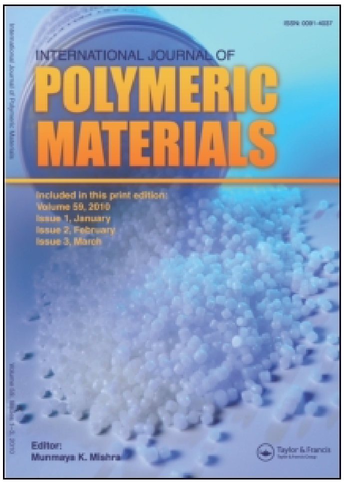

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Melt Flow Behavior of Short Coir Fiber Reinforced Natural Rubber Composites

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The melt flow behavior of natural rubber composites containing untreated, acetylated and gamma ray irradiated coir fibers was studied using an Instron Capillary Rheometer. Coir fibers of length 10 mm were used at different loading, viz., 5, 10, 20 and 30 phr. The dependence of melt viscosity, flow behavior index (n') and extrudate deformation of the composites with shear rate, shear stress and loading of fibers was analyzed. The pseudoplasticity of the melt increased with fiber loading. At low level of fiber loading, the compound exhibited increased viscosity but at high fiber loading the compound showed low viscosity. This may be attributed to the formation of a sheath-core structure at high loading where fiber is concentrated at the core of the extrudate. Incorporation of fibers decreased the extrudate deformation and die swell and this improvement was more prominent at higher fiber loading.

KEY WORDS Rheology, coir fiber, natural rubber composites.

INTRODUCTION

The modulus of an elastomeric compound can be increased by the addition of reinforcing fillers such as carbon black and silica or by the incorporation of continuous or discontinuous fibers. But compared to short fiber reinforced rubber composites, carbon black filled elastomeric compounds possess enhanced processing difficulty at high loadings and the latter contributes lesser to tensile reinforcement both in cured and uncured state. The penetration resistance will also be lower for black filled compounds. In addition to these favorable factors, short fiber reinforced rubber composites are widely accepted because of their processing advantages, design flexibility, creep resistance, damping and anisotropy in the mechanical properties.[1–4]

A thorough knowledge about the processability of a rubber compound is essential for the fabrication of an end product, because the rubber compound undergoes different types of shear during every stage of its processing. Brydson[5] has made a thorough investigation about the need for rheological studies in making a logical choice of the polymer and its processing conditions. White and Tokita[6–8] reported

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on the relationship between rheology and processing. Crowson and Folks[9] reported that fiber orientation takes place only at high shear rates and it occurs as a result of the convergent, divergent and shear flows.

Setua studied the rheological behavior of different rubber composites of natural rubber, nitrile rubber and chloroprene rubber containing short silk fibers.[10] Murty *et al.* reported on the rheological behavior of natural rubber compounds reinforced with short jute fibers[11] and observed that these compounds behave as pseudoplastic materials. The melt behavior of short aramid fiber reinforced thermoplastic polyurethane has been studied by Kutty *et al.*[12] The rheological and extrusion behavior of short fiber reinforced rubber compounds have been reported by Goettler *et al.*[13]

The elastic properties and extrudate behavior of polymer melts are of prime importance in the processing and thereby on the fabrication of polymer products. Information about these properties can be obtained from the die swell ratio, principal normal stress difference, recoverable shear strain and elastic shear modulus. The die swell ratio has a relation with the principal normal stress difference.[14] Recently, in this laboratory studies were carried out on the rheology of composites of natural rubber[15] and low density polyethylene[16] containing short sisal fibers. Geethamma *et al.*[17] have reported on the use of short coir fibers as reinforcing agent in natural rubber. The mechanical properties and swelling behavior of these composites were analyzed.[17,18] However, no study has been reported on the flow behavior of these composites. Hence this study was aimed at the rheological measurements of natural rubber composites reinforced with untreated, acetylated and gamma irradiated short coir fibers. The effects of chemical treatment and loading of coir fibers on the flow behavior of these composites were analyzed.

EXPERIMENTAL

Materials

Coir fiber was purchased from the local processing unit in Kollam, India. The composition of coir fiber is reported[19] as cellulose 36–43%, lignin 40–45%, hemicellulose 0.15–0.25% and pectins 3–4% together with some water soluble materials. The specific gravity and average diameter of coir fiber are 1.15 and 100–400 μm respectively. Natural rubber used for the experiments was ISNR 5 (light color) grade supplied by Rubber Board, Kottayam, India. All other ingredients used were of commercial grade.

Fiber Preparation

Coir fiber was first separated from undesirable foreign matter and pith material. Then it was chopped to 10 mm length. Acetylated coir fiber was prepared by first soaking the coir fiber in 5% sodium hydroxide solution for 48 h at ambient temperature. Then fibers were taken out and washed with water and soaked in glacial acetic acid for 1 hour, decanted and soaked in acetic anhydride containing 1 ml of concentrated sulphuric acid for 5 min. The fibers were then repeatedly washed with water and dried. Treated fibers were kept in polythene bags to prevent moisture absorption.

TABLE I

Formulation of mixes

Ingredients	Gum	A	B	C	D	E	F	G
Natural rubber	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
Zinc oxide	5	5	5	5	5	5	5	5
T.D.Q.[a]	1	1	1	1	1	1	1	1
Resorcinol	-	-	-	-	8.75	8.75	8.75	8.75
Hexa[b]	-	-	-	-	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
Sulphur	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
Coir fiber (untreated)	-	30	-	-	-	-	-	-
Coir fiber (acetylated)	-	-	5	10	20	30	-	-
Coir fiber (gamma irradiated)	-	-	-	-	-	-	30 (0.5 MRad)	30 (4 MRad)

[a] 2,2,4-Trimethyl-1,2-dihydroxy quinoline polymerised.

[b] Hexamethylenetetramine

[c] N-Cyclohexyl-2-benzothiazyl sulphenamide

Compounding

The compounds were prepared on a two-roll laboratory mixing mill (150 × 330 mm). Table I gives the formulation of the mixes used in the present study. Since the rheological behavior of natural rubber compound is very much dependent on its mastication time, this was kept almost constant for all mixes. Fibers were added at the end of mixing. To achieve maximum fiber orientation, the direction of compound flow was maintained.

Rheological Measurements

Measurements of melt flow properties of the compounds were carried out in an Instron Capillary Rheometer Model 3211. The capillary die used was made up of tungsten carbide and having an L/D of 20.27, with an angle of entry 90°.

The compound was cut into small pieces, then placed inside the barrel of the extrusion assembly and tightly packed without air gaps. The sample was preheated for 3 min and then extruded through the capillary at five preselected speeds of the crosshead ranging from 0.6–200 mm per minute at 90°C.

The diameter of the extrudates was obtained as they emerged out of the capillary. Maximum care was taken to avoid any further deformation or stretching of the extrudates. The equilibrium die swell measurements were carried out after a relaxation period of 24 hours at room temperature (30°C). An average of four readings was taken as the diameter of the extrudate. The rheological properties of the composites were also studied in an oscillating disc Monsanto Rheometer R-100 at 150°C.

The cross-section and surface characteristics of the extrudates were studied using optical and scanning electron microscopes. Photomicrographs of the extrudates emerged at the shear rates 3.67 and 1224.66 $\sec^{-1}$ were taken.

Calculations

Shear stress at the wall, τ_w, of the capillary rheometer was calculated using the equation[20]

$$\tau_w = \frac{F}{4A_p(l_c/d_c)} \tag{1}$$

where F is the force, A_p is the area of the plunger, and l_c and d_c are the length and diameter of the capillary, respectively.

The apparent shear rate $\dot{\gamma}_{w,a}$ was calculated using the equation

$$\dot{\gamma}_{w,a} = \frac{2d_B^2}{15d_c^3} Vxh \tag{2}$$

where Vxh is the crosshead speed and d_B is the diameter of the barrel. From the apparent shear rate values, the true shear rate at the wall, $\dot{\gamma}_w$ was evaluated using the following equation which includes the Rabinowitsch correction

$$\dot{\gamma}_w = \frac{(3n' + 1)\dot{\gamma}_{w,a}}{4n'} \tag{3}$$

where n' is flow behavior index obtained from the slope of the linear plot of log τ_w vs log $\dot{\gamma}_{w,a}$.

The melt viscosity, η, was obtained from

$$\eta = \frac{\tau_w}{\dot{\gamma}_w} \tag{4}$$

The die swell ratio can be obtained from the formula

$$\text{Die swell ratio} = \frac{d_e}{d_c} \tag{5}$$

where d_e and d_c are the diameter of the extrudate and the capillary, respectively.

The first normal stress differences ($\tau_{11} - \tau_{22}$) was determined using Tanner's equation[14]:

$$\tau_{11} - \tau_{22} = 2\tau_w[2(d_e/d_c)^6 - 2]^{1/2} \tag{6}$$

Recoverable shear strain (γ_R) and apparent shear modulus (G) were estimated from the following expressions

$$\gamma_R = \frac{(\tau_{11} - \tau_{22})}{2\tau_w} \tag{7}$$

$$G = \frac{\tau_w}{\gamma_R} \tag{8}$$

RESULTS AND DISCUSSION

Effect of Fiber Damage on Rheological Properties

Reinforcing fibers may be damaged during the two roll mill mixing and the subsequent fabrication stages. But this damage is minimum for lignocellulosic fibers.[21,22] In the case of short coir fiber reinforced natural rubber composites also, the breakage during mill mixing was minimum and is reported elsewhere.[17] A comparison of the fiber length before and after extrusion through the capillary rheometer is shown in Figure 1. During two-roll mill mixing the fibers are subjected to shear forces and this causes the breakage of fibers. The mill mixed natural rubber compound containing coir fiber was cut into small pieces in order to facilitate easy filling of the barrel of the capillary rheometer. Thus the length of the fibers is reduced again. This can be understood from Figure 1. So even though coir fibers having a length of 10 mm is used for the present study, majority of the fibers present in the compound which is fed into capillary rheometer, was in the range of 5–6 mm. From the curves corresponding to the composites extruded at shear rates 3.64 and 1224 s^{-1} it is clear that most of the fibers are of length in the ranges 3–5 and 1–3 mm respectively.

Effect of Shear Rate on Viscosity

Figure 2 shows the variation of melt viscosity, η, with shear rate, $\dot{\gamma}_w$, at 90°C. It can be seen that gum compound has got the highest viscosity among the three rubber compounds, gum, A and E. This is in contrary to the behavior of other natural fiber reinforced rubber composites. For example, Murty *et al.*[11] showed that the melt viscosity of natural rubber composite increases with the concentration of short jute fiber. They explained this in terms of fiber-fiber interaction rather than the fiber-matrix interaction. Similarly Varghese *et al.*[15] reported that the melt viscosity, of natural rubber composites increases with short sisal fiber loading. However, in the present case the decrease in viscosity upon the addition of fiber is associated with the fiber migration which is explained in the later section.

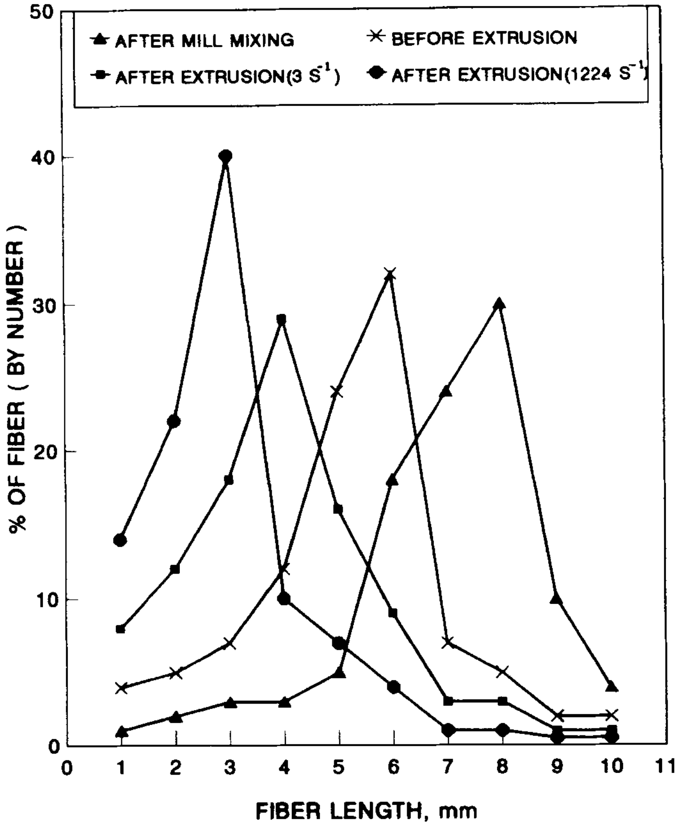

FIGURE 1 Effect of processing and rheological studies on fiber breakage.

It can be noticed from Figure 2 that the melt viscosities of all compounds decrease with increasing shear rate. This indicates the pseudoplastic behavior of the composites because of the orientation of fiber along the direction of rubber molecules.

Effect of Shear Stress on Viscosity

Figure 3 gives the effect of shear stress on the melt viscosity of natural rubber compounds containing acetylated coir fibers at different loading, viz., B (5 phr), C (10 phr), D (20 phr) and E (30 phr).

The viscosity of composite B is higher than that of gum compound for all the five shear rates. This high viscosity is due to the restriction of molecular mobility by the presence of short fibers. This phenomenon can be explained on the basis of scanning electron micrographs of the extrudates of this composite. Figures 5a and 5b show the surface and cross-section of the extrudates of composite B containing 5 phr of acetylated coir fiber at high shear rate viz., 1224 sec^{-1}. It can be observed that the distribution of fibers is not uniform and the fibers tend to be present on the outer surface layers of the extrudate.

Figure 4 shows the effect of shear stress on viscosity of natural rubber composites

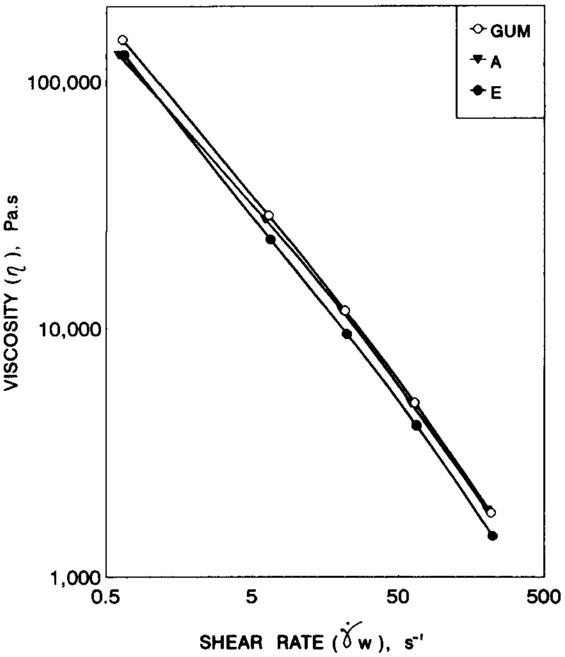

FIGURE 2 Effect of shear rate on melt viscosity.

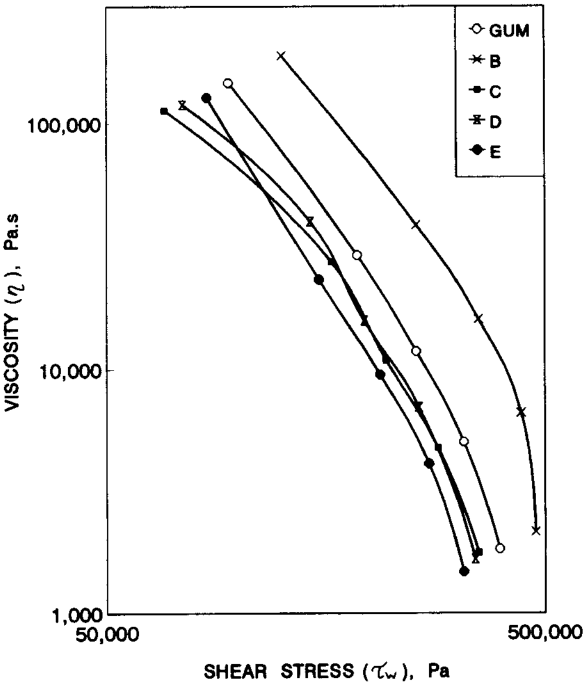

FIGURE 3 Effect of shear stress on melt viscosity.

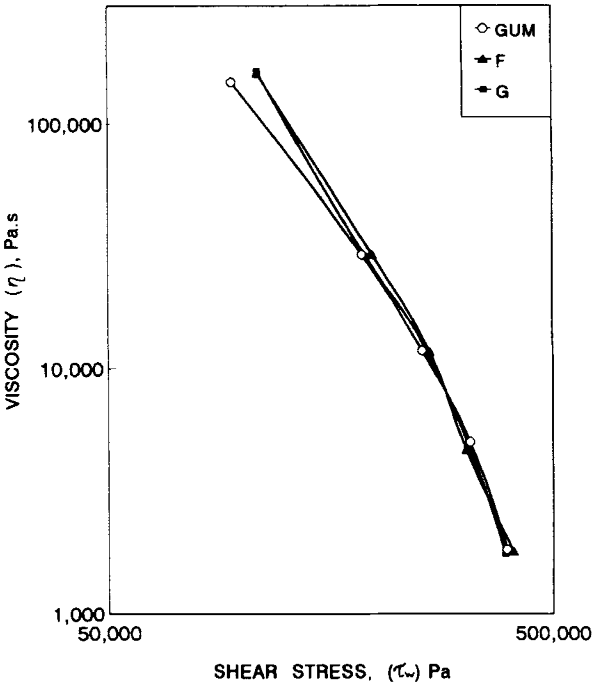

FIGURE 4 Effect of shear stress on melt viscosity of composites containing γ-irradiated coir fibers.

containing gamma irradiated coir fiber. Mix 'F' and 'G' containing 30 phr coir fibers subjected to 0.5 MRad and 4 MRad, respectively. Except at high shear rate, the melt viscosity of both of these composites is almost similar. This shows that the time of gamma irradiation has only little effect on melt viscosity of the composite. However, the viscosity of the irradiated fiber compounds is higher than that of the gum compound at low shear rates. This may be associated with the improved interaction between the fibre and matrix as a result of γ-radiation. However, at high shear stress, this interaction disappears.

But the viscosity of composites C, D and E is lower than that of gum compound (Figure 3). This can be explained in terms of a sheath-core structure in the extrudate where the less viscous natural rubber matrix encapsulates the hard coir fibers. At higher fiber loading (20 phr) majority of the fibers are concentrated in the central portion of the extrudate forming a sheath-core structure (Figure 6). At still higher fiber loading (30 phr) also, the same phenomenon can be observed (Figure 7).

Thomas *et al.*[24] and Crowson *et al.*[28] have reported the stratification phenomena in polymer blends and composites. They concluded that the differences in shear viscosity between the two components lead to interface distortion and this results in the encapsulation of the high viscous melt by the low viscous melt. At high shear rates, the stratification of the extrudates of coir fiber reinforced natural composites is associated with the migration of the low viscosity natural rubber constituent to the surface of the extrudate forming a sheath around the more stiff coir fibers.

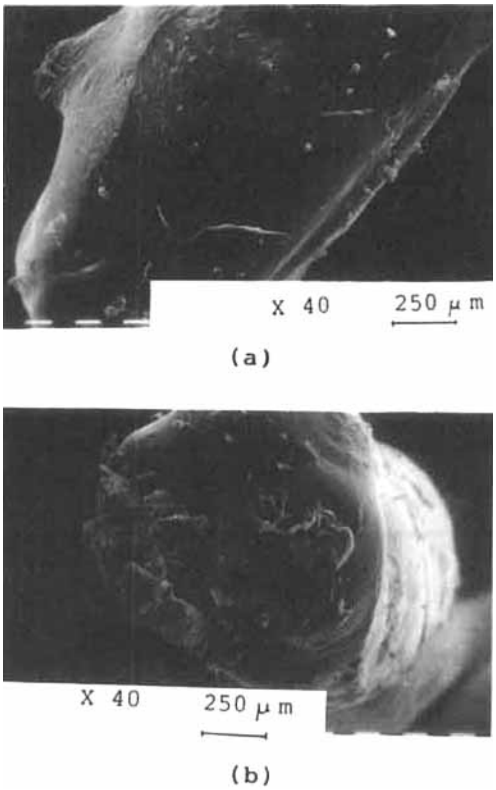

FIGURE 5 Scanning electron micrographs of the cross-section of composite B which contains·5 phr of acetylated coir fiber extruded at a shear rate of 1224 sec^{-1}: (a) surface, (b) cross section (magnification ×40).

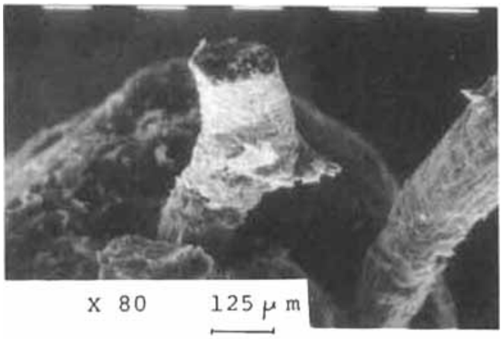

FIGURE 6 Scanning electron micrograph of natural rubber composite D which contains 20 phr acetylated fiber extruded at a shear rate of 1224 sec^{-1} (magnification ×80).

MacLean[25] and Everage[26] explained the stratification of multiphase systems based on the tendency to attain a configuration with the lowest rate of viscous dissipation.

The rheometric studies also support the same phenomenon. In Figure 8 the Monsanto Rheographs of the gum compound containing natural rubber alone and that of

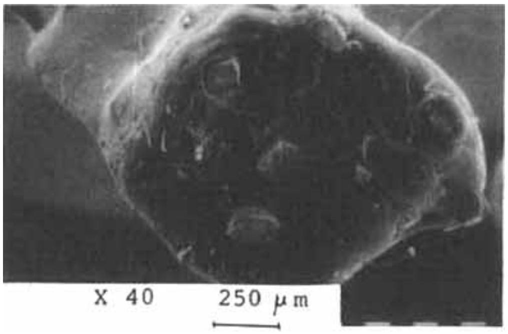

FIGURE 7 Scanning electron micrograph of the cross-section of composite E containing 30 phr acetylated coir fiber extruded at a shear rate of 1224 sec^{-1} (magnification ×40).

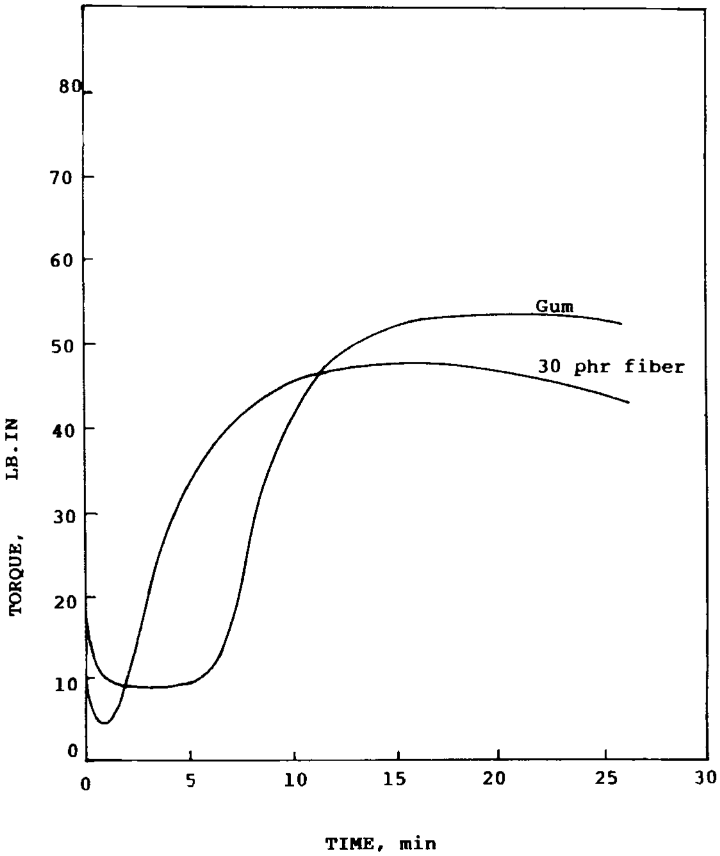

FIGURE 8 Monsanto rheograph of gum compound containing natural rubber alone and composite containing 30 phr fiber.

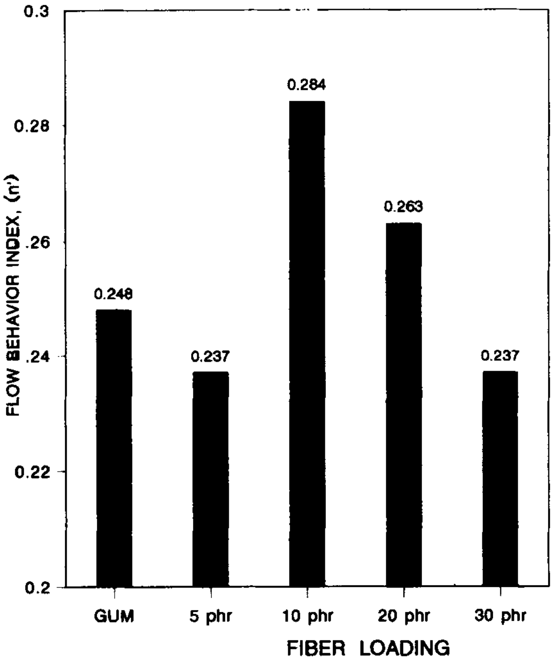

FIGURE 9 Effect of fiber loading on flow behavior index (*n'*).

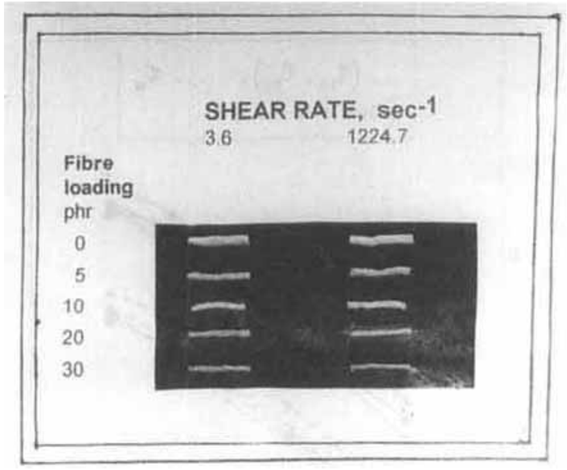

FIGURE 10 Photomicrograph of extrudates fibers of different loading at two shear rates 3.67 and 1224.66 sec^{-1}.

the composite containing 30 phr acetylated coir fiber are shown. It is clear that the maximum torque is lower in the case of the fiber containing composite.

Goettler[27] reviewed the studies of Dzyura *et al.*[23] who reported that the fibers reduce the viscosity of composite materials because of the higher pseudoplasticity induced by the fibers. More studies about the anomalous melt behavior of coir fiber reinforced natural rubber composites are under progress.

TABLE II

Variation of die swell of compounds with fiber loading at different shear rates

	d_e/d_c					
$\dot{\gamma}_w$ (sec^{-1})	Gum	C	D	E	F	G
3.6740	1.47	1.30	1.02	1.02	1.12	1.14
36.7399	1.52	1.33	1.15	1.16	1.18	1.06
122.4663	1.58	1.37	1.15	1.18	1.20	1.19
367.3990	1.60	1.37	1.17	1.12	1.24	1.02
224.6633	1.64	1.22	1.10	1.02	1.29	1.09

TABLE III

Variation of principal normal stresses with fiber loading at different shear rates

	$\tau_{11} - \tau_{22} \times 10^{-6}$, Pa					
$\dot{\gamma}_w$ (sec^{-1})	Gum	C	D	E	F	G
3.6740	1.90	2.71	1.49	1.70	2.17	2.17
36.7399	3.73	3.26	2.91	3.05	3.94	3.80
122.4663	5.09	4.34	3.86	4.20	5.29	5.22
367.3990	6.52	5.70	5.15	5.93	6.3	7.47
1224.6633	7.87	7.05	6.92	6.51	8.9	7.80

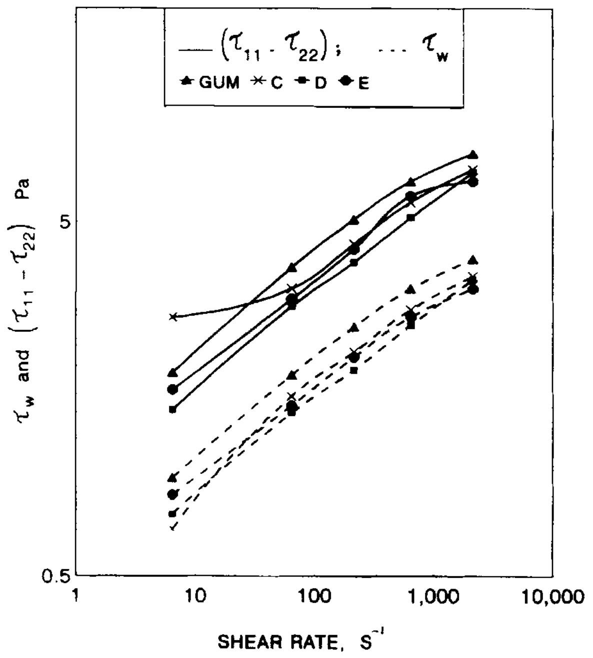

FIGURE 11 Variation of principal normal stress ($\tau_{11} - \tau_{22}$) and shear stress (τ_w) with shear rate.

TABLE IV

Variation of recoverable elastic shear strain (γ_R) with fiber loading

	R					
$\dot{\gamma}_w$ (sec^{-1})	Gum	C	D	E	F	G
3.6740	4.30	2.78	1.24	0.50	1.40	1.60
36.7399	4.76	2.43	1.63	1.72	1.89	0.98
122.4663	5.39	3.42	1.66	1.89	2.01	1.92
367.3990	5.61	3.35	1.78	1.42	2.32	0.50
1224.6633	6.09	2.16	1.30	0.50	2.69	1.24

TABLE V

Variation of elastic shear modulus (G) with fiber loading

	$G \times 10^{-6}$, Pa					
$\dot{\gamma}_w$ (sec^{-1})	Gum	C	D	E	F	G
3.6740	2.2	4.8	6.0	16.9	7.7	6.7
36.7399	3.4	5.4	8.9	8.8	10.3	19.3
122.4663	4.7	6.3	11.6	11.0	13.1	13.5
367.3990	5.8	8.4	14.4	19.0	13.7	74.1
1224.6633	6.45	1.6	26.5	64.8	15.1	31.4

The flow behavior index, n', values give an indication about the non-Newtonian or pseudoplastic nature of a polymeric material. The composite E containing acetylated coir fibers and resorcinol and hexa as bonding agent has a lower n' value (0.237) than that of composite A (0.275) which contain untreated fiber but no bonding agent. This indicates the higher pseudoplastic nature of compound E.

Figure 9 shows the variation of n' values with fiber loading. It can be seen that n' first decreased with the addition of 5 phr coir fiber but then showed a sharp increase on adding 10 phr fiber. Again the n' values decreased gradually for natural rubber compounds containing 20 and 30 phr acetylated coir fiber.

Melt Elasticity

Information about the elasticity of the melts of coir-natural rubber composites can be obtained from the extrudate distortion, die swell ratios (d_e/d_c), first normal stress difference ($\tau_{11} - \tau_{22}$), recoverable shear strain (γ_R) and the elastic shear modulus (G) of coir-rubber composites. These properties were evaluated according to the Equations 5, 6, 7 and 8 respectively.

Extrudate Distortion

Figure 10 shows the photograph of the extrudates obtained from the capillary for various mixes at two different shear rates, viz., 3.67 and 1224.66 sec^{-1}. The extrudate distortion of the natural rubber compound which does not contain fibers is significant at high shear rates. Incorporation of fibers reduces the extrudate distortion and this phenomenon becomes pronounced as the loading of fibers is increased. But the distortion increases with the increase in shear rates for a particular fiber loading.

Die Swell

This phenomenon occurs as a result of the orientation of polymer molecules as they are sheared while passing through the die of an extruder. On emerging from the die, they tend to recoil with shrinkage in the direction of flow but expand at right angles to the flow, because of the absence of continuing shear stresses. This results in the die swell.

Table II shows that the die swell values of gum rubber compound increases with increasing shear rates and it decreases with increase in fiber loading. For a particular shear rate the decrease in elasticity with increase in fiber concentration was only marginal after 20 phr fiber loading. The short coir fiber creates discontinuity in the natural rubber matrix. Hence the stress transfer will not be continuous for elastic recovery. These fibers are oriented as a result of extrusion and are incapable to disorient on relaxation. This provides a hindrance for the rubber matrix to relax and hence die swell will be lower.

Table III shows the variation of the principal normal stress difference ($\tau_{11} - \tau_{22}$) of natural rubber composites with loading of coir fiber at different shear rates. The gum compound exhibits higher $\tau_{11} - \tau_{22}$ values than those of natural rubber composites (C, D and E) containing acetylated coir fiber at all shear rates except at very small shear rate 3.67 s^{-1}. Crowson *et al.*[28] reported that elasticity is indicated by large die swell, large Weissenberg rod climbing effect and large normal stress differences. Thus it is clear that the elasticity of gum compound is higher than that of natural rubber compounds containing short coir fibers. Also it can be seen that the $\tau_{11} - \tau_{22}$ values increase with both shear rate and shear stress. The increasing melt elasticity with increasing shear rate is evident from these values.

Figure 11 represents variation of the principal normal stress difference and shear stress with shear rate. It can be seen that the magnitude of $\tau_{11} - \tau_{22}$ is much higher than that of shear stress for each rubber compound.

The recoverable elastic shear strain (γ_R) of all the composites are lower than the gum compound (Table IV). The elastic shear modulus (G) of the gum compound and the fiber composites are shown in Table V. These values are higher for composites containing coir fibers.

CONCLUSION

The rheological behavior of natural rubber composites containing short coir fibers was investigated. These composites obey the power law model for fluids. At low level of fiber loading the shear viscosity of the composite was found to be higher than the unfilled rubber compound. But at higher fiber concentrations the change in viscosity with fiber loading showed some anomalous behavior. This is explained on the basis of sheath-core structure formation at high fiber loading. The soft natural rubber present as the sheath facilitates the easy extrusion of the composite and thus a decrease in viscosity was observed. The effect of fiber loading on first normal stress difference, recoverable shear strain and the elastic shear modulus was analyzed. The change in pseudoplasticity index with the loading of acetylated coir fibers was investigated. Incorporation of fibers enhanced the resistance to shape distortion of the extrudates. This property increases with increase in fiber loading. The die swell of composites containing coir fibers was lower than that of gum compound and it was negligible for composites containing higher amount of coir fibers viz., 20 and 30 phr. Further studies are in progress on the anomalous rheological behavior of short coir fiber reinforced natural rubber composites.

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