



Compatibility Studies on Sulphur Cured EPDM/CIIR Blends

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ABSTRACT

The principal objective of this study was to explore the compatibility of a blend of two synthetic elastomers viz., ethylene-propylene-diene rubber (EPDM) and chlorobutyl rubber (CIIR). Various commercial grades of EPDM were blended with a specific grade of CIIR at different proportions. The mechanical properties such as tensile strength, tear strength, ageing resistance, etc. were studied. On the basis of the observed physical properties, two particular grades of EPDM were found to be compatible with CIIR. Differential scanning calorimetry and scanning electron microscopy confirmed the results. Chlorosulphonated polyethylene was added as a compatibilizing agent to overcome the phase separation of the other two incompatible grades of EPDM in blending with CIIR. The results revealed that the addition of compatibilizer greatly improves the compatibility and thereby the properties of the blends.

Key Words:

EPDM;
chlorobutyl rubber;
blend;
curing;
compatibilizer.

INTRODUCTION

Blending of two or more elastomers is carried out for specific objectives such as enhancement of technical properties, improvement of ageing resistance, processing characteristics, etc. In the case of miscible blends, the overall physico-mechanical properties depend on two structural parameters: (a)

proper interfacial tension which leads to a phase of sufficiently small size to allow the material to be considered macroscopically homogeneous, and (b) an inter-phase adhesion strong enough to assimilate stresses and strains without disruption of the established morphology. Unfortunately, this is

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not the case for most of the polymer blends where the components are found to be immiscible and incompatible resulting in poor mechanical properties [1-6]. Several attempts have been made to minimize phase separation and to increase interfacial adhesion [7-13]. These include the addition of a compatibilizing agent such as a third polymer, which is, a graft or block copolymer that reduces the interfacial tension between the two phases, thereby, develops satisfactory network structure in each of the phases as well as the interphase. Consequently, compatibility is a fundamental property in polymer blends, deciding their practical utility.

Although a lot of work has been done on elastomer blends, studies on the blends of ethylene-propylenediene rubber (EPDM) and chlorobutyl rubber (CIIR) are meager. In comparison, EPDM possesses better general physical properties such as high heat resistance, ozone, cold temperature, and moisture resistances than CIIR. Because of the low gas and moisture permeability, good weathering resistance, and high thermal stability of CIIR, its blends with EPDM would be attractive, provided they are compatible. These blends may find application in the manufacture of curing envelopes/bladders and other high temperature products. Hence the main objective of this work is to explore the compatibility of EPDM blends with CIIR. Initially, four commercial grades of EPDM were blended with a selected grade of CIIR at various proportions. Cure characteristics and mechanical properties such as tensile strength and tear strength of these blends were studied. In addition to the physical property measurements, the extent of compatibility of these rubber blends was also studied with different methods such as glass transition measurements using differential scanning calorimetry (DSC) and dynamic mechanical analysis (DMA) and morphological characterization using scanning electron microscopy (SEM) [14-18].

Due to the differences in viscosity and extent of unsaturation of EPDM and CIIR, the amount of curatives may not be optimum in the two rubbers resulting to inferior mechanical properties [19-21]. It is now well established that the phase morphology of immiscible polymer blends can be controlled by the addition of compatibilizers [22-24]. The effect of chlorosulphonated polyethylene (CSPE) as a compatibilizer

in phase separated EPDM/CIIR blends is also investigated.

EXPERIMENTAL

Materials

EPDM rubber with dicyclopentadiene as a diene (301-T, Mooney viscosity 47.8 [ML(1+8) at 100°C], ethylene/propylene weight ratio 68/32 and iodine value 10.5) was supplied by Herdillia Unimers Limited, EPDM rubber with ethylidene norbornene as a diene (NDR-4640, Mooney viscosity 53.4 [ML(1+8) at 100°C], ethylene/propylene weight ratio 55/45 and iodine value 16.0) was purchased from Nodal Dupont, EPDM rubber with ethylidene norbornene as a diene (MRD-10, Mooney viscosity 14 [ML(1+8) at 100°C], ethylene/propylene weight ratio 64/36 and iodine value 7.2) was supplied by Uniroyal Inc., EPDM rubber with ethylidene norbornene as another diene (EP-96, Mooney viscosity 74 [ML(1+8) at 100°C], ethylene/propylene weight ratio 63/27 and iodine value 19.0) was purchased from Japan EPR Co., Chlorobutyl rubber (Exxon 1066 having Mooney viscosity 56.3 [ML(1+8) at 100°C], iodine value 8.4 and chlorine content 1.2%) was supplied by Exxon Chemical Co. and chlorosulphonated polyethylene with specific gravity 1.12, sulphur content 1.4 % and chlorine content 26% was supplied by Aldrich. Compounding additives such as zinc oxide, stearic acid, carbon black (HAF-N330), paraffinic oil, mercapto benzthiazole (MBT), tetramethyl thiuram disulphide (TMTD), zinc diethyl dithiocarbamate (ZDC), *N*-cyclohexyl-2-benzthiazyl sulphenamide (CBS) and sulphur used in the study were of commercial grades.

Preparation of EPDM/CIIR Blends

EPDM was thoroughly masticated on a laboratory size two-roll mixing mill (Santosh, model SMX lab 613, India) at a friction ratio of 1:1.25 and was then blended with CIIR at different compositions. The blends were compounded according to ASTM-D 3182 (1982) as per the formulations given in Tables 1 and 2 (Table 1 represents the recipe for EPDM and Table 2, the recipe for CIIR). The samples were kept overnight for maturation. Cure characteristics were investigated

Table 1. Compounding recipe 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

using a rubber process analyzer (RPA 2000, Alpha Technologies, USA). 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 degree. The compounds were vulcanized up to their respective optimum cure times in an electrically heated laboratory hydraulic press (Santosh, model SMP-50, India) at 170°C and 200 kg cm⁻² pressure.

Mechanical Properties

Dumb-bell shaped tensile specimens and angular tear specimens were punched out from the vulcanized sheets and the mechanical properties were studied using a Shimadzu Universal testing machine (model- AG1, Japan) with a load cell of 10 kN capacity. The measurements were carried out according to the relevant ASTM standards at a crosshead speed of 500 mm/min.

Table 2. Compounding recipe for CIIR.

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

Thermal Properties

The glass transition temperature of pure elastomers and their blends was determined by differential scanning calorimetry (DSC Q-100, TA Instruments, USA) and dynamic mechanical analyzer (DMA Q-800, TA Instruments, USA). For DSC analysis, each sample with a weight of about 20 mg was sealed in standard aluminium pan, equilibrated at -80°C, and then heated to 20°C at a constant rate of 10°C/min in nitrogen atmosphere. This allowed the identification of the glass transition temperatures (T_g)s of CIIR, EPDM, and their blends. Specimens for DMA were punched out from the compression moulded sheets and the measurements were carried out in bending mode at a dynamic strain of 4% and at a frequency of 1 Hz. The temperature was increased at a constant rate of 3°C min⁻¹ from -90 to 20°C.

Morphological Studies

The microstructure of tensile fracture surfaces of the blends at varying compositions was studied using a scanning electron microscope (Jeol JSM 35C, Japan). The fracture surfaces of the test specimens were carefully cut from the test pieces and were then sputter coated with gold before they were examined through the SEM. The acceleration voltage used for image acquisition was 15 kV.

Effect of Compatibilizer

In order to improve the properties of the blends of EP-96 and MRD-10 grades of EPDM with CIIR, a compatibilizer such as chlorosulphonated polyethylene (CSPE) was added with varying proportions. The compatibilization effect was studied by following the improvements in mechanical properties (such as tensile strength, tear strength, etc.) as well as fracture surface morphology.

RESULTS AND DISCUSSION

Cure Characteristics

Cure characteristics of EPDM/CIIR blends are given in Table 3. The optimum cure time of the blends of 301-T and NDR-4640 with CIIR increases as the EPDM content of the blend increases. However, beyond 60% EPDM in the blend, the trend reverses.

Table 3. Cure characteristics of EPDM/CIIR blends.

Elastomer specification	Blend percentage of EPDM	Cure time (min)	Scorch time (min)	Maximum torque (Nm)	Minimum torque (Nm)
EPDM - 301 T	20	12.2	1.0	0.339	0.042
	40	14.6	1.3	0.302	0.036
	60	18.8	1.4	0.335	0.033
	80	13.0	1.2	0.324	0.037
	100	11.0	1.2	0.346	0.041
EPDM-NDR 4640	20	11.6	1.2	0.324	0.038
	40	14.4	1.3	0.329	0.039
	60	15.6	1.3	0.332	0.038
	80	12.4	1.2	0.327	0.041
	100	10.2	1.2	0.334	0.041
EPDM-EP 96	20	9.8	1.0	0.293	0.038
	40	9.1	1.0	0.287	0.045
	60	11.6	1.2	0.307	0.033
	80	12.4	1.4	0.287	0.038
	100	8.6	1.1	0.286	0.036
EPDM-MRD 10	20	13.4	1.4	0.336	0.035
	40	12.9	1.1	0.317	0.045
	60	16.9	1.4	0.333	0.046
	80	15.2	1.5	0.325	0.033
	100	18.1	1.7	0.339	0.031
CIIR	0	11.8	1.2	0.349	0.046

This may be due to the unequal distribution of ingredients in the blend components [20,21]. In the blends containing up to 60% EPDM, it can be assumed that preferential curative migration occurs towards the EPDM phase. Thus, the formation of greater numbers of interface cross-links is prolonged. As the EPDM content increases further, it forms the continuous phase and the effect of preferential migration is not very significant. The homogeneous distribution of curatives enhances the extent of cross-linking thereby causing a decrease in the optimum cure time. For the EP-96 and MRD-10 grades of EPDM blends with CIIR there is not any regular variation in optimum cure time which is an indication of inhomogeneity of these systems.

Mechanical Properties

Figure 1 shows the variation in tensile strength of the blends of different grades of EPDM with CIIR in var-

ious proportions. It is evident that the tensile strength is manifested as a synergistic behaviour for the grade NDR-4640 and an additive behaviour for 301-T. Hence these two grades of EPDM are considered as compatible with CIIR. As it is known that compatible

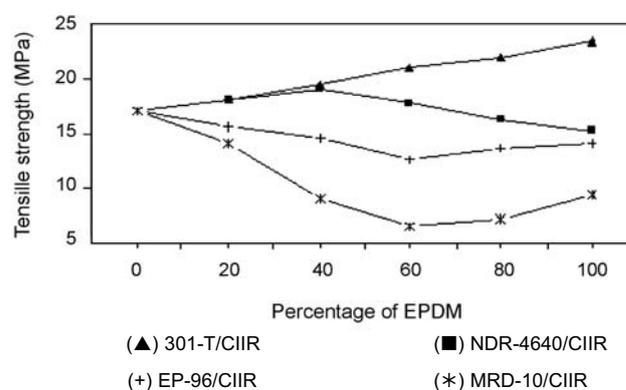


Figure 1. Variation of tensile strength of EPDM/CIIR blends vs. percentages of different grades of EPDM.

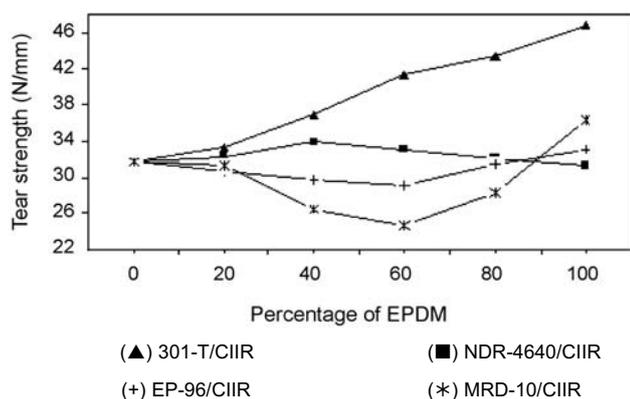


Figure 2. Variation of tear strength of EPDM/CIIR blends vs. percentages of different grades of EPDM.

systems exhibit mechanical properties as a function of blend composition that are at least a weight average of the values corresponding to the two components [25]. Better co-curing might be taking place in the case of NDR-4640/CIIR blends as the viscosity of NDR-4640 is more comparable with CIIR leading to better molecular packing and resulting in synergistic behaviour of mechanical properties. The viscosity of 301-T is slightly lower than that of CIIR, therefore, a perfect co-vulcanization probably is not taking place, which results in additive like behaviour in mechanical properties. For the remaining two different grades (EP-96 and MRD-10) the tensile properties show non-uniformity with respect to composition, in other words a non-additive behaviour is observed. Hence these are considered incompatible with CIIR [26,27]. Since both EPDM and CIIR are non-polar rubbers, the main reason for the incompatibility of these grades might be the viscosity mismatch, which results in curative migration, thus co-curing of the two phases of the blend is not taking place. Based on these observations, it is possible to correlate tensile strength versus composition plots with blend homogeneity.

Figure 2 shows the variation of tear strength of the blends of different grades of EPDM with CIIR at various proportions. It is evident that tear resistance is the maximum for blends of CIIR with 301-T and NDR-4640 grades. This also confirms a rather increased compatibility between the two phases of the blends of these two grades. For the EPDM grades, EP-96 and MRD-10, a gradual variation in tear properties is not observed indicating their non-compatibility with CIIR.

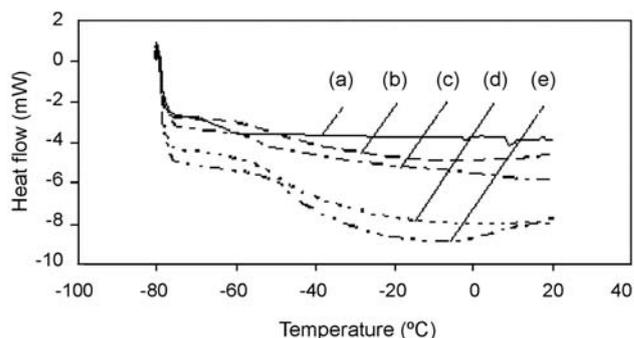


Figure 3. DSC Plots of various grades of EPDM and CIIR: (a) CIIR, (b) EP-96, (c) MRD-10, (d) 301-T, and (e) NDR-4640.

Thermal Analysis

Figure 3 shows DSC thermograms of four grades of EPDM and CIIR and Figure 4 shows the DSC thermograms of their 50/50 blends. For the 50/50 EPDM/CIIR blends, there is only a single T_g which indicates molecular level homogeneity. Although the occurrence of a single T_g may be a definitive test of compatibility of a system, there are certain circumstances, however, under which the T_g criterion may be inapplicable or misleading. One such criterion requires the T_g s of the components to be sufficiently displaced by each component therefore, resolution would take place. The resolution of T_g s of the components under roughly 20°C apart would be poor [28,29]. For all the 50/50 EPDM/CIIR blends, the differences between the components' T_g s lie below 15°C and therefore they appear as a single transition irrespective of the fact that whether they are compatible or not. Also, if there is significant degree of intermixing of both components in the interfacial regions, the

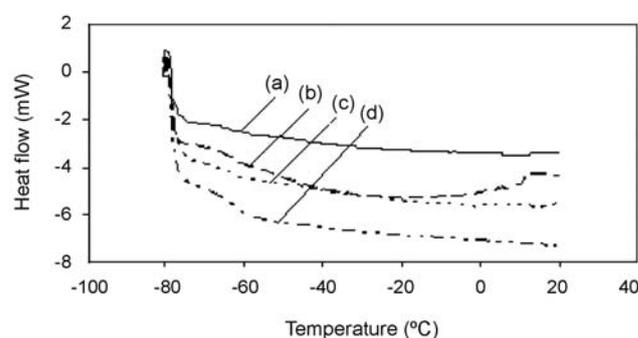


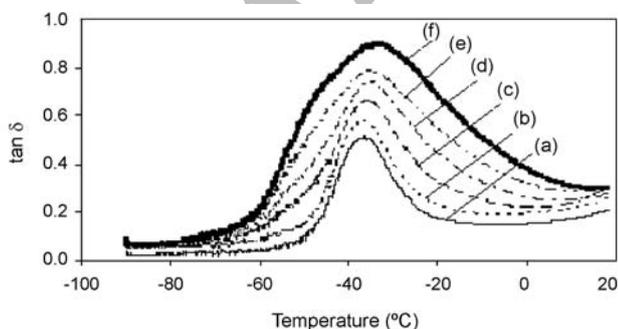
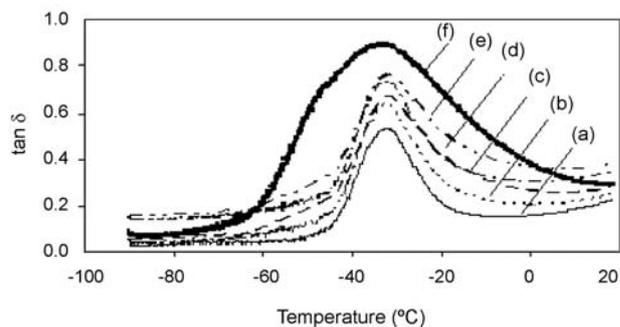
Figure 4. DSC Plots of 50/50 blends of various grades of EPDM and CIIR: (a) 301-T/CIIR, (b) NDR-4640/CIIR, (c) EP-96/CIIR, and (d) MRD-10/CIIR.

Table 4. T_g values of pure elastomers and their 50/50 blends.

Elastomers	T_g (°C)	50/50 blends of EPDM/CIIR	T_g (°C)
CIIR	-61.8	301-T/CIIR	-58.8
301-T	-54.4	NDR-4640/CIIR	-56.9
NDR-4640	-49.4	EP-96/CIIR	-60.8
EP-96	-58.1	MRD-10/CIIR	-58.4
MRD-10	-54.4	-	-

individual transitions are significantly reduced in intensity resulting in a single transition [30]. This may be due to the molecular migration from well defined phases into broad variable composition of interfacial regions, whose detection, because of the broad dispersion of the transition, may not be evident. The glass transition temperatures obtained for pure elastomers and their 50/50 blends are given in Table 4.

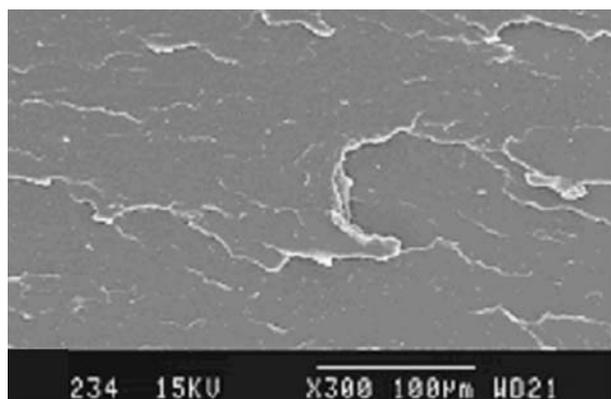
Figures 5 and 6 show the temperature dependence of the loss tangent ($\tan \delta$) at 1 Hz for EPDM, CIIR and their blends. The loss tangent, which indicates the damping ability of the material, is the ratio of the mechanical dissipation energy to the storage energy. Thus, a high $\tan \delta$ value is essential for good damping materials. CIIR shows a broad $\tan \delta$ peak, in which the efficient damping ($\tan \delta > 0.5$) has a wide temperature range. The different values of $\tan \delta$ maximum in EPDM/CIIR blends may be attributed to the dissimilar interactions between the chains of EPDM and CIIR. As shown in the figures, the damping efficiency decreases with increase in EPDM content in the blends. The peak area under the $\tan \delta$ -temperature curves can be considered as the damping index, which

**Figure 5.** Variation of $\tan \delta$ with temperature for 301-T grade EPDM, CIIR and their blends EPDM/CIIR: (a) 100/0, (b) 80/20, (c) 60/40, (d) 40/60, (e) 20/80, and (f) 0/100.**Figure 6.** Variation of $\tan \delta$ with temperature for NDR-4640 grade EPDM, CIIR and their blends EPDM/CIIR: (a) 100/0, (b) 80/20, (c) 60/40, (d) 40/60, (e) 20/80, and (f) 0/100.

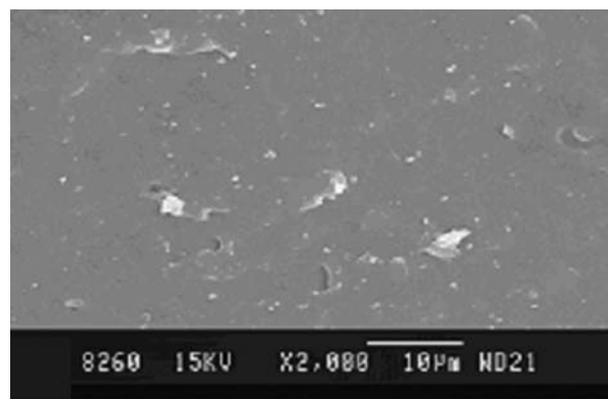
is a measure of the energy dissipation during a transition process [31]. These values are also higher for CIIR and decreases with decrease in the chlorobutyl content in the blends. In estimations of dynamic mechanical properties over a temperature range, the appearance of intermediate $\tan \delta$ peaks and the concomitant decrease of the $\tan \delta$ peaks of the individual polymers in a blend is a useful criterion of blend compatibility. It can be seen from the figures that blending of EPDM with chlorobutyl causes a shift in the position of T_g . Further more, increasing the CIIR content in the blend gradually causes a corresponding continuous shift in the position of T_g and a change in the half peak width. The presence of single $\tan \delta$ peak coupled with the intermediate $\tan \delta$ value of the blends points towards the compatibility between EPDM and CIIR [32]. Similar observations are obtained with respect to storage modulus and loss modulus confirming compatibility between the two elastomers.

Morphological Characterization

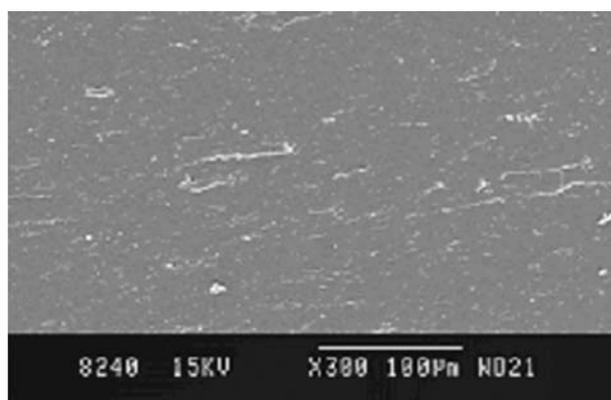
Figures 7-10 show the SEM images of the fractured surfaces of blends of CIIR with various grades of EPDM at selected proportions. The tensile fractured surfaces of 301-T/CIIR and NDR-4640/CIIR blends show smoother and more homogeneous patterns, justifying their higher tensile strength and other mechanical properties. This compatible morphological behaviour may be attributed to their comparable viscosities and intermediary surface tensions. The micrographs of EP-96/CIIR and MRD-10/CIIR blends confirm two phases with irregular domain sizes and shapes. The



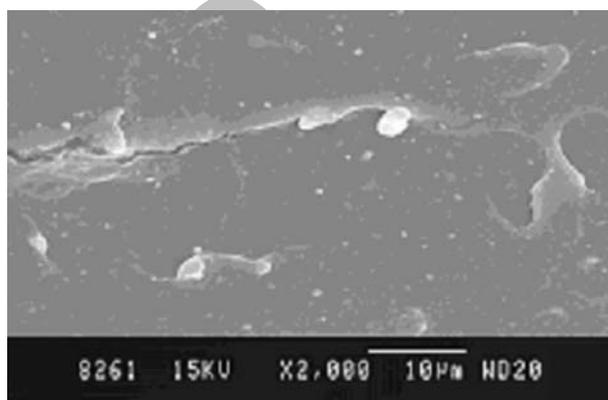
(a)



(a)



(b)



(b)

Figure 7. SEM Images of 301-T/CIIR: (a) 40/60 and (b) 60/40 blends.

gross-phase separated morphology of these blends indicates that these blends are totally immiscible, as large EPDM domains being dispersed in CIIR matrix [33].

Effect of Compatibilizer

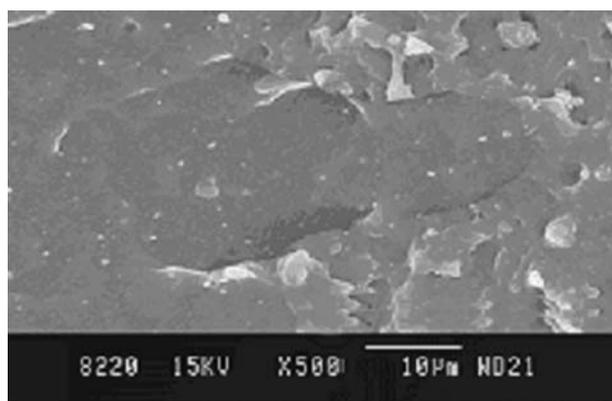
The variation of mechanical properties with varying dosage of chlorosulphonated polyethylene (CSPE) in 50/50 EPDM/CIIR blends is presented in Table 5. It is evident that the compatibilizer has not any significant

Figure 8. SEM Images of NDR-4640/CIIR: (a) 40/60 and (b) 60/40 blends.

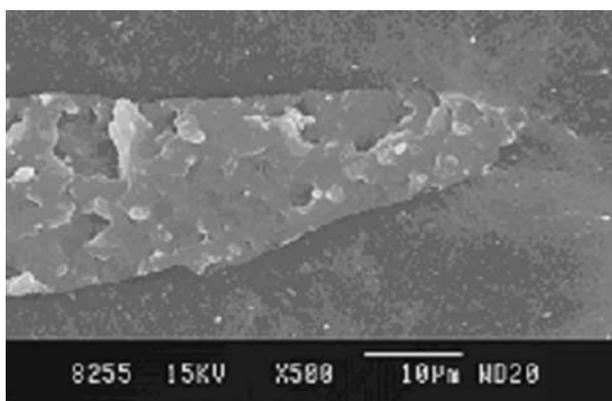
effect on MRD-10/CIIR blends. In the compatibilized blends of EP-96, tensile strength increases with increase in concentration of compatibilizer, up to a concentration of 10 phr, whereas it drops beyond this concentration. This may be due to the super-saturation of the interface with the compatibilizer, which increases the interfacial tension. Moreover, with increased concentration, the uniformly distributed domains may tend to agglomerate forming bigger aggregates. The mechanical properties, especially

Table 5. Physical properties of EPDM/CIIR blends containing varying amounts of chlorosulphonated polyethylene.

Blend ratio and tests	50/50 EP-96/CIIR blends				50/50 MRD-10/CIIR blends			
	0	5	10	15	0	5	10	15
Chlorosulfonated polyethylene (phr)	0	5	10	15	0	5	10	15
Tensile strength (MPa)	13.8	14.7	15.3	14.5	8	8.4	7.8	6.2
Tear strength (N/mm)	29.4	31.8	33.4	32.1	25.8	27.1	26	22.3
Modulus (at 300%) (MPa)	6.6	7.2	7.9	6.8	4.7	5.2	4.8	3.2
Elongation-at-break (%)	680	696	718	702	524	602	586	512



(a)



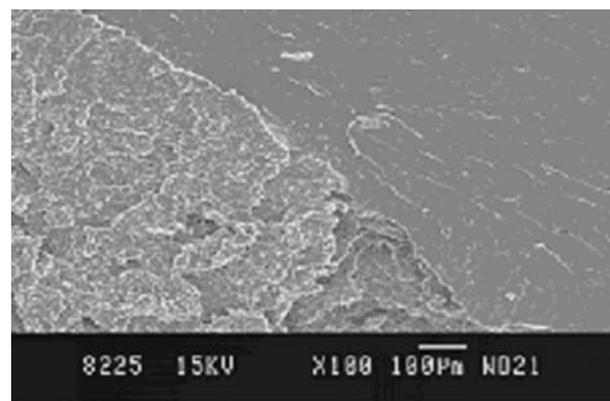
(b)

Figure 9. SEM Images of EP-96/CIIR: (a) 40/60 and (b) 60/40 blends.

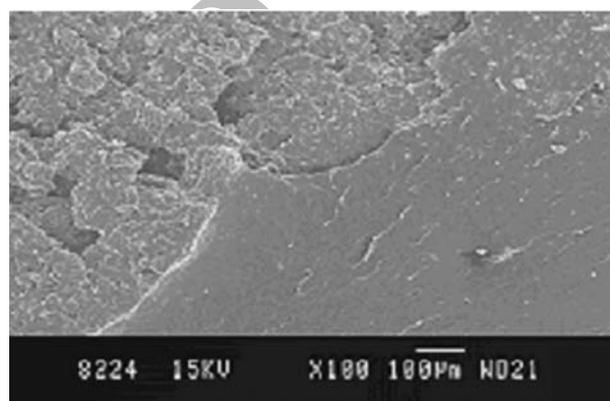
tensile strength are not just governed by overall cross-linking, but they depend on cross-link distribution and phase size as well.

As in the case of tensile strength, tear resistance also improves with the addition of chlorosulphonated polyethylene. The value reaches a peak at a concentration of 10 phr of the compatibilizer. Formation of uniformly distributed finer domains tends to elongate to higher strain and effectively prevents tear propagation. With increase in the concentration of compatibilizer above 10 phr, tear strength decreases. Modulus and elongation-at-break also follow the same trend.

The effect of concentration of compatibilizer (CSPE) on the morphology of 50/50 EP-96 grade EPDM/CIIR blend is shown in Figure 11. These SEM micrographs show changes in the phase morphology of the blends containing 0, 5, 10 and 15 phr compatibilizer. It is apparent that, with the addition of compatibilizer a more homogeneous surface texture is



(a)



(b)

Figure 10. SEM Images of MRD-10/CIIR: (a) 40/60 and (b) 60/40 blends.

observed up to 10 phr and afterwards the homogeneity decreases. This is due to the reduction of interfacial tension between CIIR and EPDM phases. The equilibrium concentration at which the dispersed EPDM domain size levels off is called critical micelle concentration (CMC). The estimation of CMC from the plot of domain size vs. concentration is reported elsewhere [34]. The CMC corresponds to the critical amount of the compatibilizer to saturate the unit volume of the interface. When compatibilizer concentration exceeds CMC, the micelles of the compatibilizer are formed in the continuous CIIR phase. Several reports are available in literature regarding the linear decrease of dispersed domain size with increasing compatibilizer concentration [35-37]. Therefore, it can be asserted that a concentration of 15 phr of compatibilizer is much above CMC and comparatively a substantial reduction of interfacial tension is achieved at a concentration of 10 phr. This is in good agreement

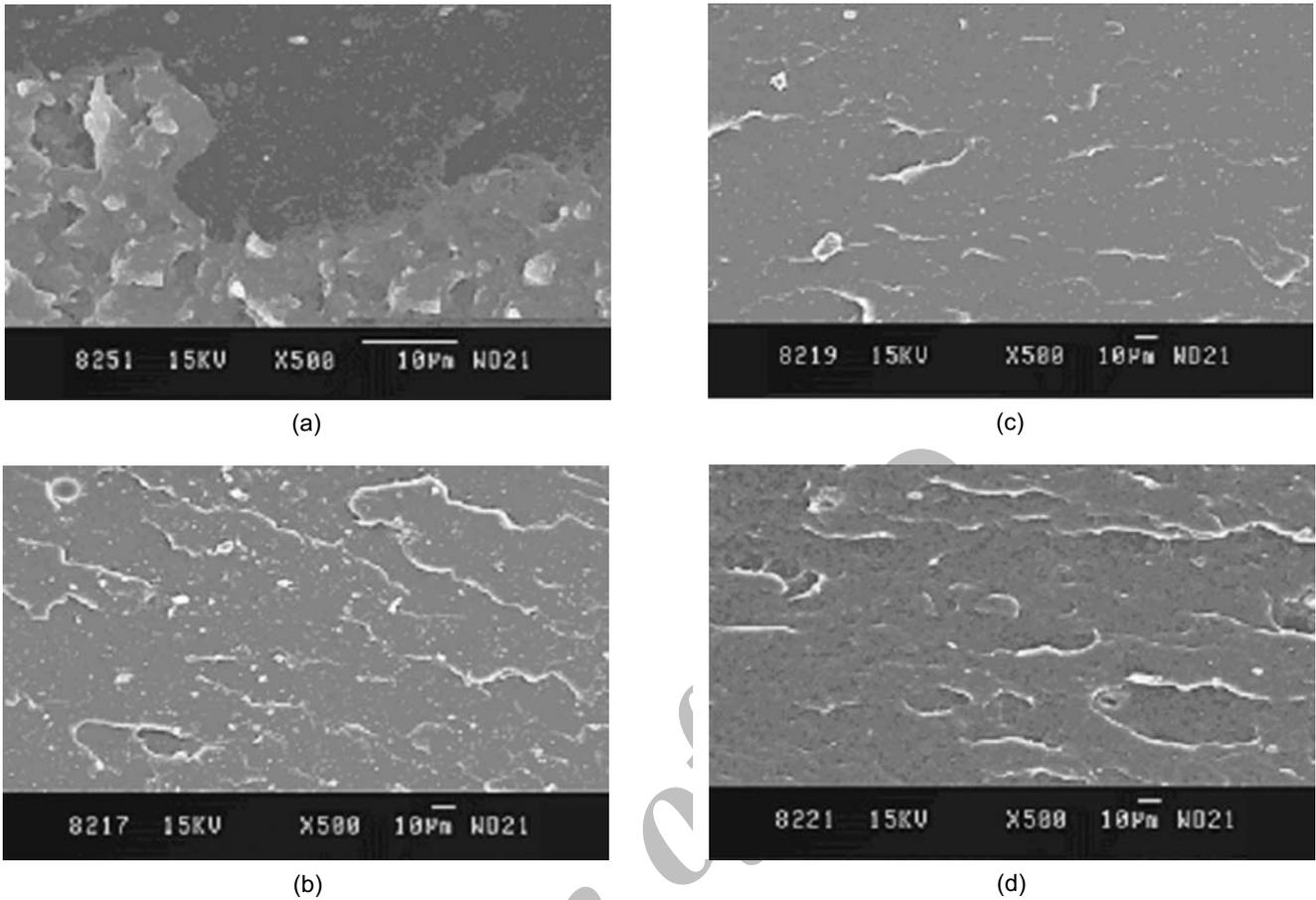


Figure 11. SEM Images of 50/50 EP-96 /CIIR blends: (a) without compatibilizer and with (b) 5 phr CSPE, (c) 10 phr CSPE, and (d) 15 phr CSPE.

with the overall improvement in mechanical properties observed.

Based on the above results, 10 phr chloro-sulphonated polyethylene is taken as the optimum amount of compatibilizer required and is used for further studies of EP-96 grade EPDM blended with CIIR. The variation in mechanical properties of the compatibilized blends is compared with those of the uncompatibilized systems at various blend compositions in Table 6. Compared to the uncompatibilized

blends, the compatibilized samples show an increase in overall mechanical properties. If the segments of the added compatibilizer are chemically identical with those in the respective phases or adhered to one of the phases, then they would act as efficient compatibilizers [38,39]. A comparatively higher percentage increase in tensile strength is observed for 20/80 and 40/60 EPDM/CIIR blends showing that compatibilizing action is efficient in these compositions. The compatibilizing action is due to the interaction of chlorine

Table 6. Comparison of physical properties of compatibilized and uncompatibilized EP-96/ CIIR blends.

Blend ratio and tests	Uncompatibilised blend				Compatibilised blend			
	20/80	40/60	60/40	80/20	20/80	40/60	60/40	80/20
Percentage of EPDM/CIIR blends	20/80	40/60	60/40	80/20	20/80	40/60	60/40	80/20
Tensile strength (MPa)	15.6	14.7	12.6	13.8	16.8	16.4	14.6	14.2
Tear strength (N/mm)	30.8	29.8	29.1	31.6	34.2	33.6	32.9	32.4
Modulus (at 300%) (MPa)	7.6	6.9	5.21	6.4	9.2	8.6	7.2	6.6
Elongation-at-break (%)	664	686	672	654	734	726	709	682

of CIIR with chlorosulphonated domain of compatibilizer. There is also structural similarity between some segments of compatibilizer and EPDM. This also promotes compatibilizing action of chlorosulphonated polyethylene in the blends. As the concentration of CIIR in the blend decreases, the probable interaction between the blend's components and compatibilizer decreases. There is higher homogeneity in mixing of rubbers in the presence of compatibilizers as the CIIR content in the blend is higher or equal to that of EPDM.

CONCLUSION

The compatibility of different grades of EPDM with a particular grade of CIIR was studied. Analysis of mechanical properties revealed that two grades of EPDM (301-T and NDR-4640) are compatible with CIIR. Thermal analysis and morphological characterization justify the results. This is explained on the basis of their viscosities and unsaturation matching, thus, co-vulcanization between the two components is possible to a great extent. The incorporation of compatibilizer, Chlorosulphonated polyethylene into incompatible EPDM/CIIR blends greatly enhances their compatibility and hence improves the mechanical properties of the systems. It is assumed that CSPE is preferentially located at the interface because of its lower viscosity, intermediary interfacial tension, and moderate polarity. The interfacial action of CSPE results in better anchorage between the phases which in turn leads to the better mechanical performance and more homogeneous morphology of the blends.

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