	0.48	15.2	8.12
H	17.42	0.67	16.75	7.18
J	18.63	0.87	17.76	6.15
K	10.65	0.33	10.32	19.98
L	19.60	1.20	18.4	18.75
N	22.09	1.04	21.05	29.72

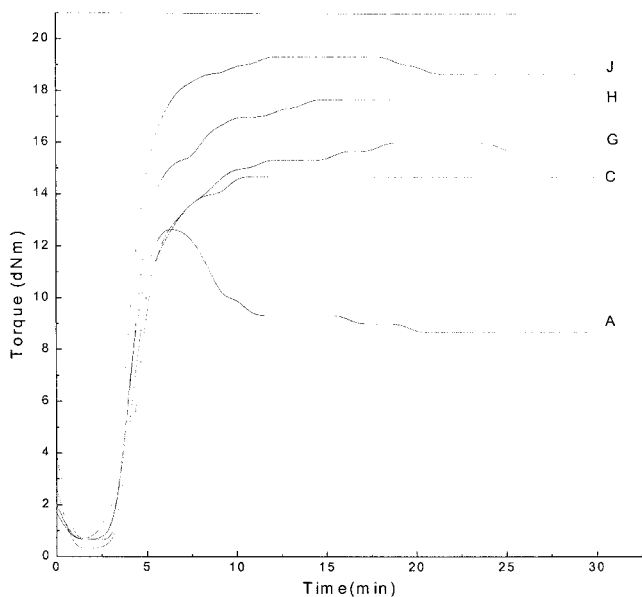


Figure 2 Rheographs of mixes A, C, G, H, and J.

Effect of fiber loading

Figure 2 shows the rheographs of mixes A, C, G, H, and J. The presence of fibers causes an increase in viscosity and torque of the mixes. This clearly suggests an increased stiffness of nylon/NBR systems with an increase in fiber loading. There is an inflexion point in the case of mix G, which may be due to the degradation of rubber and/or due to the thermal degradation of fibers. Table IV shows the cure time of mixes A, C, G, H, and J. It can be seen that the increase in fiber loading from 6 to 15 phr has no considerable effect on the cure time in the case of sulfur-cured mixes.

Effect of curing system

Figure 3 shows the rheographs of the mixes J and N, representing samples cured by sulfur and DCP system, respectively. It is seen that the maximum torque is greater in the DCP system than the sulfur system at a fiber loading of 15 phr. The cure time of DCP mix is also higher than the corresponding sulfur system, as shown in Table IV. The difference in the torque values and cure time can be explained on the basis of the different crosslinks introduced between the macromolecular chains of NBR during vulcanization. The reinforcement of fillers varies with the flexibility of the macromolecular networks due to the different crosslinks present. DCP forms rigid C—C linkages, whereas sulfur introduces flexible mono, di, and polysulfidic linkages as shown in Figure 4(a) and (b). In the DCP-cured system, there is more restriction to chain deformation due to the rigid C—C linkages, whereas in the sulfur system the restriction to deformation is less because of the flexible nature of the polysulfidic

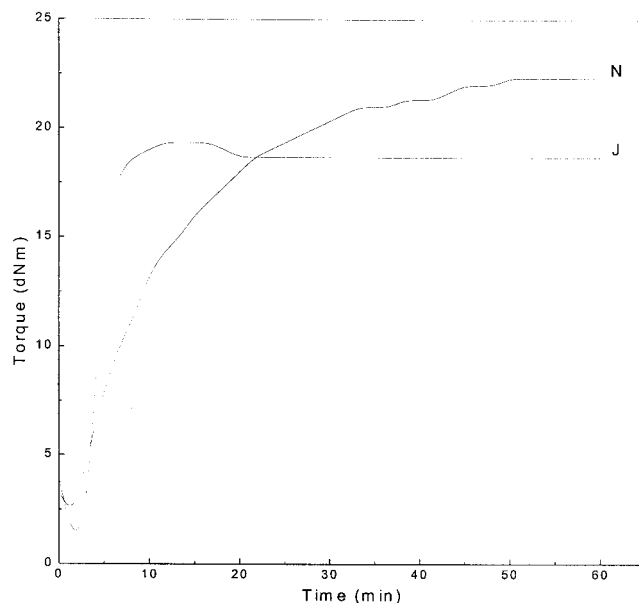


Figure 3 Rheographs of mixes J and N.

linkages. Thus, nylon/NBR systems cured by DCP will be stiffer than that by sulfur. Therefore, the variation in torque is more marked in the case of DCP-cured mixes.

Mechanical properties

Effect of fiber length

The properties of short fiber-reinforced elastomer composites depend on the degree to which an applied

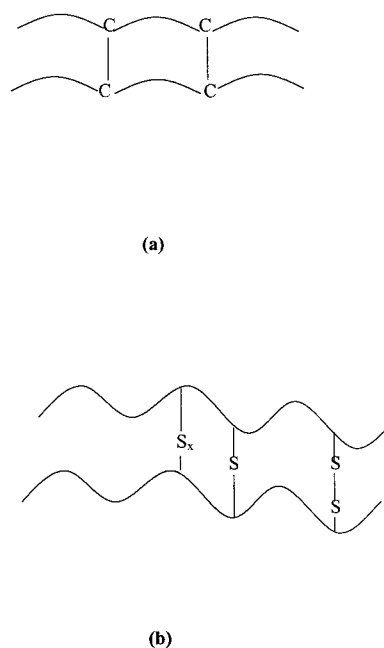


Figure 4 (a) C—C linkages in DCP cured system. (b) Poly sulfidic linkages in sulfur cured system.

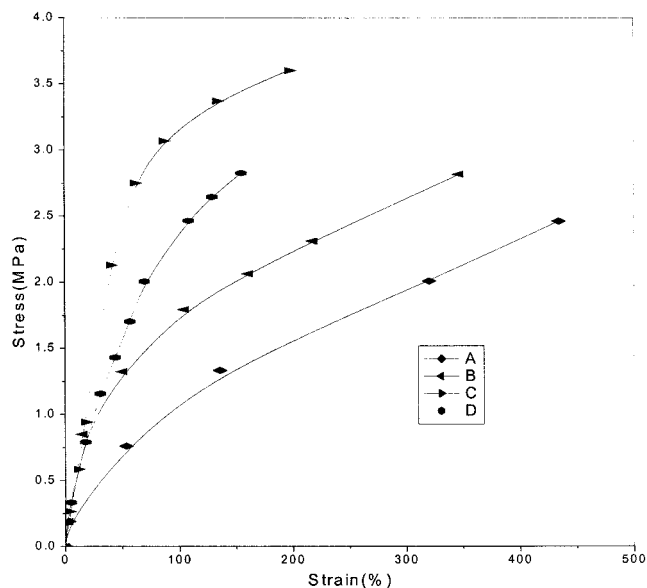


Figure 5 Stress vs. strain curves of mixes A, B, C, and D.

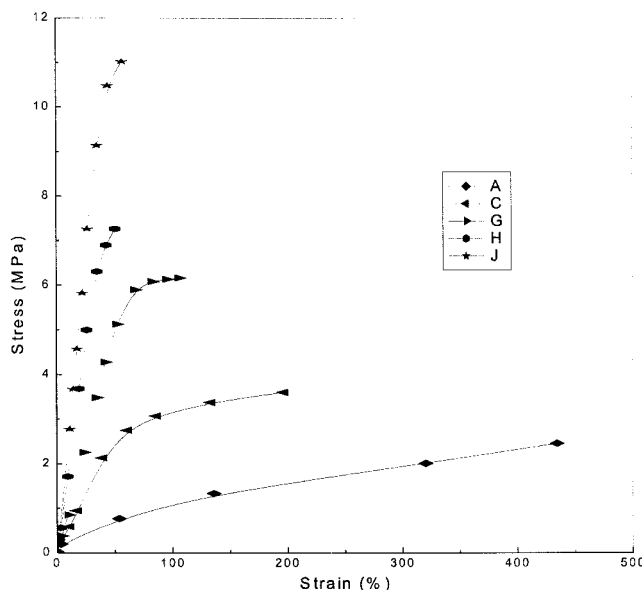


Figure 6 Stress vs. strain curves of mixes A, C, G, H, and J.

load is transmitted to the fibers. Fiber length influences the extent of load transmittance. At a critical fiber length the load transmittance from the matrix to the fiber is maximum. If the critical fiber length is greater than the length of the fiber, the stressed fiber will debond from the matrix and the composite will fail at low load. If the critical fiber length is less than the length of the fiber, the stressed composites will lead to breaking of the fiber.

The stress–strain curves of mixes A (gum), B, C, and D are shown in Figure 5. The deformation of composites can be understood from the stress–strain curve. Addition of fibers increases the modulus of the composite. Table V shows the effect of fiber length on the mechanical properties of the composites. The moduli at 20 and 40% elongation show a maximum value for the composite containing nylon fiber of length 6 mm. The composite with nylon fibers of length of 6 mm shows the maximum tensile strength. An increase in fiber length beyond 6 mm reduces the strength of the composite system, probably due to fiber entanglement. Composite with fibers of length 6 mm shows maximum tear strength and hardness. An increase in

fiber length beyond 6 mm reduces the tear strength and hardness of the composites. The elongation at break reduces with increase in fiber length because the increase in fiber length decreases the possibility and probability for rearrangement and deformation of the matrix under an applied load.

Effect of fiber loading

The stress–strain behavior of short nylon fiber–NBR composites at different fiber loadings is shown in Figure 6. It can be seen that the addition of fibers results in an increase in modulus and reduction in elongation at break. The stress–strain curves of composites at high fiber loading show brittle-type character, and therefore, the loading was restricted to 15 phr for the present work.

Table V shows the effect of fiber loading on the mechanical properties of the composites. Moduli at 20 and 40% elongation show a continuous increase up to a fiber loading of 15 phr. The reinforcement of polymers with short fibers usually leads to an increase in

TABLE V
Mechanical Properties of Composites

	A	B	C	D	G	H	J	K	L	N
Tensile strength (MPa)	2.46	2.82	3.60	2.82	6.15	7.25	11.01	2.22	5.82	11.72
Tear strength (MPa)	7.25	20.61	25.71	24.46	48.77	59.42	63.31	7.31	37.25	66.56
Modulus (MPa)										
20% Elongation	1.75	2.50	5.40	4.50	9.25	17.5	25.75	2.35	5.0	21.5
40% Elongation	1.50	2.11	5.05	3.45	10.62	16.62	25.12	1.7	5.25	24.12
Elongation at break (%)	469.6	366.6	217.6	181.0	105.6	70.6	56.6	256.3	97.7	48.0
Hardness-Shore A	53.0	62.0	71.0	61.0	73.0	78.0	81.0	56.0	75.0	84.0

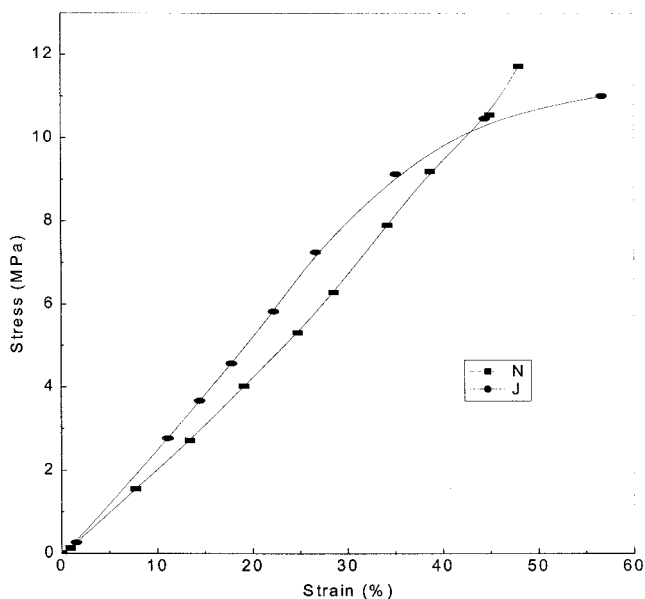


Figure 7 Stress vs. strain curves of mixes J and N.

tensile strength of the amorphous rubber.³¹ The tensile strength has been found to increase with an increase in fiber concentration. As the fiber concentration increases, the stress is more evenly distributed and the strength of the composite increases. The composite with a fiber loading of 15 phr shows highest modulus and tensile strength. Tear strength shows the same gradation as that of tensile strength, and a composite with 15 phr fiber loading shows the highest tear strength. Hardness also increases with increases in fiber loading. Elongation at break of the mixes falls sharply at low fiber loading and then decreases gradually. With an increase in fiber loading the stiffness and brittleness of the composite increased gradually with an associated decrease in elongation at break.

Effect of crosslink system

Figure 7 shows the stress–strain curve of the composites J and N. It shows that at optimum fiber loading, the composite with the DCP system shows more brittle-type behavior than that with the sulfur system. Table V shows the effect of crosslink systems on the mechanical properties of the composites. Comparison has been made with gum vulcanizates and composites at 6 and 15 phr fiber loadings at optimum fiber length. It can be seen that there are differences in the tensile strength, tear strength, and modulus of the composites. Composites cured by DCP system were found to have comparatively higher tensile, tear strength, and modulus. The elongation at break shows significant difference in the two crosslink systems. Composites with DCP as the crosslinking agent were found to have lower elongation at break than that with sulfur

crosslinking agent. This difference in elongation at break is due to the difference in the crosslinks introduced by sulfur and DCP during vulcanization. Hardness is comparatively higher for composites cured by the DCP system.

Morphology of the composites

SEM studies reveal that fiber-filled composites exhibit a marked change in fracture topography. The presence of fibers in the mix changes the fracture modes. The fracture of fiber-reinforced composite occurs in two modes:³² (1) breakage of fibers leading to fracture, and (2) pullout of several fibers from the composite. Figure 8(a) shows how fibers are longitudinally oriented in the composite. In longitudinally oriented fiber composites the fibers are oriented perpendicular to fracture point and so pullout of the fiber takes place. Due to good bonding between the fiber and the matrix, fibers are broken during tensile failure. The broken ends of the fibers protruding in the fracture surface can be seen in the photomicrograph [Fig. 8 (b)].

Swelling studies

Swelling is a uniform restrictive force induced on the vulcanizate samples. Das³³ has studied swelling behavior of short fiber-reinforced elastomeric composites. These studies provide information on the strength of the fiber–matrix interface, degree of dispersion of fibers, and their alignment in the elastomeric matrix. The swelling behavior of composites can be analyzed from the swelling coefficient values as calculated by the following equation:³⁴

$$\text{Swelling coefficient } (\alpha) = \frac{M_{\infty} - M_0}{M_0 d} \quad (3)$$

where M_{∞} denotes the weight of the solvent at the equilibrium swelling, M_0 is the initial weight of the sample, and d is the density of the solvent. Table VI shows the variation of swelling coefficients of the composites with fiber length. The swelling coefficient value of pure NBR cured by sulfur is lower compared to other mixes by sulfur system with nylon fibers of lengths of 2, 6, and 10 mm. The distribution of the crosslinks in the rubber matrix and their chemical nature contributes to the liquid sorption behavior of rubber.³⁵ In addition, when fibers are incorporated in the NBR matrix, due to polar–polar interaction between the nylon and *N,N*-dimethyl formamide the solvent uptake increases, resulting in comparatively higher values of swelling coefficient for mixes containing fibers than the gum sample. The swelling coefficient value is minimum for composites with 6-mm fiber compared to composites containing fibers of

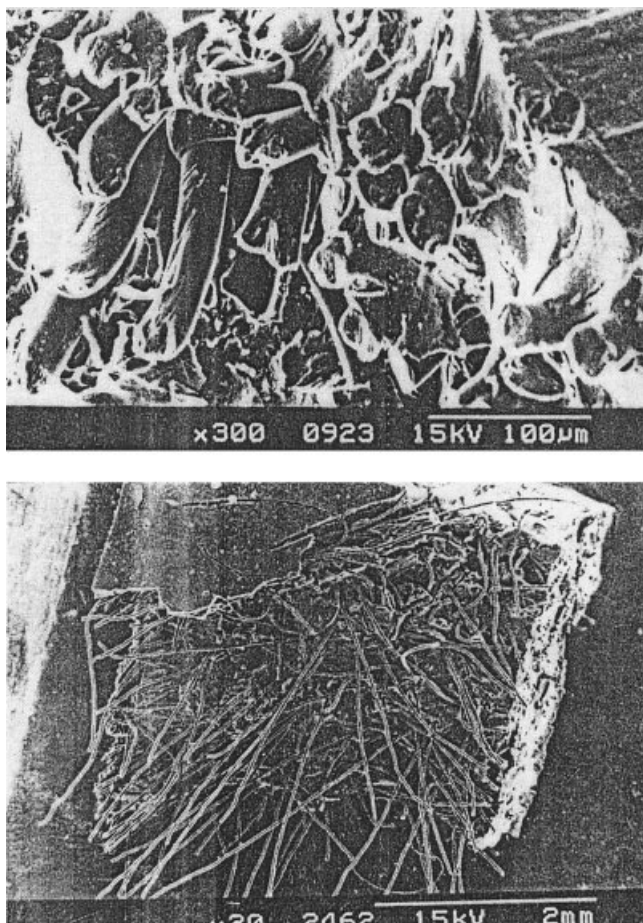


Figure 8 (a) SEM photograph showing longitudinal orientation of fibers in NBR Composite (Mix N). (b) SEM photograph showing fibers protruding in the fracture surface of tensile failure sample (Mix N).

lengths of 2 and 10 mm. It indicates that the solvent uptake behavior of the composite is minimum at a fiber length of 6 mm. This observation parallels our findings that composite with 6 mm fiber is having better mechanical properties. The effect of fibers on swelling coefficient is more marked when DCP is used as the curing system. When the mixes are cured by the DCP system, rigid vulcanizates are formed due to C—C linkages. Hence, the macromolecular chains are

TABLE VI
Swelling Coefficient

Mixes	Weight of the sample	Weight of the solvent imbibed	Swelling coefficient
A	0.461	1.686	2.803
B	0.482	1.414	3.094
C	0.462	1.263	2.884
D	0.458	1.314	3.026
K	0.495	1.238	2.638
L	0.470	0.827	1.856
N	0.597	0.814	1.438

TABLE VII
Volume Fraction (V_r) and Apparent Crosslinking ($1/Q$)

Mix	Volume fraction of rubber	Apparent crosslinking ($1/Q$)
A	0.2391	0.5109
B	0.2144	0.4646
C	0.2201	0.4995
D	0.2169	0.4772
J	0.2331	0.4875
K	0.2620	0.5442
N	0.3369	1.0020

not as flexible as in sulfur-cured system. Due to the rigidity of the network, polar-polar interaction may not be predominant in the DCP system as in the sulfur system. Therefore, in the DCP system the swelling coefficient value decreases significantly with the incorporation of fibers.

Table VI shows the swelling coefficients of composites with different fiber loadings. For mixes K, L, and N vulcanized by DCP, the swelling coefficient value decreases as the fiber loading increases. It indicates that the solvent uptake behavior of the composites is reduced at higher fiber loadings.

The volume fraction V_r of NBR in the specimen swollen in *N,N*-Dimethyl formamide was determined at room temperature using the following equation³⁶ to establish the extent of crosslinking.

$$V_r = \frac{(D - fT)\rho_r^{-1}}{(D - fT)\rho_r^{-1} + A_0\rho_s^{-1}} \quad (4)$$

where D is the weight after drying out, f is the fraction of insoluble components, T is the weight of the sample, ρ_r is the density of the rubber (NBR = 1.00 gcm⁻³), ρ_s is the density of the solvent (*N,N*-Dimethyl formamide = 0.948 gcm⁻³) and A_0 is the weight of the solvent imbibed. Table VII shows the volume fraction of rubber V_r in mixes cured by the sulfur and DCP systems. The composite vulcanized by the DCP system shows a higher V_r value, clearly indicating its better fiber-rubber interaction. This result supports our observation of better mechanical properties for DCP vulcanized composites. A comparison of crosslink density in mixes cured by sulfur and DCP systems has been made from the reciprocal swelling values, $1/Q$, where Q is calculated³⁷ as follows.

$$Q = \frac{\text{Solvent swollen weight} - \text{dried weight}}{\text{Original weight} \times 100/\text{formula weight}} \quad (5)$$

Table VII shows the apparent crosslinking values of mixes A, B, C, D, J, K, and N. Generally, the crosslink density increases with the incorporation of fibers into the rubber matrix. However, for the present system,

the apparent crosslinking calculated based on the reciprocal swelling values for the sulfur-cured system shows a difference from the general trend. It may be due to the predominance of polar-polar interaction in the sulfur-cured system, with flexible polysulfidic linkages, compared to the rigid vulcanizate formed when cured by DCP. The mixes cured by the DCP system show higher crosslink densities, which clearly indicate higher fiber-rubber interaction in those systems.

CONCLUSION

The processing characteristics and mechanical properties of short nylon fiber-reinforced NBR composites have been analyzed as a function of the fiber length, fiber loading, and crosslink system. Addition of nylon fiber to NBR offers good reinforcement, and causes improvement in mechanical properties. A fiber length of 6 mm has been found to be optimum for the best balance of properties. It has been found that the cure time and maximum torque is lesser in the sulfur system than in the DCP system. Composites vulcanized by the DCP system show better mechanical properties than those by the sulfur system. Swelling studies conducted supports the observations of mechanical properties. The solvent uptake behavior is found to be minimum in composites with a fiber length of 6 mm. The crosslink density and volume fraction of rubber were found to be higher in composites cured by the DCP system than those by the sulfur system, indicating better interaction between fibers and matrix in the former.

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