

Natural Rubber/Ethylene Propylene Diene Blends for High Insulation Iron Crossarms

Suda Kiatkamjornwong,¹ Kornteen Pairpisit^{2,*}

¹Department of Imaging and Printing Technology, Faculty of Science, Chulalongkorn University, Phyathai Road, Bangkok 10330, Thailand

²Multidisciplinary Program of Petrochemistry and Polymer Science, Chulalongkorn University, Phyathai Road, Bangkok 10330, Thailand

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ABSTRACT: This research studied the composition and behavior of natural rubber (NR) and ethylene propylene diene monomer (EPDM) blends at various carbon black concentrations (0–30 phr) in terms of electrical resistivity, dielectric breakdown voltage testing, and physical properties. The blends having electrical properties suitable for application in high-insulation iron crossarms were selected for investigation of compatibility and increased physical properties. The effect of the homogenizing agent concentration on improvement of compatibility of blends was studied by scanning electron microscopy, pulsed nuclear magnetic resonance spectroscopy, and rheology techniques. We also examined mechanical properties such as tensile strength, tear strength, elongation at break, and hardness. The NR/EPDM blends filled with a fixed concentration of silica were investigated for ozone resistance. A carbon black content as high as 10 phr is still suitable for the insulation coating material, which can withstand electrical voltage at 10 kVac. Addition of the homogenizing agent at 5 phr can improve the me-

chanical compatibility of blends, as evidenced by the positive deviation of shear viscosity of the rubber blend, that is, the calculated shear viscosity being higher than that of experimental data. Moreover, the pulsed NMR results indicated that the spin-spin relaxation (T_2) of all three components of the rubber blend was compressed upon the addition of the homogenizing agent. The ratio of NR/EPDM in the blend to best resist the ozone gas is 80/20 with the addition of silica of 30 phr into the blend. Also, the NR/EPDM filled with silica had a decreased change in thermal and mechanical properties of blends after thermal aging. The synergistic effect of silica content and high NR content (80) in 20 phr EPDM could improve antioxidation by ozone in the absence of a normal antioxidant for natural rubber. © 2004 Wiley Periodicals, Inc. *J Appl Polym Sci* 92: 3401–3416, 2004

Key words: rubber; blends; mechanical properties; viscoelastic properties; dielectric properties

INTRODUCTION

NR and EPDM are blended to combine the excellent physical properties of NR with the ozone-resistant properties of EPDM. The principal target application for such blending has for many years been pneumatic tire sidewalls,¹ where ozone cracking was seen as a factor limiting the lifetime of pneumatic tires. As a consequence, many products dominated by NR, such as domestic appliances and weather seal profiles for vehicles, are almost entirely superseded by EPDM. Sulfur and peroxide as curatives accelerated by dibenzothiazole disulfide (MBTS) and diphenylguanidine (DPG) to improve covalcanization of NR/EPDM blends were studied.² The NR : EPDM ratio of 40 : 60 yielded good tensile properties, excellent heat aging, and static

ozone resistance resulting from the synergistic sulfur/peroxide cure system. High molecular weight EPDM was combined with NR to produce better ozone resistance. The liquid EPDM improved blend processing as a result of a lower Mooney viscosity of the compound. When liquid EPDM was combined with the conventional EPDM and antioxidant, a higher level of NR (up to 85% by weight) could be used to achieve good physical property and excellent static ozone resistance.³ The effect of electric noise of carbon black-filled chloroprene rubber was investigated by Bengtsson.⁴ They found that the rubber samples exhibited a wide range of resistivity from over 107 ohm cm to under 100 ohm cm when carbon black, namely high-abrasion furnace black (HAF), fast-extrusion furnace black (FAF), or medium thermal black (MT) was incorporated in the EPDM/NR blend of a 75 : 25 ratio. The addition of carbon black enhanced both mechanical and electrical properties.⁵ Zaharescu et al.⁶ investigated thermal and oxygen uptake properties of NR/EPDM blends. The presence of NR increased thermal instability and the high rate of

*Present address: Telephone Organization of Thailand, Bangkok, Thailand.

Correspondence to: S. Kiatkamjornwong (ksuda@chula.ac.th).

oxygen consumption because of the higher content of double bonds in NR.

In this research, two substances, namely silica and carbon black, are filled in NR and EPDM blends. In the compounding, the ratio of NR used is always higher than that of EPDM. We use only EPDM to improve the ozone resistance of NR; therefore, the antioxidant is not used in this study. The properly compounded rubber will be used as an electric insulator material for coating on the Telephone Organization of Thailand's (TOT) channel steel crossarms.^{7,8}

EXPERIMENTAL

Materials

The rubber used was standard Thai natural rubber (STR 5L), manufactured by Teck Bee Hang Co., Ltd. (Songkla, Thailand), and EPDM (Nordel IP 4640), manufactured by Chemical Innovation Co., Ltd. (Bangkok, Thailand). Dibenzothiazole disulfide (MBTS) and tetramethyl thiuram disulfide (TMTD) used as accelerators and homogenizing agent (Ultra-blend 4000) was manufactured by Behn Meyer & Co., Ltd. (England), carbon black (HAF N330, surface area of $83 \text{ m}^2 \text{ g}^{-1}$) was manufactured by Thai Carbon Black Public Co., Ltd. (Chonburee, Thailand), and silica (Ultrasil VN 3 Granulate, surface area of $155\text{--}195 \text{ m}^2 \text{ g}^{-1}$) was manufactured by J. J. Degussa Chemicals Ltd. (Germany). A processing additive for natural rubber and synthetic rubber, EF44, containing 8.3% zinc in fatty acid, was used to enhance viscosity compatibility.

PROCEDURES

Control of rubber viscosity

Because the Mooney viscosity of the NR is higher than that of EPDM, and to avoid viscosity incompatibility between NR and EPDM, mastication of NR is necessary to reduce its Mooney viscosity to match that of EPDM. The NR was masticated on a two-roll mill (model R11-3FF, Kodaira, Japan) for 0, 2, 4, or 6 min, and the viscosity of the masticated NRs was measured using a Mooney viscometer (Techpro, Viscotech, USA). After mastication, the masticated NRs were set aside for 30 min and cut to give NR specimens with a diameter of 44.5 mm. Then a specimen was placed in a heated chamber under pressure with a disc-shaped rotor turning inside a cylindrical chamber. Shear stress was produced between surfaces of the rubber and disc. The measured torque yielded the Mooney viscosity of the rubber. It is necessary to strictly control the rotor speed (normally within $2 \pm 0.02 \text{ rpm}$) at a temperature of $373 \pm 0.5 \text{ K}$. The specimens were warmed up for 1 min in the chamber before rotating the motor. A reading of viscosity was taken after 4 min. The

typical Mooney viscosity is reported as ML1+4 (373 K). This test was performed according to ASTM D 1646-87.

Rubber compounding

In this study, a common mixing method was used by mixing two rubbers (NR/EPDM) in a homogenizing agent (Ultra-blend 4000) and followed by addition of ZnO (Metoxide, Thailand Ltd., Bangkok), stearic acid (Pan-Century Edible Oil, SSDN, BHD), fillers, and other curing agents, such as the above-mentioned accelerators and sulfur (Siam Chemicals Ltd., Bangkok), and Struktol EF44, a processing additive to enhance viscosity compatibility. The sequential addition and time schedule were as follows: the NR was first masticated for 4 min by the two-roll mill. The masticated NR, EPDM, and homogenizing agent were mixed together for 3 min in an internal mixer (Farrell, UK) with a capacity of 3.5 L. The temperature of this step was controlled between 373–393 K because the softening point of the homogenizing agent is 368–383 K. Zinc oxide, stearic acid, and a half quantity of carbon black (N330) were added and mixed for 3 min. If other fillers, for example, silica (1/3 quantity of silica) and poly(ethylene glycol) or (PEG 3350) from Kijpaiboon Chemical Ltd., Bangkok, Thailand, were required, they could be added at this step. To avoid cure-rate incompatibility, two accelerators were used. Both worked in a synergistic manner to offer a wide range of curing rate characteristics and scorching. Then 2/3 quantity of the residual filler was added and mixed for 3 min and the accelerators (MBTS and TMTD) were added and mixed for 2 min. The compounded rubber was removed from the internal mixer, and it was mixed with sulfur on the two-roll mill at 363 K for 4 min. Compounded rubbers were sheeted out and left for 24 h before vulcanization in a hot press (Wabash, Charernchai, Karnchang Factory, Thailand).

An oscillating disk rheometer (ODR from Techpro, Model Rheotech, USA) was utilized to determine a vulcanization time of the compounded rubbers. The temperature was set at 423 K, with a testing time of 20 min and a rotation amplitude of 3° , according to ASTM D2084-95. The compounded rubbers were tested under pressure in the die cavity located in the electrically heated plates. The cavity was formed by a fixed lower die and a movable upper die. The ODR maintained a contact with the specimen until the cure reaction was completed.

Preparation of blend compounds

The rubber masterbatch was compounded with the ingredients, for example, homogenizing agent, activators, fillers, and accelerators, using the internal mixer. The temperature of mixing was 363–393 K. Then cura-

TABLE I
Recipe of Rubber Compounds/Blends in the Presence of Carbon Black (N330) in NR/EPDM Blends

Compound ingredients (phr)	NEC0	NEC3	NEC5	NEC7	NEC10	NEC20	NEC30
NR	70	70	70	70	70	70	70
EPDM	30	30	30	30	30	30	30
Ultrablend 4000	5	5	5	5	5	5	5
ZnO	5	5	5	5	5	5	5
Stearic acid	1.5	1.5	1.5	1.5	1.5	1.5	1.5
Carbon Black (N330)	0	3	5	7	10	20	30
MBTS	1	1	1	1	1	1	1
TMTD	0.3	0.3	0.3	0.3	0.3	0.3	0.3
Sulfur	2	2	2	2	2	2	2

tives were added on the two-roll mill to protect scorching of the compound.⁹ The important parameters under study are as follows.

Effect of carbon black concentration on electrical properties of rubber compounds/blends

This study concentrates on electrical conductivity of the rubber blends upon the addition of carbon black (N330). The formulations used are shown in Table I. Volume resistivity and surface resistivity and dielectric strength are then tested.

Effect of homogenizing agent concentration on the compatibility of NR/EPDM blends

The homogenizing agent (Ultrablend 4000) was used to improve the blend homogeneity. It is a low molecular-weight polymeric resin blend, and has components compatible with aliphatic, naphthenic, or aromatic parts of the rubber. Also, Ultrablend 4000 can also function as a processing aid. Thus, the effects of homogeneity and physical properties on the final products were studied. The formulations of NR/EPDM blends with various Ultrablend 4000 concentrations are given in Table II. Mechanical properties of the blends were investigated.

TABLE II
Recipe of NR/EPDM Blends in the Presence of the Homogenizing Agent Ultrablend 4000

Compound ingredients (phr)	NEH0	NEH3	NEH5	NEH7
NR	70	70	70	70
EPDM	30	30	30	30
Ultrablend 4000	0	3	5	7
ZnO	5	5	5	5
Stearic acid	1.5	1.5	1.5	1.5
Carbon Black (N330)	3	3	3	3
MBTS	1	1	1	1
TMTD	0.3	0.3	0.3	0.3
Sulfur	2	2	2	2

Effect of silica on aging

Tensile properties obtained from the recipes of NR/EPDM blends in Table II were unsatisfactorily because tensile strength decreased after aging. Apart from carbon black, the addition of reinforcing silica was thus necessary to improve its properties, as shown in Table III. Aging behavior in terms of mechanical properties was characterized.

Effect of NR/EPDM ratios on ozone resistance

To study the effect of the NR/EPDM blend ratio in the presence of Ultrablend 4000 and silica on properties required, Table IV shows the varied concentrations of both NR/EPDM and Ultrablend 4000 in each formulation.

Electrical property testing of the blends

Volume resistivity and surface resistivity

A standard size specimen, according to ASTM D 257-99, was placed between two electrodes. A voltage of 500 volts was applied for 60 s and the resistance was measured in a high resistance meter (Hewlett Packard,

TABLE III
Recipes of NR/EPDM Blends Containing Fillers (Carbon Black and Silica)

Compound ingredients (phr)	NEC3	NES30	NEC3S30
NR	70	70	70
EPDM	30	30	30
Ultrablend 4000	5	5	5
ZnO	5	5	5
Stearic acid	1.5	1.5	1.5
Carbon Black (N330)	3	—	3
Silica	—	30	30
PEG	—	1	1
EF44	—	1	1
MBTS	1	1	1
TMTD	0.3	0.3	0.3
Sulfur	2	2	2

TABLE IV
Recipes of NR/EPDM Blend Ratios and Ultrablend 4000

Compound ingredients (phr)	NS	N8E2S	N7E3S	N6E4S	ES
NR	100	80	70	60	—
EPDM	—	20	30	40	100
Ultrablend 4000	0	5	5	5	0
ZnO	5	5	5	5	5
Stearic acid	1.5	1.5	1.5	1.5	1.5
Carbon Black (N330)	3	3	3	3	3
Silica	30	30	30	30	30
PEG	1	1	1	1	3
EF44	1	1	1	1	1
MBTS	1	1	1	1	1
TMTD	0.3	0.3	0.3	0.3	0.3
Sulfur	2	2	2	2	2

Model HP 4339D, Japan). Surface or volume resistivity was then calculated. Surface resistivity is expressed in ohms, and volume resistivity is expressed in ohm cm. The connections for volume and surface resistivity measurements are shown in Figure 1. Volume resistivity, that is electrical resistivity, was also measured by the method described in ASTM D 991-89.

Dielectric strength

In this study, the test method applied the maximum voltage required, 10 kVac, for 1 min by a high-voltage tester (Hipotronics, Model HD100, U.S.A.) is the TOT's specification.⁸ The voltage was applied across the two electrodes and raised from 0 to 10 kVac at a uniform rate. A breakdown was observed when an electrical burn-through punctured the sample, or decomposition occurred in the specimen. If it did not cause any damage to the specimen, this implied that the specimen could withstand the voltage applied. The specimen size for this test was a 4-in or somewhat larger plaque. Although any specimen thickness could be used, the most common thicknesses were between 0.8 and 3.2 mm (0.032 to 0.125 in), according to ASTM D 149-97.

Determination of the NR/EPDM compatibility

Glass transition temperature by differential scanning calorimetry (DSC)

A sample of 8 to 10 mg in an aluminum sample pan was placed into the differential scanning calorimeter (DSC7, Perkins Elmer, UK). The sample was cooled at a controlled rate of 10 K min⁻¹, and the temperature was varied from 173 to 273 K. A plot of heat flow versus temperature was produced. The resulting thermogram was then analyzed.

Glass transition temperature by dynamic mechanical thermal analysis (DMTA)

A sample size for the usual flexural test mode of 1 × 10 × 40 mm is required in the parallel plate shear mode. The sample was cooled at a controlled rate of 5 K min⁻¹, for frequencies from 1.66 to 3.33 Hz and temperatures from 173 to 293 K in a dynamic mechanical thermal analyzer (DMA242, Netzsch, Germany). Thermograms are plotted to show elastic modulus, E' , and $\tan \delta$ vs temperature. The T_g value of sample can be measured from the peak in $\tan \delta$.

Scanning electron microscopy

A scanning electron microscope (JSM-5800LV) was used to observe the blend morphology of vulcanized rubber. The sample was prepared by staining with osmium tetroxide, which reacted with unsaturated carbon-carbon bonds. Then the sample was placed on the stub by double-sided sticky tape and was coated by sputtering of carbon or another conductive material such as gold to provide an electrically conductive layer, to suppress surface charges, to minimize radiation damage and to increase electron emission. NR/EPDM blends in the absence and presence of the homogenizer were viewed.

Compatibility study by rheological characterization

The blend compatibility of NR/EPDM blends was studied through the capillary and dynamic flow of a single bore capillary rheometer (Rosand Precision, England). Testing conditions were carried out at a wide range of shear rates (10–1500 s⁻¹) at 373 K.¹⁰ The

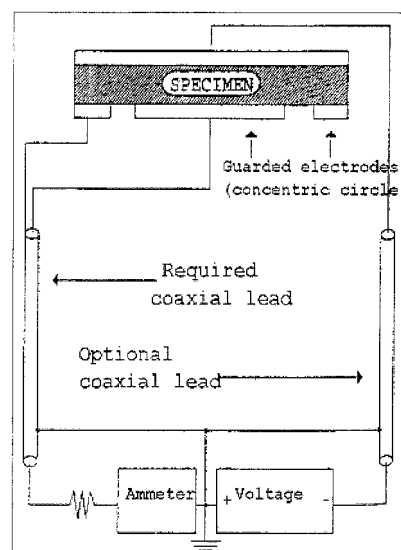


Figure 1 Connections to guarded electrodes for volume and surface resistivity measurements.

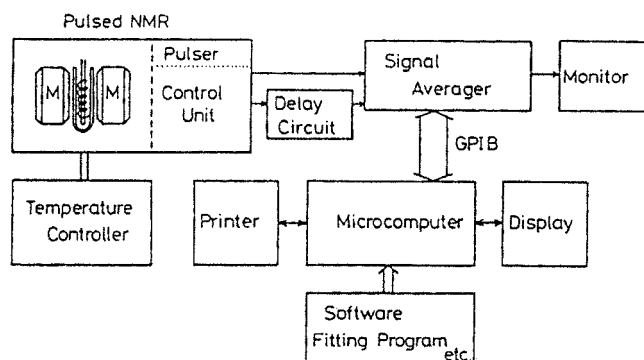


Figure 2 Block diagram of the real-time pulsed NMR measurement system controlled by a microcomputer.

capillary die used was 2 mm in diameter and 32 mm in length.

Homogeneity of the blend by pulsed nuclear magnetic resonance

The NMR measurements were performed under the control of a microcomputer (Bruker PC-20, Germany, with a resonant frequency for protons of 20 MHz). The sample (30–50 mg) was dissolved in the solvent and transferred into an NMR sample tube. Then the sample tube was set in an NMR sample holder, and a real-time measurement was started. After the measurement, data were analyzed with a microcomputer by a nonlinear least-squares fitting method.

A block diagram of a real-time pulsed NMR measurement system is given in Figure 2.¹¹ The H signal of NMR was fed into the signal averages and averaged for certain times to increase the signal-to-noise ratio if necessary. The temporal changes of both the fraction and the spin-spin relaxation time T_2 signals from the solid echo and spin echo method were obtained. From

this information, the degree of heterogeneity from the difference in mobility of the polymer can be obtained.

Mechanical testing

Mechanical properties of the vulcanized rubbers, that is, tensile strength, hardness, and tear resistance, before and after aging, were measured according to ASTM and ISO test methods.

Tensile properties

Tensile strength, tensile modulus, and elongation at break of a specimen were measured in a universal testing machine (Lloyd LR5K, England). A dumbbell specimen is required (die C), according to ASTM D 412-87. The specimens were placed in the grips at a specified grip separation and pulled until failure. Six specimens were required for each measurement. The test speed was 400 mm min⁻¹ for measuring strength and elongation, and 1 mm min⁻¹ for measuring the modulus.

Hardness measurement

A standard specimen of 6.4 mm (0.25 in) thickness was either molded or cut from a sheet, according to ASTM D 2240-97 with the Shore A scale. The standard specimen was placed on the surface of the Rockwell Hardness tester (Shore A, Instron MFG, England). A minor load was applied and the gauge was set to zero. The major load was applied by tripping a lever. After 15 s, the major load was removed. The specimen was allowed to recover for 15 s and then the hardness was read off from the dial with the minor load still applied.

TABLE V
Effect of High Concentration Carbon Black on Vulcanization Properties

Vulcanization characteristics and properties tested	Formulation ^a			
	NEC0	NEC10	NEC20	NEC30
ODR at 423 K, t ₉₀ (min)	7.8	7.1	5.7	4.9
Mooney viscosity at 373 K, MU	36.3	36.5	37.5	52.9
Tensile strength (MPa)	12.7	15.8	13.8	14.8
Tear strength (N mm ⁻¹)	25.1	30.4	32.3	35.7
Hardness (Shore A)	40.6	48.3	53.4	56.4
Electrical properties:				
Before aging				
Volume resistivity (ohm cm)	2.7 × 10 ¹⁵	3.3 × 10 ¹⁵	1.8 × 10 ¹⁰	b
Surface resistivity (ohm)	7.5 × 10 ¹⁵	4.2 × 10 ¹⁵	5.5 × 10 ¹⁰	b
After aging (at 373 K for 22 h)				
Volume resistivity (ohm cm)	2.7 × 10 ¹⁵	3.7 × 10 ¹⁵	5.2 × 10 ¹⁰	b
Surface resistivity (ohm)	2.8 × 10 ¹⁶	6.8 × 10 ¹⁵	3.7 × 10 ¹⁰	b

^a NEC0, NEC10, NEC20, and NEC30 contain 0, 10, 20, and 30 phr of carbon black, respectively.

^b The resistivities were lower than the limit of testing equipment.

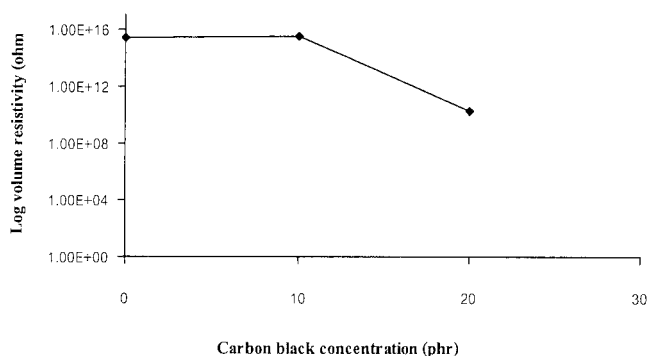


Figure 3 Effect of carbon black concentration on volume resistivity of the natural rubber.

Tear resistance

The specimen was die cut to the appropriate shape (Die C) from a sheet, according to ASTM D 624-81. The specimen was then placed in the grips of the testing machine and pulled at a rate of 500 mm min⁻¹ until rupture. The resistance was measured in Newtons.

Aging tests

Thermal aging

In this study the specimen was tested in an air oven (Ueshima, Japan) at 373 K for 22 h, according to ISO 188 Standard or ASTM D 573-99. The results of the aging test were expressed as a percentage of the change in each physical property and calculated as follows:

$$P = [(A - O)/O] \times 100, \quad (1)$$

where P is the percentage change in property, O is the original value, and A is the value after aging.

Ozone resistance test

The strip specimen size was 10 × 40 × 2 mm³. Then the specimen was stretched up to 20% elongation and it was hung in a controlled chamber at 313 K (Ozone test chamber, Hampden, England). The ozone concentration was set at 50 ppm and was left for 72 h, according to ASTM D 1149-99. Cracking on the specimen surface was then examined.

RESULTS AND DISCUSSION

Based on the work of Lewis et al.,¹² examination of NR (STRL 5L)/EPDM blends and NR/Brominated EPDM blends by rheology and the log additive rule, along with thermal properties and pulsed NMR techniques, indicate that the blends are thermodynamically incompatible. This research tried to reduce the viscosity

incompatibility and cure-rate incompatibility due to the dissimilar nature of the two rubbers. A sequential procedure was carried out as mentioned above.

Control of rubber viscosity

Because the Mooney viscosity of NR is always higher than that of EPDM, the mixing of the two rubbers induced a viscosity mismatch problem. NR must be then softened by mastication on a two-roll mill before mixing with EPDM. The Mooney viscosities of the unmasticated rubbers in this research were 71 and 59 MU at ML 1+4 (373 K), respectively. Therefore, NR was masticated and sampled for Mooney viscosity measurements at different times to find the proper mastication time for viscosity matching. The Mooney viscosity of NR after 4 min of mastication is close to that of EPDM. The viscosity for a mastication time of 0, 2, 4, or 6 min is 71, 63, 60, or 58 MU, respectively. Therefore, the mastication time of 4 min is good for the subsequent experiments to control the NR viscosity.

Vulcanization characteristics

After mixing, the compounded rubber blends were made into sheets for vulcanizing in the rolls. The temperature and the appropriate cure time of this compound were determined by ODR. The cure time at 90% or t₉₀ of the masterbatch in this study was varied from 5–8 min; therefore, another 2 min are needed for a complete cure. The torque against time slope on a recorder is proportional to the measured stiffness or shear modulus of the rubber.¹³

Effect of carbon black concentration on electrical properties of rubber compounds/blends

Although carbon black dominates the conductive electrical properties of filled rubber,¹⁴ the primary purpose of using carbon black in this research is to reinforce the rubber blends. Inclusion of rigid filler particles in the soft matrix (NR) contributes additional filler–rubber interactions resulting in more crosslinking in the flexible network. Significant changes in the overall properties could be obtained from filler–elastomer and filler–filler interaction. It is essential that the carbon black concentration used in these applications be investigated, to obtain an appropriate reinforced and insulation rubber for coating on an iron crossarm.

For formulations NEC0, NEC10, NEC20, and NEC30, concentrations of carbon black at 0–30 phr were added. Based on the formulations, NEC0 was used for comparison with the carbon black filled compounds. Surface and volume resistivity of the blends were investigated. The higher the surface/volume resistivity, the lower the leakage current and the less

TABLE VI
Effect of Low Concentration Carbon Black on Vulcanizate Properties

Vulcanization characteristics and properties tested	Formulation ^a				
	NEC0	NEC3	NEC5	NEC7	NEC10
ODR at 423 K, t ₉₀ (min)	7.8	7.4	7.7	6.9	7.1
Mooney viscosity at 373 K, MU	36	35	33	37	36
Tensile strength (MPa)	12.7	14.7	11.5	12.0	15.8
Tear strength (N mm ⁻¹)	25.1	26.1	26	29.8	30.4
Hardness (Shore A)	40.6	42.6	44	44.9	48.3
Electrical properties:					
Volume resistivity (ohm cm)	2.7×10^{15}	2.2×10^{15}	3.8×10^{15}	3.5×10^{15}	3.3×10^{15}
Surface resistivity (ohm)	7.5×10^{15}	6.8×10^{15}	1.6×10^{15}	1.2×10^{16}	4.2×10^{15}
Dielectric breakdown voltage at 10 kVac for 1 min	b	b	b	b	b

^a NEC0, NEC3, NEC5, NEC7, and NEC10 contain 0, 3, 5, 7, and 10 phr of carbon black, respectively.

^b The specimens can withstand the dielectric breakdown at 10 kVac for 1 min.

conductive the material is. From Table V, the formulation NEC30 was found to be overloaded in terms of both the volume resistivity and surface resistivity. That is, the resistivity of this formulation was lower than the limit of the testing equipment. This is an effect of the quasi-graphitic microstructure of the carbon black, which makes the blend more electrically conductive. The results of accelerated aging tests on the resistivity of blends containing of carbon black of various concentrations are also given in Table V. The effect of aging on the resistivity of carbon black-filled rubbers is a slight increase. This effect is very small, for most applications; a small change in resistivity is useful for outdoors applications.¹⁵

The effect of carbon black concentration on the resistivity of blends is shown in Figure 3. This plot shows that increasing the carbon black concentration resulted in decreased resistivity, which was caused by the increase of the number of carbon black aggregates per unit weight. The decreased distance between black aggregates facilitated the electron transfer from aggregate to aggregate.^{16,17} According to the TOT's specification for the insulation coating, the materials for iron crossarm coating must have a volume resistivity

higher than 10^{11} ohm cm.⁸ Thus, the carbon black concentration is critical, which indicates that at most 10 phr are required for a suitable insulation compound. The volume resistivities of natural rubber and EPDM are 10^{15} and 10^6 ohm cm, respectively. As a consequence, the concentrations of carbon black at 0–10 phr (formulations NEC0, NEC3, NEC5, NEC7, and NEC10) were studied as given in Table VI. The major application of carbon black in this recipe is for antistatic protection. The small amount of carbon black used in this application is to achieve a suitable resistivity before a significant drop in resistivity is noted. The dielectric breakdown voltages were investigated to determine *in situ* dielectric failure arising under electrical field application.

The antistatic resistivity of all formulations in this recipe could not be measured. In addition, no standard resistance tests have been specified for antistatic materials, which have a resistance above 3×10^8 ohms.¹⁵ After applying the required high voltage at 10 kVac for 1 min to the specimens, we did not find any electrical burn-through, punctures, or electrical discharges. The results of dielectric breakdown voltage tests indicated that the appropriate concentration of carbon black is lower than 10 phr for insulation compounds. Too high a carbon black content (>10 phr) produced conductive compounds, which are dangerous for the telephone system and any human beings nearby.

TABLE VII
Effect of Homogenizing Agent Concentration on Vulcanizate Properties of NR/EPDM Blends

Vulcanization characteristics and properties tested	Formulation ^a			
	NEH0	NEH3	NEH5	NEH7
ODR at 423 K, t ₉₀ (min)	6.9	7.8	7.4	7.8
Mooney viscosity at 373 K, MU	43	41	35	37
Tensile strength (MPa)	4.53	11.6	14.7	11.2
Tear strength (N mm ⁻¹)	28.4	26.9	26.1	29.2
Hardness (Shore A)	41.9	41.8	42.6	42.2

^a NEH0, NEH3, NEH5, and NEH7 contain 0, 3, 5, and 7 phr of Ultrablend 4000, respectively.

Effect of the homogenizing agent concentration on the compatibility of NR/EPDM blends

Using a homogenizing agent is a convenient way to improve the properties of immiscible blends. This investigation attempts to improve adhesion between phases of the NR/EPDM blend system by incorporation of a suitable homogenizing agent. The structure of a prospective homogenizing agent should have the

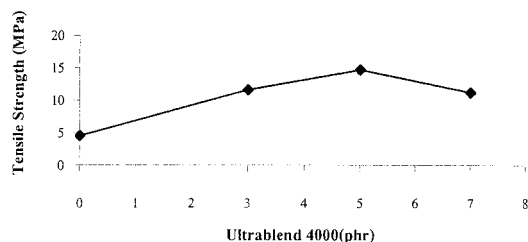


Figure 4 Effect of the homogenizing agent concentration on tensile strength of NR/EPDM blends.

same or a similar chemical nature to those of the two rubbers. One can obviously observe the improved properties after a proper homogenizing agent has been incorporated.¹⁸

The homogenizing agent selected contains portions that are compatible with aliphatic, naphthenic, and aromatic parts of the elastomer in the blend. It is absorbed quickly by the rubber components to wet out the surfaces, inducing a fast incorporation of other compounds. It improves the homogeneity of the rubber blends of different polarities or viscosity, and increases mechanical properties of the vulcanizates.

Effect of the homogenizing agent concentration on mechanical properties of NR/EPDM blends

The concentrations of Ultrablend 4000 homogenizing agent of 0, 3, 5, and 7 phr were added into NR/EPDM blends (NEH0, NEH3, NEH5, and NEH7, respectively), as summarized in Table II. Curing characteristics and mechanical properties of the NR/EPDM blends homogenized with Ultrablend 4000 are given in Table VII and Figures 4 to 6 in terms of tensile strength, tear strength, and hardness, respectively.

It was found that the tensile strength of blends was increased when a small amount of Ultrablend 4000 (3 phr) was added. It can be seen that tensile strength increases in the blends containing 3 (NEH3) and 5 phr (NEH5) Ultrablend 4000. However, this decreased

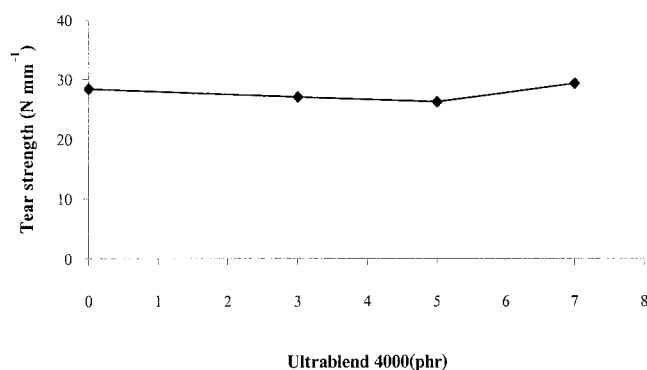


Figure 5 Effect of the homogenizing agent concentration on tear strength of NR/EPDM blends.

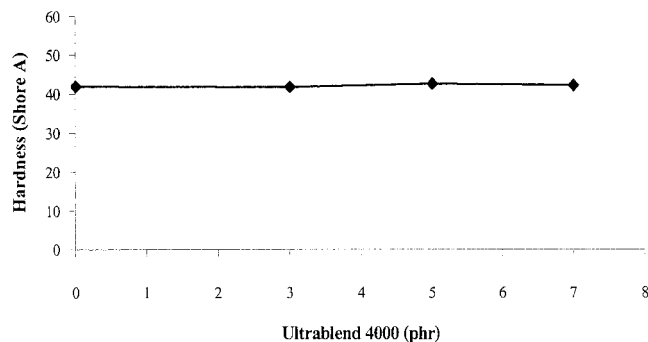


Figure 6 Effect of the homogenizing agent concentration on hardness of NR/EPDM blends.

when more Ultrablend 4000 (7 phr) was added (formulation NEH7). This result can be explained in terms of the compatibility gained by the addition of Ultrablend 4000, which improves the compatibility between the matrix phase (NR) and dispersed phase (EPDM). When the sample was stretched in the tester, the stress was transferred from the matrix phase (NR) to the dispersed phase (EPDM) through the homogenizing agent. The mechanical properties were gradually improved when 3 and 5 phr of Ultrablend 4000 were added. On the contrary, when Ultrablend 4000 was further added to 7 phr, tensile strength was poor. This could be the agglomeration of the excess amount of Ultrablend 4000 to become another phase. This new phase induces slippage or weak points between the matrix and dispersed phases, yielding a lower tensile strength. The tear strength and hardness of blends were not significantly different when Ultrablend 4000 was added.

In addition to the tensile strength, elongation at break was measured, as shown in Table VIII. The high value of elongation at break and tensile strength of NR/EPDM blends found in the presence of 5 phr Ultrablend 4000 can explain the good adhesion between the phases of NR and EPDM.¹⁹ A concentration of Ultrablend 4000 of 5 phr should be thus used for the blends of NR/EPDM.

Effect of silica on mechanical properties of NR/EPDM blends

Silica (Ultrasil VN3, commercial silica without silane as a coupling agent) was incorporated to further im-

TABLE VIII
Dependence of Tensile Strength and Elongation at Break of NR/EPDM Blends (70/30) on the Concentrations of the Homogenizer

Homogenizing level (phr)	Tensile strength (MPa)	Elongation at break (%)
0	4.5	383
3	11.6	544
5	14.7	609
7	11.2	589

TABLE IX
Comparison of Fillers (Silica and Carbon Black) on
Vulcanizate Properties of NR/EPDM Blends

Vulcanization characteristics and properties tested	Formulation ^a		
	NEC3	NES30	NEC3S30
ODR at 423 K, t ₉₀ (min)	7.4	4.3	5.4
Mooney viscosity at 373 K, MU	35	67	71
Physical properties:			
Before aging			
Tensile strength (MPa)	14.7	10.9	13.9
Tear strength (N mm ⁻¹)	26.1	30.5	30.4
Hardness (Shore A)	42.6	56	54.5
After aging (at 373 K for 22 h)			
Tensile strength (MPa)	3.72	8.1	11.2
Tear strength (N mm ⁻¹)	16.8	31.5	34.2
Hardness (Shore A)	45.6	58.5	57.3
% Change in tensile strength after aging	75	26	20

^a NEC3, NES30, and NEC3S30 contain 3% carbon black, 30% silica, and 3% carbon black and 30% silica, respectively.

prove the mechanical properties, such as tear strength and hardness of NR/EPDM blends. The effectiveness of fillers (Ultrasil VN3 and N330) on vulcanizates and mechanical properties of NR/EPDM blends is shown in Table IX. Figures 7 to 9 illustrate the effects of the fillers (silica and carbon black) on the mechanical properties of NR/EPDM blends.

Dispersion of silica particles in NR/EPDM is a major concern. Although both are reinforcement fillers, the surface area of the silica is much higher (155–195 m² g⁻¹) than that of carbon black and their surface chemistry is different (see Tables V and IX). We found that silica increases the Mooney viscosity as expected, and reduces the ODR. When the amount of Ultrasil 4000 was kept constant at 5 phr, we anticipated that the silica particles absorbed a greater concentration of Ultrasil 4000 than did the carbon black particles,

resulting from their higher surface area. However, the surface of N330 carbon black contains basic groups, whereas the silica surface is dense with hydroxyl functional groups, which are relatively acidic and hydrophilic. The homogenizer (the nonpolar compound) should thus be strongly adsorbed on the carbon black surface because of the similar nonpolar functional groups, although the acidity of the silica surface may contribute to better particle wetting. The absorbed homogenizer reduces the filler–filler interaction and prevents agglomeration by themselves or coagglomeration. Before aging, the tensile strength of NEC3 is thus rather higher than those of NES30 and NEC3S30.

Because of the high temperature resistance of silica, the presence of silica in the blends produces a marked increase in mechanical strength after aging by 120%. The main effect of silica additive alone in NR/EPDM is thus very attractive. More interestingly, the synergistic improvement in tensile strength of the blend was obviously seen in the presence of both carbon black (3 phr) and silica (30 phr), especially regarding properties after aging, with another increase of tensile strength by 38%. Considering the ODR and Mooney viscosity, one could observe a high viscosity when silica was added. The silica filler contains the hydroxyl function group on its surface. The intermolecular bonding between the hydroxyl group in silica and the NR/EPDM might be able to take place. The network structure further developed when carbon black was added, as evidenced by the increased Mooney viscosity. However, silica has been known for quite a long time to affect the crosslinking density in vulcanization systems by accelerator/sulfur curing. Bokobza and Rapoport²⁰ proved that in the absence of a silane coupling agent, silica in the mentioned curing system reacts with chemical ingredients in the formulation leading to a lower overall cure, which can be quantified by measurements of polymer chain orientation. Upon heating, intermolecular forces responsible for

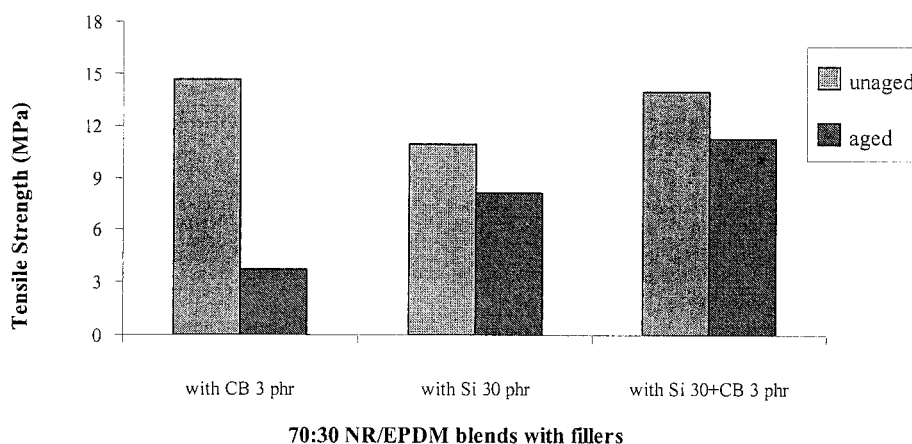


Figure 7 Comparison of tensile strength of the NR/EPDM with carbon black-, silica-, and carbon black/silica-filled blends.

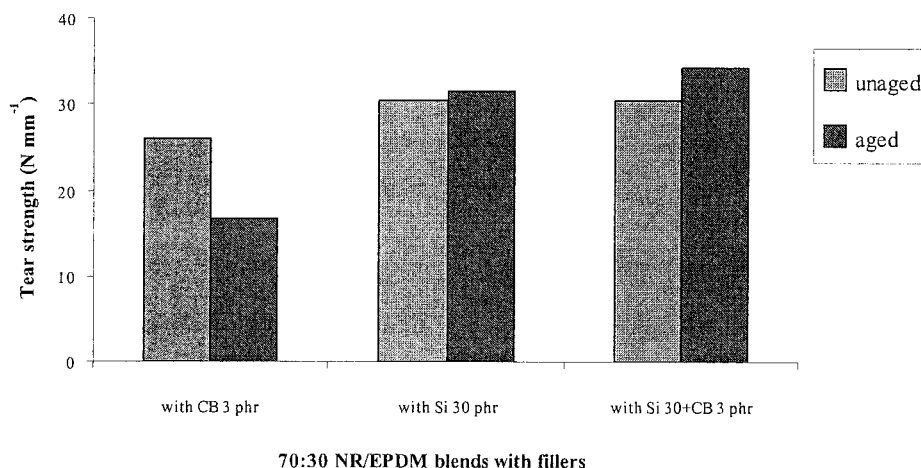


Figure 8 Comparison of tensile strength of the NR/EPDM blends containing carbon black (3%), silica (30%, and carbon black/silica 3 : 30).

miscible behavior tend to disappear as the internal energy of the molecules is high enough to overcome forces from the intermolecular forces. Some physical structure was thus destroyed to result in a lower tensile strength. It is obvious that silica enhances the tensile strength of NR/EPDM blends after thermal aging, because silica improves heat resistance of the material, and it does not promote curing.¹³ The synergistic effect of carbon black and silica is likewise seen in the tear strength and hardness. This effect enhances the blend heat resistance by a substantial increase in tear strength and hardness.

Characterization of blends by differential scanning calorimetry (DSC) and dynamic mechanical thermal analysis (DMTA)

The DSC and DMTA thermograms of the cured NR, EPDM, and NR/EPDM of various Ultrablend 4000 con-

centrations are shown in Table X. It is found that the glass transition temperatures (T_g) of the vulcanized NR and EPDM, and Ultrablend 4000 were -62°C (211 K), -49°C (224 K), and 59°C (332 K), respectively.

Detection of glass transitions by DSC is sometimes uncertain if the transition occurs over a broad range of temperatures or if the component is present at a low level. The DSC T_g will be lower than that of DMTA by an amount that depends on the specific polymer.²¹ DMTA is the method of choice to give a more accurate values of T_g or other transitions also observed for all components in a system than DSC, because the properties measured are the dynamic modulus and damping, which change substantially between the glassy and rubbery states.²² The glass transition temperatures (T_g) of all formulations measured from the DMTA were shifted between those of NR and EPDM (see Table X). One can conclude

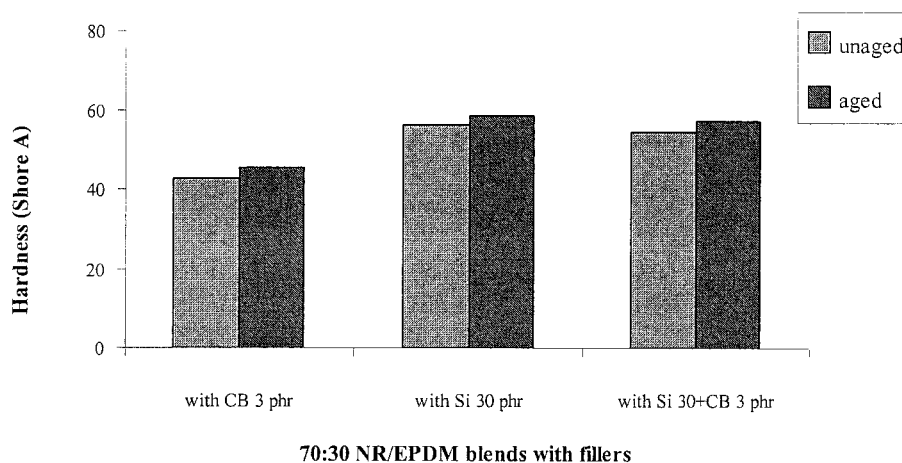


Figure 9 Comparison of tear strength of the NR/EPDM blends containing carbon black (3%), silica (30%), and carbon black/silica (3 : 30).

TABLE X
Glass Transition Temperatures from DMTA Measurement of Cured NR and EPDM, Homogenizing Agent, and NR/EPDM Blends

Component sample	T_g , K (°C)				Compatibility	
	NR	EPDM	NR shift	EPDM shift	Partial	Total
NR	211 (-62)	—	—	—	—	—
EPDM	—	224 (-49)	—	—	—	—
NEH0	213 (-60)	235 (-38)	217 (-56)	228 (-45)	✓	—
NEH3	—	237 (-36)	217 (-56)	230 (-43)	✓	—
NEH5	—	237 (-36)	217 (-56)	227 (-46)	✓	—
NEH7	210 (-63)	—	215 (-58)	230 (-43)	✓	—

NEH0, NEH3, NEH5, and NEH7 contain 70 phr NR, 30 phr EPDM, and 0, 3, 5, and 7 phr Ultrablend 4000, respectively.

that all the blends are partially miscible. The NR/EPDM (NEH5) containing Ultrablend 4000 of 5 phr had T_g values shifted closer together than the other formulations. Ultrablend 4000 at this particular concentration increased the compatibility of the NR/

EPDM blends. Considering the solubility parameter of $19.4 \text{ (MPa)}^{1/2}$ for NR and of $18.4 \text{ (MPa)}^{1/2}$ for EPDM and averaged molecular weights of NR and EPDM of 2×10^6 and 3×10^3 , respectively, the components in the homogenizing agent, having

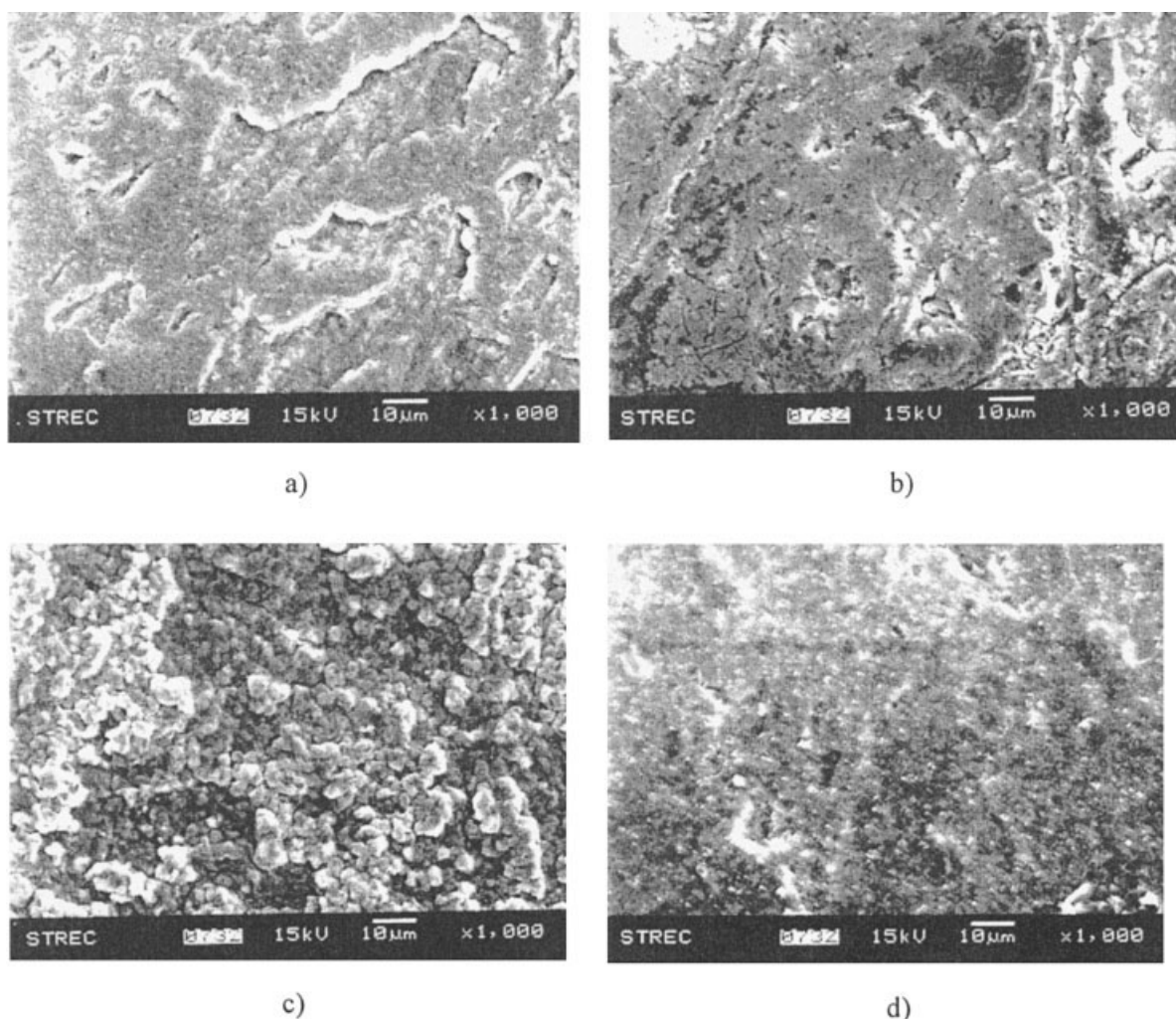


Figure 10 SEM micrographs of (70 : 30) NR/EPDM blends containing (a) 0, (b) 3, (c) 5, and (d) 7% phr of the homogenizing agent, stained with osmium tetroxide: (a) without the homogenizing agent (Ultrablend 4000); (b) filled with the homogenizing agent (Ultrablend 4000), 3 phr; (c) filled with the homogenizing agent (Ultrablend 4000), 5 phr.

TABLE XI
Shear Viscosity of (70 : 30) NR/EPDM Blends with the Homogenizing Agent from Experiment vs. Log Additivity Rule Model

Shear rate (s ⁻¹)	Shear viscosity (Pa s)			Concentration of Ultrablend 4000 (phr)							
	NR	EPDM	Ultrablend 4000	0		3		5		7	
				log η blend experiment	log η [eq(2)]	log η blend experiment	log η [eq(2)]	log η blend experiment	log η [eq(2)]	log η blend experiment	log η [eq(2)]
40	4967	8066	1849	3.64	3.76	3.74	3.74	3.83	3.74	3.71	3.73
90	2590	4241	1449	3.38	3.48	3.44	3.47	3.55	3.46	3.42	3.46
180	1438	2414	1072	3.12	3.23	3.19	3.22	3.30	3.22	3.16	3.21
350	816	1349	739	2.87	2.98	2.93	2.97	3.08	2.97	2.91	2.97

broad solubility parameter values of 16.7 to 23.5 (MPa)^{1/2}, are anticipated to give a better compatibility. Broadly speaking, it is a better homogenizing agent for the blend.^{23,24} Because the Ultrablend 4000 is a low molecular weight polymeric resin blend or a higher molecular weight homolog of the plasticizer, it is compatible with aliphatic, naphthenic, or aromatic pairs of the rubber, as indicated by the T_g shifts.

Characterization of blends by scanning electron microscopy (SEM)

Scanning Electron Microscopy (SEM) has been used to study the phase morphology of NR/EPDM blends. Staining the NR/EPDM blends by osmium tetroxide is an easy technique, because NR contains a greater degree of unsaturation, which reacts with osmium tetroxide. The stained region of NR phase

will appear brighter than that of the EPDM phase. Figure 10(a)–(d) show the surface morphology of NR/EPDM blends containing 0, 3, 5, and 7 phr homogenizing agent (Ultrablend 4000). It is clear that the homogenizing agent addition has a remarkable effect on phase dispersion.²⁵ The phase size of blends was reduced after an appropriate amount of the homogenizing agent level was added, leading to fine dispersion of one phase in another. The compatibility of NR/EPDM blends was thus improved. We found a relationship between the effect of the optimum concentration (5 phr) of the homogenizing agent, morphology of the blends, and tensile strength and elongation at break. One can observe a reduced phase size of the second polymer after an optimum homogenizer concentration was added and upon a finer dispersion of EPDM domains into the NR matrix. At the suitable concentration of the

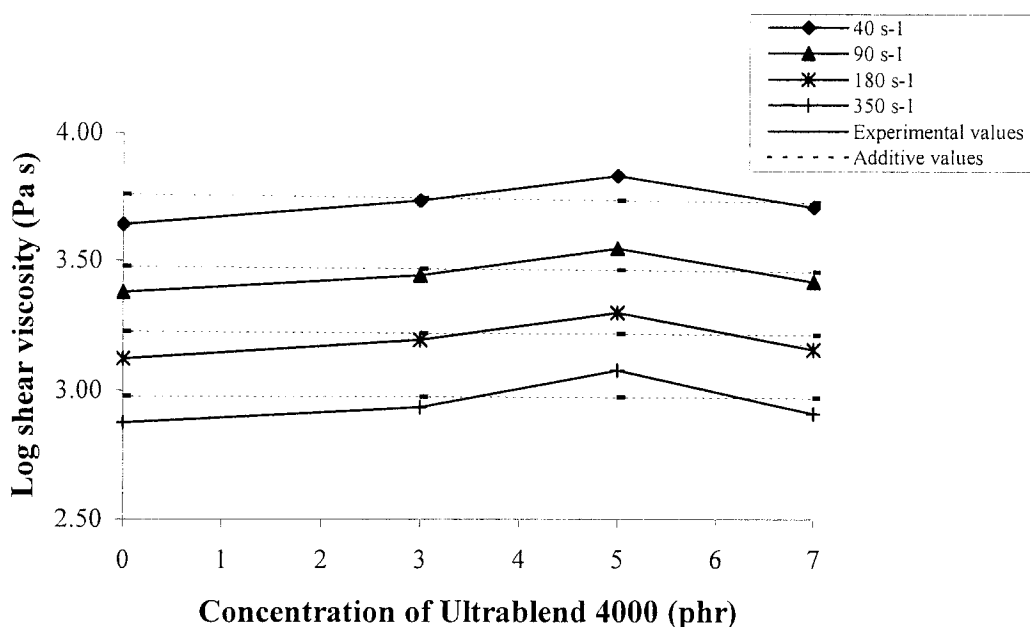


Figure 11 Comparison of apparent shear viscosity at apparent shear rates of 40, 90, 180, and 350 s⁻¹ for (70 : 30) NR/EPDM blend with the homogenizing agent.

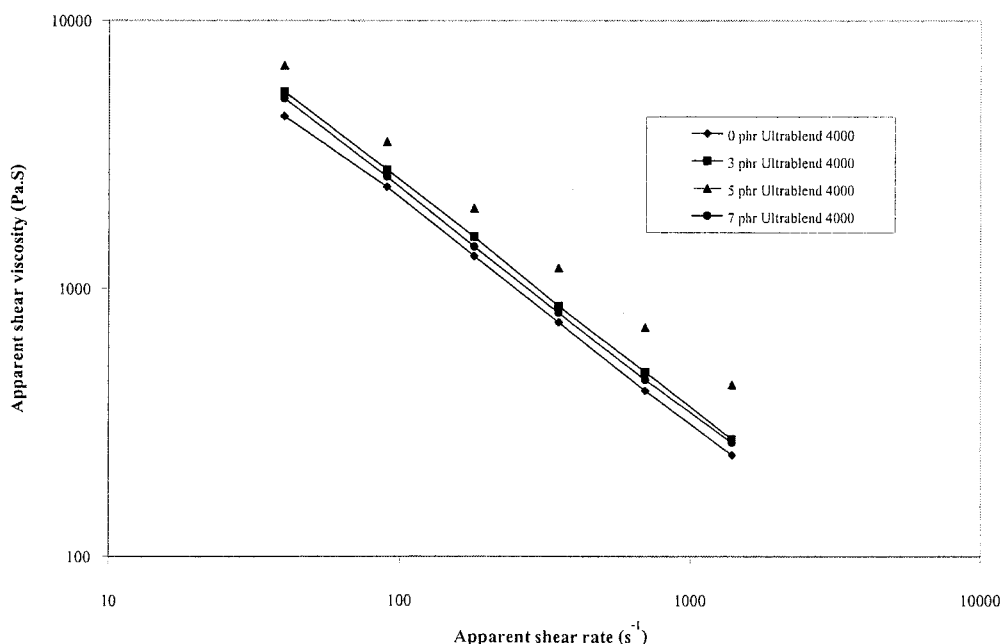


Figure 12 Relationship between apparent shear rate and apparent shear stress of (70 : 30) NR/EPDM blend with various amounts of the homogenizing agent.

homogenizing agent, the better adhesion between phases yields improved tensile properties.

Rheological characterization

In this section, the blend compatibility of NR and EPDM was studied through capillary and dynamic flow. The shear stress and shear viscosity of rubber blends were measured using a Rosand single bore capillary rheometer.

The compatibility of the blends can be determined by the positive or negative deviation of the measured viscosity calculated by the log additivity rule model [eq. (2)].¹⁰ It is used for the evaluation of the thermodynamic compatibility of rubber blends and the devi-

ations of the blend viscosities from ideal behavior. The log additivity rule is

$$\log(\eta_{blend}) = \sum_i x_i \log(\eta_i), \tag{2}$$

where η_{blend} and η_i are the shear viscosity of the blend and phase i , and x_i is the weight fraction of phase i . The results of a compatibility analysis by the log additivity rule model are shown in Table XI. Shear viscosity and shear rates are plotted against the content of the homogenizing agent (%Ultrablend 4000) for the 70 : 30 NR/EPDM blends as shown in Figure 11. A positive deviation of the blends was only found when around 5 phr of the homogenizing agent was added, which confirmed the blend compatibility of NR and EPDM. Linear flow curves from NR/EPDM blends with various amounts of the homogenizing agent are shown in Figure 12. Pseudoplastic behavior was observed in the flow of blends. That is, the apparent shear viscosity decreased with an increase in the apparent shear rate. Therefore, the shear stress produced became smaller when the rate of shear increased. This flow behavior indicates a pseudoplastic fluid or shear thinning behavior of the blends.¹⁰

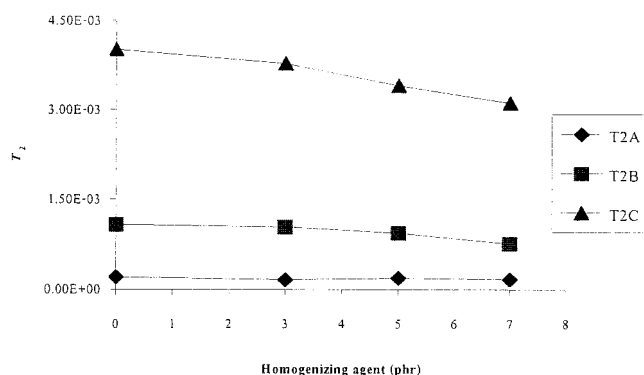


Figure 13 Temporal change of the T_2 value for NR/EPDM blends vs the homogenizing agent concentration.

Homogeneity of NR/EPDM blends by pulsed NMR

The phase separation of NR/EPDM blends was investigated by the pulsed NMR technique. After the measurement, the signals were analyzed by use of the

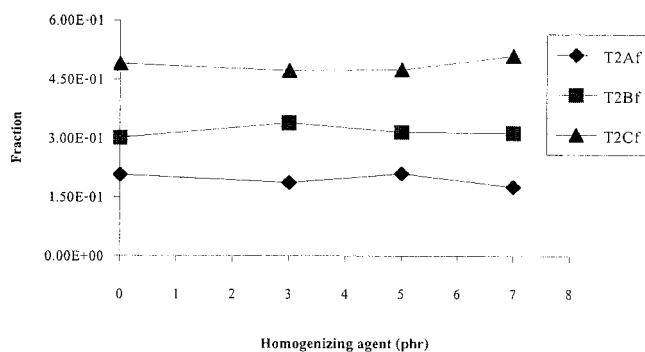


Figure 14 Fractions of NR/EPDM blends vs the homogenizing agent concentration.

nonlinear least-squares fitting method. The decay signal in the T_2 measurement is fitted by the summation of Gaussian, Weibull, and exponential functions.^{11,26} The temporal change of T_2 and fractional amounts of blend components are displayed in Figures 13 and 14. Three components with different T_2 values were observed, where T_{2A_f} and T_{2B_f} are related to the fraction of the EPDM component in the blends and T_{2C_f} is related to NR. The T_2 value is the spin-spin relaxation time, directly reflecting the mobility of molecules. We can obtain information on the degree of heterogeneity from the difference in mobility and temporal change of both fractional and T_2 values.

T_{2A} and T_{2B} are the temporal change of T_2 of the EPDM component in blends and T_{2C} is for NR. The degree of molecular motion is highest for T_{2C} (NR), followed by the T_{2B} (EPDM) and T_{2A} (homogenizer). There is a slight decrease in the three T_2 values for an increasing amount of the homogenizing agent (see Fig. 13). These results indicate that more homogenizing agent suppresses molecular mobility in all the three components.²⁶ We may anticipate that the amorphous region in the NR

matrix decreases, perhaps with some crystallization. The figures of fractional change of T_{2C} , T_{2A_f} and T_{2B_f} cannot clearly show the influences on filled and unfilled NR/EPDM due to the homogenizing agent. More investigation is required to clarify the effect of various amounts of the homogenizing agent, and it is necessary to apply this technique in combination with the results from other methods for determining the phase separation of the blends.

Effect of natural rubber content on ozone resistance

One of the objectives of this study is to find the proper ratio of NR/EPDM blends for ozone resistance. The blend ratios of NR/EPDM used in this study were 100/0, 80/20, 70/30, 60/40, and 0/100 (coded as NS, N8E2S, N7E3S, N6E4S, and ES, respectively). To improve the NR/EPDM blends for ozone resistance, the amount of EPDM cannot exceed 40% by weight. The results of vulcanizate characteristics and mechanical properties are shown in Table XII; as expected, the natural rubber specimens could not withstand the ozone gas, which is the nature of the NR polymer. One can see cracks in the vulcanized rubber after tests. When 20 phr of EPDM and higher concentrations were blended, the specimens could withstand the ozone gas. The specimens did not crack. The dispersed EPDM domains reduced the crack length and increased the critical energy for macroscopic cracks.²⁷ The EPDM domains function as crack dissipation centers, which delays the crack appearance. In the case of silica-filled blends, they enhance both mechanical properties (as reinforcement filler) and static ozone resistance.

Table XIII shows the result of ozone resistance of unfilled and silica-filled NR/EPDM blends, especially at the high NR content of 80 phr. The silica-filled

TABLE XII
Effect of NR Concentration on Properties of NR/EPDM Blends

Vulcanization characteristics and properties tested	NR/EPDM blend ratio				
	100/0	80/20	70/30	60/40	0/100
ODR at 423 K, t ₉₀ (min)	5.7	5.9	5.4	6.1	14.2
Mooney viscosity at 373 K, MU	88	68	71	70	101
Physical properties:					
Before aging					
Tensile strength (MPa)	28.9	19.4	13.9	8.8	16.6
Tear strength (N mm ⁻¹)	80	35.0	30.4	27.5	44.3
Hardness (Shore A)	57	53.6	54.5	53.6	59.7
After aging (at 373 K for 22 h)					
Tensile strength (MPa)	23.9	13.5	11.2	6.7	9.1
Tear strength (N mm ⁻¹)	65	37.4	34.2	28.0	52.2
Hardness (Shore A)	57.5	58.2	57.3	59	67.3
Ozone testing	-	+	+	+	+

The specimens cannot (-) or can (+) withstand the ozone gas of 50 ppm at 313 K for 72 h.

TABLE XIII
Effect Of Silica Filler on NR/EPDM Blends

Properties tested of vulcanizates	NR/EPDM blend ratio					
	Unfilled silica			Filled silica		
	80/20	70/30	60/40	80/20	70/30	60/40
Before aging						
Tensile strength (MPa)	17	14.7	8.9	19.4	13.9	8.8
Tear strength (N mm ⁻¹)	29.0	26.1	21.4	35.0	30.4	27.5
Hardness (Shore A)	41.4	43.1	42.2	53.6	54.5	53.6
After aging (at 373 K for 22 h)						
Tensile strength (MPa)	5.6	3.7	4.1	13.5	11.2	6.7
Tear strength (N mm ⁻¹)	22	16.8	17.1	37.4	34.2	28.0
Hardness (Shore A)	42.3	45.8	43.4	58.2	57.3	59
% Change in tensile strength after aging	67	75	53	30	20	23
Ozone testing	-	+	+	+	+	+

The specimens cannot (-) and can (+) withstand the ozone gas of 50 ppm at 313 K for 72 h.

80/20 (NR/EPDM) blend could withstand ozone gas of 50 ppm. In addition, other mechanical and physical properties of the silica-filled NR/EPDM blends at any blend ratio are overwhelmingly favorable compared with the unfilled blends. Losses in tensile strength after aging in unfilled blends were higher than 50%, which is a very good indicator that silica can also withstand heat deterioration.

CONCLUSIONS

The objectives of this research are to study compositional effects of NR/EPDM blends for coating on iron crossarms. The vulcanized blends must be highly insulating, and must withstand ozone and thermal aging to meet the suitable physical and mechanical properties for the TOT's network. The compositional effect of NR and EPDM was determined by investigating the effects of the carbon black concentration on electrical properties of rubber blends, of the homogenizing agent concentration on compatibility between NR/EPDM blends, and of the ratio of NR/EPDM on the extent of ozone resistance. The insulation coating must have a volume resistivity of more than 10¹¹ ohm cm. The 3 phr of carbon black was found to give a higher tensile strength and suitable volume resistivity.

Incompatibility between inorganic silica and organic rubber gives rise to aggregation of the silica particles in the rubber matrix, which often results in a poor dispersion of silica particles. Therefore, mechanical properties of the silica-filled rubber (NES30) are often inferior to those of carbon black-filled rubber (NEC3). We found that after 30 phr of silica was added, the mechanical properties of the blend after aging were greatly improved. The compatibility of NR/EPDM blends was improved when the homogenizing agent was added, as indicated by

partial miscibility by the log additive rule. The positive deviation of the blends was only found when 5 phr of the homogenizing agent (Ultrablend 4000) was added, which confirmed the blend compatibility of NR and EPDM. The phase dispersion of blends can be observed by SEM. Moreover, the pulsed NMR results indicated that the spin-spin relaxation times (T_2) of all three components of the rubber blend were reduced upon the addition of the homogenizing agent. The gradual increases in tensile strength and elongation at break of blends were observed when 3 and 5 phr of the homogenizing agent were added, respectively. On the contrary, mechanical properties were poor when 7 phr of the homogenizing agent was added. The 80 : 20 ratio of NR/EPDM blends filled with silica can withstand ozone gas at 50 ppm. For aging applications, inclusion of silica not only increased thermal and mechanical properties of the blends, but also improved the ozone resistance of NR/EPDM blends.

The proper composition of NR and EPDM, which possesses the requirements of high insulation, antiozone, and proper physical and mechanical properties suited for the TOT's network is as follows: Natural Rubber, 80 phr EPDM, 20 phr; Ultrablend 4000, 5 phr; ZnO, 5 phr; stearic acid, 15 phr; carbon black (N330), 3 phr; silica, 30 phr; PEG, 1 phr; EF44, 1 phr; MBTS, 0.3 phr; and sulfur, 2 phr.

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