

Effect of Fillers on Morphological Properties and Wear Characteristics of Xnbr/NR Blends

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ABSTRACT: The blends of carboxylated acrylonitrile butadiene rubber (XNBR) and natural rubber (NR) were prepared using a blending technique in the presence of different types of carbon black. The effect of filler on morphological and wear characteristics was studied. ISAF N234 carbon black showed a significant effect on curing, mechanical, and thermal studies. The DIN abrader results showed high abrasion resistant properties of 80 wt % NR

and 20 wt % XNBR with ISAF N234. The rubber compound containing 40 wt % of NR and 60 wt % of XNBR with ISAF N234 is found to be the toughest rubber against all types of rock. © 2010 Wiley Periodicals, Inc. *J Appl Polym Sci* 120: 710–718, 2011

Key words: elastomer; composites; morphology; friction; wear; rock

INTRODUCTION

Polymer blends are being used extensively in numerous applications; this statement is also true with rubber blends, especially for the manufacturing of tire. Natural rubber (NR) is known to exhibit various outstanding properties due to its ability to crystallize under stretching; reinforcing fillers are necessarily added into NR in most cases to gain the appropriate properties for specific applications. A wide variety of particulate fillers are used in the rubber industry for various purposes, of which the most important are reinforcement, abrasion resistance, hardness, modulus, reduction in material costs and improvements in processing.^{1–3} The use of carbon black as reinforcing filler is identical with the history of tires. Although it has lost some ground to other reinforcing fillers such as silica, by virtue of its unrivalled performance, it is still the mostly cheap, popular, and widely used reinforcing filler. However, the primary properties of carbon blacks are normally controlled by particle size, surface area, structure, and surface activity and they are in most cases interrelated.⁴ Apart from blends of common rubbers, specialty rubber is also being utilized, depending on service demands and components of the tire.^{5,6} These compounds are capable of forming

a chemical link between these dissimilar rubbers to produce a technologically compatible blend. The blend vulcanizates thus produced exhibit enhanced physical properties by judicious selection of the natural rubber (NR) and carboxylated acrylonitrile butadiene rubber (XNBR) ratio.⁷ The effort toward improving the efficiency of such transportation system is essential for reduce running costs and cutting down the end cost figures by using the XNBR and NR rubber blends.

Tires used in mining vehicles are very costly and need regular maintenance, as it is impossible to accept its replacement expense within a very short term. The rugged working conditions in mining industries reduce the life span of tires on account of cuts, contamination with rock, abrasion, wear, speed fluctuations, etc. The simplest form of wear, which is particularly important in the friction of rigid materials, is abrasive wear and this had also been observed previously by Ratner and Sakhnovoski as the wear governed by the abrasion of the surface layer of materials by the sharp edges of hard projections from the rough surface of the abradant.^{8–11} The wear of rubber is a complex phenomenon and is dependent on a combination of mechanical, mechanochemical, and thermo-chemical processes. Schallamach¹² and later Grosch¹³ reviewed the abrasion of rubber and tire wear in detail. Schallamach¹⁴ observed that during intense abrasion in sliding contact, a high temperature is developed, and consequently the abrasion resistance of the rubber depends, to a large extent, on its resistance to high

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temperature and heat. An increase in the abrasion resistance of rubber products can be achieved by studying the mechanism of wear of rubber under different operating conditions. The mechanism of wear provides a link between the abrasion resistance of rubber and its mechanical properties, which will predict the life of a product in service, and also to develop the method of testing abrasion.

Blending of elastomers has been often used to obtain an optimum number of desirable combinations, physical properties, processability, and cost. In this study, the cure characteristics and the morphological, mechanical, and wear properties of NR and XNBR were investigated. Depending upon the service demands and to withstand with the rugged working condition in the mines NR/XNBR blends have been chosen for this study. Also, this is impossible to sustain with this condition for any single blend alone. XNBR rubber provides extra crosslinking, better tensile properties, higher continues service temperature and chemical resistance than other single rubber. These rubber compounds were examined in a specially fabricated experimental set-up for evaluating their wear resistance properties when abraded against various rock types. The parameters such as influence of composition of rubber vulcanizates on wear characteristics and the mechanism of wear of these compounds against different rocks are reported. A literature search shows a lack of studies about XNBR with NR blends alone. Thus, the work involving the intermixing of NR and XNBR with carbon black except any other filler has been conceptualized for the first time. But the authors have done some study with XNBR-NR blends with N550 type of carbon black in the recent past.¹⁵

EXPERIMENTAL

Materials used in rubber preparation

The carboxylated acrylonitrile butadiene rubber used was Nipol N-34 grade, of Nippon Zeon Co. Ltd. Japan. Its specific gravity is 0.98, Mooney viscosity at 100°C is 45. Bound nitrile content is 27%. Natural rubber (NR - RMA1X) was supplied by the Rubber Board, Kottayam, Kerala. Zinc oxide, stearic acid, *N*-cyclohexyl-2-benzothiazyl sulfonamide (CBS), and antioxidant/antiozonant (IPPD) were supplied by Bayer (India) Ltd. Standard rubber grade process oil (Elasto 710) was purchased from the local market. Carbon black was supplied by Birla Carbon.

Preparation of raw rubber

The compounding formulation for the XNBR and NR blends with its various ingredients were mixed in a two roll mill at a friction ratio of 1 : 2 following

standard mixing sequence. The reinforcing filler (carbon black) was added along with the process oil followed by curatives.

Cure characteristics of rubber compound

The cure characteristics of the rubber compound were studied with the help of a Monsanto Oscillating Disc Rheometer (ODR-100s) at 150°C as per ASTM D-2084-07. From the graphs, the optimum cure time, scorch time, and rate of cure (min^{-1}) could be determined. Also, in rubber manufacture, the time during which a rubber compound can be worked at a given temperature before curing begins, known as scorch time (T_{55} , min), could also be obtained.

Determination of crosslink density

The crosslink density was determined by immersing a small amount (known mass) of sample in 100 mL toluene to attain equilibrium swelling. After this the sample was taken out from toluene, the solvent was blotted from the surface of the sample and weighed immediately. This sample was then dried out at 80°C to constant weight. Then the chemical crosslink density was calculated by the Flory-Rehner equation.¹⁶

Mechanical characterization (tensile and tear)

Vulcanized slabs were prepared by compression molding, and the dumb-bell shaped specimens were punched out from a molded sheet by using ASTM Die C. The tests were done by means of a universal tensile testing machine (Hounsfield H10KS) under ambient condition ($25 \pm 2^\circ\text{C}$), following the ASTM D 412-06 and ASTM D 624-00(2007). The moduli at 100% elongation, tensile strength, tear strength, and elongation at break (%) were measured at room temperature. The initial length of the specimens was 25 mm, and the speed of the jaw separation was 500 mm/min.

Five samples were tested for each set of conditions at the same elongation rate. The values of the tensile strength, modulus at 100% elongation, and elongation at break (%) were averaged. The relative error was below 5%. The hardness was measured by Shore A hardness tester.

Differential scanning calorimetry

Differential scanning calorimetry (DSC) measurements were carried out using a Perkin-Elmer PYRIS Diamond DSC instrument. The samples (≤ 10 mg), sealed under aluminum pans, were scanned in the temperature range of -100 to 100°C . The heating rate was $10^\circ\text{C min}^{-1}$ under the nitrogen atmosphere with a flow rate of 40 mL min^{-1} .

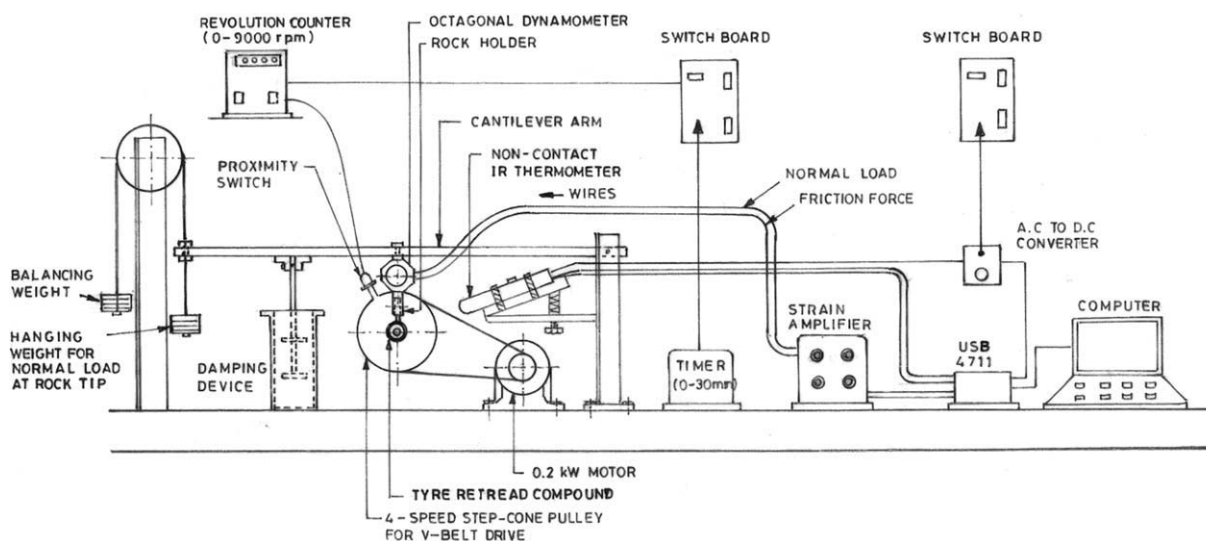


Figure 1 Schematic diagram of experimental set-up for rubber wear measurement.

Thermal characterization (DTA-TGA)

TGA studies were carried out on a Shimadzu-DT-40 instrument in the presence of air at a rate of $10^{\circ}\text{C}/\text{min}$ from 50 to 600°C temperature. Degradation temperature of the composites was calculated by the TGA plot.

Scanning electron microscopy

The tensile fracture surface of the samples were studied in a scanning electron microscope (JSM-5800 of JEOL Co.; Acceleration voltage: 20kV ; type of coating: gold) at 1000 times zooming. Scanning electron microscopy (SEM) has been used to study the morphology and filler dispersion of the abraded surface of the samples prepared.

DIN abrasion test

DIN abrasion test was done by the DIN abrasion tester for determining the abrasion resistance of compounds of vulcanized rubber recommended by the Indian Standards Institution vide IS:3400 (Part 3), 1987.

The experimental procedure for investigation of wear of rubber

In our previous literature, we have already given detailed discussions and pictographs about the preparation of rubber specimen, rock sample, and full arrangement of the experimental setup, during wear testing of the rubber sample.¹⁷ The pictograph of the experimental setup is shown in Figure 1. Total weight of 450, 900, and 1350 g was placed on the hanger of the cantilever beam, so as to produce a normal load of 4.41, 8.82, and 13.23 N at the rock-rubber contact and the test run was conducted for

500 revolutions with sandstone sample placed in the rock holder. The temperature, normal load, and frictional force were recorded on the computer during the wheel rotation. The above procedure was repeated on rubber disks of different XNBR-NR combination with carbon black variation using other types of rocks as abradant. The mass loss of the rubber samples after 500 revolutions (M500) was measured at room temperature, and dynamic coefficient of friction (μ) and abrasion loss (V) were computed.

RESULTS AND DISCUSSIONS

Compound formulation

Compounding formulations based on changing of the carboxylated nitrile rubber (XNBR) and natural rubber (NR) contents are shown in Table I. Also, three types of carbon blacks and one semireinforcing filler are used, such as SAF (N110), ISAF (N231 and N234), and SRF (N774). For vulcanization, the amounts of additives such as sulfur, process oil, and CBS were based on 100 wt % of rubber and the samples have the code name NX-1, NX-2, NX-3, NX-4, NX-5, NX-6, NX-7, NX-8, and NX-9, respectively.

The physical properties of the different types of carbon black used in this study have been already discussed in the previous literature.¹⁷ The main difference between the carbon black was their particle size, tensile strength, and relative road wear abrasion (1.25 for SAF, for 1.15 ISAF and 0.60 for SRF). The reinforcing filler (carbon black) was added along with the process oil followed by curatives.

Cure characteristics of the rubber vulcanizates

The optimum cure time for NX-2, NX-5, and NX-8 rubber sample is higher than other rubber

TABLE I
Compound Formulations

Compounds	Sample codes (wt%)								
	NX-1	NX-2	NX-3	NX-4	NX-5	NX-6	NX-7	NX-8	NX-9
Carboxylated acrylonitrile butadiene rubber (XNBR)	20	20	20	40	40	40	60	60	60
Natural rubber (NR)	80	80	80	60	60	60	40	40	40
Carbon blacks									
SAF ^a (N110)	20	–	–	20	–	–	20	–	–
SRF ^b (N774)	20	–	–	20	–	–	20	–	–
ISAF ^c (N234)	–	40	–	–	40	–	–	40	–
ISAF (N231)	–	–	40	–	–	40	–	–	40
Stearic acid	2	2	2	2	2	2	2	2	2
Antioxidant (IPPD)	1	1	1	1	1	1	1	1	1
Accelerator (CBS)	0.7	0.7	0.7	0.7	0.7	0.7	0.7	0.7	0.7
Zinc oxide	5	5	5	5	5	5	5	5	5
Process oil-elasto 710	2	2	2	2	2	2	2	2	2
Sulfur	2	2	2	2	2	2	2	2	2

^a SAF, super abrasion furnace.

^b SRF, semireinforcing filler.

^c ISAF, intermediate super abrasion furnace.

vulcanizates due to the mixing of ISAF N234 carbon black with XNBR and NR as shown in Table II. The torque difference ($M_H - M_L$), which showed the extent of crosslinking,¹⁸ is found to have lesser variation from one compound to the other. The obtained crosslink density values get corresponded with the variation in torque differences. It is also found that crosslink density increased with increase in torque difference.

Faster cure rate index observed in Table II for the compounds containing ISAF N234 type carbon black may be due to the rise in temperature at the time of mixing, whereas moderate cure rate is found for the compounds that contain ISAF N231 type carbon black. The decrease in cure rate may be due to the greater thermal history formed during mixing, as a result of their higher compound viscosities. The possible formation of a Zn complex in which sulfur and ammonium modifier participate may facilitate for the increase in rate of cure.¹⁹

Mechanical properties of the rubber samples

Tensile strength, modulus, elongation at break, and tear strength for all the compounds are shown in Ta-

ble III. The tensile strength of NX-5 and NX-8 is much higher with the addition of carbon black ISAF N234 in the system. The tear strength for all the samples is moderate, but it varies with varying the matrix ratio. So the system with carbon black ISAF N231 giving better reinforcing effect as well as tear strength may be due to the outstanding reactivity with the rubber matrix. The modulus of all the NR vulcanizates increased with increasing concentration of NR. This was for one or two possible reasons: the restriction of molecular chain mobility and an increase in the crosslink density. Elongations at break values are higher for the blends enriched with NR. The hardness value of the vulcanizates indicates the same trend as the modulus values.

While for other blend types, mainly where NR percentage increased (Sample NX-1, NX-2, NX-4 and NX-6), the tensile and tear strength decreased due to the effect of carbon black which suppresses the XNBR effect in the system and the filler is uniformly dispersed in the natural rubber matrix that can be attributed to the aggregation of XNBR.²⁰ It has also been proved from the SEM studies that the aggregation leads to the formation of weak point in the NR matrix, and accordingly reduces the elastomeric

TABLE II
Cure Characteristics of the Rubber Vulcanizates

Cure characteristics	NX-1	NX-2	NX-3	NX-4	NX-5	NX-6	NX-7	NX-8	NX-9
Minimum torque (dN.m)	13.56	15.07	16.79	17.51	20.59	13.56	16.57	30.13	16.82
Maximum torque (dN.m)	21.47	23.16	28.62	29.57	29.31	21.03	39.17	42.18	34.4
$M_H - M_L$ (dN.m)	7.91	8.09	11.83	12.06	8.72	7.47	22.6	12.05	17.58
Scorch time (min)	10.13	12.57	7.28	7.53	12.13	12.90	6.32	11.35	7.13
Optimum cure time (min)	16.52	31.35	15.12	16.12	23.65	22.5	17.82	27.03	17.85
Cure rate index (min^{-1})	9.08	4.03	9.57	8.96	5.97	9.84	7.47	5.05	7.78
Crosslink density (moles/g) $\times 10^{-5}$	6.36	7.18	11.78	15.31	15.27	9.71	28.17	24.83	36.64

TABLE III
Mechanical Properties

Sample code	Tensile strength (MPa)	Elongation at break (%)	100% Modulus (MPa)	Tear strength (N/mm)	Hardness (Shore A)
NX-1	5.83	475	0.694	25.44	40 ~ 50
NX-2	4.44	213	2.556	27.73	45 ~ 55
NX-3	9.54	398	3.292	35.33	60 ~ 65
NX-4	6.14	230	3.069	25.49	60 ~ 62
NX-5	8.03	229	4.083	48.40	55 ~ 60
NX-6	6.31	300	2.600	25.12	55 ~ 60
NX-7	4.94	166	3.863	16.67	60 ~ 65
NX-8	7.98	216	4.358	10.50	65 ~ 70
NX-9	5.23	166	4.250	13.72	66 ~ 70

strength.^{21,22} Also, it has been proved by the increasing the hardness of the rubber samples. From NX-7 to NX-9 the hardness of the rubber samples has been increased up to 70 Shore A due to mixing of 60 phr of XNBR in the matrix. Generally XNBR has various butadiene monomers (including 1,3-butadiene, 1,2-butadiene) responsible for producing higher hardness.

Differential Scanning Calorimetry study

To study the thermal response of the blends, differential scanning calorimetry (DSC) has been performed. The DSC curves for all rubber vulcanizates are shown in Figure 2. The criterion for compatibility or incompatibility in polymer blends is the presence of a single glass transition temperature T_g for the material, which is intermediate between the T_g s of the pure components and the existence of two T_g s in the DSC thermograms for incompatible polymer blends. All traces show a reasonably sharp T_g transition dependent on composition; however, the graphs for the blend DSC traces are not shown for brevity.

From Figure 2, most of the sample showed the T_g at around -60°C but in case of few samples like NX-1, NX-5, and NX-8 the T_g shifted to the upper

side ($\leq 5^\circ\text{C}$). All the samples have showed only one melting peak on the DSC curve, and this is attributed to the same backbone structure of the matrix and the carbon black reinforcement. But in case of 80 wt % of NR, 20 wt % of XNBR with ISAF N234, the melting peak appreciably shifts to the lower side. This may be due to the dilution effects.

Thermal analysis

High temperature TGA (50–650°C) curves for the sample are shown in Figure 3. The temperature for the onset of degradation (T_1), the temperature at which 10% degradation occurred (T_{10}), the temperature at which 50% degradation occurred (T_{50}), and the temperature at which 90% degradation occurred (T_{90}) were calculated from the TGA plots and are given in Table IV.

It was observed that the onset degradation temperature was more or less the same for all the samples. The onset degradation temperature thereby probably decreased in the case of rubber samples containing ISAF N234 type of carbon black, due to a decrease in crosslink density. Crosslinking increased the rigidity of the system, which in turn increased

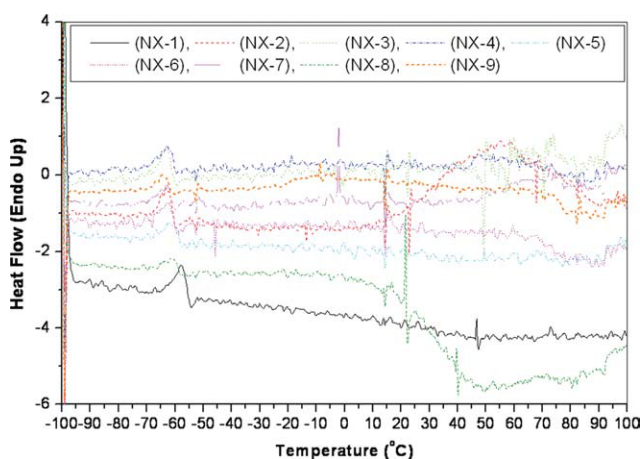


Figure 2 DSC study of different types of blends. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

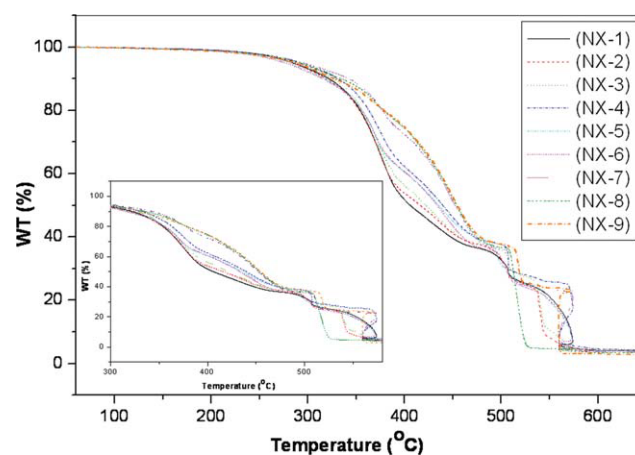


Figure 3 TGA curves of different types of blends. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

TABLE IV
Thermal Properties of the Rubber Samples

Sample code	T_{10} (°C)	T_{50} (°C)	T_{90} (°C)
NX-1	317	404	572
NX-2	305	414	543
NX-3	311	421	551
NX-4	315	439	561
NX-5	301	435	570
NX-6	298	432	572
NX-7	324	452	561
NX-8	316	452	521
NX-9	311	454	559

the thermal stability.^{23,24} The rate of degradation was almost the same up to 90% degradation for all the samples. However, with the presence of carbon black N110 and N774 there is further increase in thermal stability. This may be due to the use of semireinforcing filler which increased the degradation temperature of the matrix.

SEM study

The interfacial linking between XNBR and NR in the presence of carbon black is apt to form a compact and coherent rubber blend matrix. So, SEM studies were considered to shed some light in this regard and thus the tensile fractured surface of the vulcanizates derived from blends of XNBR-NR were chosen for this study. The tensile fracture samples were scanned after gold coating and are represented in Figure 4. The smooth fracture surfaces and smooth filler dispersion and unidirectional tear path oriented along the direction of flow, which is smooth rubbery in nature observed for all rubber samples.²⁵ The micrographs of the NX-1, NX-2, NX-6, NX-7, and NX-9 rubber sample is characterized by a smooth, rubbery failure (which is a smooth failure in the case of rubber samples without the formation of necking) where the additives are clearly seen; the appearance is associated with a low tensile strength.

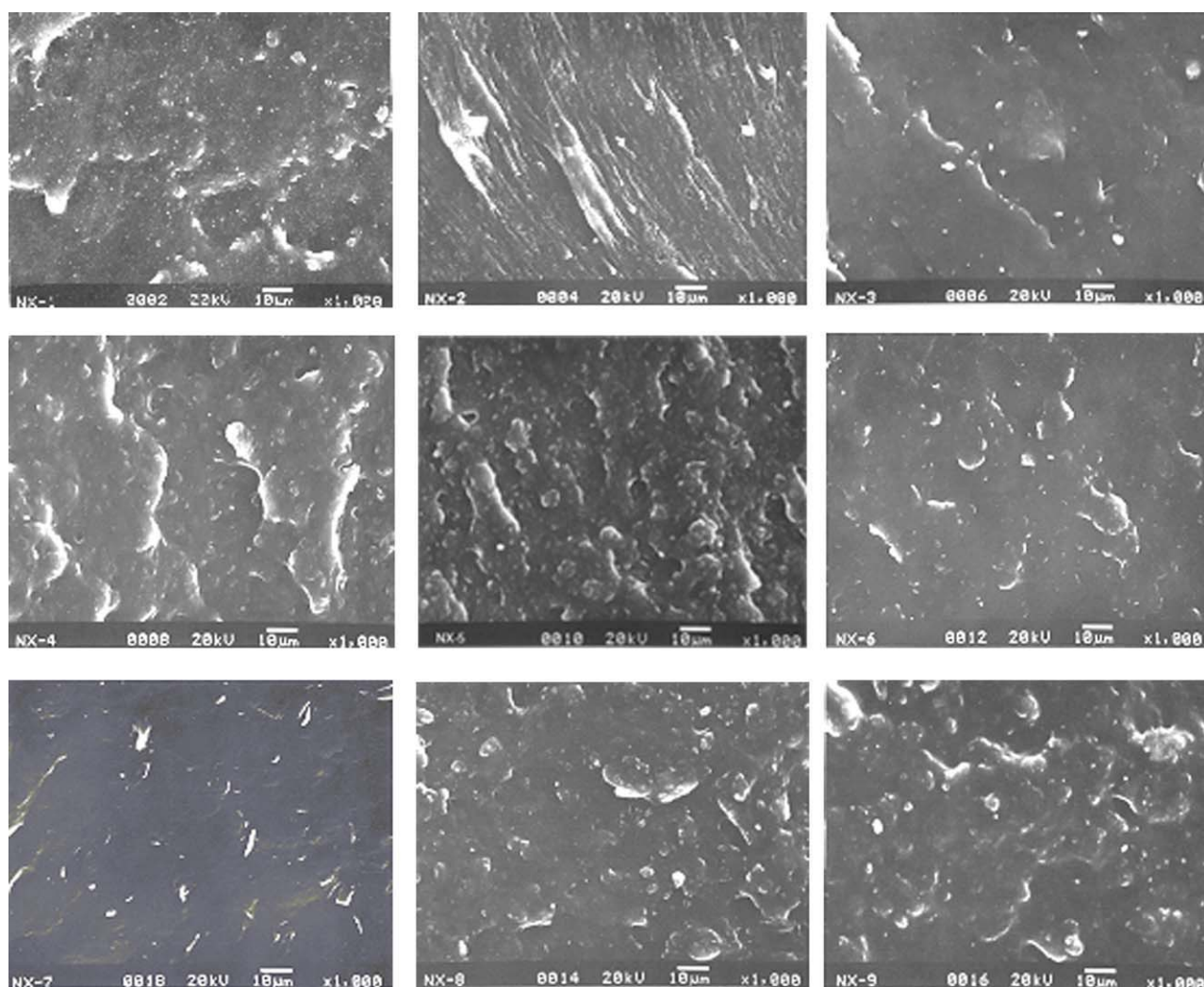


Figure 4 SEM pictures of different types of blends (a) NX-1, (b) NX-2, (c) NX-3, (d) NX-4, (e) NX-5, (f) NX-6, (g) NX-7, (h) NX-8, and (i) NX-9. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://www.interscience.wiley.com).]

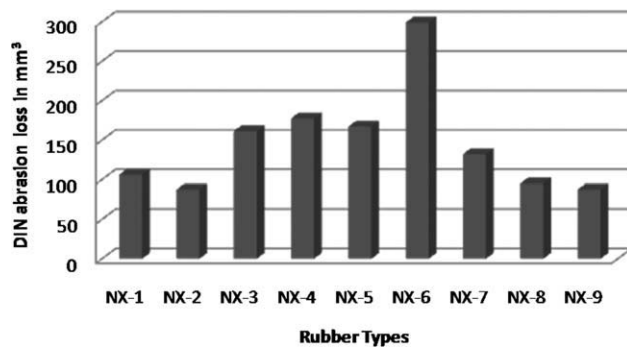


Figure 5 DIN abrasion results of all the samples.

But for NX-3, NX-4 and NX-5, and NX-8, fatigue type of failure is clearly observed from the figure. The SEM study reveals that it is possible to form a coherent blend of NR and XNBR in the presence of carbon black. It may also be concluded that the rough surface has been seen in the sample containing ISAF N234 type of carbon black.

DIN abrader test results

Figure 5 refers to the DIN abrasion test result of the rubber compounds. NX-6 compound showed high abrasion against DIN abrader. The compound NX-1 consisting of SAF N110 and SRF N774 type of carbon black exhibited high abrasion resistance due to the nanosize particle ($\leq 20\text{--}22\text{ nm}$) which entered in to the void of the matrix. So, the dispersion of the CB is good in the matrix, which can be seen from the SEM pictures, and plays a vital role for increase in the abrasion resistance. Also, it is seen that NX-7 to NX-9 overall shows the average abrasion resistance like NX-1 to NX-3.

Rock-rubber abrasion results

Figure 6 shows the abrasion loss of different rubber compounds when abraded against various rocks at different loads. Out of all types of rocks, coal being the softest rock has been identified as the major abrader for almost all types of rubber compounds under the present study. This may be due to the smaller interfacial site of the molecule of coal which results in high abrasion and causes a tarry oily nature of the abraded surface at rock rubber interface. The rubber compound containing 40 wt % of NR and 60 wt % of XNBR are found to be the toughest rubber against all rock types under the present study. Concrete and granite is another rock which has abraded almost all the rubber compounds except 60 wt % of NR and 40 wt % of XNBR extensively and may be considered as an important rock for rock-rubber abrasion studies.

Figure 7 shows the comparison between the DIN abrasion test results with mass loss of rubber compounds by rocks. NX-6 showed high abrasion against DIN abrader and rocks. The compound NX-2, NX-8, and NX-9 exhibited moderate abrasion resistance due to the presence of ISAF type of carbon black, which is mainly used in the OTR tire.

Temperature generation at rock-rubber interface

Since the low thermal conductivity of rubbers can result in a very high temperature at the interface, it is important to investigate the effects of frictional heating on the sliding friction of rubbers.²⁶ Ettles and Shen²⁷ have presented a paper concerning the effects of heat generation on the level of friction at the interface. Table V shows the range of temperature generated during friction of rocks with different rubber compounds. In general the temperature

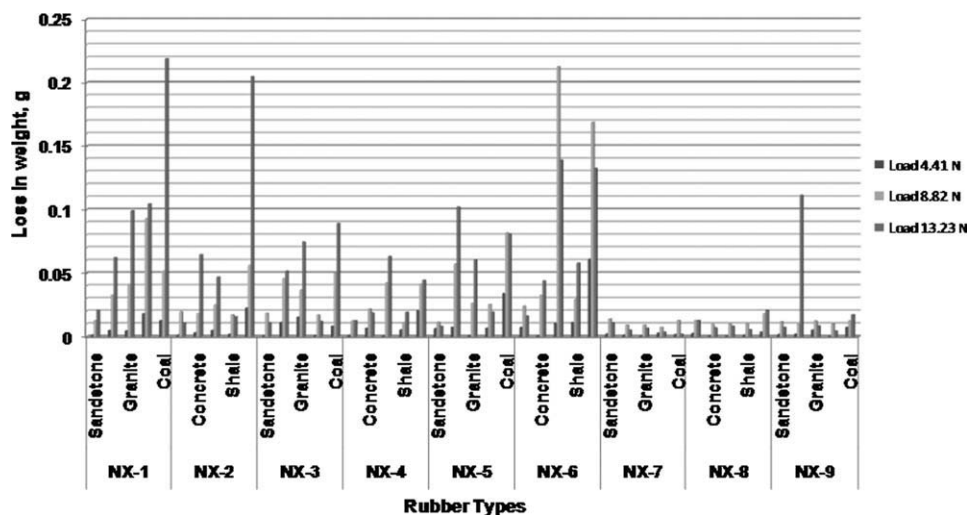


Figure 6 Rubber samples abraded against different rocks at different loads.

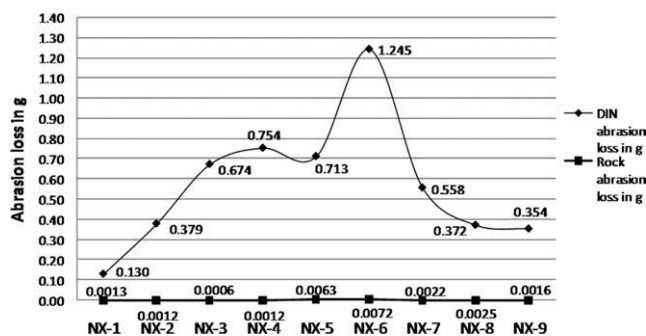


Figure 7 Comparison of mass losses of rubber compounds in DIN abradant.

generation has been found to be higher in case of the rubber compounds containing ISAF N234 type of carbon black, because it is the high structure carbon black. Generally, in the high structure carbon black the particle size is bigger so the dispersion of the CB is not good as well as it deteriorates the abrasion resistance and mechanical properties and generates more heat.

The temperature generation during friction in rubber compound NX-7 and NX-8 is slightly lower, which may be attributed to the presence of SRF N774 and ISAF N234 type of carbon black in the matrix. The highest temperature was observed when rubbers are abraded against coal surfaces whereas the lowest temperature could be noticed for shale. For other rocks the temperature generation was found to be in the moderate range.

CONCLUSIONS

Preparation of abrasion resistant tire tread rubber with the help of an open two-roll-mixing mill represents a novel method for making cost-effective rubber products. The XNBR with addition of NR with ISAF type of carbon black obtained from this process have very good mechanical properties compared to

TABLE V
Temperature Ranges of Different Rubber Samples During Abrasion Against Rocks

Sample code	Temperature range (°C) upto 500 revolutions at 4.41 N load				
	Sandstone	Concrete	Granite	Shale	Coal
NX-1	22–73	20–93	22–73	22–77	20–100
NX-2	22–72	22–89	23–77	21–75	22–106
NX-3	21–71	22–94	22–71	22–76	21–106
NX-4	22–78	22–92	22–73	23–79	22–104
NX-5	22–73	23–96	22–74	22–73	23–107
NX-6	23–76	21–93	21–73	22–75	22–101
NX-7	22–72	22–83	22–74	22–74	24–92
NX-8	22–76	21–83	21–75	23–79	23–95
NX-9	22–70	22–98	22–78	22–74	23–97

other rubbers. It is found that the XNBR has potential as an effective modifier for natural rubber since its incorporation into rubber enhances the plastication of NR during mastication in the mill.

Faster cure rate index and tensile and tear strengths are observed for the compounds containing ISAF N234 type carbon black. The higher modulus is obtained for the compounds under the whole ISAF carbon black groups.

It was observed that the onset degradation temperature was higher for samples containing carbon black ISAF N110 and SRF N774. In the DSC study of the samples containing carbon black ISAF N234 the melting peak appreciably shifts to the lower side due to the dilution effects.

It may also be concluded that rough surface has been seen in the sample containing ISAF N234 type of carbon black after the tensile failure, which is brittle in nature. ISAF N234 type of carbon black with 80 wt % of NR and 20 wt % of XNBR showed higher abrasion resistance property against DIN abradant. Of all types of rocks, coal has been identified as the major abradant for almost all types of rubber compounds in the experimental setup. The rubber compound containing 60 wt % of NR and 40 wt % of XNBR is found to be the toughest rubber against all rock types under the present study.

In the sample containing ISAF N231 type of carbon black temperature generation is higher at the time of abrasion against rock surfaces.

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