Flow Properties of Unvulcanised Natural Rubber/Carboxylated Styrene Butadiene Rubber Latices and their Blends

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ABSTRACT: The rheological behavior of natural rubber (NR) and carboxylated styrene butadiene rubber (XSBR) latices and their blends was investigated, with special reference to the effect of shear rate, temperature, and blend ratio. Arrhenius plots of the blends were drawn and the temperature sensitivity of different blends was determined. All the blends exhibit pseudoplastic behavior, i.e., the viscosity decreases with the increase in shear rate because of the break down of total networks within the system. The viscosity–composition curve showed negative deviation on account of the interfacial slip between the phases. The interfacial slip is occurred with the lack of interaction between the polar XSBR and nonpolar NR phases. For predicting the flow behavior of the blends at a particular temperature and the shear rate, master curves were drawn for the blend systems. The pseudoplasticity index values were determined by Power law analysis. Attempts have been made to correlate the experimental values with theoretical predictions, using Haschin, Heitmiller, and Mashelker–Sood models. © 2007 Wiley Periodicals, Inc. J Appl Polym Sci 104: 2528–2535, 2007

Key words: latex blends; rheology; shear rate; pseudoplasticity; zero shear viscosity

INTRODUCTION

Understanding the rheological properties of the colloidal dispersions is industrially important, both during processing and for end product properties. The flow properties of latices possess considerable importance from fundamental and applied points of view. At the fundamental level the rheology of latices is a direct manifestation of the various interaction forces that occur in the system. At an applied level the rheology studies are vital in many applications, such as food, emulsion, paints, inks, adhesives, and many others.[1–4] These properties of latices are dependent on its component structure, morphology, and distribution of the latex particles. He et al.[5] studied the effect of these factors on the rheological properties of the acrylate emulsion latices.

The knowledge of flow properties of latices is of great relevance, in the field of surface coatings, adhesives, textile, and paper treatments, and in the manufacture of latex-dipped products and latex foam rubber. They are also important for the transportation and handling of latices. From the application point of view the colloid stability and flow properties of latices are equally important, and it is difficult to say which is prominent.[6] The flow properties of latices are dependent on various factors like, the volume fraction of dispersed phase, particle size and particle-size distribution, presence of surface-active agents, and bound electric charges and other additives.[7–13] The viscosity of the system on the other hand, depends on factors like hydrodynamic interaction between the particles or droplets and the liquid, particle–particle interactions, and interparticle attractions that promote the formation of aggregates, flocs, and networks. Recently, Thomas and coworkers[14,15] investigated the influence of surface-active agents, shear rates, temperature, blend composition, prevulcanisation, and maturation time on the rheological behavior of natural rubber (NR) and styrene butadiene rubber latices and their blends.

NR latex is a stable colloidal dispersion of *cis*-1,4-polyisoprene obtained from the Hevea Brasilinesis tree on tapping. NR latex is preserved as low ammoniated latex by adding secondary preservatives. The latex obtained from the tree is having a hydrocarbon

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content of ~33%. Before transportation, the crude latex is concentrated to a rubber content of 60%, mainly by centrifugation to avoid the transport of water, to increase the rubber hydrocarbon content and to improve the uniformity of the product. Moreover, NR latex possesses better mechanical properties, flow properties, and film formation characteristics. So it can be widely used in industry for the manufacture of various products like gloves, balloons, condoms, etc.

The synthetic latices are usually prepared by the emulsion polymerization of its monomers. The latices, such as carboxylated styrene butadiene rubber (XSBR), are prepared by the emulsion polymerization of styrene and butadiene followed by carboxylation. The carboxylation of synthetic latices will enhance the colloid stability. The XSBR latices are resistant to water. They have good mechanical and adhesive properties.

The blending of XSBR with NR will improve the properties of NR latex. The carboxylation of synthetic latices like styrene–butadiene rubber will impart desirable mechanical or optical properties. The blending of hard and soft latex particles is an attractive strategy to develop new durable coatings with very good mechanical properties, and with zero volatile organic compounds.[16] It is envisioned that the latex with low T_g will deform and form a continuous film with embedded high T_g latex particles, whose presence will impart desirable mechanical or optical properties.[17–19] The rheology and the structural analysis of these latices and their blends have relevance in various zones like dipping, coating, etc. Many

TABLE I
Characteristics of NR and XSBR Latex

Centrifuged NR latex	
Supplied by	Gaico Rubbers Ltd., Kuravilangadu, Kottayam, Kerala, India
DRC (%)	60
TSC (%)	61.25
Alkalinity (%)	0.75
VFA number	0.05
Mechanical stability	Good
Manganese content	Traces
Magnesium (ppm)	29
XSBR latex (PLX-802)	
Supplied by	Apar Industries Ltd., Bombay, India
DRC (%)	47
TSC (%)	50.66
Styrene content (%)	52
PH value	8.60
Brookfield viscosity (CPS)	59.40
Mechanical stability	Good

NR, natural rubber; DRC, dry rubber content; TSC, total solid content; VFA, volatile fatty acid; XSBR, carboxylated styrene butadiene rubber.

TABLE II
Mixing Ratio of NR and XSBR latex

	N_{100}	N_{70}	N_{50}	N_{30}	N_0
60% Centrifuged NR latex	100	70	50	30	–
47% XSBR latex	–	30	50	70	100
10% KOH solution	1	1	1	1	1

researchers have studied the latex particles with surface functional groups, with a broad spectrum of applications, such as their use as high-tech and biomedical materials.[20] There are reported works on the properties of carboxylated copolymer latices that are widely used for the production of paper coatings, textile coatings, and adhesives.[21]

The purpose of the present study is to investigate the flow properties of NR and XSBR latex, with special reference to the effect of blend ratio, shear rate, and temperature. The viscosity measurements were theoretically predicted, using Haschin upper and lower bound, Heitmiller, and Sood–Mashelker model.

EXPERIMENTAL

Materials used

Centrifuged NR latex with 60% dry rubber content (DRC) was supplied by Gaico Rubbers, Kuravilangadu, Kerala, India. The XSBR latex was supplied by Apar Industries, Bombay, India. The basic characteristics of two latices are given in Table I.

Blend preparation

The blending of two latices was carried out, using mechanical stirrer of 1 L capacity at 2000 rpm with sigma type rotor. The two latices were mixed together in accordance with the DRC and stirred for half an hour at ambient temperature. It was then kept for 1 h to ensure homogenization and also for the sedimentation of impurities. It is used for the rheological measurements. All the samples were kept for 1 h uniformly before rheological measurements. The blends were designated by N_{70}, N_{50}, and N_{30}, respectively, where N denotes NR and the subscript denotes the weight percent of NR. Details of blending are shown in Table II.

Rheological measurements

The rheological measurements of latex blends were done, by using a Haake Rotovisco RV-12 Serial-type rotational viscometer with M-500 measuring head and NV sensor system (double gap sensor). The measurements were done at different shear rates and at temperatures 30, 40, and 50°C.

RESULTS AND DISCUSSION

Influence of shear rate

Blend rheology of polymer latices depend on many variables, such as blend composition and viscosity of homopolymers. Shear rate is one of the parameters for the understanding of flow behavior of latices. The effect of shear rate on the viscosity of NR and XSBR latices and their blends are shown in Figure 1. The individual components and their blends show a decrease in viscosity with increase in shear rate. But for most of the systems the viscosity levels off at a particular shear rate and further increase in shear rate does not affect the flow, indicating Newtonian behavior. The rubber particles are dispersed in the aqueous medium of latex. The rubber particles often form network structure, which is schematically represented in Figure 2. As the shear rate is increased, the particles in the network are destroyed and as a result the viscosity decreases.[14,22] Infact at high shear rates the rupturing of all networks takes place inside the system. NR and 70/30 NR/XSBR blends show a slight increase in viscosity at high shear rate. This may be due to the flocculation of latex particles by destabilization of the colloid. It is important to mention that at rest, the colloid particles come together to form network structures. Due to the presence of more solid content (Table I), NR shows the highest viscosity than XSBR and blends. Due to the breaking down of total entanglements within the system, NR shows a sharp decrease in the viscosity up to a shear rate of 173.1 s^{-1}. From Figure 1, it is clear that there is a large difference between the viscosity of N_{100} and N_0 at lower and higher shear rates. This is because of the difference in total solid content of the two latices. The viscosity of 70/30, 50/50, and 30/70
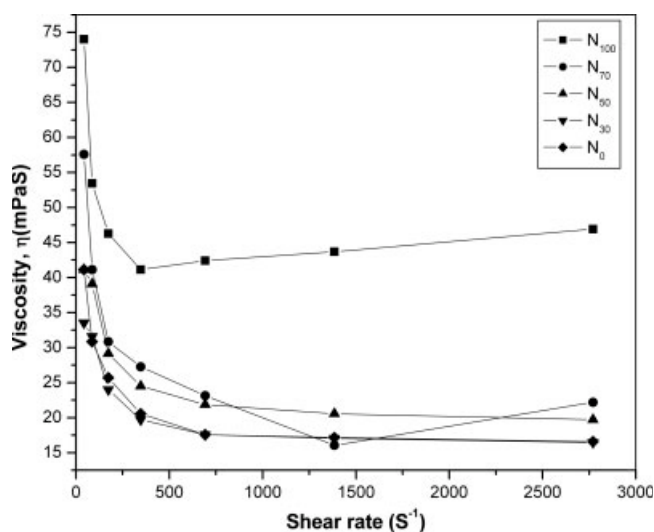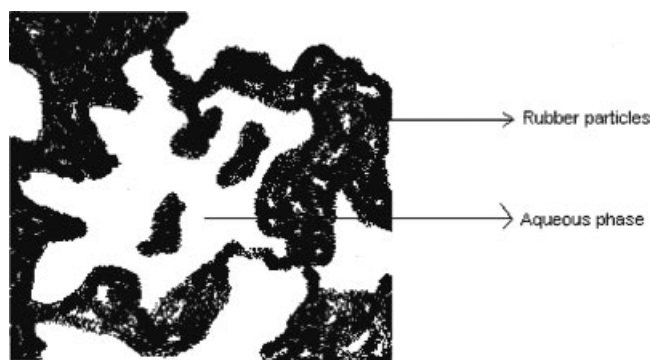

Figure 2 Schematic representation of network formation in rubber particles.

NR/XSBR blends are close to N_0. Individual latices and their blends exhibit pseudoplastic behavior at low shear rates, while at higher shear rates all the systems show Newtonian nature. The viscosities of the polymeric materials are also influenced by the particle size. As compared with XSBR (around 3 μm) the particle size of NR is high (around 5 μm), therefore the interphase area will be small. As a consequence the stabilizers have concentrated more on the aqueous phase than the surface of rubber and form a network. The rubber particles get occluded in this network structure leading to agglomeration of rubber particles and an increase in viscosity. In a latex system, associated particles shows higher viscosity than unassociated particles.

Influence of temperature

Viscosity is highly sensitive to temperature. Most materials show decrease in viscosity as temperature increases. This has an important implication for polymer processing. Temperature has a strong effect on the viscosity of the dispersions.[22–24] In all cases, the viscosity of individual latices and their blends decrease with increase in temperature. Figure 3 gives the effect of temperature on the latices and their blends at low shear rate. NR shows sharp decrease in viscosity with increase in temperature. As the temperature increases the interaction between the particles decreases, resulting in the rupture of links formed between the particles. Hence the viscosity of the latices and their blends decreases with increase in temperature. In N_{50} and N_{30} blends the effect of temperature is negligible. We can understand that the rheology of N_{50} and N_{30} blend are almost independent of temperature. Generally, latices have two types of flow units, one is the Newtonian flow unit (water) and the other is the non- Newtonian flow unit (rubber). Maron and Fok[25] studied the flow behavior of butadiene–styrene latex between 20 and 50°C. They found that the decrease of the viscosity

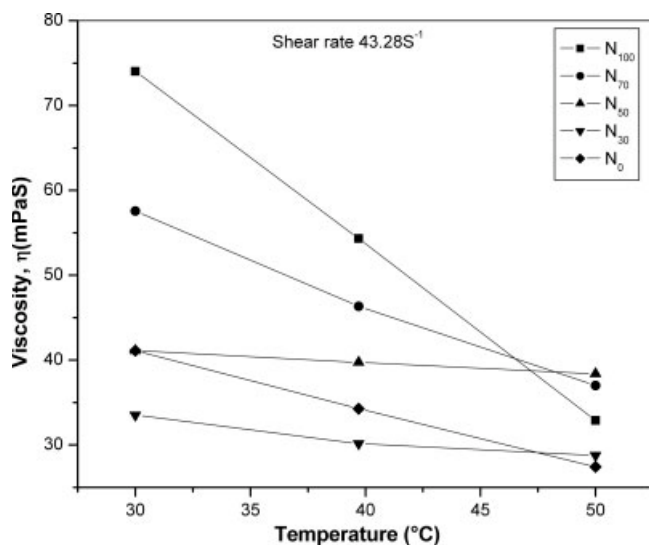**Figure 1** Effect of shear rate on the viscosity of NR, XSBR, and NR/XSBR blends at 30°C.

Figure 3 Temperature effect on the viscosity of NR, XSBR, and NR/XSBR blends.

of the latex with temperature results primarily from the decrease of the viscosity of the water medium. The activation energy needed for the flow differs only slightly from that of pure water.[26] In the case of synthetic latex the deviation from Newtonian behavior has been found to be essentially independent of temperature and to be a function of concentration and shear stress.

The temperature effect can be more understood from the activation energy values of these blends given in Table III. The temperature sensitivity of a material is obtained from the activation energy values. The activation energy is determined using Arrehenius equation,

$$\eta = \eta_0 e^{-E_a/RT} \tag{1}$$

Activation energy is obtained from the slope of the plot η versus $1/T$. Among blends N_{70} shows higher activation energy, indicating the high temperature sensitivity of the material. This is because of the higher viscosity of the NR phase (continuous phase). Therefore it needs more energy for the breaking of all networks within the system.

Influence of blend ratio

In the case of blends, depending on the viscosity of two phases and the weight percentage, one of the phases is dispersed in the other. If the dispersed particles are attracted to each other, they tend to flocculate. Review of literature[27] reveals that in NR and SBR latex blends micro flocculation occurs. Figure 4 shows the effect of blend ratio on the viscosity of latex blends at a shear rate of 43.28 s^{-1}. The breakage of networks starts at lower shear rate. Therefore, lower shear rate of 43.28 s^{-1} is selected for studying the influence of blend ratio. At higher shear rates the viscosity of the latex systems levels off due to the breaking down of all networks. Negative deviation can be observed in NR/XSBR system. The system N_{100} shows maximum viscosity in this system, i.e., 74.03 mPaS while that of XSBR is 41.13 mPaS. The negative deviation of viscosity from the additivity line is due to the lack of interaction between the polar XSBR and nonpolar NR phases. The viscosity of the polymer blends depends on the interfacial adhesion in addition to the characteristics of individual components. On applying shear stress to systems with weak interfaces the interlayer slippage occurs and as a result the viscosity decreases. In the case of immiscible polymer blend system, the continuous phase viscosity determines the flow behavior.

The deviation from the additivity line increases with increase in XSBR content in the system. The observed fall in viscosity on blending is due to the incompatibility and interlayer slippage of the two phases. The incompatibility is due to the polarity difference of the two phases. Due to the lack of interfacial interaction diluents effect will occur upon blending, because of the viscosity difference of two components. The morphology of blends has been examined from scanning electron micrographs of cryogenically fractured surfaces. In the case of heterogeneous blends the properties depend largely on its morphology. In these blends NR is nonpolar and XSBR is polar in nature. These are highly immiscible. Let us now examine the morphology of blends. The morphology development in polymer blends depends

TABLE III
Activation Energy, Pseudoplasticity Index Value, and Zero Shear Viscosity
of NR and XSBR Latices and their Blends

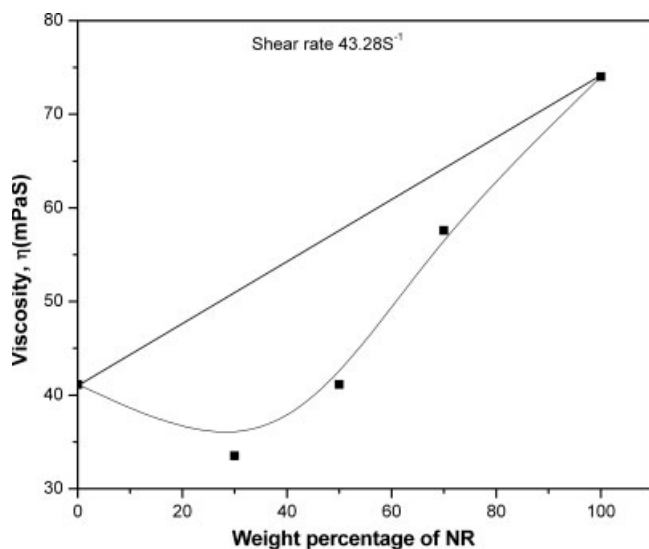| Sample	Activation Energy (kJ/mol) at Shear Rate (43.28 s^{-1})	Pseudoplasticity Index (n)			Zero Shear Viscosity (η_0 at 30°C) (mPa s)
		30°C	40°C	50°C	
N_{100}	6.35	0.91	0.81	0.96	81.10
N_{70}	3.47	0.74	0.78	0.80	64.46
N_{50}	0.54	0.81	0.78	0.74	42.31
N_{30}	0.72	0.82	0.79	0.77	40.56
N_0	3.18	0.78	0.78	0.81	48.73

Figure 4 Variation of viscosity with blend ratio of NR/XSBR at 30°C.

on the blend ratio, viscosity of the components, the interfacial tension between the two phases, and the shear applied during processing. In polymer blends the morphology development takes place as a consequence of both droplet break-up and coalescence. According to Taylor[28,29] the two opposing forces, i.e., the applied shear force that tend to break-up the droplet and the counteracting interfacial forces that results in coalescence will affect the drop dimension and domain stability. One can also explain this based on the capillary number,

$$Ca = \frac{\dot{\gamma}\eta_m D}{2\Gamma} \tag{2}$$

where D is the diameter of the droplet, γ is the shear rate, η_m is the matrix viscosity, and Γ is the interfacial tension. Ca is a dimensionless number, which indicate the formation of droplet size in relation to the applied shearing forces and the counteracting interfacial forces. Figure 5(a) is the scanning electron micrograph of 70/30 NR/XSBR blend. In this figure the dark portion indicates the dispersed XSBR particles in the continuous NR matrix. From Figure 5(b) it is clear that the two phases exhibit cocontinuous morphology at 50/50 blend ratio, i.e., both the phases are continuous. Morphology of 30/70 NR/XSBR blend system is obtained from Figure 5(c), in which NR is dispersed in the continuous XSBR phase in accordance with blend ratio. The size of the domains present in N_{70} and N_{30} polymer blends are given in Table IV. It depends on the viscosity of the continuous phase. If the matrix is highly viscous the coalescence of dispersed particles decreases and *vice versa*. The size of dispersed particles in N_{70} and N_{30} blends are 1.7 and 2.4 μm, respectively. This is in accord-

ance with the capillary number as given in Eq. (2). According to the capillary number as the matrix viscosity increases the domain size decreases. It is found that the domain size of 30/70 (c) blend are higher than 70/30 (a), it can be explained in terms of the lower viscosity of the continuous XSBR phase.
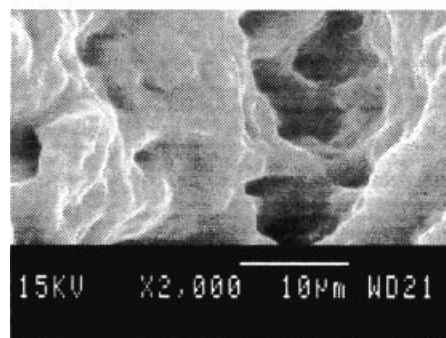
Utracki and Sammut[30] studied the variation of viscosity in polymer blends. According to them,

$$\ln \eta_{i\text{app(blend)}} = \sum W_i \ln (\eta_{\text{app}})_i \tag{3}$$
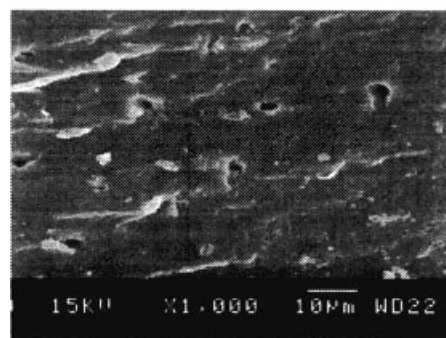
where W_i is the weight fraction of the ith component of the blend and η_i is its viscosity. An immiscible blend can exhibit three types of behavior: positive
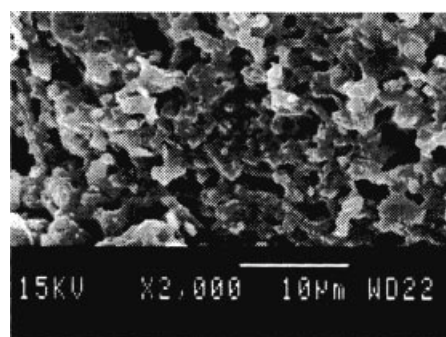
Figure 5 Scanning electron micrographs of (a) N_{70}, (b) N_{50}, and (c) N_{30}.

TABLE IV
Domain Diameter of Dispersed Phases in N_{70} and N_{30} Blends

Sample	$\overline{D}_n$ (μm)	$\overline{D}_w$ (μm)
N_{70}	1.7	2.0
N_{30}	2.4	2.6

deviation, as in a homogeneous blend, in which there is a large interaction between the phases; negative deviation, when the interaction is small; and a positive–negative deviation, when there is a concentration-dependent change of structure. In two-phase polymer blends, the viscosity depends on the characteristics of the components and as well as on the interfacial thickness and interfacial adhesion. In the present blend system, the negative deviation is due to the lack of interaction between the two components owing to its difference in polarity. Under these circumstances, the viscosity of the continuous phase determines the viscosity of blend system. Thomas and coworkers[31,32] reported the rheological behavior of several immiscible blend system.

Master curve

Shear rate–shear stress master curves are useful in the prediction of flow behavior of the polymeric materials at a particular shear rate.[33] There are many reports in the literature[34] on the viscosity shear–stress master curves over a range of temperatures. Viscosity shear–stress master curves will give an idea about the viscosity of polymers at a given temperature.[35–37] To construct a master curve it is necessary to pick one temperature, suppose T_1, as the reference temperature. The shift factor is a function of shear rate and designated as $f\dot{\gamma}$. All other experimental curves, each at a particular temperature are then shifted horizontally, that is, along the shear rate axis until it overlaps the curve with the reference temperature, T_1.

The master curve was plotted for N_{70} blend, using shift factor at 30, 40, and 50°C. For plotting this, the flow curve at 50°C was taken as the reference. The horizontal shift $f\gamma$ of the curves at 30 and 40°C of N_{70} were determined. Figure 6 is the flow curve of N_{70} blend at different temperatures. Using these shift factors the master curve of N_{70} blend was drawn (Fig. 7). It is very interesting to note that all the curves coalescence into a single master curve. The shear thinning behavior of these blends with temperature and shear rate can be obtained from the master curve. Master curve is an important tool for predicting the flow behavior of polymeric solutions at a particular temperature and shear rate.
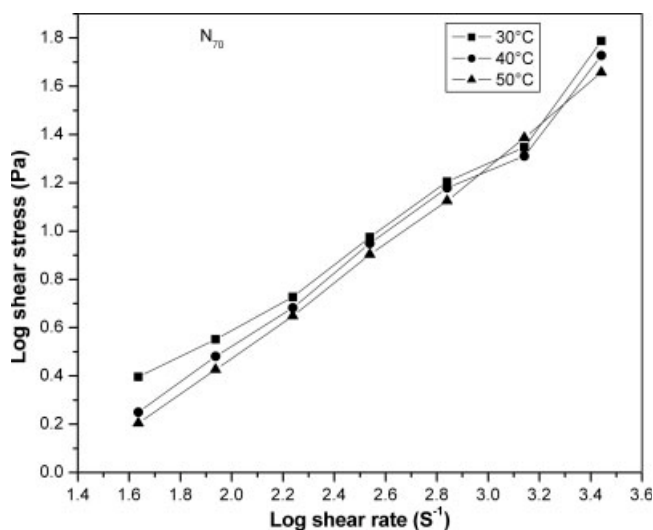

Figure 6 Shear rate–shear stress curve of N_{70} blend at different temperatures.

Pseudoplasticity, activation energy, and zero shear viscosity

The flow properties of latices were analyzed by the Power law:

$$\tau = k\,\dot{\gamma}^n \qquad (4)$$

where, ι is the shear stress (Pa)

  k is the viscosity index
  $\dot{\gamma}$ is the shear rate (s^{-1})
  n is the pseudoplasticity index

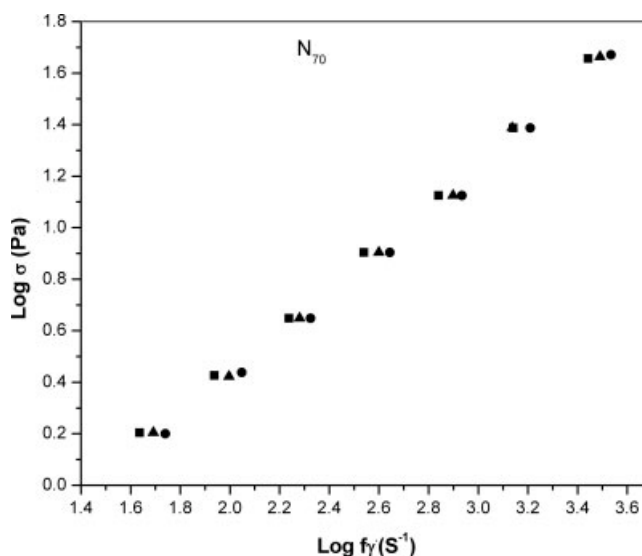Power law is widely used as a model for non-Newtonian fluids.[14,15,24] It holds for many polymer

Figure 7 Master curve of N_{70} blend.

solutions and can be described by Newtonian, shear-thinning, and shear-thickening behavior in terms of the power factor n. The pseudoplasticity index values, i.e., n, can be calculated from the power law equation by plotting log ι against log $\dot{\gamma}$.

The results obtained for various blends at different temperatures are given in Table III. It is clear from the values that XSBR is more pseudoplastic than NR. The pseudoplasticity values of blend systems are intermediate between the values of individual components. The n values obtained from power law can be used to explain the extent of non-Newtonian behavior of NR, XSBR and their blends.

The activation energy for the blends decreases with increase in XSBR content up to N_{50}. Except N_{70}, the activation energy of all the blends is less than that of its components. Table III shows the zero shear viscosity (η_0) of NR, XSBR, and their blends. NR shows the highest zero shear viscosity due to the high total solid content in the system. The η_0 decreases with increase in XSBR content till N_{50}. This can be explained in terms of the less miscibility of the two systems and also because of the less total solid content of the other component, XSBR latex.

Theoretical comparison of viscosity

The experimental viscosities of NR/XSBR blends are compared with several models of viscosity of polymer blends. The following models have been used to describe the viscosity of binary blend,

a. Haschin[38] model.
   Upper bound:

$$\eta*_{\text{blend}} = \eta_2 + \frac{\phi_1}{\frac{1}{(\eta_1-\eta_2)} + \frac{\phi_2}{2\eta_2}} \tag{5}$$

   Lower bound:

$$\eta*_{\text{blend}} = \eta_1 + \frac{\phi_2}{\frac{1}{(\eta_2-\eta_1)} + \frac{\phi_1}{2\eta_1}} \tag{6}$$

where, η_{blend} is the viscosity of the blend
η_1 and η_2 are the viscosities of Components 1 and 2, and
ϕ_1 and ϕ_2 are the volume fractions of Components 1 and 2, respectively.
b. Heitmiller model,[39]

$$\frac{1}{\eta_{\text{blend}}} = \frac{w}{\eta_1} + (1-w)\eta_2 \tag{7}$$

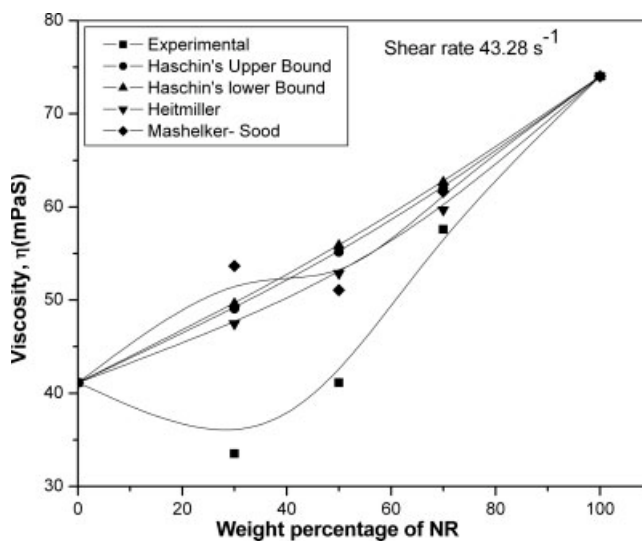where, w is the weight fraction of Component 1.

Figure 8 Comparison of theoretical and experimental viscosities of NR/XSBR latex blends.

c. Sood–Mashelker model[40]

$$\ln \eta_{\text{mix}} = \frac{\phi_1(\alpha - 1 - \gamma\phi_2)\ln \eta_1 + \alpha\phi_2(\alpha - 1 + \gamma\phi_1)\ln \eta_2}{\phi_1(\alpha - 1 - \gamma\phi_2) + \alpha\phi_2(\alpha - 1 - \gamma\phi_1)} \tag{8}$$

The α and γ in the eq. (7) are calculated using the following equations:

$$\gamma = \frac{\beta}{f_1} \tag{9}$$

$$\alpha = \frac{f_2}{f_1} \tag{10}$$

where f_1 and f_2 are free volume fraction of Components 1 and 2

$$f = f_g + \alpha_f(T - T_g) \tag{11}$$

where, $f_g = 0.025$

$$\alpha_f = \frac{B}{2.303C_1C_2} \tag{12}$$

where, $B = 0.9 \pm 0.3 \approx 1$, $C_1 = 17.44$ K, and $C_2 = 51.6$ K

$$\gamma = \frac{\beta}{f_1} \tag{13}$$

where, β is the interaction parameter.
   The value of γ in Sood–Mashelkar model varied to get the best-fit value with the experimental values. The value of γ used for the calculation of blend

viscosity is −0.8 and the interaction parameter, β corresponds to this γ value is −0.005176.

Comparison of theoretically predicted and experimentally determined viscosities of NR/XSBR system at a particular shear rate (43.28 s^{-1}) are given in Figure 8. There is no remarkable difference between the Haschin's lower and upper bound model. All the theoretical models are below the additivity line, indicating the heterogeneous nature of the blend system. For 70/30 NR/XSBR blend system, the Heitmiller model agrees more with the experimental results. They derived the blend viscosity equation by considering the morphology as concentric layers of one fluid in another fluid. In this blend system, XSBR is dispersed in continuous NR phase.

CONCLUSIONS

The flow behavior of NR, XSBR, and their blends at varying shear rate, temperature, and blend ratio were studied. The NR/XSBR system showed shear-thinning behavior. For all systems the viscosity decreased with increase in shear rate, resulted from the breaking down of networks within the system. At higher shear rate flow resistance decreased and the viscosity levels off. It means that the system was Newtonian at higher shear rate. A negative deviation of viscosity from the additivity line is obtained. For blends such a deviation of the viscosity of NR/XSBR system was an evident support to the lack of interaction between the polar XSBR and the nonpolar NR. Due to the weak interfaces, inter layer slippage occurred in the system with shear and consequently the viscosity decreases. One obvious reason for the negative deviation was that the continuous phase viscosity was known to dominate the viscosity of an immiscible system at low disperse phase volume fraction. As the temperature increases, the activation energy for the breaking of total entanglements within the system decreases, along with the viscosity. The temperature sensitivity of the blends was analyzed from the activation energy values. The 50/50 NR/XSBR blend was found to be more sensitive to temperature than other blends and individual components. The relevance of master curves in rheological properties of NR/XSBR blends were explained by plotting curves of N_{70} and N_{30} blends. The viscosity of the system at any particular temperature and shear rate can be obtained from these master curves. The degree of shear-thinning of the blends and its individual components were determined by power law. The zero shear viscosity of blends and individual latices was analyzed. The experimental results were theoretically correlated, using Haschin's upper and lower bound model, Heitmiller and Sood–Mashelkar models. Among these Heitmiller model fits well with the experimental values for 70/30 NR/XSBR blend.

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