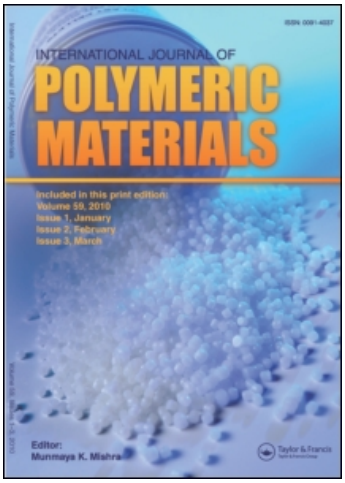

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Characterization of Short Nylon Fibre Reinforced Natural Rubber Composites

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Characterization of Short Nylon Fibre Reinforced Natural Rubber Composites

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The study undertaken corroborates two important aspects of the short nylon-6 fibre reinforced natural rubber composites. Effect of fibre loading on the tear, hardness and abrasion resistance is positive in the sense that it enhances these properties where as tensile strength increases only in the longitudinal orientation of fibres. The hysteresis properties and ultimate elongation are adversely affected. Secondly, an increase in the size of the fibres has mixed effect on the mechanical properties like tensile, abrasion, set and heat build up, where as tear strength improves in longitudinal orientation of the fibres. There exists a critical loading of fibres beyond which the mechanical properties of the composites decrease because of ineffective bonding of fibres with the rubber matrix.

INTRODUCTION

The importance of short fibre reinforcement in rubbers have been realised only recently because of its varied advantages like design flexibility, processing economy and high strain moduli over the other conventional reinforcing fillers. Most of the earlier work has

† M/s J.K. Industries Ltd., Jaykaygram, Kankroli, Rajasthan.

been confined to the reinforcement of rubbers by treated discontinuous cellulose fibres (Santoweb).[1–4] Very little work has been done on fibres other than cellulose. Derringer and O'Connor[5–7] decades back made an attempt to utilize nylon yarns as short fibres in improving the modulus of rubbers. Basically they made a comparison of the fibre properties and stress-strain behaviour of the rubber-fibre composites. The other fibres they made use of were glass and polyester fibres.

Very recently, De *et al.*[8–10] studied elaboratedly the effect of short jute, glass and silk fibres in the reinforcement of natural and synthetic rubbers. They established that the extent of fibre reinforcement depends on the factors like, fibre-matrix adhesion, fibre length, fibre dispersion and varies from fibre to fibre.

The present study deals with the processing behaviour and the physicomechanical properties of short nylon-6 fibre reinforced natural rubber composites. Main investigation parameters are as follows: (i) Effect of loading of short nylon-6 fibres on composite properties, (ii) Effect of change in the size of the fibres on the properties and (iii) Effect of ageing on the fibre-matrix properties.

The nylon cords and fabric available in bulk in the scrap tyres, belts and hoses may be recovered mechanically and reutilized as short fibres in many plastics and rubber industries. With this idea the present work has been undertaken in order to utilize the waste nylon cords and fabrics in rubber.

EXPERIMENTAL PROCEDURE

Materials used

Crumb natural rubber (ISNR-5), supplied by Rubber Research Institute of India, Kottayam, Kerala, was used from a single bale throughout the investigation, since variation in bale to bale may vary the molecular weight affecting ultimately the physical properties of the vulcanizates. Nylon-6 fibres; derived from Caprolactum and treated with RFL bonding system, was supplied by Messrs Dunlop India Ltd., Sahaganj, Calcutta (Table I). Vulkanox HS, Vulkanox 401 0NA and Vulkacit-MOZ were supplied by Messrs Bayer India Ltd., Thane, Maharastra.

TABLE I

Specifications of nylon-6 yarn[2]

Type of fabric	Heat set and dipped Nylon-6 cord
1. Construction in raw stages	1260 Denier/2
2. Breaking strength, N	196 min.
3. Elongation at break, %	22
4. Dip pick-up, %	4.5
5. chemical treatment	RFL dope

[2] Chemically treated Nylon-6 of given specification was supplied by M/s Dunlop (India) Ltd., Sahaganj, Hooghly.

Processing details

The continuous cords were chopped to short lengths of approximately 6.25 mm and 12.25 mm with a semi automatic chopping machine. The composites were prepared on a two-roll mixing mill (152 mm × 330 mm) at a constant friction ratio 1:1.24 as per ASTM D3182-74. The chopped nylon yarns were incorporated into the rubber matrix just after mastication and then all other ingredients were added. During mixing, the spun and twisted nylon yarns underwent severe cleavage due to shearing action at the nip of the rolls, resulting in excessive heat generation. The rolls were continuously cooled and the temperature was kept constant at 35–40°C throughout the mixing cycle. As a result of cleavage, the twisted nylon yarns were separated into its individual filaments. The mixing was more of dispersive nature at the initial stage of mixing. Subsequently the individual filaments underwent distributive mixing and in the process tended to orient themselves in the direction of the applied stress i.e. the mill direction. Finally, the compounded stock was passed through the tight nip of the mill without banding once or twice to effect orientation of the fibres in the mill direction. The nip gap, the friction ratio, time of mixing and the sequence of addition of ingredients into rubber were all kept identical for all the mixes during the mixing cycle.

According to Moghe,[11] the alignment of fibres is expected to be in one direction, but this is never achieved. First a random distribution of fibres occur, then 60–70% of the fibres orient themselves along the mill direction after final pass through the rolls.

TABLE II

Aspect ratio of nylon-6 fibres

Mix No.	Length of the yarn before mixing, (mm)	Length of fibres after mixing, (mm)	Diameter of yarn before mixing, (mm)	Diameter of fibre (filament) after mixing, (mm)	Aspect ratio (L/D) before mixing	Aspect ratio (L/D) mixing
A–E	6.32	5.89	0.846	0.035	7.5	170.0
F	11.87	11.75	0.846	0.063	14.0	185.6

Note: The quantities refer to average values over the samples.

The short nylon yarns consisting of several spun filaments open out to individual filaments during mixing. The extent of fibre breakage was determined by dissolving the compounded rubber in benzene and extracting the fibres and measuring the fibre length and diameter by an optical microscope. The length, diameter and the aspect ratio of the original yarn and the individual filaments (fibres) are shown in Table II.

The formulations of the mixes are given in Table III and the curing characteristics are shown in Figures 2 and 3. Mix F contains 30 phr of nylon fibres of higher length than those studied under mixes A to E. Fibre incorporation in general improves the overall processing behaviour. This is no doubt an attractive property of the fibre reinforced natural rubber composites, but higher fibre loading pose several problems during mixing, limiting the extent of fibre loading to a maximum of about 40 phr. This phenomenon was also observed by De *et al.*[10,12] in their studies on short jute and glass fibre reinforced rubber composites.

The optimum cure time of the mixes were determined by a Monsato Rheometer (Model R-100) at 150°C. The samples were moulded by an electrically heated single daylight hydraulic press at 150°C and 4.5 N mm^{-2} pressure to their respective optimum cure times. However, thicker samples were vulcanized for longer time depending on their thickness to obtain equivalent degree of cure.

The stress-strain properties of the composites (ASTM D412-80, using A) and the tear strength (ASTM D624-81) were evaluated by using an Instron Universal Testing Machine (Model 1195) at a

TABLE III

Composition of the mixes in phr

Ingredients	Mixes					
	A	B	C	D	E	F
Natural rubber[a]	100	100	100	100	100	100
Zinc oxide	5	5	5	5	5	5
Stearic acid	2	2	2	2	2	2
IPPD[b]	1	1	1	1	1	1
Vulkanox-HS[c]	1.5	1.5	1.5	1.5	1.5	1.5
TMTD[d]	1.0	1.0	1.0	1.0	1.0	1.0
MOZ[e]	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
Nylon-6 (L = 6.5 mm)	0	10	20	30	40	0
Nylon-6 (L = 12.5 mm)	0	0	0	0	0	30

[a] Crumb natural rubber obtained from RRII, Kottayam.
[b] N-isopropyl-N'-phenyl-p-phenylene diamine.
[c] 1,2-dihydro-2,2,4 trimethyl quinoline.
[d] Tetramethyl thiuram disulphide.
[e] Benzothiazyl-2-sulphene morpholide.

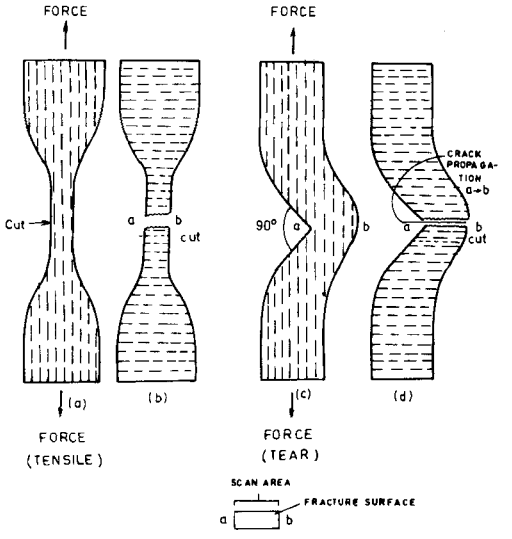

FIGURE 1 Fracture surfaces and scan areas of tensile and tear test specimens.

crosshead speed of 500 mm per minute at room temperature (30°C). Heat build up property (ASTM D623-78, method A) was measured by a Goodrich flexometer. Rebound resilience was determined as per BS 903, A8 using a Dunlop tripsometer at 35°C and shore hardness (ASTM D2240-81) using Shore A Durometer. Abrasion resistance was determined by Croydon–Akron abrasion tester (BS 903, Pt. A9, 1957). The tensile, tear and heat build up properties of the composites were determined both along longitudinal and across transverse orientations of the fibres.

The fracture surfaces of the tested specimens were sputter coated with gold within 24 hours of testing and examined under a Scanning Electron Microscope (Model ISI-60). Orientation of the photographs were kept constant and the angle of tilt was 0° for all the samples. The fracture surfaces and scan areas are shown in Figure 1.

RESULTS AND DISCUSSION

The modulus of the short nylon fibre reinforced natural rubber composites increase with an increase in the concentration of the fibres (Figures 2 and 3). The initial viscosity, thermoplasticity and

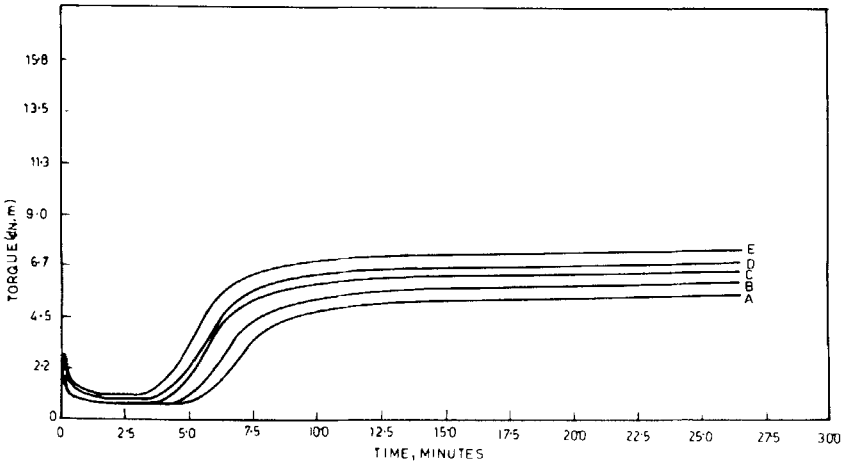

FIGURE 2 Rheographs of the mixes A–E at 150°C.

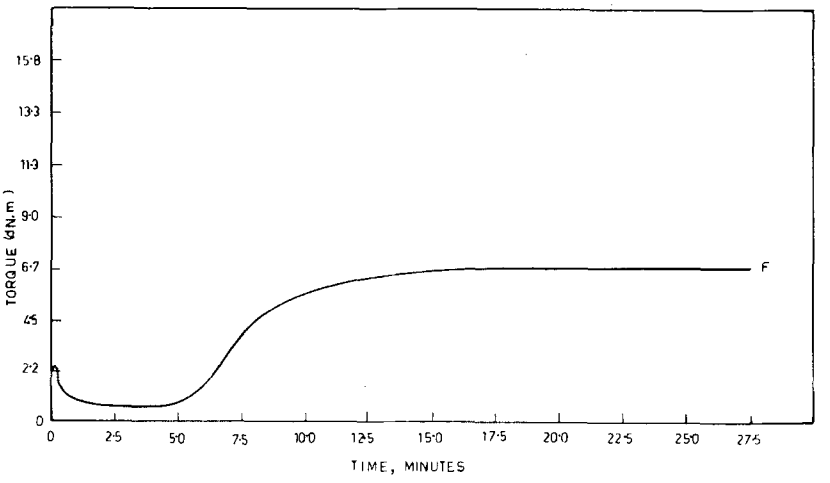

FIGURE 3 Rheograph of mix F at 150°C.

the maximum torque increase with loading of fibres. However, the scorch safety of the mixes gradually decrease with loading of fibres, possibly due to the loss in the heat stability of the compounds. The optimum cure time of the mixes remain more or less the same. The physicomechanical properties of the composites are given in Table IV.

SEM study of a typical dipped nylon twine, at a magnification of 143× is shown in Figure 4. The photomicrograph of the chopped edge of the twisted yarn shows the uneven cut edge which is responsible for the variation in length of individual fibres in the matrix. Also seen is the roughness of the surface of the twisted yarn with the coated adhesive system on the yarn. SEM photomicrograph (Figure 5) of the extracted fibres presents a general view of the filaments extracted in benzene from the rubber matrix showing the individual filaments of much reduced diameter as appear in the matrix.

Effect of concentration of short nylon-6 fibres on composite properties

The stress–strain properties of short nylon fibre reinforced natural rubber composites at various concentrations of the fibres are shown

TABLE IV

Physicomechanical properties of the mixes

Property		Mixes					
		A	B	C	D	E	F
1. Tensile strength, MPa	L	16	10	16	26	23	22
	T	15	7	7	8	9	6
2. Elongation at break, (%)	L	725	50	35	30	43	40
	T	800	200	150	90	62	150
3. Tear strength (N/mm)	L	13	66	84	114	108	136
	T	13	60	80	107	129	64
4. Abrasion loss, (cc/min × 10^2)		2.0	0.86	0.62	0.54	0.96	0.70
5. Goodrich heat build up, Δ°C, 50°C (Longitudinal)		11	25	34	—	—	—
a. Time to blowout, min		—	—	—	12	5	11
b. Temp. at blowout, total °C		—	—	—	96	91	95
6. Goodrich heat build up, Δ°C, 50°C (transverse)		11	32	—	—	—	—
a. Time to blowout, min.		—	—	7	5	4	6
b. Temp. at blowout, total °C		—	—	83	78	85	96
7. Permanent set, %	L	0.98	2.6	5.0	—	—	—
	T	—	2.5	—	—	—	—
8. Compression set, Method A, %	L	9.3	7.4	6.5	5.7	5.3	5.5
	T	9.3	6.6	6.6	4.7	3.7	5.6
9. Rebound resilience, %		55	54	50	46	43	46
10. Hardness, shore A		41	75	80	86	88	85

L = Longitudinal orientation of fibres, T = Transverse orientation of fibres.

in Figures 6 and 7, for longitudinal and transverse orientations respectively. It is evident from these figures that there is reasonably good reinforcement between natural rubber and nylon fibres and the influence of rubber matrix on the stress–strain relationship is gradually replaced by the properties with increasing concentration of the fibre.

The tensile strength and ultimate elongation of the composites at various loadings of nylon fibres are shown in Table IV. The tensile strength first decreases upto 10 phr fibre loading, then increases till 30 phr loading of the fibres in the longitudinal orientation. Further loading of fibres reduces the tensile strength (Mix E) of the composites. The increase in the tensile strength is explained as due to the reinforcing action of nylon in natural rubber matrix, whereas

FIGURE 4 Photomicrograph of chopped edge of a twisted Nylon yarn (143×).

a decrease in the tensile strength at 40 phr and higher loadings is due to dilution of the rubber matrix with the fibres. As the chemical treatment is on the surface of the yarn, and not on the surface of individual fibres, it has little effect in bonding fibres with the rubber. This is evident from the SEM photomicrograph of the fracture surfaces of tensile test specimen (Figure 8) showing the tensile

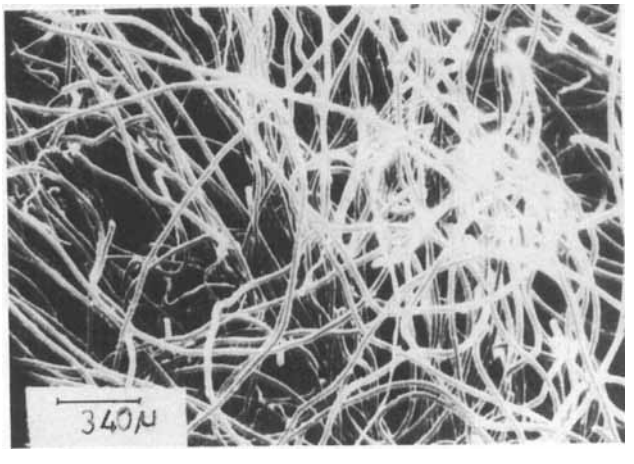

FIGURE 5 Photomicrograph showing general topography of the extracted Nylon fibres (33×).

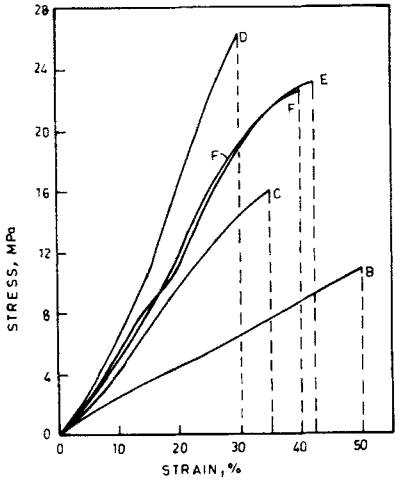

FIGURE 6 Stress–strain relations for Mixes B to F with fibres oriented in the longitudinal direction.

fracture surface of Mix D (30 phr) with the fibres oriented in the longitudinal direction of the applied stress. Figure 8 shows the torn out fibres almost engulfing the rubber matrix. The tensile strength registers a higher value and the elongation at break a lower value because of buckling effect of the fibres. The ultimate elongation

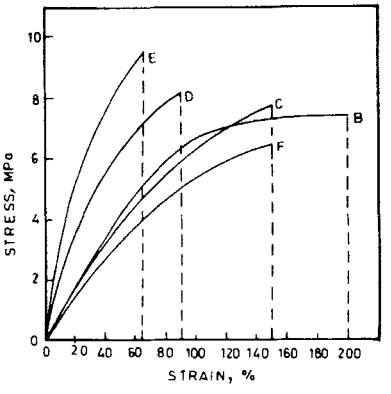

FIGURE 7 Stress–strain relations for Mixes B to F with fibres oriented in the transverse direction.

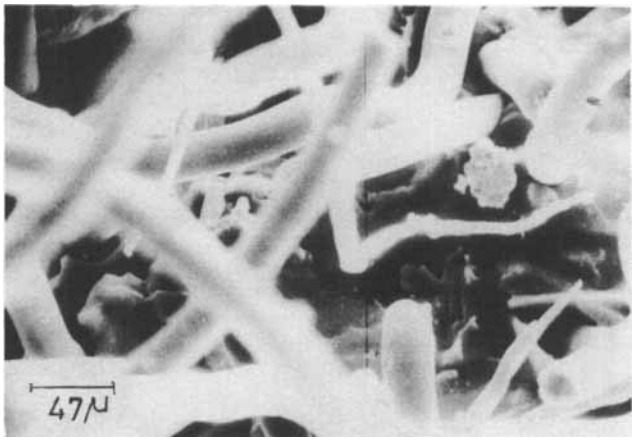

FIGURE 8 SEM photograph of tensile fracture surface of Mix D in longitudinal orientation of the fibres (240×).

drops drastically from 725% to 50% at 10 phr fibre loading and further decreases at higher loadings.

The tensile strength in the transverse orientation of fibres decreases sharply at 10 phr loading of the fibres and then marginally improves on incorporation of more and more fibres upto 30 phr (Mix E) loading. However the improvement is not remarkable so as to reach the value of the reference stock (Mix A). On the otherhand elongation at break drops drastically from 800% to 200% at 10 phr fibre loading and thereafter the decrease is more slow. In the transverse orientation, the fibres are oriented perpendicular to the applied stress and the matrix reflects the rubbery properties to some extent, exhibiting relatively higher elongation.

Figure 9 shows the SEM photograph of tensile fracture surface of mix D with the fibres in the transverse orientation of the applied stress. The fracture surface shows less number of fibres and less fibre breakage as compared to that in case of longitudinal orientation of fibres (Figure 8). It is evident that there exists a strong anisotropy in the tensile properties of the composites and the nylon fibres act as efficient reinforcing filler in natural rubber at a loading of nearly 30 phr.

The tear strength of the composites (Table IV improve significantly with fibre incorporation and the increase is more in the

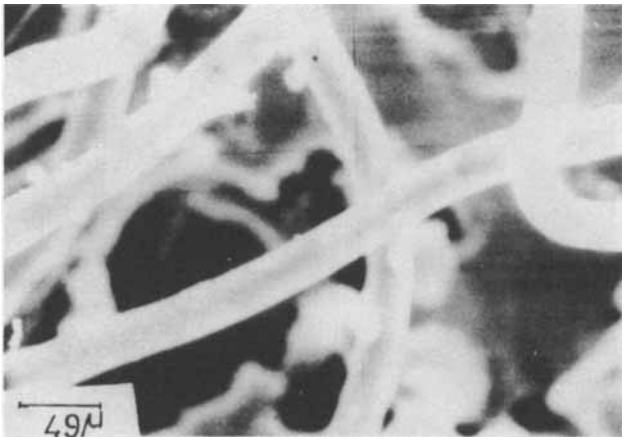

FIGURE 9 SEM photograph of tensile fracture surface of Mix D in transverse orientation of the fibres (230×).

longitudinal orientation than in transverse orientation, at the same concentration of the fibres. But the rate of increase in tear strength after 10 phr loading is much slower than that observed upto 10 phr loading. At very high loadings, for instance at 40 phr (Mix E) the tear strength again falls by 7% in longitudinal orientation of fibres. The critical fibre loading of nylon fibres in natural rubber is adjudged to be around 30 phr. The higher values of tear strength in the longitudinal orientation of fibres is due to the obstruction of the random tear path by the presence of fibres across it. More the concentration of fibres, greater is the resistance to tear. Conversely, in transversely oriented fibres, the tear proceeds parallel to the fibre length, thus facing less resistance to its propagation (fibres are parallel to the direction of tear path). This is fairly evident from the SEM study of the torn surface (Figure 10), which shows the absence of regular tear path on the fracture surface of longitudinally oriented fibres with the torn out fibres and the matrix. There exists no definite tear path which is totally different from the tear paths observed for natural rubber gum and filled torn fracture surfaces[13] earlier. SEM photomicrograph (Figure 11) of the tear fracture surface of Mix D with fibres oriented in the transverse direction show a distinct tear path on the rubber matrix with the fibres on either side of it. The tear progresses more easily along its tear path,

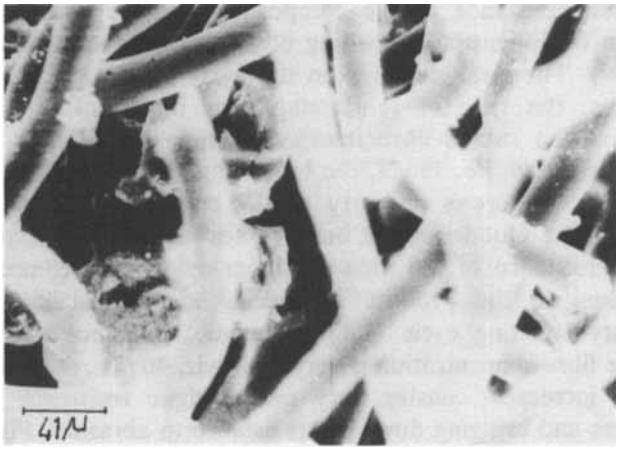

FIGURE 10 Photomicrograph of tear fracture surface of Mix D in the longitudinal orientation of the fibres (270×).

resulting in relatively lower tear strength of the matrix as compared to that for the longitudinally oriented fibres. Fibres overlap because of the ploughing effect during tearing and the matrix emerges out. The presence of rare holes in the matrix is the result of pulling out of fibres.

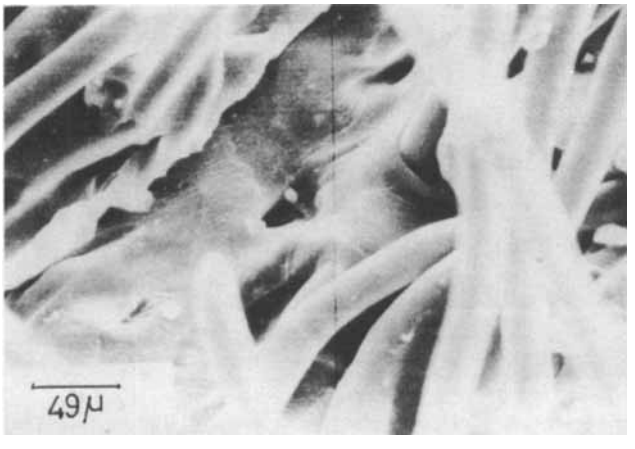

FIGURE 11 Photomicrograph of torn surface of Mix D in the transverse orientation of the fibres (230×).

Abrasion resistance of the composites increase with the concentration of fibres upto a loading of 30 phr and then it decreases (Table IV). The wear depends on the hysteresis properties of the composites, the type of reinforcing filler incorporated and the bonding at the rubber-fibre interface. Since the strength of the nylon fibre is high i.e. 196 N, the loss is mainly assumed to be due to the poor hysteresis property of the composites. This is also reflected in the Goodrich heat build up test data. As expected, the abrasion resistance of the natural rubber-nylon fibre composite is proportional to the product of specific mechanical loss in an elementary stressing cycle and the fatigue resistance of rubber. When the fibre concentration is very high, viz. 40 phr, the hysteresis loss also increases, causing very poor fatigue resistance of the composites and bringing down the resistance to abrasion. Figure 12 shows the general topography of the abraded surface of Mix B showing the mutilated fibres throughout the matrix. The abraded surface for 30 phr loaded fibres is shown in Figure 13. The surface do not exhibit any wear pattern, but shows broken waves of wear debris still anchored to the matrix by the embedded fibres. Because of high friction, fibres also undergo mutilation and fatigue, resulting in sections resembling cracks.

The compression set property of the composites is reduced with

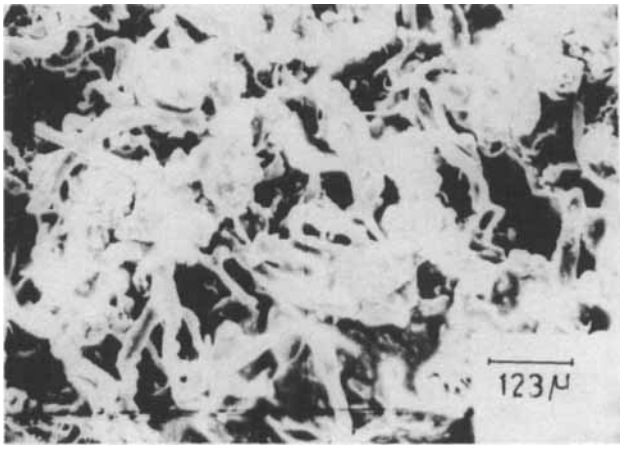

FIGURE 12 SEM photograph of abraded surface of Mix B (91×).

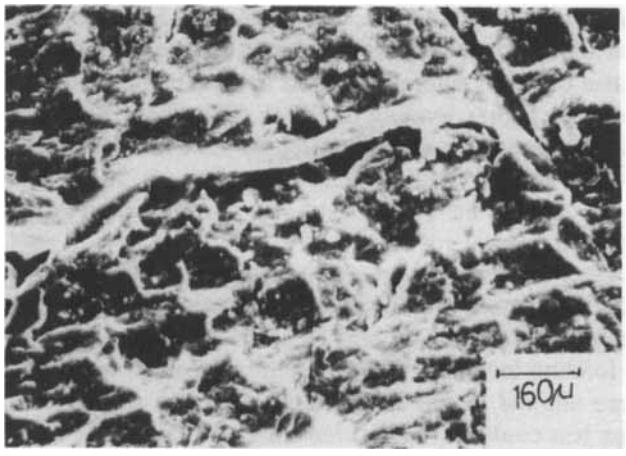

FIGURE 13 SEM photograph of abraded surface of Mix D (70×).

an increase in fibre concentration, in both longitudinal as well as in transverse orientation of the fibres (Table IV). This shows that the set properties of the composites are very good. However, the compression set in the longitudinal direction of fibres is higher than that in the transverse direction of fibres at a particular concentration.

The rebound resilience of the composites decrease with increase in fibre concentration (Table V). This is due to high loss modulus at

TABLE V

Percent retention of properties after ageing

Properties		Mixes					
		A	B	C	D	E	F
Tensile strength	L	95	93	87	70	101	134
	T	92	93	112	175	92	167
Tear strength	L	91	112	102	87	105	99
	T	103	153	108	91	79	132
Elongation at break	L	86	253	116	119	95	58
	T	82	315	72	64	137	40

L = Longitudinal orientation of fibres, T = Transverse orientation of fibres.

higher concentration of fibres. The resilience decreases at a lower pace upto 10 phr loading, beyond which the rate of decrease is very fast because of heavy energy loss and decrease in the rubber matrix volume due to higher loading of fibres. The shore hardness of the rubber-fibre composites increase steeply with an increase in fibre concentration upto a loading of 20 phr, after which the increase is rather slow. This is an evidence of the reinforcement of the short nylon fibres in natural rubber.

The composites were subjected to compression fatigue cycles by a Goodrich flexometer and the heat hysteresis were recorded. The composites exhibit a steady and rapid increase in heat buildup as the fibre loading increases. One interesting feature is that when the fibres were aligned in the longitudinal direction of the stress cycle the fatigue test could be carried out only upto a loading of 20 phr of fibres (i.e. Mix C) whereas for the transversely oriented fibres the test could be carried out only upto a loading of 10 phr (Mix B). Beyond these loadings the samples cracked and blew out with sudden and tremendous rise in temperature rendering the tests difficult to continue. The blowout time and temperature were recorded. In general, it was observed that for transversely oriented fibres the blow out times were less than that for the longitudinally oriented fibres at the same fibre concentration. On the other hand, the temperature rise is more in the former than in case of the later. Blow out time decreases and temperature rise increases with increase in fibre loading. Figures 14a [(i) and (ii)] show the test

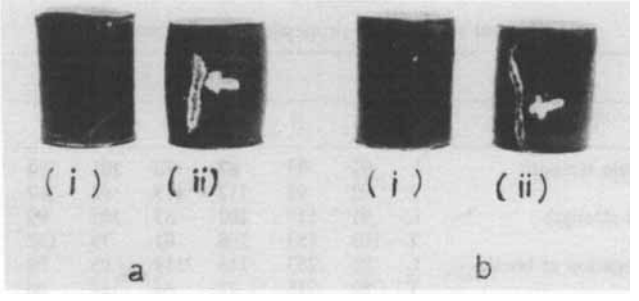

FIGURE 14 Mode of failure of the heat build up samples of Mix D during Goodrich flexometer testing—(a) with fibres in the longitudinal direction and (b) with fibres in the transverse direction.

samples before and after the test. In case of longitudinally oriented fibres, a single crack (Figure 14a) appears on one side and grows along the direction of the stress cycle. That means the stress distribution in the direction of the application of force show less energy dissipation during the stress–strain cycle. This may be due to the alignment of fibres end to end and to the friction between these ends resulting in heat generation. In case of transversely oriented fibres (Figure 14b) the crack propagation is at an angle to the application of stress.

Permanent set of the samples increase steadily from 0.8 to 5.0 with an increase in fibre loading from 0 to 20 phr, when the fibres are oriented in the longitudinal direction of the stress cycle (Table IV). However as soon as the fibre loading is increased to 30 phr, the samples develop cracks after a few minutes of start of the Goodrich heat buildup experiment and permanent set can not be accounted for. For transversely oriented fibres, permanent set of composites can be measured only up to 10 phr fibre loading beyond which the samples blow out, disrupting the conduction of the test. The permanent set values remain the same for 10 phr loading in both the directions of the fibres.

Effect of change in the size of fibres

Two different sizes have been chosen to study the effect on overall physicomechanical properties of the composites. Mixes B to E contain different concentrations of nylon fibres having aspect ratio of 7.5 (length = 6.32 mm) and the Mix F contain 30 phr nylon fibre with aspect ratio of 14.0 (length = 11.87 mm).

An examination of the stress–strain curves of Mixes B to F (Figures 6 and 7) show that Mix F behaves similar to Mix E when the fibres are oriented in the longitudinal direction and similar to Mix C when the fibres are oriented in the transverse direction in the matrix. This implies that the stress–strain properties exhibit greater anisotropy when the size of the fibres are increased keeping the fibre loading constant. When we look at the tensile properties of the Mix F (length = 11.87 mm) the tensile strength decreases and ultimate elongation increases compared to that of Mix D (length = 6.32 mm). This occurs in both the orientations of fibres, although the extent of variation is small. This predicts that the tear strength

of Mix F containing longer fibres should improve. Table IV shows that tear resistance increases to 131 N/mm from 95 N/mm in the longitudinal orientation of fibres, whereas it falls down to 54 N/mm in the transverse orientation of fibres. This probably is due to the higher length of the fibres which obstructs obtrusively the random tear path in the longitudinal direction, where as in the transverse orientation, tear simulates more or less peeling conditions and less energy is required to propagate the tear once it is initiated.

The heat build up property do not change much with the change in the length of the fibres. Only in case of transversely oriented fibres heat development of Mix F is more than that of Mix D. This is because, longitudinal orientation of the fibres show better flexibility than the transverse orientations as a result, cracking occurs more easily and rapidly in the latter than in the former (Figure 15). Abrasion loss is a bit higher for Mix F than for Mix D. The extra loss is due to excessive wear of the fibres.

The compression set, rebound resilience and hardness properties of the mixes for both the sizes of fibres (Mixes D and F) remain the same. Thus it can be concluded that, at the same loading of fibres, lower the size of the fibres (Mix D) higher the total surface area, hence better is the reinforcement. This is responsible for higher tensile properties (Mix D) as compared to the Mix F containing longer fibres at the same fibre loading.

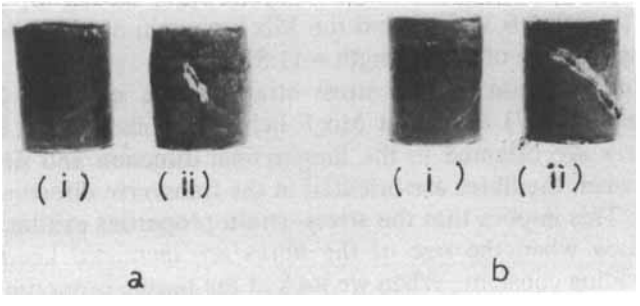

FIGURE 15 Mode of failure of heat build up specimens of Mix F after Goodrich flexometer testing—(a) in the longitudinal orientation of the fibres and (b) in the transverse orientation of the fibres.

Effect of ageing on the fibre-matrix properties

Percent retention properties of the composites after ageing for 48 hours at 100°C in an air ageing oven are given in Table V. The retention of tensile strength in the transverse orientation and tear strength in both the orientations of the fibre are higher in fibre filled vulcanizates than that of Mix A containing no fibres. However, the tensile strength in longitudinal orientation deteriorate continuously as the fibre loading increases. Incorporation of the fibres arrests the fall in properties because of improved reinforcement of the fibres in the rubber matrix and higher resistance of fibres to air ageing.

Mix F shows a high degree of retention of properties after ageing compared to that for Mix D. Thus the fibres play a positive role in retention of properties of the rubber composites after ageing.

CONCLUSIONS

1) A fibre loading of 30 phr is found to impart optimum physico-mechanical properties to the composites.
2) Lower the size of the short fibres better is the reinforcement.
3) Hysteresis properties are adversely affected on incorporation of short nylon fibres.
4) Greater anisotropy is observed in almost all physicomechanical properties of the short nylon fibre natural-rubber composites, as the fibre length increases.
5) The aging properties improved on incorporation of short nylon fibres.

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