

EPDM/CIIR Blends: Effect of EPDM Grade on Mechanical Properties

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Blends of chlorobutyl rubber (CIIR) with two grades of ethylene-propylene diene monomer rubber (EPDM) were prepared and the effect of blend ratio on the cure characteristics, hot air ageing resistance, steam ageing resistance, and mechanical properties were evaluated. The blend of CIIR with EPDM grade 301 T showed additive behavior and the blend with the other grade of EPDM (NDR 4640) showed synergistic behavior.

Keywords: ageing resistance, blends, CIIR, EPDM, mechanical properties

INTRODUCTION

Efforts to develop blends of elastomers for diverse applications have continued to burgeon for several decades. Elastomer blends are of great importance for industrial applications because of the great regulatable variability of their properties despite the limited number of initial components [1–3]. Although many immiscible systems have been successfully commercialized, the applicability of such blends is limited because of their inferior macroscopic properties. Hence, successful blending of incompatible elastomers is gaining more importance. Tires probably represent the largest use of rubber blends. In non-tire industries, blends have been introduced for critical applications. A blend can offer a set of properties that may give it the potential of entering application areas not possible with either of the

Received 6 October 2006; in final form 13 October 2006.

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polymers comprising the blend. By blending different polymers, it is possible to combine the properties of the individual components in a single material. The process of blending can be implemented rapidly and economically. This technique has helped to develop many new materials, which are of good quality and are cheaper in market. Several reviews on rubber-rubber blends are available [4–5]. Kim and Hamed prepared a vulcanizate based on a 50/50 natural rubber/cis-butadiene rubber [6]. This blend has been found to retain the rupture resistance property of NR vulcanizate and resistance to slow fatigue crack growth property of cis-BR vulcanizate. Botros et al. studied the properties of nitrile rubber (NBR)-chloroprene rubber (CR) blends [7]. The blend was found to possess the thermal resistance of CR and the oil resistance of NBR. Saha and Swapan studied the rheological and morphological characteristics of PVC/CR blends [8]. Experimental results showed that the CR can promote the processability of PVC. It was also found that the mixing sequence has a profound influence on the processability and compatibility of the melt blend system.

EPDM possess good physical properties, such as high heat resistance, ozone resistance, and cold and moisture resistance. Because of the low gas and moisture permeability, good weathering resistance and high thermal stability of CIIR, blends of EPDM with CIIR may be attractive, if they possess sufficient mechanical strength and ageing resistance. In this work, two grades of EPDM, that is, 301 T and NDR 4640, were blended separately with CIIR at different blend ratios. Cure properties of the blends were investigated. The physical properties such as tensile strength, tear strength, abrasion resistance, tension set, hardness, heat and steam ageing resistance, flex crack resistance, and rebound resilience were also evaluated.

EXPERIMENTAL

Materials

EPDM rubber with dicylopentadiene (301 T), Mooney viscosity [ML(1 + 8)@100°C] 47.8, ethylene/propylene weight ratio 68/32 and iodine value 10.5 was kindly supplied by Herdillia Unimers Limited. EPDM rubber with ethylidene norbornene (NDR 4640), Mooney viscosity [ML(1 + 8)@100°C] 53.4, ethylene/propylene weight ratio 55/45 and iodine value 16.0 was kindly supplied by Nodal Dupont. In this study hereafter the 301 T grade EPDM is referred as EPDM (A) and NDR 4640 grade EPDM is referred as EPDM (B). Chlorobutyl rubber (Exxon Chlorobutyl 1066) having Mooney viscosity [ML(1 + 8) 100°C] 56.3, iodine value 8.4 and chlorine content (%) 1.2, was

TABLE 1 Compounding Formulations for EPDM

Ingredients	Concentration (phr)
EPDM rubber	100
ZnO	4.0
Stearic acid	1.5
MBT	1.0
TMTD	0.5
ZDC	0.5
Carbon black	40
Paraffinic oil	7.0
Sulphur	1.0
Antioxidant	1.0

supplied by Exxon Chemical Company, Baton Rouge, USA. Rubber additives such as zinc oxide, stearic acid, carbon black (HAF-N330), paraffinic oil, dibenzthiazyl disulphide (MBTS), tetramethyl thiuram disulphide (TMTD), zinc diethyl dithiocarbamate (ZDC), 2-(4-morpholinyl mercaptobenzthiazole) (MOR), and sulphur used in the study were of commercial grade.

Compounding of Rubber

EPDM was thoroughly masticated on a laboratory size (16 × 33 cm) two-roll mixing mill at a friction ratio of 1:1.25 and was blended with CIIR at different percentages. The blends were compounded according to ASTM-D 3182 (1982) as per formulations given in Tables 1 and 2. After complete mixing the stock was passed six times through tight nip and finally sheeted out at a fixed nip gap. The samples were kept overnight for maturation.

TABLE 2 Compounding Formulations for CIIR

Ingredients	Concentration (phr)
CIIR rubber	100
ZnO	4.0
Stearic acid	1.5
MOR	1.0
Carbon black	40
Paraffinic oil	5.0
Sulphur	1.5
Antioxidant	1.0

Determination of Cure Time and Crosslink Density

Cure characteristics have been studied using a Rubber Process Analyzer (RPA 2000, Alpha Technologies) as per ASTM D 1646 (1981). The die type used was biconical and the die gap was 0.487. The optimum cure time of the samples was determined at 170°C at a frequency of 50.0 cpm and a strain of 0.20 deg. The total crosslink density was determined from the swelling data in toluene [9–11]. The individual crosslink densities of the EPDM and CIIR phases could not be measured because both have common true solvents [12]. The concentration of chemical crosslink was estimated from the equilibrium swelling data as follows. Samples of approximately 1 cm diameter, 0.20 cm thickness and 0.20 gm weight were punched out from the vulcanizate and allowed to swell in toluene. The swollen sample was taken out of the solvent after 48 h and weighed. Solvent was then removed in vacuum and the sample weighed again. The volume fraction of rubber (V_{ro}) in the swollen network was then calculated by the method reported by Ellis and Welding [13–14].

The crosslink density ($1/2 M_C$) was then determined using the Flory-Rehner equation [15]

$$\frac{1}{2} M_C = \frac{-[\ln(1 - V_{ro}) + V_{ro} + \chi V_{ro}^2]}{2\rho_r V_s (V_{ro})^{1/3}}$$

where V_s = molar volume of solvent and χ = parameter characteristic of interaction between rubber and solvent [16]. Values of interaction parameters taken for calculations are the following: for CIIR-toluene = 0.568, EPDM-toluene = 0.490 [9–10].

Compression Molding

Blanks cut from unvulcanized sheets marked with the machine direction were vulcanized at a temperature of $170 \pm 2^\circ\text{C}$ and at a pressure of 200 Kgcm^{-2} in an electrically heated hydraulic press (Santosh, SMP-50), to their respective optimum cure times. Rectangular moldings were cooled quickly in water at the end of each curing cycle and were used for subsequent property measurements.

Mechanical Properties

Dumbbell-Shaped tensile specimens were punched out from the vulcanized sheets and the mechanical properties were studied using a Universal Testing Machine (Shimadzu-AG1) with a load cell of 10 kN capacity, as per ASTM D 412-68. The gauge length between

the jaws at the start of each test was adjusted to 30 mm and the measurements were carried out at a cross-head speed of 500 mm/min. An average of at least six sample measurements was taken to represent each data point. Angular test pieces were punched out of the compression-molded sheets and tear resistance measured according to ASTM D 624. Samples for abrasion resistance were molded and volume loss/h was determined on a Zwick abrader according to DIN 53514. Hardness of the samples was measured and expressed in shore A units. Samples for tension set, hardness, flex crack resistance, and rebound resilience were molded and tested as per the relevant ASTM standards.

Ageing Resistance

The ageing resistance and tension set of the vulcanizates were determined after ageing the samples at 100°C for 24, 48, and 72 h in a laboratory air oven. The samples used were dumbbell-shaped tensile and angular tear specimens that were subsequently used for the property measurements to evaluate the effect of ageing on the mechanical properties. The steam ageing resistance of the vulcanizates was also studied using a steam chamber maintaining at a pressure of one atmosphere and temperature 100°C for the time intervals 6, 18, and 36 h.

RESULTS AND DISCUSSION

Cure Characteristics

The cure rates of the blends of EPDM (A) and EPDM (B) grades of EPDM with CIIR corresponding to various compositions are shown in Figure 1. When compared with EPDM (A)/CIIR blends, cure rate is higher for EPDM (B)/CIIR blends, which is probably due to the higher unsaturation of EPDM (B) as well as the presence of ethyldene norbornene (ENB) as the diene. In the case of EPDM (A), it is less unsaturated and the diene is dicylopentadiene (DCPD), which has the slowest cure rate among the termonomers used in EPDM. The cure rates of pure EPDMs and CIIR are almost in the same range. The cure rate decreased with increase in EPDM content in the blends up to blend ratio of 60% EPDM and then the cure rate increased. This may be attributed to a nonuniform distribution of curatives in the blend components [17,18]. Due to preferential migration of curatives from CIIR to EPDM owing to the low viscosity and more unsaturated nature of EPDM, blends containing higher percentage of CIIR require more time for attaining optimum crosslink densities [19]. Thus cure time increases, resulting in a decreased cure rate. For blends containing

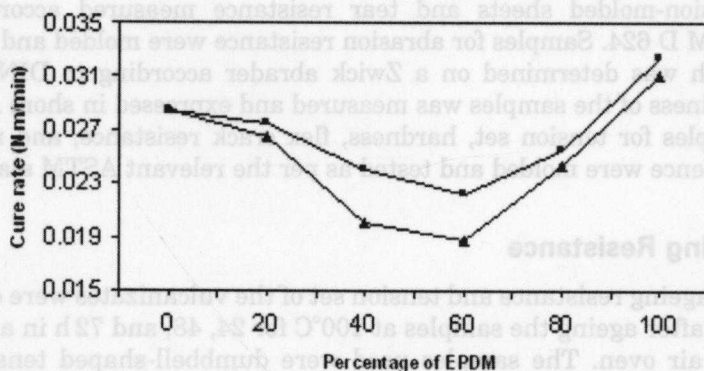


FIGURE 1 Cure rate vs. percentage of EPDM in blends. ■, EPDM (B)/CIIR blends and ▲, EPDM (A)/CIIR blends at 170°C.

higher percentage of EPDM, EPDM forms the continuous phase and the effect of preferential migration is not so significant [20–21].

Crosslink Density

Figure 2 shows the variation of the crosslink density of the vulcanizates. The additive variation of crosslink density is indicative of blend homogeneity. The total crosslink density increases with increase in EPDM content in the blends. Due to the more unsaturated nature of EPDM, the vulcanizable sites will produce a higher crosslink density. The more homogenous nature of EPDM (B)/CIIR blends results in higher crosslink density.

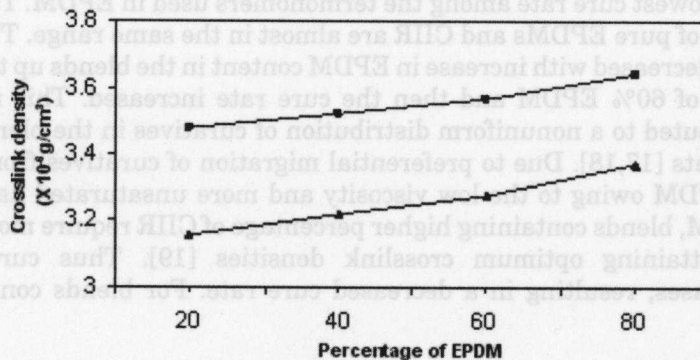


FIGURE 2 Variation of crosslink density with percentage of EPDM. ■, EPDM (B)/CIIR blends and ▲, EPDM (A)/CIIR blends.

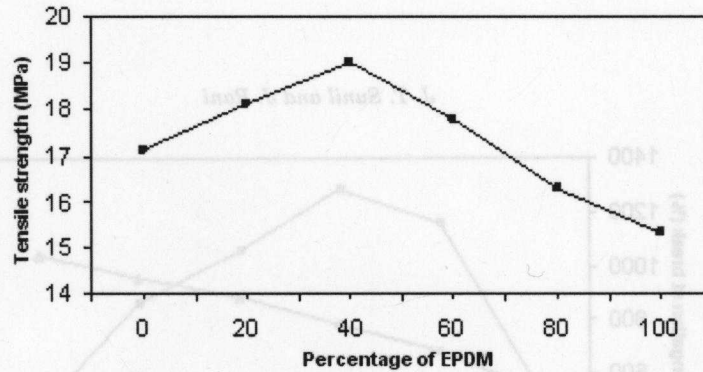


FIGURE 3 Variation of tensile strength with percentage of EPDM for EPDM (B)/CIIR blends.

Mechanical Properties

The variations in tensile strength and elongation at break with percentage of EPDM for the vulcanizates are shown in Figures 3, 4 and 5. The tensile strengths of the EPDM (A)/CIIR blends under unaged condition are close to the additive behavior while EPDM (B)/CIIR blends are exhibiting a synergistic behavior indicating that EPDM (B)/CIIR blends are more compatible than EPDM (A)/CIIR blends. Similar result is observed for elongation at break, too (Figure 5). The elongation at break is close to the arithmetic average for EPDM (A)/CIIR blends and synergistic behavior for EPDM

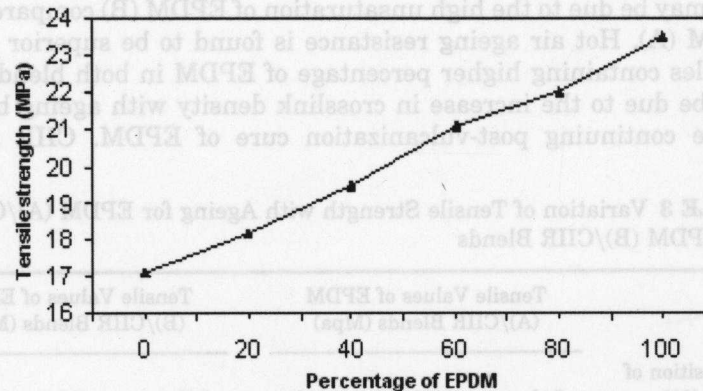


FIGURE 4 Variation of tensile strength with percentage of EPDM for EPDM (A)/CIIR blends.

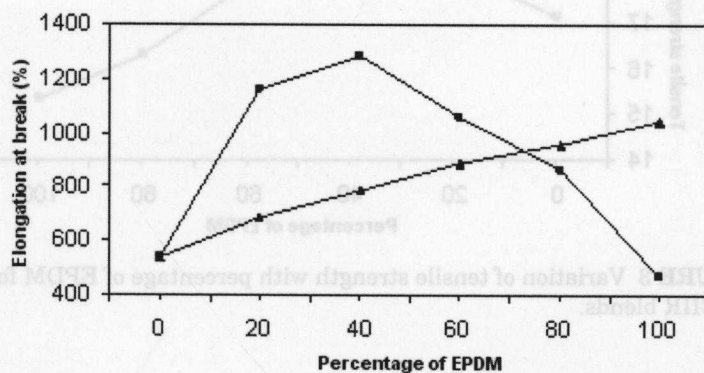


FIGURE 5 Variation of elongation at break with percentage of EPDM. ▲, EPDM (A)/CIIR blends and ■, EPDM (B)/CIIR blends.

(B)/CIIR blends. As the rate of vulcanization of the two phases are comparable, significant amount of interphase crosslinking may take place and this might be the reason for the better values of tensile strength and elongation at break [22].

The tensile strength and elongation at break of the vulcanizates under hot air ageing conditions are respectively shown in Tables 3 and 4. A drop in tensile strength and elongation at break are observed for EPDM (B)/CIIR blends compared with EPDM (A)/CIIR blends. This may be due to the high unsaturation of EPDM (B) compared with EPDM (A). Hot air ageing resistance is found to be superior for the samples containing higher percentage of EPDM in both blends. This may be due to the increase in crosslink density with ageing because of the continuing post-vulcanization cure of EPDM. CIIR attains

TABLE 3 Variation of Tensile Strength with Ageing for EPDM (A)/CIIR and EPDM (B)/CIIR Blends

Composition of EPDM (%)	Tensile Values of EPDM (A)/CIIR Blends (Mpa)			Tensile Values of EPDM (B)/CIIR Blends (Mpa)		
	24 h	48 h	72 h	24 h	48 h	72 h
100	24.4	23.2	21.8	14.2	13	11.6
80	22.8	21.7	20.5	14.8	13.4	12.1
60	21.4	20.6	19.8	15.6	14.8	13.7
40	19.1	18.2	17.2	18	16.8	14.8
20	17.2	16	14.6	16.5	15.2	13.6
0	15.9	14.5	12.9	15.9	14.5	12.9

TABLE 4 Variation of Elongation at Break with Ageing for EPDM (A)/CIIR and EPDM (B)/CIIR Blends

Composition of EPDM (%)	Elongation at Break Values of EPDM (A)/CIIR Blends (%)			Elongation at Break Values of EPDM (B)/CIIR Blends (%)		
	24 h	48 h	72 h	24 h	48 h	72 h
100	928	860	812	412	356	278
80	864	786	708	528	343	242
60	812	718	654	582	396	316
40	676	584	532	608	412	338
20	570	472	418	520	406	326
0	474	397	306	474	397	306

maximum torque faster than EPDM and then it reverts. As ageing continues, the tensile strength values decrease. The tensile values of the vulcanizates are found to depend on the proportions of poly-, di-, and mono-sulphidic linkages in addition to the crosslink density. A higher percentage of polysulphidic crosslinks is found to result in higher tensile strength. The decrease in values on continued ageing might be due to the decrease in the concentration of polysulphidic crosslinks [23–25]. The deterioration in the tensile strength of the vulcanizates with ageing may be principally due to main chain scission. The chain shortening of polysulphidic crosslinks may also be contributing to this effect [26–29].

Tables 5 and 6 represent the steam ageing resistance of EPDM (A)/CIIR and EPDM (B)/CIIR blends, respectively. As in the case of hot air ageing, the steam ageing resistance is also higher for blends

TABLE 5 Effect of Steam Ageing on Tensile Strength and Elongation at Break for EPDM (A)/CIIR Blends

Composition of EPDM (%)	Tensile Strength Values after Steam Ageing (Mpa)			Elongation at Break Values after Steam Ageing (%)		
	6 h	18 h	36 h	6 h	18 h	36 h
0	16.1	14.2	11.5	488	412	334
20	17.4	16.1	14.2	576	516	443
40	18.3	17.3	16.1	690	645	584
60	19.6	18.8	17.5	822	786	710
80	21.3	20.8	19.7	895	823	786
100	23.6	23.4	22.6	910	886	854

TABLE 6 Effect of Steam Ageing on Tensile Strength and Elongation at Break for EPDM (B)/CIIR Blends

Composition of EPDM (%)	Tensile Strength Values after Steam Ageing (Mpa)			Elongation at Break Values after Steam Ageing (%)		
	6h	18h	36h	6h	18h	36h
0	16.1	14.2	11.5	488	412	334
20	16.7	14.7	12.1	526	459	376
40	17.4	16.1	14.8	580	492	422
60	16.5	15.2	14.4	485	426	368
80	14.5	13.8	12.7	432	362	302
100	13.8	12.6	11.7	388	331	314

with higher percentage of EPDM, probably due to the post vulcanization reaction strengthening the network. Among the two grades of EPDM, EPDM (A)/CIIR blend is exhibiting better retention in properties owing to its more saturated nature. A comparison of tensile strength values of vulcanizates of varying compositions subjected to hot air ageing and steam ageing shows that a drastic loss in property is happened during steam ageing and the loss was high for 100% CIIR and blends containing higher percentage of CIIR. The comparative result points to the fact that dehydrohalogenation, which is the main problem for halogenated butyl compounds, is more severe during steam ageing than under thermal ageing conditions.

Tear strength is an important property that contributes toward resistance to crack growth. It can be seen from Table 7 that the variation of tear strength with the addition of EPDM is very similar to that observed in the case of tensile strength. That is, tear strength

TABLE 7 Variation of Tear Strength with Blend Composition for EPDM (A)/CIIR and EPDM (B)/CIIR Blends

Composition of EPDM (%)	Tear Strength Values of EPDM (A)/CIIR Blends (N/mm)				Tear Strength Values of EPDM (B)/CIIR Blends (N/mm)			
	Unaged	24h	48h	72h	Unaged	24h	48h	72h
0	31.8	28.6	24.1	21.2	31.8	28.6	24.1	21.2
20	33.4	30.1	28.5	24.8	32.4	29.4	25.7	22.0
40	37.0	33.6	30.7	28.0	34.0	31.8	29.4	23.6
60	41.2	38.8	35.0	32.1	33.2	31.1	29.0	25.9
80	43.4	41.4	40.8	37.2	32.1	30.2	28.7	26.7
100	46.7	45.8	43.6	40.8	31.3	29.6	28.8	27.3

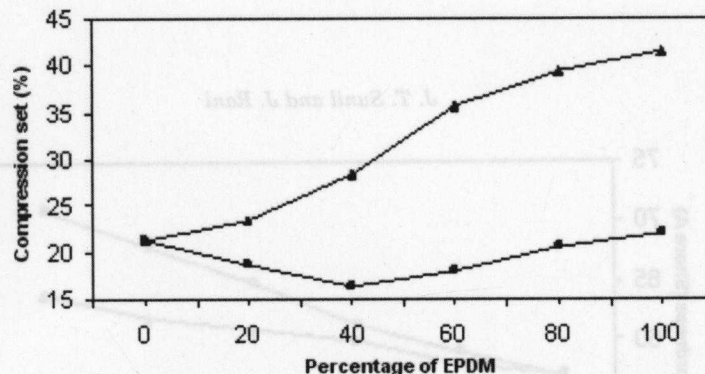


FIGURE 6 Variation of compression set with percentage of EPDM. ■, EPDM (B)/CIIR blends and ▲, EPDM (A)/CIIR blends.

increases gradually with an increase in percentage of EPDM in the vulcanizate. This observation is associated with the ability of EPDM molecules to transfer the tearing force, and also probably because of the optimum crosslink densities in the rubber phases and at the inter-phase. The variation of tear strength followed an additive behavior that points toward partial miscibility. Upon ageing, the tear strength values decrease primarily due to the breakage of crosslinks.

Compression set and tension set values depend strongly on the elastic recovery of the sample. Figure 6 represents the variation of compression set properties and Table 8 represents the variation of tension set properties with blend composition for EPDM (A)/CIIR and EPDM (B)/CIIR blends. In general, the compression set and tension set values decrease with increase in CIIR content in the

TABLE 8 Variation of Tension Set for EPDM (B)/CIIR and EPDM (A)/CIIR Blends

Composition of EPDM (%)	Tension Set Values for EPDM (A)/CIIR Blends (%)			Tension Set Values EPDM (B)/CIIR Blends (%)		
	24 h	48 h	72 h	24 h	48 h	72 h
0	—	—	—	—	—	—
20	76	—	—	97.5	—	—
40	102	—	—	117	—	—
60	116	125	—	128	—	—
80	123	131	144	136	—	—
100	130	136	141	141	158	—

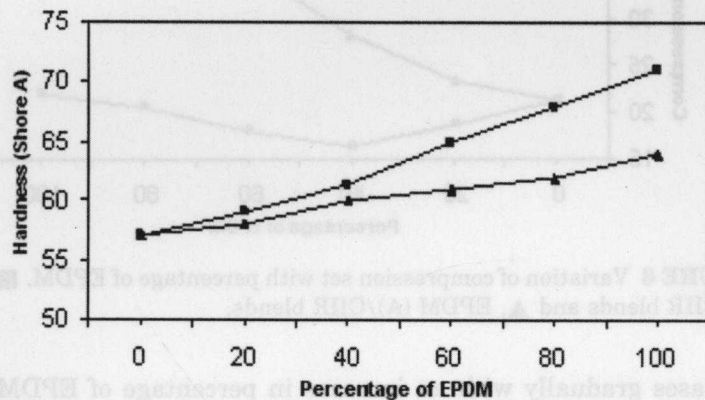


FIGURE 7 Variation of hardness with percentage of EPDM. ▲, EPDM (A)/CIIR blends and ■, EPDM (B)/CIIR blends.

blends. This is due to the more elastic nature of CIIR so that even after stress-relaxation process, there is no significant change in dimensions resulting in lower set values. When compared with EPDM (A)/CIIR blends, the compression set values are better for EPDM (B)/CIIR blends due to its more compatible nature, but tension set values show a drastic fall probably due to the highly unsaturated nature of EPDM (B).

Figure 7 shows the variation of hardness with increasing percentage of EPDM. It is evident from the figure that the rubber vulcanizates become stiffer and harder as the EPDM content increases. This result is expected because, as more EPDM get into the blend matrix, the elasticity of the rubber chains is reduced, resulting in more rigid vulcanizates. Hardness values are higher for EPDM (B)/CIIR blends, probably due to their high crosslink density.

The results from Figure 8 reveal that EPDM as well as the samples containing higher percentage of EPDM exhibit higher abrasion resistance. It has been reported that crosslink density, hardness, and modulus of the vulcanizate are important factors controlling the abrasion resistance [30–34]. The increased crosslink density that results in increased hardness and modulus ultimately gives rise to the enhancement of abrasion resistance. On the contrary, the lower the friction coefficient, the higher the abrasion resistance. From the results obtained in this study, the greater abrasion resistance of EPDM (B)/CIIR blend vulcanizates when compared with EPDM (A)/CIIR blends can be attributed to their higher crosslink density.

Figure 9 shows the variation of rebound resilience with percentage of EPDM. The rebound resilience decreases gradually with increase in

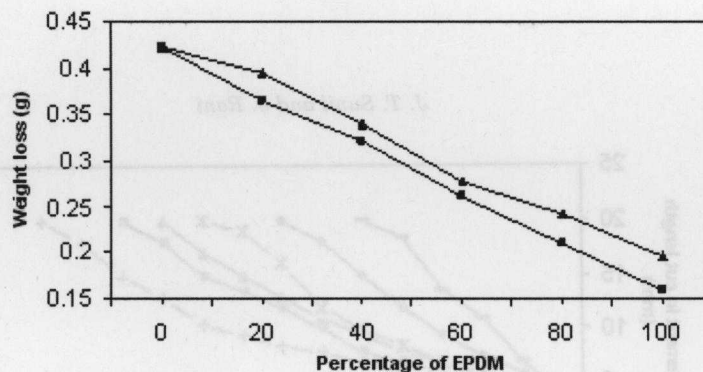


FIGURE 8 Effect of EPDM content on abrasion resistance. ▲, EPDM (A)/CIIR blends and ■, EPDM (B)/CIIR blends.

chlorobutyl content in the blends. The reason can be explained on the basis of morphological features and high strain energy density of chlorobutyl rubber.

During service, the rubber articles pass through repeated stressing conditions. It is desired that under these severe conditions the crack propagation should be minimum or negligible to have a better service life. Increment in cut length against number of flexing cycles is shown in Figures 10 and 11 for EPDM (A)/CIIR blends and EPDM (B)/CIIR blends, respectively. The samples were tested at room temperature. It has been observed that, with the increase in chlorobutyl rubber content in the blends, the rate of crack growth decreased, which is

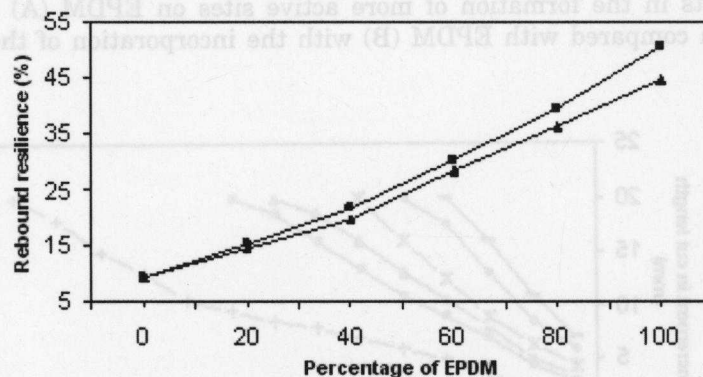


FIGURE 9 Variation of rebound resilience with percentage of EPDM. ▲, EPDM (A)/CIIR blends and ■, EPDM (B)/CIIR blends.

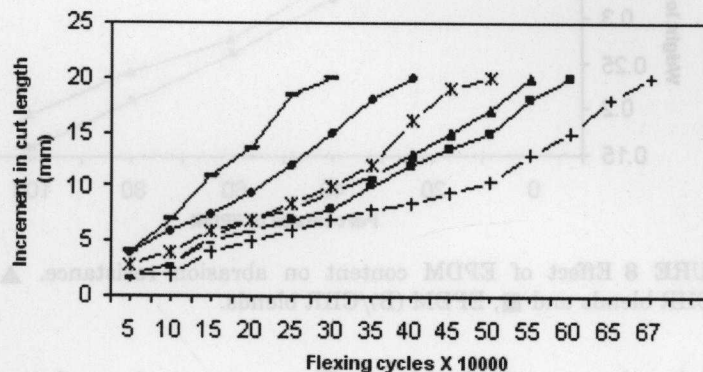


FIGURE 10 Plot of increment in cut length against flexing cycles for EPDM (A)/CIIR blends at a precut of 2.0 mm. ○, 100/0; ◆, 80/20; ×, 60/40; ▲, 40/60; ■, 20/80; +, 0/100.

due to the inherently superior viscoelastic nature of CIIR. This is valid for both EPDM (A)/CIIR and EPDM (B)/CIIR blends. The resistance for cut growth is better for EPDM (A)/CIIR blends. This may be due to different extent of filler distribution in the two grades of EPDM. It is expected that reinforcing fillers like carbon black will distribute more in the EPDM phase for the case of EPDM (A)/CIIR as compared with EPDM (B)/CIIR, due to the low viscosity of EPDM (A) [35]. This results in the formation of more active sites on EPDM (A) surface when compared with EPDM (B) with the incorporation of the filler.

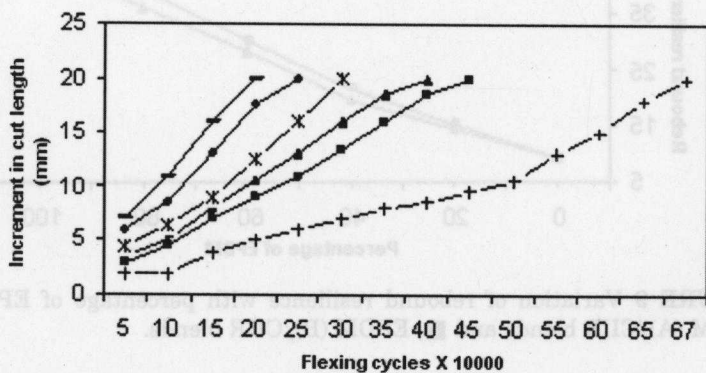


FIGURE 11 Plot of increment in cut length against flexing cycles for EPDM (B)/CIIR blends at a precut of 2.0 mm. ○, 100/0; ◆, 80/20; ×, 60/40; ▲, 40/60; ■, 20/80; +, 0/100.

When the two surfaces are brought into molecular contact, adsorption takes place in addition to the bonds formed by crosslinking. These bonds can absorb high stress before rupture leading to better flex crack resistance for EPDM (A)/CIIR blends.

CONCLUSIONS

The sulphur cured EPDM/CIIR vulcanizates at varying blend compositions were found to exhibit good physical properties at all compositions. The properties are found to be an average of the component elastomers. Although better mechanical properties are exhibited by NDR/CIIR blends, they show a drastic drop under severe conditions of thermal and steam ageing. On the other hand, resilience, flex crack resistance, and ageing resistances are better for 301 T/CIIR blends, which is probably due to the more saturated nature of 301 T. Based on the physical property studies, 301 T/CIIR blends can be used for high-temperature applications such as for use in products like curing envelopes, conveyor belts, and so on.

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