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Mechanical and Rheological Properties of Short Nylon Fibre NR/SBR Composites

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NR/SBR composites were prepared with virgin and waste short nylon fibre using a two-roll mill. The effects of the fibres and bonding agent content on the microstructure, mechanical and rheological properties of the composites were investigated. The curing characteristics of the composites were determined and subsequently vulcanised at 150ºC using a hot press. The cure and scorch times of the composites decreased while the maximum torques increased with increasing fibre loading. The adhesion between the fibre and rubber was enhanced by using a dry bonding agent consisting of resorcinol, hexamethylenetetramine and hydrated silica (HRH). The bonding agent provided a shorter curing time and enhanced mechanical properties. The fracture surfaces of samples were examined by scanning electron microscopy (SEM). The presence of fibres in the matrix influenced the fracture modes leading to pull out and breakage of fibres. Anisotropic swelling studies were carried out to study the fibre matrix interaction and fibre orientation. The restriction to swelling is higher for composites containing bonding agent and the preferential orientation of the fibres is in the milling direction. For composites prepared with virgin and waste fibres, the dependence of the storage modulus (G') on angular frequency followed a clear non-terminal behaviour.

Keywords: NR/SBR composites; waste short nylon fibre; dry bonding agent; mechanical properties; morphology; rheology

Short fibre-rubber composites have achieved significant importance both in the industrial field and in the area of research and development[1–4]. Reinforcement of rubber with short fibre combines the elasticity of rubber with strength and stiffness of the fibre. Since this material is also light weight, there is tremendous potential in strength/weight or stiffness/weight ratios over conventional materials[5]. Moreover, the short fibres can be incorporated directly into the rubber compound along with other additives hence producing composites that are amenable to standard rubber processing techniques of extrusion, calendaring and various types of moulding operations. This is in contrast to the slower processes required for incorporating and placing continuous fibres[6,7]. Recently short fibre reinforced rubber has gained importance due to its advantages for instance, design flexibility, anisotropy in technical properties, stiffness, damping and processing economy[8–12]. Elastomers reinforced with continuous fibres are well-known, but is limited mainly to application in tyres, belts and hoses. The manufacture of articles

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of complex shapes and high performance applications (thermal insulators) cannot be easily accomplished with a continuous fibre reinforced elastomer[5]. On the other hand, the preparation of a complex engineering component can be accomplished by using short fibres as the reinforcing medium for an elastomer, which can be processed by the well-known techniques used in the rubber industry. Design of short fibre-reinforced rubber composites mainly depends on several factors such as aspect ratio (L/D) of the fibre, control of fibre orientation as well as dispersion and existence of a strong interface between fibre and rubber. Chemical structures of both the fibre and the matrix as well as the presence of a bonding system determine the extent of interfacial adhesion and thus the strength of the composites[13–15]. Many researchers have used short glass fibres for reinforcing rubbers because of their high modulus, high strength and low creep[2,10]. Manchado and Arroyo[4] studied the effects of aramid, glass and cellulose short fibres on the processing behaviour, crosslink density and mechanical properties of NR, SBR and EPDM rubber composites. Furthermore, recent studies[5,16] on natural fibres have shown that it is possible to obtain good performing materials by using environmentally friendly reinforcement agents[17–21]. The effect of fibre–matrix adhesion, aspect ratio of filler, fibre dispersion and orientation, nature of matrix and type of fibre on the extent of reinforcement has also been studied[22–25]. In recent years, many researchers have been occupied with solving the disposal problems created by waste rubber vulcanisates, as they do not decompose easily. Waste fibres represent another type of environmental problem when subjected to expensive recycling processes. In the process of tyre recycling, approximately 10% waste fibres are obtained. A process that could use these types of wastes would represent an important environmental benefit and great economic savings for communities[26–28]. In this study, NR/SBR composites were prepared using waste and virgin short nylon 66 fibres with bonding agents such as resorcinol, hexamethylenetetramine and hydrated silica (HRH). The effect of fibre loading and the bonding agent on cure characterisation, mechanical properties, morphology and rheology of the composites were investigated as well.

EXPERIMENTAL

Materials

Natural rubber (SMR 20) and also Styrene-Butadiene Rubber (SBR 1502) with ML (1+4) at 100ºC = 54 (Bandar-Emam petrochemical company, Iran) were used. Virgin nylon 66 fibres in yarn form were obtained from Iran Saba Fibre Co., with 1260 denier and diameter of 0.18 mm which were chopped to approximately 6 mm in length. The waste nylon fibres in cord form were the by-product of Yazd Tyre Reclamation with diameter of 0.68 mm which were chopped to approximately 6 mm in length. N-cyclohexyl-2-benzothiazyl sulfenamide (CBS), sulfur (S), zinc oxide (ZnO) and stearic acid were used for the curing system. A combination of resorcinol, density = 2.36 g/cm^3, hexamethylenetetramine, density = 1.33 g/cm^3 and hydrated silica (Vulcasil S), density= 2 g/cm^3 were used as the bonding agent between rubber and fibre from Bayer company.

Preparation

The sample formulations employed in this study are given in *Table 1*. The composite materials (both virgin and waste fibres) were prepared in a two-roll mill model (Polymix 200L) at a roll speed of 50 r.p.m. and temperature 50ºC. The nip gap, mill-roll speed ratio, mixing time and temperature, and sequence of mixing were kept constant for all compounds. The orientation of fibre in the

TABLE 1. SAMPLE NAMES AND COMPOSITIONS

Ingredients	Sample code								
	B_oF_o (1)	BF_o (2)	BF_{10} (3) BWF_{10} (10)	BF_{20} (4) BWF_{20} (11)	BF_{30} (5) BWF_{30} (12)	BF_{40} (6) BWF_{40} (13)	B_oF_{30} (7) B_oWF_{30} (14)	$B_{1/2}F_{3o}$ (8)	B_2F_{30} (9)
NR	40	40	40	40	40	40	40	40	40
SBR	60	60	60	60	60	50	60	60	60
ZnO	5	5	5	5	5	5	5	5	5
Stearic acid	2	2	2	2	2	2	2	2	2
Resorcinol	0	2.5	2.5	2.5	2.5	2.5	0	1.3	5
Hexamine	0	1.6	1.6	1.6	1.6	1.6	0	0.8	3.2
Silica	0	5	5	5	5	5	0	2.5	10
Virgin Fibre	0	0	10	20	30	40	30	30	30
Waste Fibre	0	0	10	20	30	40	30	0	0
CBS	0.8	0.8	0.8	0.8	0.8	0.8	0.8	0.8	0.8
Sulfur	2	2	2	2	2	2	2	2	2

B: bonding agent,
F: virgin fibre,
WF: waste fibre

uncured compound was achieved in rolling direction by passing the sample through a controlled nip. The rolling direction was kept constant to promote enhanced fibre orientation, (shear forces occurring during milling operations orient most of the fibres along the grain direction). The sheeted rubber compound was conditioned at room temperature (25ºC) for 24 h before vulcanisation.

Characterisation

Zwick rheometer, model 4308, was used to determine cure characteristics data for composites at 150ºC according to *ASTM D2084*. The samples were vulcanised at 150ºC in a hydraulic press 25 ton (Davenport Ltd., England) under 180 kg/cm^2 pressure for a period, based on data obtained from the rheometer. Stress-strain measurements were carried out by using a tensile testing machine, HIWA 200 at a crosshead speed of 500 mm/min. Dumb-bell shaped samples (thickness = 2 mm) were cut along (longitudinally) and across (transversely) in the grain direction. Mechanical properties were determined according to *ASTM D412*. The hardness was measured at room temperature using a shore-A hardness Tester (Zwick Company) according to *ASTM D2240*. Resilience was determined at 28ºC using the Resilience Tester Frank according to *ASTM D1054*. Abrasion loss was measured with the Frank Abrasion Tester as *ASTM D5963*. Anisotropic studies were carried out according to *ASTM D3616* using rectangular samples with dimensions of 12.52 mm cut from the tensile sheets in both directions and swollen in toluene at room temperature for 2 days. The morphology of cryogenically fractured composite surfaces were observed under a Cambridge Stereo scan 360, scanning electron microscope (SEM). The rheological characterisation of samples was carried out using a stress controlled rheometer (RPA 2000); experiments were performed at a temperature of 80ºC and in the frequency range of 0.01–30 Hz.

RESULTS AND DISCUSSION

The results of rheometric studies for the samples are given in *Figures 1–3* and the cure characteristics of the compounds are summarised in *Table 2*. *Figure 1* shows that the minimum and maximum torques of compounds increased with the incorporation of virgin nylon fibre to the bonding system. The addition of fibre alone into the matrix (compound 7) caused a decrease in the scorch time and optimum cure time with an increase in the optimum cure rate of the composite in comparison with compound 1 as shown in *Figures 1* and *2* respectively. It can be seen that the maximum-minimum torque of the compounds, which is an indication of the optimum crosslink density[6], increased with fibre content, suggesting that the fibre plays a role in the curing process of the composite. The incorporation of compound no 2 alone into the matrix of the dry bonding system gave a decrease in the scorch time and optimum cure time but caused no significant change in the cure rate in comparison with compound 1. According to Bhowmick *et al.*[29] the silica in the bonding system may retard the cure rate. The addition of fibre to the virgin matrix (compound 7) caused an increase in the cure rate compared to that of the compound 1, due to the presence of amine groups on the fibre surface, which is in accordance with the results reported in the literature[28–29]. Therefore, nylon fibres itself have a role in the sulfur curing process of NR/SBR rubber. When the fibre and dry bonding system were added to the matrix simultaneously (compounds 3, 4, 5, and 6), the scorch time and optimum cure time were decreased; the cure rate was increased with respect to the compound containing either the fibre or dry bonding system alone

TABLE 2. CURE CHARACTERISTICS OF COMPOSITES

Sample Code	Max. Torque (dNm)	Min. Torque (dNm)	Cure Time (t_{90}) (min)	Scorch Time (t_5) (min)	Cure Rate (dNm)/(min)
1	7.92	1.05	13.8	7.25	1.06
2	8.8	1.51	10.61	3.91	1.08
3	13.05	1.9	9.29	3.47	1.86
4	15.9	2.2	8.96	3.38	2.5
5	21.17	2.57	8.32	3.25	3.26
6	27.2	3.35	7.97	3.14	4.93
7	18.9	2.2	12.58	5.12	2.51
8	20.44	2.32	10.39	4.2	3.04
9	22.29	2.86	7.31	2.2	3.8
10	11.26	1.53	10.19	3.8	1.5
11	12.86	1.82	9.2	3.71	2.1
12	14.03	2.1	8.8	3.5	2.4
13	15.69	2.46	8.1	3.3	3.1
14	12.5	2.56	7.31	1.95	2.79

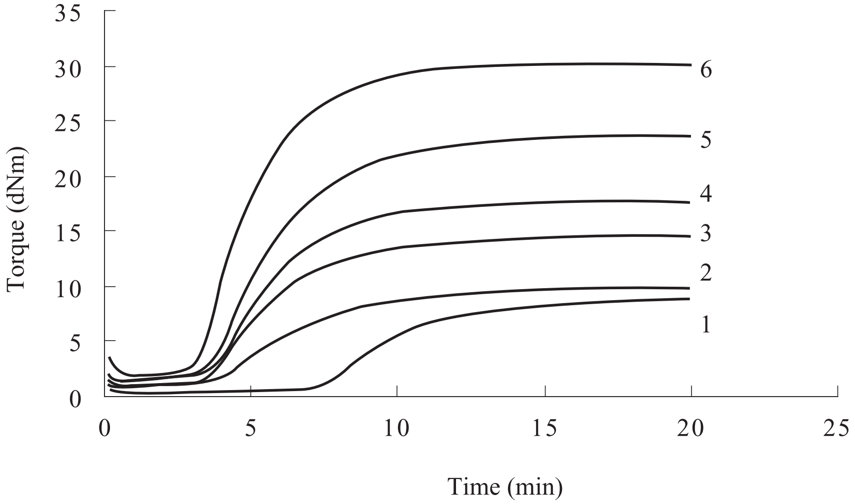

Figure 1. Rheographs of composites prepared with virgin fibres at 150ºC. (Numbers to curves represent compounds)

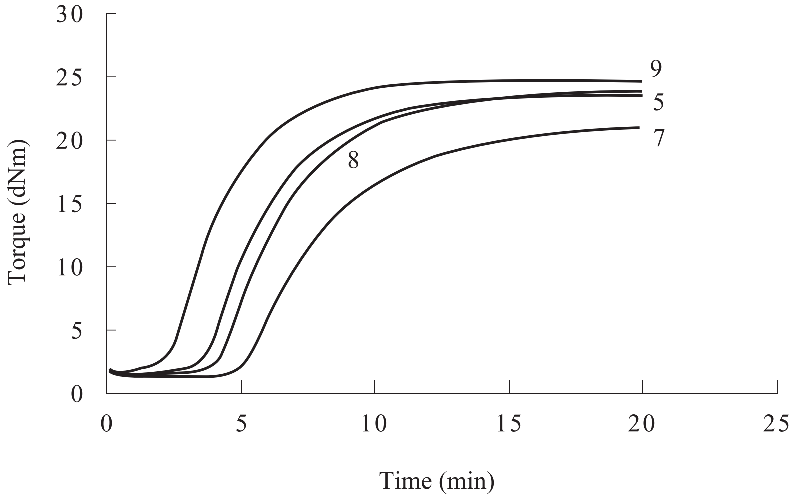

Figure 2. Rheographs of composites prepared with virgin fibres and different bonding agent content at 150ºC.
(Numbers to curves represent compounds)

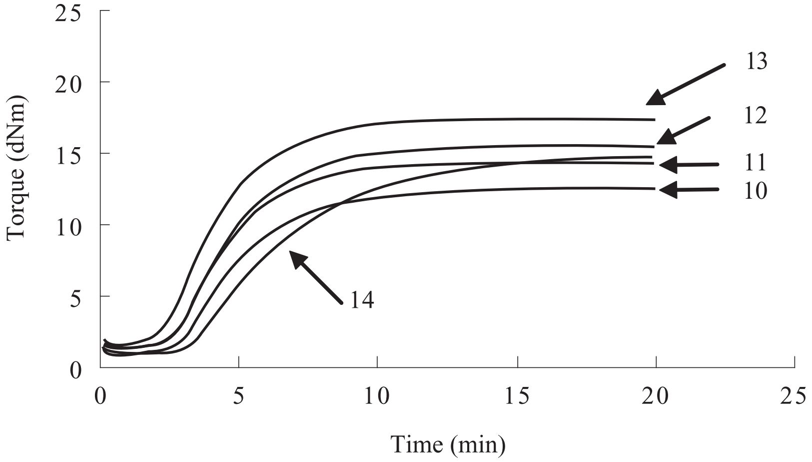

Figure 3. Rheographs of composites prepared with waste fibres at 150ºC.
(Numbers to curves represent compounds)

(compounds 7 and 2). This observation suggests that the amine groups in the bonding agent (hexamine) and in nylon fibres (generated from the degradation of fibres at the curing temperature[28]), establishing an alkaline pH media, accelerate the curing reaction of the sample. In fact, the concentration of amine groups in this compound is high enough to neutralise the retarding effect of silica[29]. *Figure 2* shows the effect of HRH bonding system concentration on cure characteristics of the composites prepared with 30 wt% of virgin short fibre. Here, the maximum torque increased and reduction of scorch time was obtained when the concentration of bonding agent was increased. *Figure 3* shows the rheographs of composites prepared with different waste fibre loadings. It could be observed that in the composites prepared with waste fibre, the reduction of cure time was less than that of the virgin nylon fibre compounds as shown in *Figures 3* and *1* respectively. It is necessary to declare that waste fibres used to prepare the composites, have been coated by the supplier before use. They may also contain some impurities remaining of the tyre rubbers, thus, the amine groups resulting in the acceleration of cure reaction have been deactivated in waste fibres. Consequently, the number of interactions in the virgin fibre composites is much more than that of the composite prepared with the waste fibres. The mechanical properties of the composites prepared with both virgin and waste fibres are shown in *Table 3*. The presence of fibres produced a marked anisotropy in tensile properties of the composites. *Table 3* also shows the variation of tensile strength in NR/SBR compounds with different fibre content. The tensile strength increased linearly with increase in fibre loading up to 30 p.h.r. and then showed a slight increase for composites containing 40 p.h.r. fibres. However, in the transverse direction, the mechanical properties of compounds improved slightly as the fibre content was increased (*Figure 4*). In addition, it was observed that the nylon fibres in the absence of a suitable bonding agent (compound 7) did not reinforce the matrix (compared with the compound including bonding agent (compound 5)). Noticeably, the increase in tensile strength in the longitudinal direction is much higher than that in the transverse direction which highlights the anisotropy of these composites. It is well known that longitudinally oriented composites show brittle behaviour while transversely oriented composites show elastic deformation and, fibres act as barriers that prevent the distribution of stress throughout the matrix[6]. It should be noted that the tensile strength of the composites prepared with virgin fibre was greater than that of waste-fibre compound (*Figure 5*). This may be attributed to the higher diameter of cord-type waste fibres (According to the data aforementioned in the Experimental section) and consequently their lower aspect ratio (L/D) in comparison with the virgin fibres as well as poor adhesion between waste fibres and the rubber matrix. *Table 3* shows the dependence of the elongation at break versus the fibre concentration in the composites. Clearly, the addition of fibre leads to a sharp decrease in the elongation at break in both directions. On the other hand, the longitudinal elongation is lower than that of the transverse direction for all concentrations. This observation indicates the fibres were well oriented in the rubber matrix in the mill direction[29]. *Table 3* affirms that the elongation at break in compounds prepared with bonding agent is lower than compounds prepared without bonding agent at similar fibre loading. This is probably due to the stronger adhesion between fibre and the rubber matrix which would consequently result in a lower elongation at break[30,31]. An increase in hardness is observed with virgin and waste fibre loading. The hardness of composites prepared with the fibres and bonding agent was higher than that of compounds prepared without bonding agent at similar fibre loading. Therefore, the

TABLE 3. MECHANICAL PROPERTIES OF COMPOSITES

Sample code	Tensile Strength (MPa)	Elongation at Break (%)	Hardness (Shore A)	Resilience (%)	Abrasion (%)
1	5.01	456.8	47	71	25.6
2	5.97	462.1	48	70	25
3	L 13.49 T 6.20	35.6 338.3	79	61	21.6
4	L 17.5 T 8.1	26.9 116.4	82	56	19.8
5	L 24 T 11.1	24.9 40.4	87	55	18
6	L 26.9 T 11.3	21.2 36.7	89	57	14.2
7	L 20.7 T 8.8	30.6 78.1	86	59	27
8	L 22.8 T 9.6	23.3 45.9	87	61	22.7
9	L 24.7 T 10.1	26.5 64.5	88	57	23.9
10	L 8.5 T 6.8	40 350	72	62	22.2
11	L 12 T 7.1	36.1 200.3	76	60	21
12	L 17.5 T 9	30 103.5	82	57	19.9
13	L 19.2 T 9.1	28 70	86	58	18
14	L 17.9 T 7	19.1 97	88	60	18

adhesion between the rubber and the fibre plays an important role in adequate fibre reinforcement in the rubber composites[22,24]. A continuous decrease in resilience was observed with increasing waste fibre concentration in all the composites (*Table 3*). When the fibre content is increased, the energy is predominantly dissipated at the fibre-matrix interface, hence the resilience of composites is reduced[28]. The abrasion loss of the fibre filled samples (virgin and waste fibre) was lower than that of the unfilled compound as shown in *Table 3*. This is due to the more restrained matrix of the composite so that fibres do not separate from the matrix easily[17]. The anisotropic solvent swelling behaviour of all the composites indicated that the restriction to swelling is higher for composites containing bonding agent. This indicates that the bonding agent improves the interfacial bond strength between the fibre and matrix. According to Kumar[12], the orientation of fibres in one direction of fibre composites leads to lower swelling indexes measured for the rectangular samples cut specially for measuring the swelling index at that direction. Solvent swelling studies also confirm the preferential orientation of the fibres in the milling direction because it is restricted to a greater extent in the longitudinal direction than in the transverse direction, which is evident from the lower swelling index in the longitudinal direction, as shown in *Figures 4* and *5*. Studies on the fibre-matrix adhesion based on SEM were carried out using composites 7, 14, 5, and 12 which contained both virgin and waste fibres. *Figures 8a* and *b* show the SEM photomicrographs for the compounds 7 and 14 in longitudinal direction (L). Long segments of fibre and de-bonding of the fibres from the matrix show poor bonding between the fibres and the matrix leaving cavities at places where the fibres were pulled out from the matrix when stress was applied. However, when the bonding agent was introduced, the morphology of the fracture surface (Compounds 5 and 12) altered significantly with the presence of short fibre broken ends protruding from the fracture surface as shown in *Figures 8c* and *d*. These observations confirmed an orientation of fibres in the milling direction. *Figures 8e* and *f* are the SEM photomicrographs of the tensile fracture surface for the samples 5 and 12 which were cut in the transverse direction (T). Here, fibres appeared to be lying mostly parallel to the fracture surface, indicating the preferential orientation of the fibres was in the opposite direction. *Figures 9* and *10* show the elastic moduli (G') and complex viscosities (η^*) of

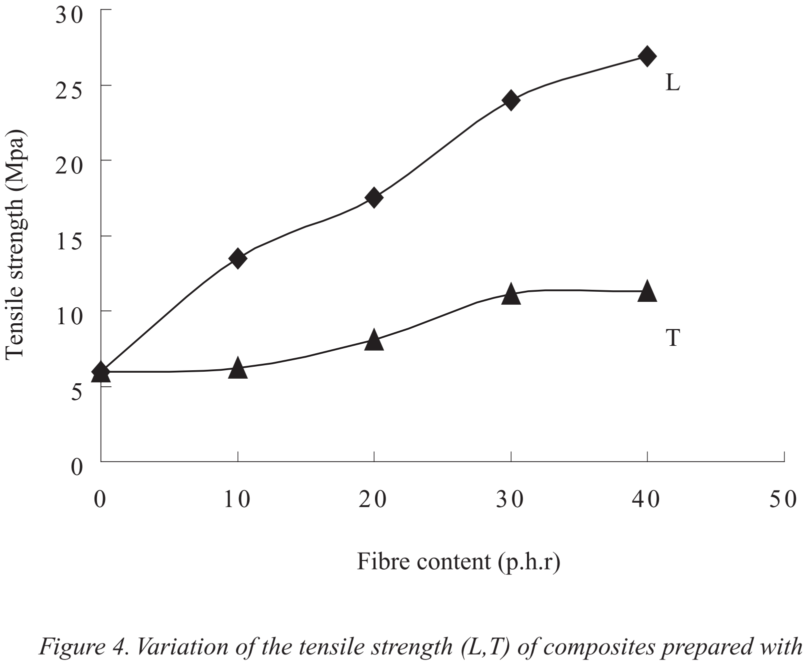

Figure 4. Variation of the tensile strength (L,T) of composites prepared with the virgin fibre loading (compounds 2-6).

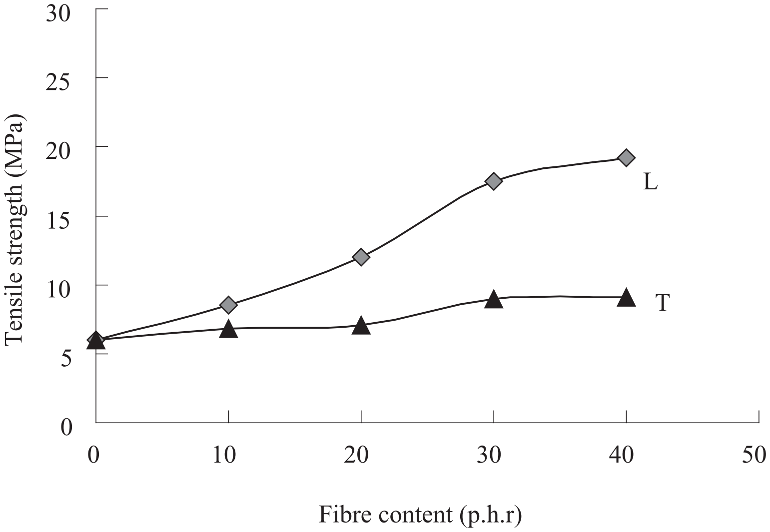

Figure 5. Variation of the tensile strength (L,T) of composites prepared with the waste fibre loading (compounds 2 and 10-13).

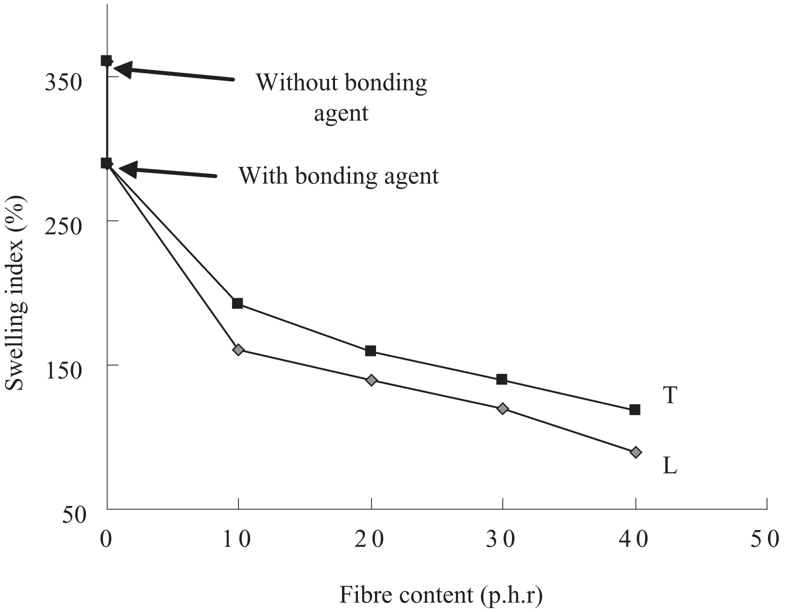

Figure 6. Variation of the swelling index (L, T) of composites prepared with the virgin fibre loading.

composites prepared with virgin and waste fibres as a function of angular frequency respectively. The NR/SBR prepared without fibres exhibited a viscosity that approaches Newtonian behaviour towards the low frequencies. The viscosity of all composites clearly showed a shear thinning behaviour over the entire range of shear rates used for the characterisation. The increase in the storage modulus and complex viscosity were found to be larger in the composites prepared with virgin fibres in comparison with the waste fibre composites.

CONCLUSION

The cure characteristics, mechanical and rheological properties of virgin and waste nylon fibre reinforced NR/SBR composites have been investigated. The introduction of fibre or dry bonding system into the compound reduced the cure and scorch times while the maximum torques and cure rate increased. The NR/SBR composites displayed anisotropic mechanical properties due to the preferential orientation of nylon fibres in the milling direction (longitudinal). In the samples prepared with the fibres (virgin and waste) and the bonding agent, mechanical properties were higher in the longitudinal direction compared to the transverse direction. The anisotropy in properties of the composites as well as the strong bonding of the fibre to the matrix in the presence of the dry bonding system was confirmed by SEM studies. The increase in the storage modulus and complex viscosity were found to be larger in the composites prepared with virgin fibres compared to the waste fibre composites.

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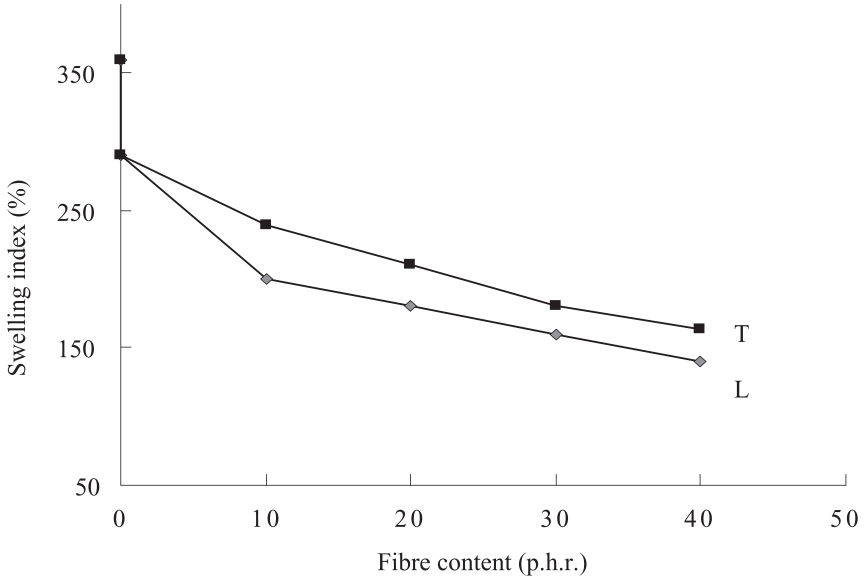

Figure 7. Variation of the swelling index (L,T) of composites prepared with the waste fibre loading.

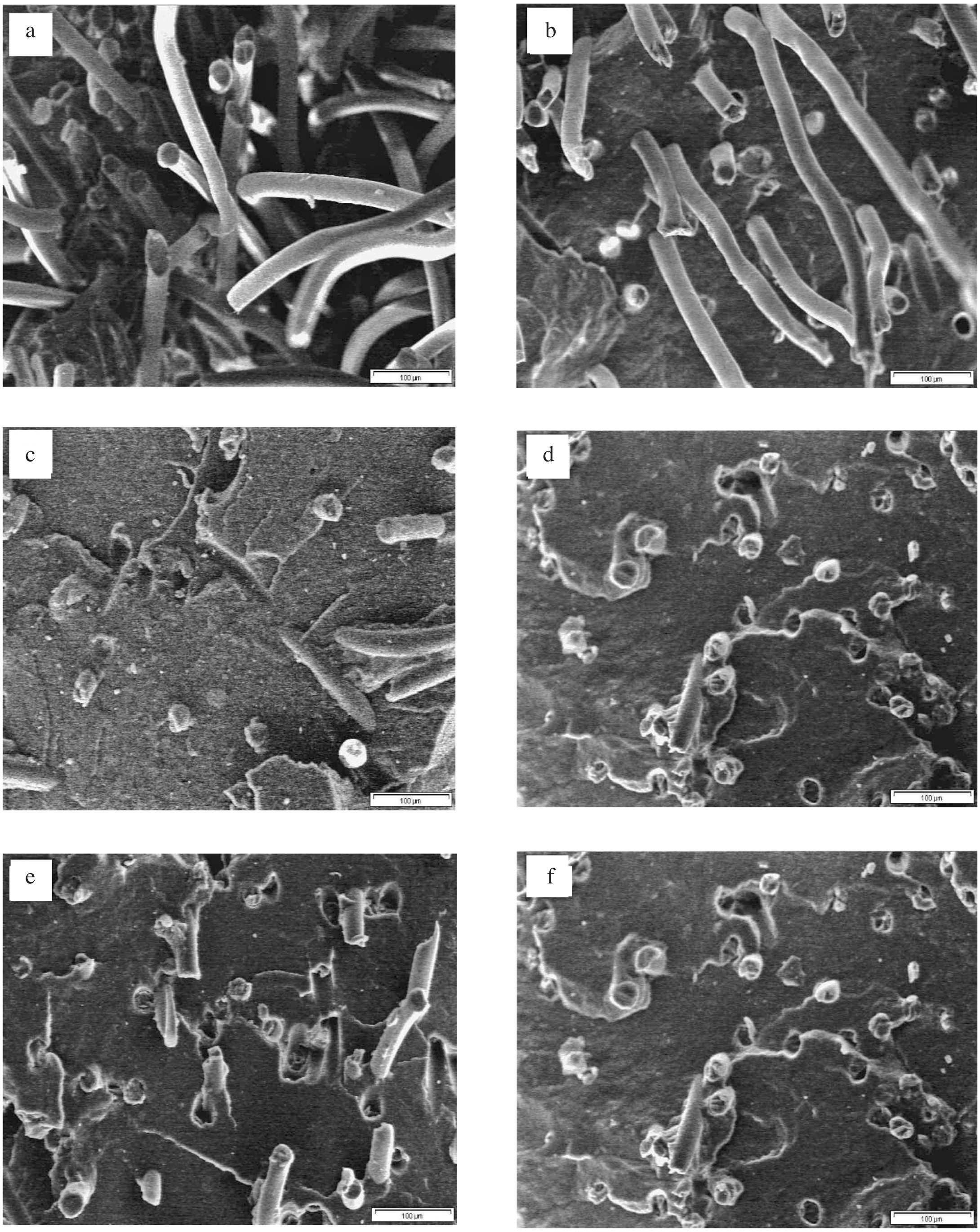

Figure 8. SEM photomicrographs of the composites 7 (a) and 14 (b) in longitudinal direction without bonding agent; composites 5 (c) and 12 (d); composites 5 (e) and 12 (f) in transverse direction with bonding agent.

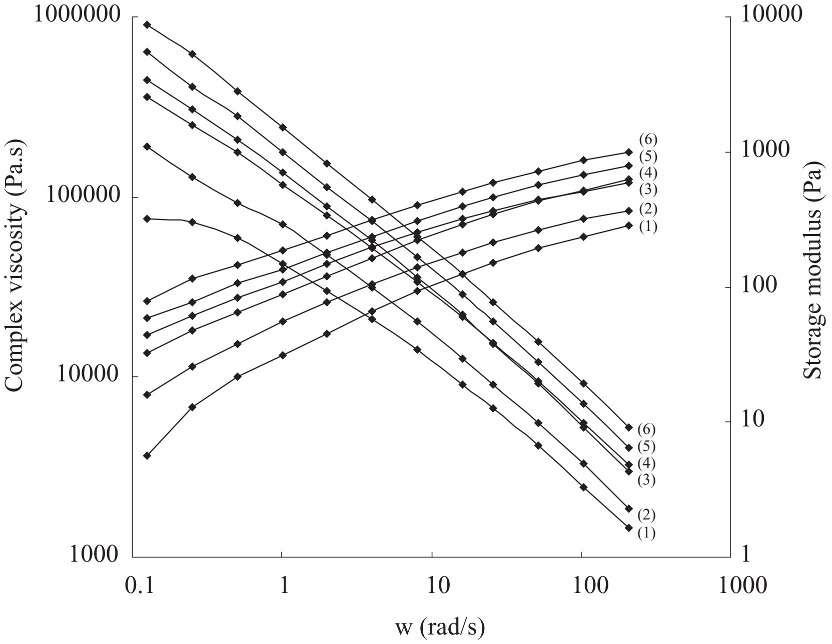

Figure 9. Complex viscosity and storage modulus as a function of frequency for various loadings of virgin fibre and bonding agent.

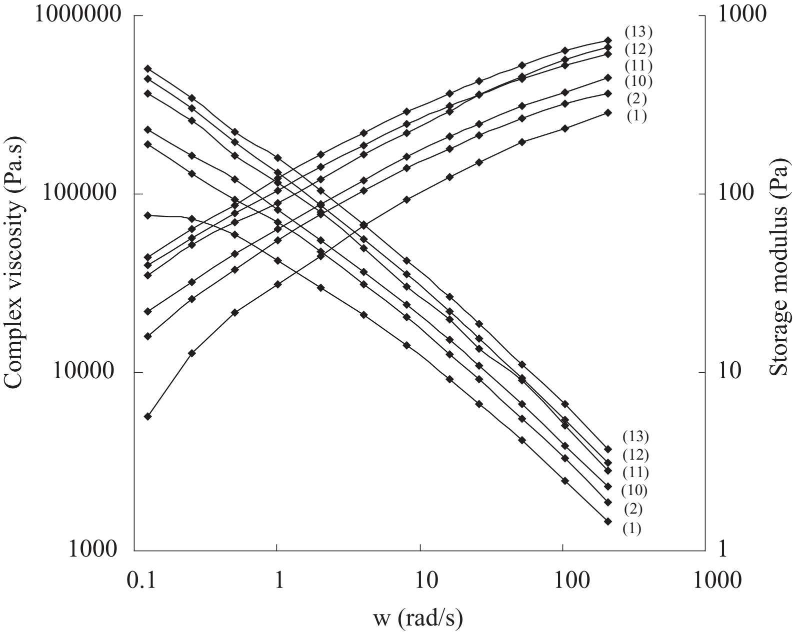

Figure 10. Complex viscosity and storage modulus as a function of frequency for various loadings of waste fibre and bonding agent.

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