Properties of Styrene Butadiene Rubber/Recycled Acrylonitrile-Butadiene Rubber (SBR/NBRr) Blends: Effect of the Addition of *trans*-Polyoctylene Rubber

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ABSTRACT: This study investigated polymer blends based on different ratios of styrene butadiene rubber (SBR) and recycled acrylonitrile butadiene rubber (NBRr) with and without *trans*-polyoctylene rubber (TOR) as a compatibilizer. Five compositions of SBR/NBRr/TOR were prepared; 95/5/5, 85/15/5, 75/25/5, 65/35/5, and 50/50/5. Rheological characteristics, tensile and physical properties, and the morphological behavior of compatibilized and uncompatibilized SBR/NBRr blends were determined. The rheological characteristics indicated that compatibilized SBR/NBRr blends had a shorter cure time (t_{90}) than uncompatibilized SBR/NBRr blends. The compatibilized SBR/NBRr blends showed lower minimum torque (M_L) when compared with as uncompatibilized SBR/NBRr blends, indicating that compatibilization improved the processability of the blends. However, the maximum torque (M_H) of compatibilized SBR/NBRr blends was higher than that of uncompatibilized SBR/NBRr blends. The incorporation of TOR improved the tensile strength, tensile modulus (M100), hardness, and crosslinked density of compatibilized SBR/NBRr blends. However, the elongations at break (E_b) and resilience of compatibilized SBR/NBRr blends were lower as compared to uncompatibilized SBR/NBRr blends. Scanning electron microscopy of the tensile fractured surfaces demonstrated that the addition of TOR in SBR/NBRr blends improved the adhesion between NBRr and the SBR matrix, thus improving the compatibility of SBR/NBRr blends. © 2012 Wiley Periodicals, Inc. J Appl Polym Sci 000: 000–000, 2012

Key words: SBR/NBRr blends; *trans*-polyoctylene; rheological characteristics; mechanical and physical properties; morphology

INTRODUCTION

Generally, compatibilization can lead to a finer phase structure and enhanced interfacial adhesion.[1] The interfacial adhesion can be improved by introducing a third component into an immiscible binary system that will either interact chemically with both phases or will have a specific interaction with one phase and a physical interaction with the other.[2] The compatibilizer may form an interface between the immiscible blend components so that imposed stresses can be transferred between the phases via the covalent bonds along the copolymer backbone. However, it is important to understand the blend morphology because it has a strong effect on the properties of polymer blends.[3] Addition of a suitable compatibilizer has been shown to result in the development of a finer scale distribution of the dispersed phase in the matrix as well as an enhancement of the physico-mechanical properties of the nitrile rubber blends.[4]

Trans-polyoctylene rubber (TOR) has been introduced as a compatibilizer for incompatible rubber blends containing polar rubbers, such as acrylonitrile butadiene-rubber (NBR) and nonpolar rubber styrene butadiene rubber (SBR), and as a processing aid for extremely stiff rubber compounds such as highly filled rubber compounds for skim-coating of steel wires for tires.[5] TOR is a low-molecular-weight polymer made from cyclo-octene by methathesis polymerization. This high-performance polymer presents a dual character during processing: it functions as a plasticizer, and after vulcanization it behaves as a rubber and has been used as a compatibilizer for incompatible blends. This polymer can provide good processability in the temperature range of rubber processing (100–150°C) as well as good collapse resistance below its melting temperature of 54°C because of recrystallization.[6] TOR also has linear and cyclic macromolecules which are unbranched and contain one double bond for every eight carbon atom with prevalently *trans*-isometric double bonds.[7] Blending of SBR/NBR blends lead to an incompatible blend.[8] It is well known that the physico-mechanical properties of the pure components SBR and NBR are very weak in the absence of

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TABLE I
Characteristics of Materials

Materials	Description	Source
Styrene butadiene rubber (SBR)	1502	Bayer (M) Ltd
Recycled acrylonitrile-butadiene rubber (NBRr)	Size: 117–334 μm	Juara One Resources Sdn Bhd, Bukit Mertajam, Penang, Malaysia.
trans-polyoctylene rubber (TOR)	Vestenamer 8012 ML_{1+4} at 100°C < 10 MW = 100,000 T_m = 51°C, T_g = −65°C Crystallinity at 23°C = 27%	Huls, Germany
Carbon black	N330	Malayan Carbon (M) Ltd.
N-cyclohexyl-2-benzothiazyl sulfenamide (CBS), zinc oxide, stearic acid, sulphur, and processing oil		Anchor Chemical (M) Ltd.

reinforcing filler, and consequently, the physico-mechanical properties of their blends are also inferior. This originates principally from the incompatibility of both components with each other in view of the fact that NBR has a greater polarity than SBR.[9]

Recycling of waste rubber has become an important global issue that can address three major problems; wasting of valuable rubber, health and environmental pollution.[10] Recycled rubber waste does not decompose easily due to its crosslinked structure and the presence of stabilizers and other additives. Compared with reclaimed rubber, recycled NBR rejects are lightly crosslinked and are made up of a high-quality rubber hydrocarbon. Large quantities of recycled rubber rejects such as recycled glove are generated worldwide daily because of extensive use of these materials; rejects include product that does not meet processing and product specifications, leftover rubber from manufacturing activities, and also old and defective rubber products such as gloves, catheters, tubes, and old tyres. Malaysia produces an abundant output of nitrile rubber gloves (NBR). Most of this material originates from medical, industrial and research activities, and after a certain period of time these polymeric materials are no longer serviceable and are mostly discarded.

To solve this environmental issue, this study uses recycled NBR glove waste obtained from an industrial floor in an effort to create value added instead of scrapping the material. Recently, we reported[11,12] a comparison between the properties of virgin and recycled NBR (NBRr) in SBR/NBRv and SBR/NBRr blends. The results indicated that the tensile strength of SBR/NBRr blends was higher than that of SBR/NBRv blends up to 15 phr of NBRr content. Also, the incorporation of ENR-50 improved the tensile strength, tensile modulus (M100, stress at 100% elongation), hardness and crosslinked density of SBR/

NBRr blends when compared to controls without ENR-50 at all blend ratios.[13] However, less attention has been given to a detailed study of the compatibilization of TOR in polar-non-polar rubber mixtures such as SBR/NBR blends, particularly for blends involving waste rubber. To the best of our knowledge, no attempts have been made to study the effect of TOR as a compatibilizer in SBR/NBRr blends. This work studies the rheological characteristics, tensile and physical properties, and also morphological behavior of SBR/NBRr/TOR.

EXPERIMENTAL

Materials and preparation of blends

The materials used in this study were SBR, recycled NBRr—a recycled product from waste gloves with a size range of 117–334 μm, TOR, carbon black N330, *N*-cyclohexyl-2-benzothiazyl sulphenamide (CBS), zinc oxide, stearic acid, sulfur and processing oil. The characteristics of these materials are shown in Table I. The SBR/NBRr blends with and without TOR (5 phr) were formulated as shown in Table II. The rubber was preblended and the mixing procedure was carried using a two-roll mill at room temperature. Cure characteristics were studied using a Monsanto moving die rheometer (MDR 2000). Roughly 4 g samples of each compound were used for testing at the vulcanization temperature (160°C). The rubber compounds were then compression molded at 160°C using a hot press for various cure times, t_{90}.

Measurement of mechanical properties

Dumb-bell shaped samples were cut from the molded sheets and the tensile testing was carried

Ingredients (phr)	Blend				
	R05	R15	R25	R35	R50
SBR	95	85	75	65	50
NBRr	5	15	25	35	50
trans-polyoctylene rubber (TOR)	0&5	0&5	0&5	0&5	0&5
Zinc oxide	5	5	5	5	5
Stearic acid	2	2	2	2	2
Sulfur	2	2	2	2	2
N-cyclohexyl-2-benzothiazole sulfenamide (CBS)	1	1	1	1	1
Processing oil	5	5	5	5	5
Carbon black (N330)	50	50	50	50	50

out according to ISO 37. Tensile testing was performed at a crosshead speed of 500 mm/min using an Instron 3366. The hardness of the sample was measured according to ISO 48 using a Wallace dead load, with the hardness ranging from 30 to 85 IRHD (International Rubber Hardness Degree). Resilience was studied using a Wallace Dunlop Tripsometer according to BS 903 Part A8. Rebound resilience was calculated according to the following equation:

Percentage resilience
$$= [(1 - \cos \theta_2)/(1 - \cos \theta_1)] \times 100\%,$$

where θ_1 is the initial angle (45°) and θ_2 is the maximum rebound angle.

Crosslink density measurements

Cured test pieces of dimension $30 \times 5 \times 2$ mm^3 were weighed using an electrical balance and each test piece was immersed in a glass vessel containing 30 mL toluene at 25°C. The vessel was kept in the dark to prevent oxidation. The samples and excess toluene were removed from the glass vial by lens blotting paper. The samples were then kept in a closed vessel to prevent toluene evaporation and the weights of the swollen samples were determined. The sample was then reimmersed in toluene and the process was repeated until a constant swollen weight was obtained. The sample was deswollen under vacuum at room temperature to a constant weight in order to find the volume fraction of toluene absorbed in the rubber. The swelling data were used to calculate the molecular weight between two crosslinks (Mc) by applying the Flory–Rehner equation[14]:

$$M_c = \frac{-\rho_p V_s V_r^{1/3}}{\ln (1 - V_r) + V_r + \chi V_r^2} \quad (1)$$

$$V_r = 1/(1 + Q_m) \quad (2)$$

where ρ is the rubber density, V_s is the molar volume of the solvent (toluene), V_r is the volume fraction of the swollen rubber, χ is the interaction parameter of the rubber, and Q_m is the weight swell of the SBR/NBR blends in toluene. The degree of crosslinking density (υ) is given by:

$$\upsilon = 1/(2 \; M_c) \quad (3)$$

The following constant values were used to determine the degree of crosslinking density of SBR/NBR.

$$\rho \; (\text{SBR}) : 0.933 \; \text{g/cm}^3; \rho \; (\text{NBR}) : \; 1.17 \; \text{g/cm}^3;$$

$$\chi \; (\text{SBR}) : \; 0.446; \chi (\text{NBR}) : 0.390;$$

$$V_s(\text{toluene}) : \; 106.35 \; \text{cm}^3/\text{mol}.$$

Fourier transform infrared spectroscopy

Fourier transform infrared spectroscopy (FTIR) spectra were obtained using Perkin-Elmer Spectrum One Series equipment and the attenuated total reflection technique. The selected resolution and scanning range were 4 cm^{-1} and 600–4000 cm^{-1}, respectively.

Scanning electron microscopy (SEM)

Scanning Electron Microscopy (VPFESEM) was conducted using model Zeiss SUPRA 35VP to analyze the surface aspects of the material, including the quality of bonding and the presence of microdefects. The sample surfaces were mounted on aluminium stubs and sputter coated with a thin layer of gold of about 2 nm thickness prior to analysis to avoid electrostatic charging and poor resolution.

RESULTS AND DISCUSSION

Rheological characteristics

The rheological characteristics of SBR/NBRr blends with and without TOR are summarized in Table III. These results show that scorch time t_2 and cure time t_{90} of uncompatibilized and compatibilized SBR/NBRr blends decrease as NBRr content increases. In our previous works,[11–13] we have reported that increasing the NBRr content in SBR/NBRr blends reduces the t_2 and t_{90} because of the existence of crosslinked precursors and unreacted curative in the recycled rubber that accelerate the vulcanization process. In SBR/NBRr blends with incorporation of TOR, the t_{90} and t_2 seems to follow the previous trends as NBRr content increased. This is likely due to the characteristic nature of TOR, including its plasticizer effect and its ability to act as an

Blend (phr/phr)	Cure time, t_{90} (min)		Scorch time, t_2 (min)		Minimum torque, M_L (dNm)		Maximum torque, M_H (dNm)	
	Without TOR	With TOR	Without TOR	With TOR	Without TOR	With TOR	Without TOR	With TOR
R05	19.43	11.60	5.98	3.15	2.31	2.21	19.58	20.27
R15	18.00	10.72	5.19	2.84	2.79	2.74	18.50	19.65
R25	16.28	10.40	3.72	2.49	3.15	2.92	17.58	18.57
R35	15.60	10.11	2.67	2.10	3.61	3.05	16.49	17.14
R50	12.28	10.02	1.82	1.55	4.07	3.69	15.60	16.45

unsaturated rubber, particularly when it is added with a polar rubber such as NBR. According to Chang et al.,[15] TOR influences the vulcanization process of the NR/EPDM blend. The variations in curing characteristics imply that TOR, having a lower degree of unsaturation than NR, participates in the vulcanization reaction and becomes a part of the network structure.

The minimum torque (M_L) increased for both uncompatibilized and compatibilized SBR/NBRr blends as NBRr content increased. The increase in the M_L value of SBR/NBRr blends is probably because of the presence of crosslinked NBRr rubber and other additives in SBR/NBRr blends. However, at a similar blend ratio, the compatibilized SBR/NBRr blends exhibit a lower M_L. The addition of TOR in SBR/NBRr blends improves the processability. This could result from a reduction in the viscosity of the SBR/NBRr because of the plasticizer effect of TOR, enhancing the interaction between the blend components.[16] Nah[5] reported that the initial torque and minimum torque of NR/NBR/TOR blends decreased with increasing TOR level because of the crystalline melting of TOR.

The maximum torque, M_H, of SBR/NBRr blends with and without TOR decreased as NBRr content increased. This was due to the poor interaction of SBR/NBRr as shown later in the morphological study. However, SBR/NBRr blends with TOR exhib-ited a higher M_H value than SBR/NBRr blends without TOR. This observation is attributed to the improvement in the crosslink density of SBR/NBRr blends in the presence of TOR as shown later in crosslink density results.

Tensile and physical properties

Tables IV and V present the effect of blend ratio on the tensile and physical properties of SBR/NBRr blends with and without TOR, respectively. As illustrated in Table IV, both SBR/NBRr blends showed a similar trend where the tensile strength decreased as NBRr content increased. These trends were probably because of the incompatibility of the blends.[17,18] The deterioration of tensile strength at higher NBRr content was due to the weak interaction between NBRr and the SBR matrix because of the increased agglomeration of NBRr. However, the tensile strength of SBR/NBRr blends increased with incorporation of TOR. This is probably due to the better dispersion of NBRr in the SBR matrix, which improved the interfacial adhesion in SBR/NBRr blends. TOR acts as a processing aid and as a rubber component, and contributes to the improved distribution of NBRr, minimizing the NBRr coalescence and resulting in a better stress transfer as shown later in the morphological study. In addition, TOR behaves as a compatibilizer via providing specific

TABLE IV
The Effect of Blend Ratio on the Tensile Properties of SBR/NBRr Blends with and Without TOR

Blend (phr/phr)	Tensile strength (MPa)		Elongation at break (%)		Tensile modulus, M100 (MPa)	
	Without TOR	With TOR	Without TOR	With TOR	Without TOR	With TOR
R05	23.47	23.86	520.33	509.00	2.80	2.83
R15	21.00	22.02	502.67	491.00	3.22	3.29
R25	17.53	18.23	452.93	439.80	3.38	3.41
R35	16.36	16.90	414.00	387.33	3.78	3.89
R50	10.55	11.14	356.20	289.00	4.23	4.30

TABLE V
The Effect of Blend Ratio on the Physical Properties of SBR/NBRr Blends with and Without TOR

Blend (phr/phr)	Hardness (Shore A)		Crosslinked density ($\times 10^{-8}$ mol/cm^3)		Resilience (%)	
	Without TOR	With TOR	Without TOR	With TOR	Without TOR	With TOR
R05	70.22	73.56	5.63	5.84	42.13	41.76
R15	71.22	75.11	6.23	6.46	38.58	37.67
R25	72.55	77.00	6.59	7.22	35.64	35.13
R35	74.66	79.22	7.70	8.27	34.45	33.41
R50	75.84	80.11	10.04	10.46	32.95	31.41

interaction between SBR and NBRr. The covulcanization process of the polymer blends in the presence of TOR enhanced the compatibility amongst them.

It can be seen from both tables; results show that both M100 and hardness of the SBR/NBRr blends with and without TOR increased gradually as NBRr content increased. This was due to the incorporation of NBRr in the SBR/NBRr blends, which enhanced the crosslinking density of SBR/NBRr blends. When more NBRr was blended with the SBR matrix, a more rigid SBR/NBRr blend was obtained which led to higher values of M100 and hardness. Nah et al.[19] reported that the increase in hardness with addition of TOR was probably due to the fact that the relative degree of crosslinking was much higher than for the other rubber components. From Table IV, it can be notify that the E_b of both SBR/NBRr blends decreased as NBRr content increased. In our previous work,[12] we reported that the increase in crosslink density contributed to the decrease in E_b. The SBR/NBRr blends with TOR have a lower E_b as compared to SBR/NBRr blends without TOR. This observation is probably because of the presence of various additives, crosslinked precursors, and unreacted curative in the NBRr itself together with TOR, which have hindered the flow and mobility of the SBR/NBRr blends. Carbon black, for example, can restrict the molecular orientation and mobility of polymer blend molecules.[20]

The resilience of SBR/NBRr blends with and without TOR decreased as NBRr content increased as tabulated in Table V. This illustrates that the SBR/NBRr blends became more rigid as more NBRr was added into the SBR matrix. Resilience is the ratio of energy consumed on recovery from deformation to the energy required to produce the deformation.[21] When more NBRr was added into the SBR matrix, the molecular mobility of the rubber chain decreased and the stiffness of the rubber vulcanizates increased.[22] As a result, there is not enough energy to recover the deformation of the rubber vulcanizates. The participation of TOR in the vulcanization process was reported to lead to the variation of the dynamic property where TOR increased the rigidity of the blend vulcanizates.[15]

Fourier transform infrared analysis

The FTIR spectra of SBR/NBRr blends with TOR, i.e., R05/TOR and R50/TOR, show nitrile (C≡N) functional group of NBRr via absorption band at 2326 cm^{-1}, Figure 1. This indicates the nitrile group NBRr in range 2100–2400 cm^{-1} was not involved as binding sites in this blend. The absence of amine groups i.e., NH$_2$ and NH in spectra was used to prove this.[23] Furthermore, the C=C bonds from TOR in the region of 1650 cm^{-1} was also absent. This indicates the diene bond of TOR has been used as a compatibilizer in the sulfur crosslinks between SBR and NBR as shown in Figure 2. The band at
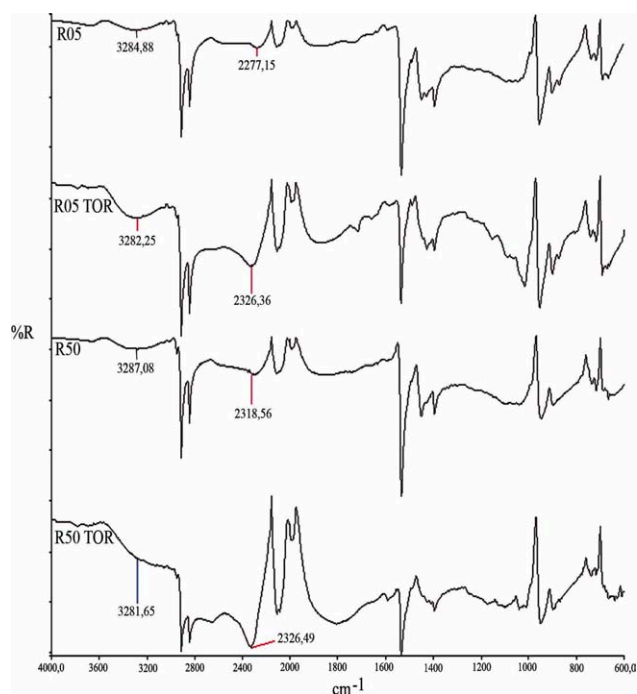

Figure 1 FTIR analysis of SBR/NBRr blend with and without TOR. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.].
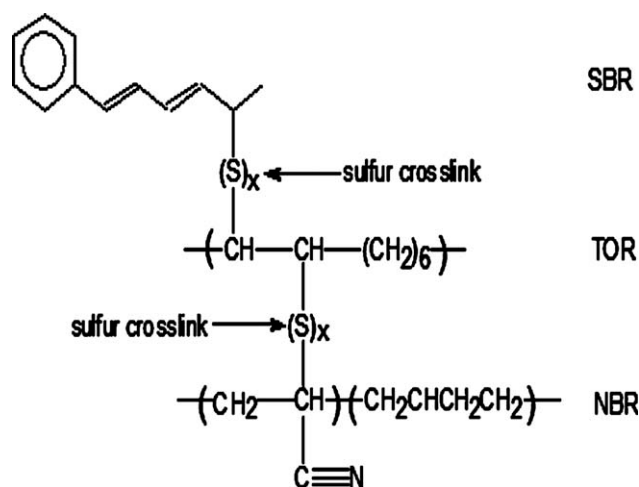
Figure 2 Propose interactions of SBR and NBRr with the presence of TOR.

3282 cm^{-1} was due to trapped moisture from the environment onto carbon black filler. This in agree with trapped CO_2 band in region 1991–2112 cm^{-1}.

Morphological behavior

Figure 3 presents SEM micrographs taken from the tensile fracture surfaces of SBR/NBRr blends with TOR [Fig. 3(a,c)] and SBR/NBRr blends without TOR [Fig. 3(b,d)]. Figure 3(a,c) shows rougher surfaces with many tear lines, meaning that more energy is needed to break the sample when compared to Figure 3(b,d), which displayed a broader tearing line. This could be explained by the good interaction between the NBRr and SBR matrix with the presence of TOR. The uniform dispersion of NBRr in the SBR matrix altered the crack path, which leads to more resistance for crack propagation and thus higher tensile strength. The presence of more tear lines on the fracture surfaces of SBR/NBRr blends with TOR indicates the effect of increased interaction between phases, thus improving the compatibility and enhancing the tensile strength.

Figure 4 displays the tensile fracture surface of SBR/NBRr blends at a 50/50 blend ratio (a) and (c) with TOR; (b) and (d) are without TOR at 200× and 2.00× magnification, respectively. Figure 4(b,d) shows a coarser fractured surfaced because of the poor distribution of NBRr
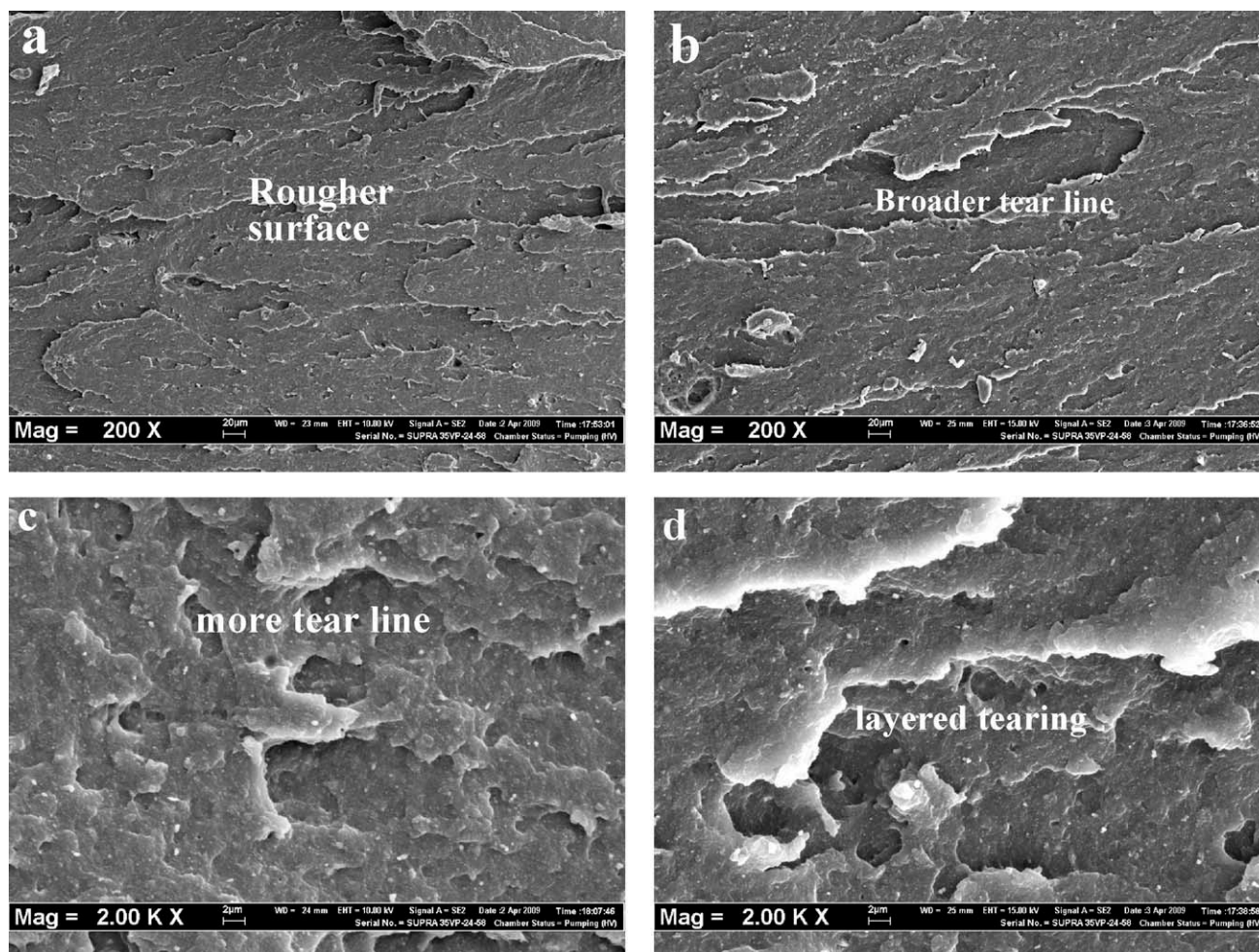

Figure 3 (a) SEM micrographs showing tensile fracture surface of SBR/NBRr blends (a) with TOR and (b) without TOR at 95/5 blend ratio at × 200 magnification; (c) with TOR and (d) without TOR at 95/5 blend ratio at × 2.00 magnification.
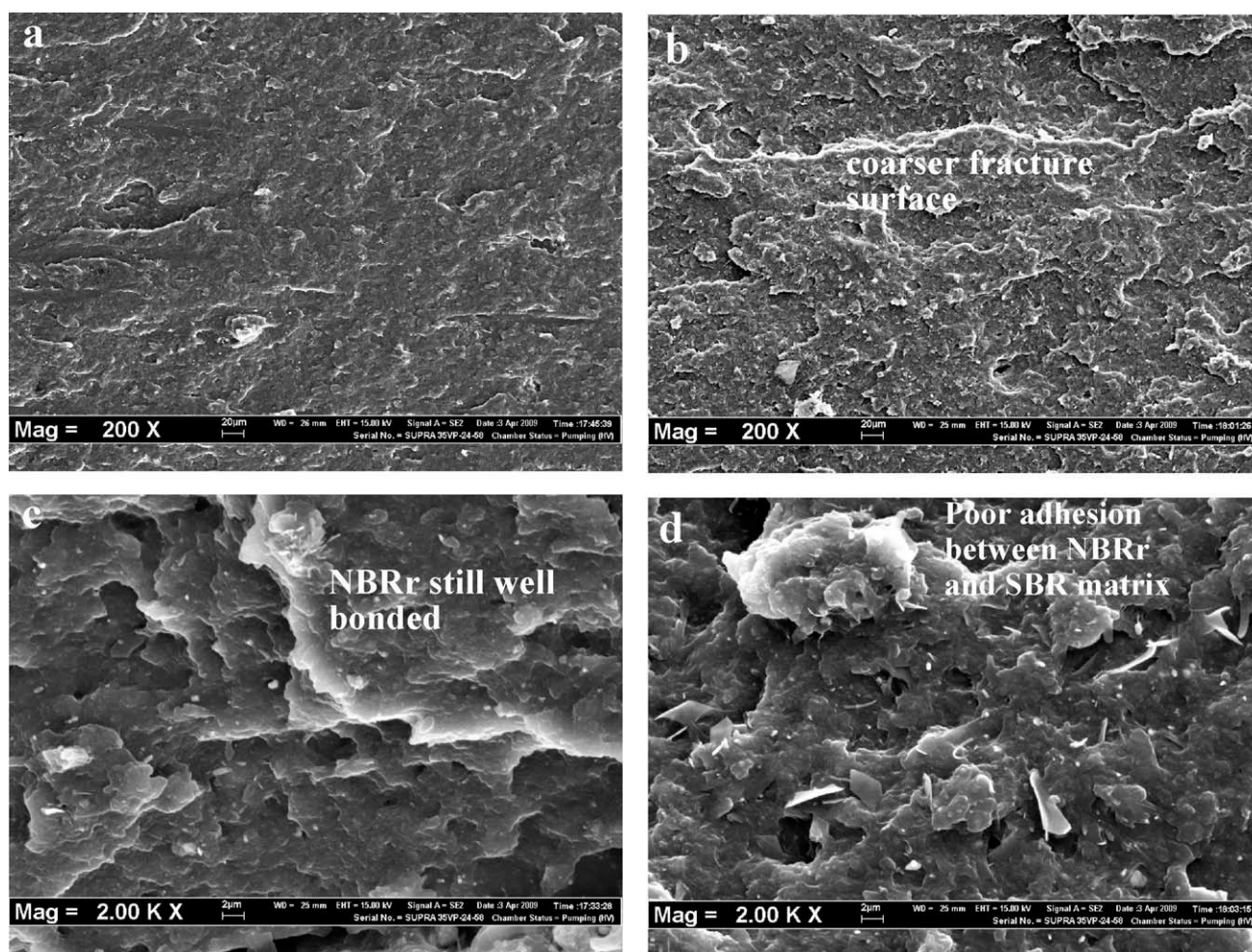
Figure 4 (a) SEM micrographs showing tensile fracture surface of SBR/NBRr blends (a) with TOR and (b) without TOR at 50/50 blend ratio at × 200 magnification; (c) with TOR and (d) without TOR at 50/50 blend ratio at × 2.00 magnification.

in the SBR matrix, particularly when more NBRr content was added to the SBR matrix. This demonstrates low-adhesion between phases, contributing to the poor stress transfer across the interface. However, the surface of SBR/NBRr blends with TOR [Fig. 4(a,c)] illustrates better adhesion between NBRr and the SBR matrix. NBRr is still well bonded with the SBR matrix, which means that stronger interfacial adhesion occurred than in SBR/NBRr blends without TOR. Lohmar[24] reported that the introduction of TOR into various polymer blends reduced the scale of the phase morphology, leading to an improvement in mechanical properties.

CONCLUSION

These results enable us to make the following conclusions:

1. The presence of TOR reduced the optimum cure time (t_{90}) and scorch time (t_2) of SBR/NBRr blends. The minimum torque (M_L) of SBR/NBRr blends with TOR was lower than that of SBR/NBRr blends without TOR, indicating that they could be proc-

essed more easily. The maximum torque (M_H) of SBR/NBRr blends with TOR was higher than that of SBR/NBRr blends without TOR.

2. Incorporation of TOR approximately enhanced tensile strength, tensile modulus, and hardness but reduced the E_b and rebound resilience of SBR/NBRr blends.

3. FTIR spectrum results indicated that the presence of TOR improved the interaction between SBR and NBRr.

4. The tensile fracture surface of SBR/NBRr blends with TOR indicated better dispersion and stronger interfacial adhesion between NBRr and the SBR matrix. In contrast, the SBR/NBRr blends without TOR showed coarser surfaces and lower adhesion between NBRr and the SBR matrix, particularly when more NBRr content was added.

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