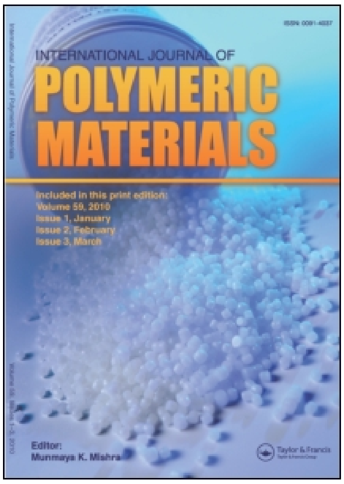

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Effect of Different Diene Monomers on the Polyblend Systems Consisting of EPDM and EVA

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Effect of third monomer (different dienes) on the cure characteristics, structures, properties, stability, fracture mechanism of melt-mixed blends of EPDM rubber and thermoplastic EVA copolymer are studied over the entire composition range in presence of Dicumyl Peroxide (DCP) as the curative 1,4 HD containing EPDM has the decreasing effect on the ultimate extent of curing the blends up to around 50% of EVA and then slightly increases; however, ENB has just opposite effect on the state of cure. But increasing effect was observed in case of DCPD containing EPDM. Presence of DCPD decreases the scorch safety as EPDM/EVA ratio decreases. Up to 40% EVA scorch time increases and then decreases at higher level for 1,4 HD. For ENB containing EPDM the scorch time initially decreases, then increases and remains constant at higher level. Activation energy of all the blends for 1,4 HD and ENB containing EPDMs and EVA lie in between that of pure EPDM and EVA. But it is true up to 40% of EVA loading in case of DCPD containing EPDM. 1,4 HD contribute least and DEPD contribute to a greater extent than ENB towards the interfacial adhesion, it seems. At a particular EPDM/EVA blend ratio, 1,4 HD-containing EPDM suffer greater weight loss than DCPD and ENB containing EPDMs. SEM study suggests the different modes of failure depending on diene content of EPDM as also confirmed by X-ray analysis.

KEY WORDS EPDM, EVA, 1.4 HD, ENB, DCPD, polymer blend.

INTRODUCTION

The scientific and commercial progress in the field of polyblends is enormous during the last two decades probably because of the realisation that new molecules may not always meet the needs of new materials so easily. Successful blending[1] can be implemented more rapidly and economically than the development of new macromolecular chemistry for a particular end use. The blend prepared by melt mixing of a polyolefin thermoplastic and elastomers have gained considerable attention in recent years.[2–6] Coran *et al.*[7–10] patented various dynamically crosslinked thermoplastic elastomer blends. Sain *et al.*[11] have shown the effect of PE loading on filled EVA with improved fire resistance. Thomas[12] have studied melt mixing of EVA and polyester. Recently Das *et al.*[13,14] have made the speciality polyblend systems of EVA and XLPE by melt mixing technique.

Here we study the blends of EPDM and thermoplastic EVA copolymer with special reference to the effect of diene types of EPDM rubber on the blend properties. Considering the results, a mechanistic scheme has been put forward for the vulcanization process in the presence of an organic peroxide.

EXPERIMENTAL

Blends are formulated by gradual replacement of EPDM by EVA as shown in Table I. Ethylene Vinyl Acetate (EVA) was of Elvax-460 variety from DuPont, USA. EPDMs were Nordel-1040 (DuPont, UK), Royalene-501 (Uniroyal Co., USA) and Royalene-400 (Uniroyal Co., USA). Blending EVA with EPDM was done in a Brabender Plasticorder at 60 rpm and 120°C for 10 min following the procedure as described earlier.[15] Dicumyl peroxide (DCP, 99%) was added at last and then sheeted out in a two roll open mixing mill. Continuous measurement of cure characteristics at 150°C, 160°C and 170°C were done in Monsanto Rheometer (R-100) in order to study the scorch safety, extent and rate of curing and activation energy etc. Curing was done at 160°C up to the optimum extent (t_{90} min from Rheometric data). Activation energy was determined from the knowledge of change in optimum cure time with temperature. Swelling coefficient and volume fraction of rubber were determined by swelling the vulcanizates in toluene at 25°C. Tensile properties were measured with the help of a universal testing machine. Aging was done in an air oven at 120°C for 50 hrs.

Thermal analysis of the samples were conducted in a Stanton Redcroft Thermal Analyser in Nitrogen at a rate of 10°C/min. X-ray analysis was done with the help of Philips PW 1729 generator with PW 1840 defractometer, Ni-filter and CuK_α radiation. Failure mechanism and the blend morphology were studied with SEM, camscan series 2 and E5200 autosputter coater.

RESULTS AND DISCUSSION

Effect of diene type on the cure characteristics of the blends

The rate of cure has been calculated from the Rheograph following the procedure as described earlier,[16] considering the first order reaction and plotted against the

TABLE I

Compounding formulations

Compound Nos.	1	2	3	4	5	6	7	8	9
EPDM (A/B/C)	100	80	70	60	50	40	30	20	—
EVA 460	—	20	30	40	50	60	70	80	100
DCP	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5

EPDM-A NORDEL-1040 (1,4 HD ≈ 4%; E:P ≈ 58:42)
EPDM-B ROYALENE-501 (ENB ≈ 4%; E:P ≈ 57:43)
EPDM-C ROYALENE-400 (DCPD ≈ 3%; E:P ≈ 68:32)
EVA-460 ELVAX-460 (18% VA; MFI ≈ 2.8)

blend ratio in Figure 1. As observed, for Hexadiene (HD) containing EPDM (A), cure rate decreases with the increase in EVA up to about 40% and then increases as more EPDM is being replaced by EVA. For ENB containing EPDM (B) the rate of cure increases up to about 40% EVA and then decreases at its higher level. However for DCPD containing EPDM (C) the cure rate gradually increases as EPDM is being replaced by EVA throughout the entire range of blend ratio studied. The state of cure, as determined by ($T_{max} - T_{min}$), has been plotted against blend ratio in Figure 2 suggesting predominant action of diene type of EPDM on the curing of blends with EVA. 1,4 HD has decreasing effect on the ultimate extent

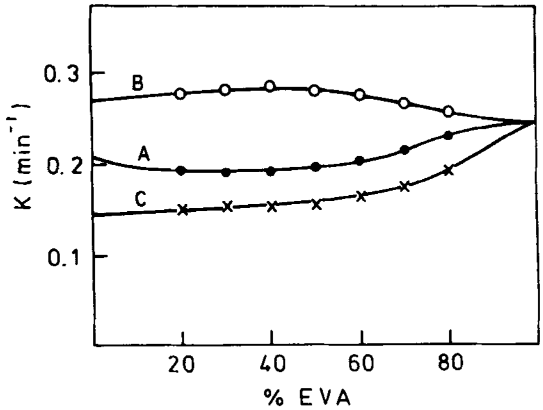


FIGURE 1 Variation of cure rate against % EVA.

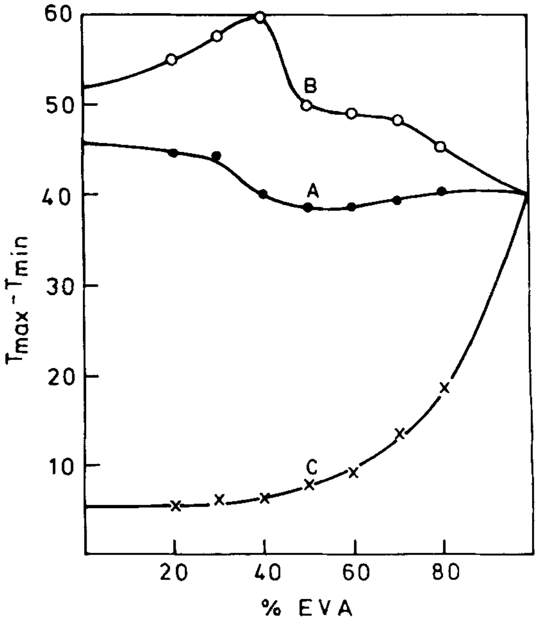


FIGURE 2 Variation of state of cure ($T_{max} - T_{min}$) against % EVA.

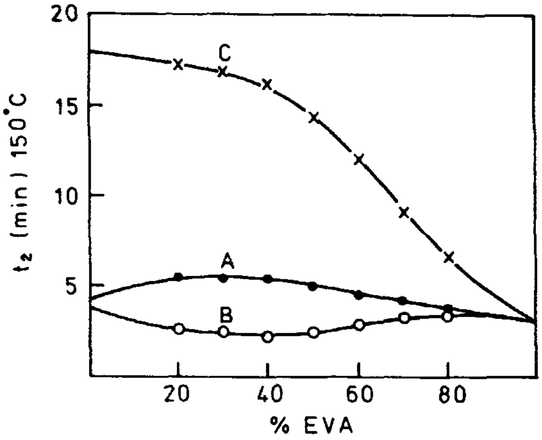


FIGURE 3 Variation of scorch time against % EVA.

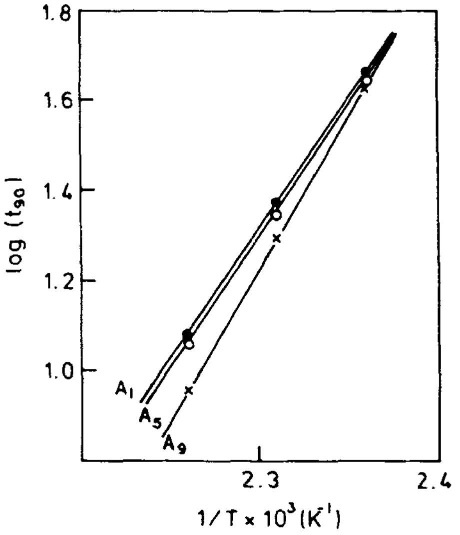


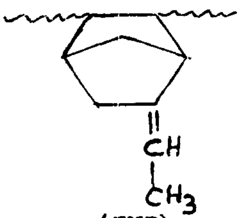
FIGURE 4 Plot of log (t_{90}) vs. 1/T.

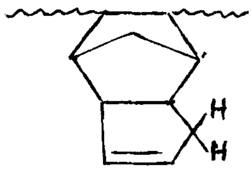
of curing of the blends as the EVA percentage is increased, attains a minimum around 50% EVA and then slightly increases whereas DCPD has increasing effect as the EPDM is being replaced by EVA. However ENB has increasing effect up to 40% EVA and then decreases the state of cure on further increase in EVA in the blend with EPDM.

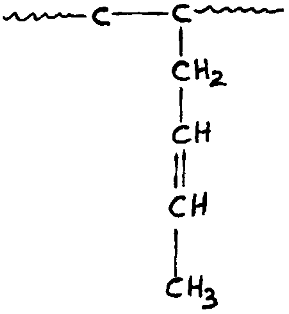
Scorch time has been shown (Figure 3) to vary with the blend ratio depending on the diene content in the EPDM rubber. Presence of DCPD decreases the scorch safety as the EPDM is being replaced by EVA. In 1,4 HD containing EPDM the scorch time increases up to 40% EVA and then decreases at higher level of EVA whereas for ENB containing EPDM the safety decreases up to 40% EVA and then increases and remains constant at higher level of EVA.

The effect of diene type on the cure activation of the blends has been shown in Figures 4 and 5. Both the 1,4 HD and ENB containing EPDMs exhibit lower activation energy of curing than that of EVA, however DCPD containing EPDM has higher activation energy than the EVA. For both the 1,4 HD and ENB containing EPDMs there is marginal increase in activation energy up to 50% EVA in the blend and further addition of EVA slowly increases the activation energy. Whereas for DCPD containing EPDM the activation energy lowers down as the EVA content increases up to about 50% and then remain more or less constant on further increase in EVA replacing EPDM. Activation energy of all the blends for 1,4 HD and ENB containing EPDM and EVA lie in between that of pure EPDM and pure EVA suggesting involvement of both the phases, independently, in the curing process. But for DCPD containing EPDM and EVA systems, the activation energy of the blends up to 40% of EVA lie in between that of pure EPDM and pure EVA suggesting independent curing of both. Beyond 40% EVA there might be some interdependent interaction during curing leading to lowering activation energy associated with high cure rate as observed in Figure 1.

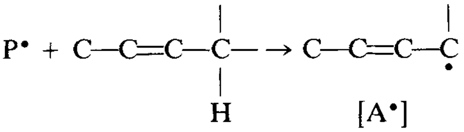
The effects of diene type in the EPDM curing alone has been established earlier[17] which is again found to be dependent on the concentration of diene, concentration of peroxide and time and temperature of curing.[18] Similarly, the peroxide curing of EVA also is dependent on the concentration of acetate content, concentration of peroxide and time and temperature of curing. The behaviour of the blends could probably be explained by the structure of diene types as


(ENB)


(DCPD)


(HD)

and also considering the number of active allylic hydrogens. DCPD has only two active allylic hydrogen and HD has five such allylic hydrogens and thus more crosslinking via coupling of allylic radials in the case of 1,4 HD containing EPDM as given below:



$$P^\bullet + C{-}C{=}C{-}\underset{H}{\overset{|}{C}}{-} \rightarrow \underset{[A^\bullet]}{C{-}C{=}C{-}\overset{|}{\underset{\bullet}{C}}} \qquad (1)$$

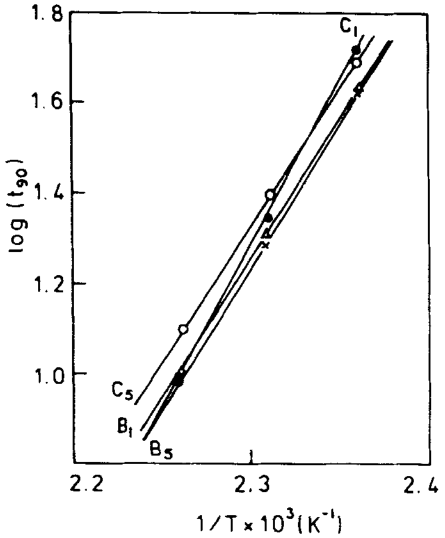
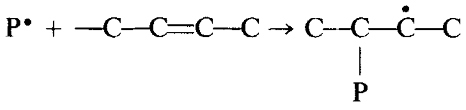


FIGURE 5 Plot of log (t_{90}) vs. 1/T.

$$A^{\bullet} + A^{\bullet} \rightarrow A{-}A \text{ (crosslink)} \qquad (2)$$

Addition reaction as shown



$$P^{\bullet} + {-}C{-}C{=}C{-}C \rightarrow C{-}\underset{\displaystyle P}{\underset{|}{C}}{-}\overset{\bullet}{C}{-}C \qquad (3)$$

is however predominant in DCPD containing EPDM and the subsequent polymerization through this resultant radical may not be occurring because of steric effect as observed from the very low cure state and cure rate.

ENB residue is however more like 1,4 HD, an internal olefin, containing active allylic hydrogens, sterically unconstrained α-methyl group and perhaps more active α-methylene group. Apart from crosslinking by coupling (Equations 1 and 2) a considerable amount of addition reaction (Equation 3) and subsequent polymerization is taking place making state of cure and cure rate comparatively more than other varieties of EPDM.

The isomerization like

$$C{-}C{=}C{-}\underset{\bullet}{C} \rightleftharpoons C{-}\underset{\bullet}{C}{-}C{=}C \qquad (4)$$

cannot be ruled out even with ENB containing EPDM, may be responsible for further polymerization process. However, coupling through allylic radicals or addition of radicals of double bond and isomerization of allylic radicals (Equation 4) may lead to polymerization. The extent of above mentioned reactions will affect the thermal stability of the compound (will be discussed) as the number of double bonds varies.

Crosslinking of EVA may proceed via hydrogen ion abstraction involving tertiary carbon atom and acetyl group.[13] Like EPDM the crosslinking efficiency seems to depend on the amount of peroxide, temperature of curing and acetate content of the EVA. This is probably reflected in the rate and state of cure of ENB and 1,4 HD containing EPDM with EVA where an inversion has been observed at 40–50% EVA level (Figure 1, curves A, B). This is worth mentioning that there is not much difference in rate and ultimate state of cure between above two pure EPDM and pure EVA. However wide differences have been observed in case of DCPD containing EPDM and EVA and no inversion is encountered. Here again the increasing trend pattern changes the slope at 50:50 blend ratio (Figure 1, curve C). The plot of activation energy of curing (Figure 6) against blend ratio also follow the same trend suggesting probably the phase inversion. It is difficult to predict whether any phase interaction is there or not but the peroxide curing efficiency seems to be changing in the EPDM/EVA blend, depending on the blend ratio as there is no linearity in the change in activation energy has been observed (Figure 6).

Effect of diene type on solvent swelling

In a binary blended cured system, condition can be established wherein a polymer representing a continuous phase is highly swollen, while the dispersed polymer phase is lightly swollen or unswollen. The unswollen or lightly swollen dispersed phase is considered as filler and reinforcing filler concept, as by Zapp,[19] can be applied to this system. If the interfacial bonds are formed during co-vulcanization the unswollen or lightly swollen dispersed phase will restrict the swelling of highly swollen continuous phase and the plot of swelling coefficient vs. blend ratio lie

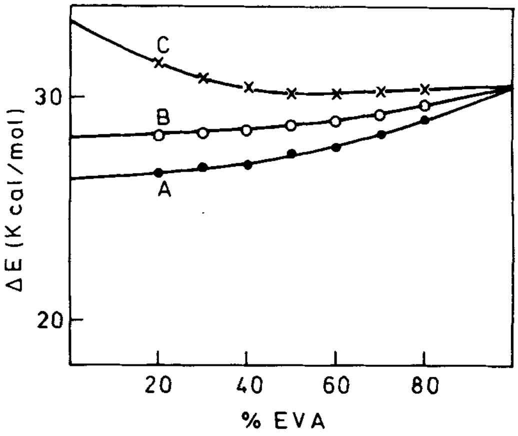


FIGURE 6 Variation of activation energy against % EVA.

below the additive average line. This can be supplemented by the Krause[20] theory through the relationship

$$\frac{V_{r_o}}{V_r} = 1 - M\,\phi/(1 - \phi)$$

V_{r_o} = Volume fraction of elastomer in the swollen gel when no dispersed phase is present.
V_r = Volume fraction of the continuous phase elastomer when a lightly swollen dispersed phase is present.
ϕ = Volume fraction of dispersed phase in the unswollen covulcanizate.

in the case of polymer blend. If the slope (M) is negative in a plot of V_{r_o}/V_r against $\phi/(1 - \phi)$ the phase adhesion is present. In this study we have considered the swelling co-efficient in toluene and restricted the Krause plot in the region of high EPDM containing blends.

As observed from the Figures 7, 8 and 9, the plots of swelling coefficient against blend ratio and V_{r_o}/V_r against $\phi/(1 - \phi)$ for all three types of EPDM in blends with EVA. In the case of EPDM containing 1,4 HD (Figure 7) the swelling co-efficient decreases as the EPDM is being replaced by EVA but the swelling co-efficient lies above the additive average line. This may be due to the presence of increasing EVA plastic phase and not dependent on the state of cure at least at initial level. The value of V_{r_o}/V_r lies much above the unity lines with higher M (slope) value. Figure 8 reveals that, for the ENB containing EPDM, there is a change in the decreasing trend of swelling coefficient at about 40% EVA which coincides with the change in state of curve shown in Figure 2 (Curve B). Here again the swelling coefficient lies above the additive average line. The value of volume fraction ratio lies above the unity line with two distinct M (slope) values. In the case of EPDM with DCPD (Figure 9) the swelling decreases as the EPDM

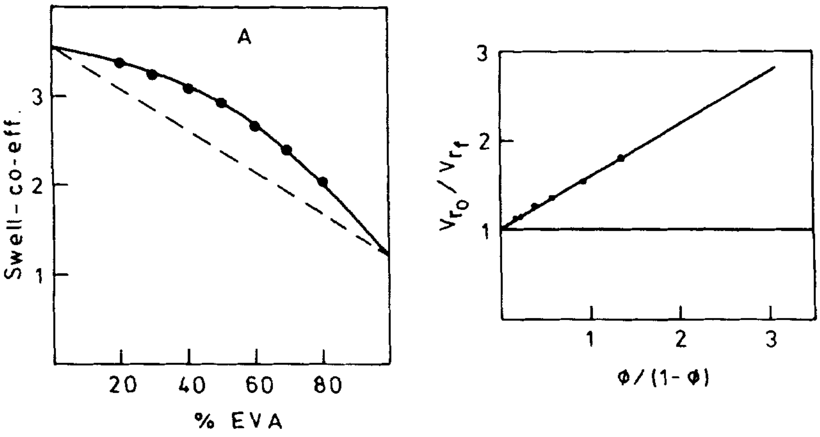


FIGURE 7 Plot of swelling coefficient vs. % EVA for the EPDM(A)/EVA blend and V_{ro}/V_{rf} vs. $\phi/(1 - \phi)$.

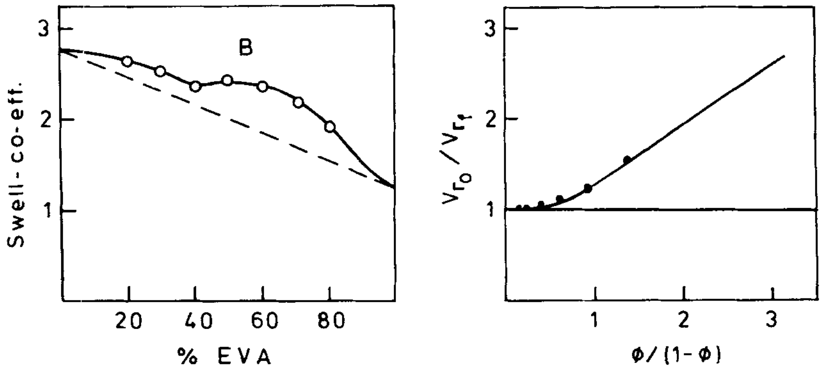


FIGURE 8 Plot of swelling coefficient vs. % EVA for the EPDM(B)/EVA blend and V_{ro}/V_{rf} vs. $\phi/(1-\phi)$.

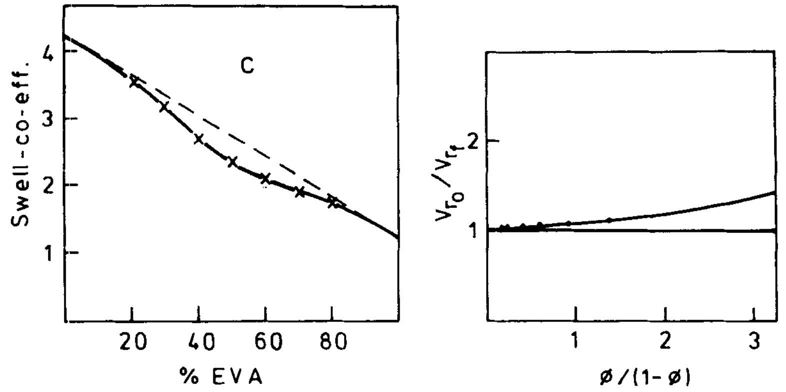


FIGURE 9 Plot of selling coefficient vs. % EVA for the EPDM(C)/EVA blend and V_{ro}/V_{rf} vs. $\phi/(1-\phi)$.

is being replaced by EVA. This is in line with the increasing ultimate extent of cure as the EPDM/EVA ratio decreases. However the values of swelling coefficient lie near the additive average line except the region of 40–60% EVA where it is less than the additive values. Surprisingly the values of V_{r_0}/V_r lie slightly above the unity line with M (slope) values changing around 50% of EVA.

It has been found that none of the slopes is negative, hence the co-vulcanization theory does not hold good in these systems in a true sense. From the slopes (M) it seems logical to assume the contribution of different diene to the different extent towards the curing process of the blends. 1,4 HD probably contribute least and DCPD contribute to a greater extent than ENB.

Thermal stability

Figure 10 depicts that the weight loss increases with the increase in temperature but the extent of weight loss depends on the diene type. Similar to XLPE/EVA blends, the EPDM/EVA blends are also characterised by two stage degradation.[14]

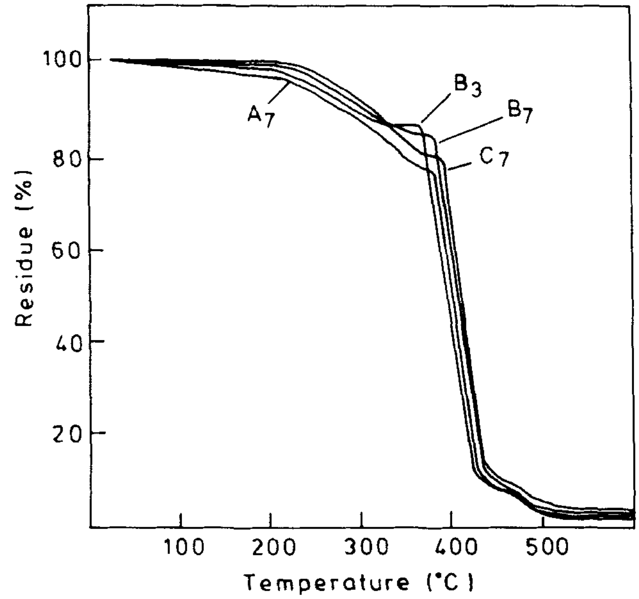


FIGURE 10 TGA plot of EPDM/EVA blend (A_7, B_7, C_7, B_3).

At higher level of EVA this becomes more prominent. At a particular EPDM/EVA blend ratio, 1,4 HD containing EPDM suffers greater weight loss than DCPD and ENB containing EPDM. The drastic weight loss is observed around 400°C. However at comparatively lower temperature (around 225°C) the DCPD containing EPDM has an edge over ENB containing EPDM. The loss is more for higher EPDM/EVA blends (curves B_3 and B_7).

DTA curves as observed in Figure 11 reveal that an endothermic peak near 75°C corresponds to the melting of EVA phase which is sharp at high EVA content, without weight loss. Second exothermic peaks are observed at around 225°C associated with the start of weight loss. Near 350°C there is another endothermic peak where loss in weight is slightly checked and around 400°C there is exothermic peak associated with drastic weight loss corresponding to the oxidative degradation. This degradation temperature is less for 1,4 HD containing EPDM. Around 475°C there is another endothermic peak in conjunction with exothermic peak around 440°C, both of which are associated with weight loss although at different rate. Beyond 425°C, the peaks are of no value for all practical purposes because of extreme degradation resulting in minute residue left over.

X-ray characterization of the blends

Analysis was conducted over the angle in between $5° < 2\theta < 60°$ and a representative example is shown in Figure 12. Comparison of the heights and widths of the characteristic peaks of the diffractograms clearly reveal that EVA is more crystalline than EPDM and the crystallinity increases as EPDM/EVA ratio decreases. At the equivalent blend ratio, the DCPD containing EPDM in blend exhibit slightly more crystallinity than other two types of EPDMs. In this case (curve C_3) the EVA peak

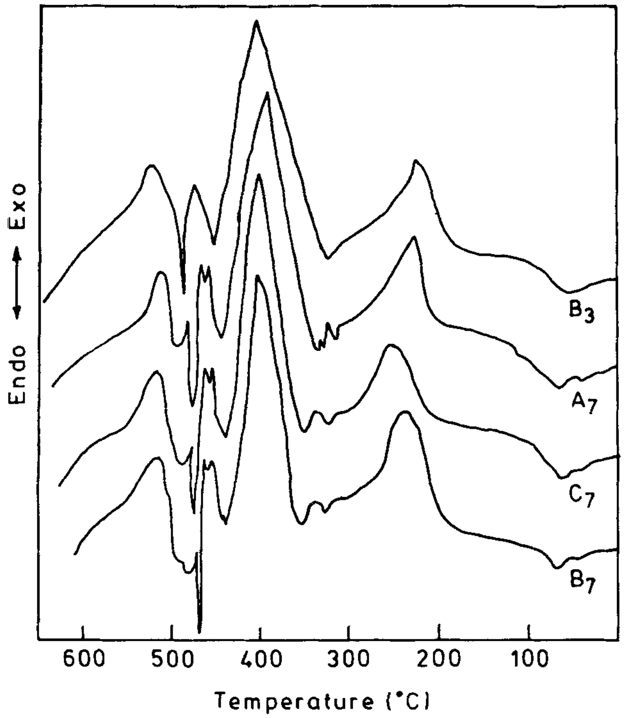


FIGURE 11 DTA plot of EPDM/EVA blend (A_7, B_7, C_7, B_3).

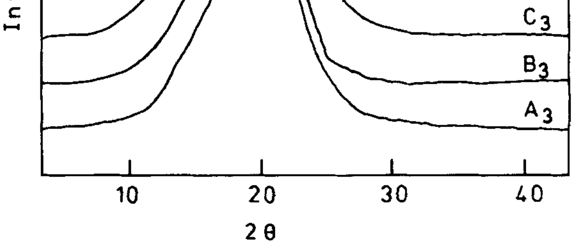


FIGURE 12 X-ray defractogram of EPDM/EVA blend (A_3, B_3, C_3, A_7).

is comparatively sharper than that of other two EPDMs. EVA peak becomes sharper as the EPDM/EVA ratio decreases and is more prominent in case of DCPD containing EPDM. Regarding crystallinity, 1,4 HD containing EPDM has slight edge over ENB containing EPDM in blend with EVA.

The interplanner distance (*r*) has been calculated from the (2θ) values at the corresponding peak points as described by the author earlier[21,22] considering the limits of experimental inaccuracy involved, it can be safely said that interchain distance (*r*) of EVA remains almost the same in all the blends for a particular EPDM but it is slightly higher than that of pure EVA suggesting the probable immigration of EPDM into its interplane spacing at the initial installment of EPDM addition. The interchain spacing (*r*) of EVA is slightly higher in the case of 1,4 HD containing EPDM than DCPD and ENB containing EPDM blends with EVA. This crystallinity and interchain spacing have predominant effects on the physical properties and swelling behaviour of blends.

Effect of diene type on the physical properties before and after ageing

A representative example of stress-strain behaviour is shown in Figure 12 and values of tensile properties both before and after ageing is given in the Table II (a, b, c). Tensile strength and modulus of the blend increases gradually as the EPDM/EVA ratio decreases, starting from pure EPDM to pure EVA vulcanizates. Hardness and elongation at break also follow the above trend and is unaffected by the type of diene monomer in the EPDM. Except for the EPDM containing DCPD, the above observations are not in conformity with the state of cure (Figure 2) achieved during vulcanization. Although an inversion in state of cure is observed for both the EPDMs containing 1,4 HD and ENB, there is gradual increase in modulus (Table II) as the EPDM is being replaced by EVA. This can be attributed to the increase in crystallinity as the EPDM/EVA ratio decreases which play a predominant role in increasing the modulus, while testing at ambient, probably the contribution of plastic phase in determining the tensile properties at ambient is more, as can be confirmed from the Figure 12. Figure 12 reveals that as the

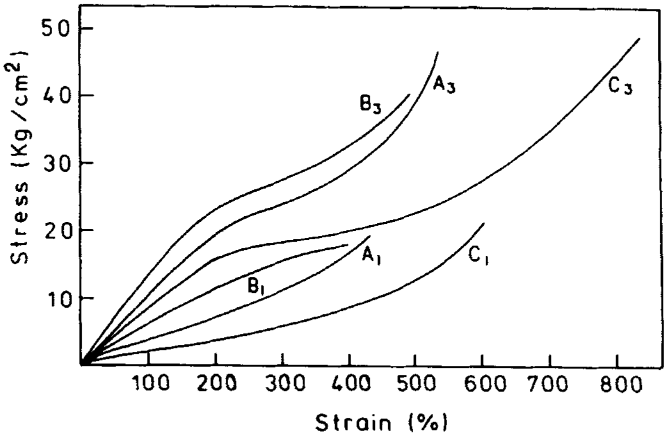


FIGURE 13 Plot of stress vs. strain of EPDM/EVA blend (A_1, A_3, B_1, B_3, C_1, C_3).

TABLE II(a)

Physical properties both before and after ageing for the EPDM (1,4 HD)/EVA blends

Compound Nos.	A_1	A_2	A_3	A_4	A_5	A_6	A_7	A_8	A_9
Modulus 300% (kg/cm^2)	13	18	24	30	41	44	49	53	60
Tensile strength (kg/cm^2)	22	28	46	57	61	81	99	124	164
Elongation at break (%)	430	450	530	700	830	900	920	950	1050
Hardness (IRHD)	50	60	65	71	78	82	85	89	90
Change in Properties afer Ageing (120°C for 50 hrs)									
Modulus	—	—	+24	+21	+17	+18	+15	+12	+14
Tensile strength	−26	−24	−21	−17	−18	−12	+2	+6	+4
Elongation at break	−36	−30	−26	−22	−23	−20	−17	−18	−21

TABLE II(b)

Physical properties both before and after ageing of EPDM(ENB)/EVA blends

Compound Nos.	B_1	B_2	B_3	B_4	B_5	B_6	B_7	B_8	B_9
Modulus 300% (kg/cm^2)	15	20	28	35	39	48	50	52	60
Tensile strength (kg/cm^2)	19	25	40	52	60	78	101	121	164
Elongation at break (%)	400	390	490	590	740	860	990	1010	1050
Hardness (IRHD)	51	61	66	71	79	81	84	87	90
Change in properties after ageing (120°C for 50 hrs)									
Modulus	—	—	+18	+16	+16	+14	+12	+12	+14
Tensile strength	−22	−20	−18	−15	−12	−10	+3	+4	+4
Elongation at break	−30	−25	−22	−20	−18	−14	−15	−17	−21

EPDM is being replaced by EVA, a necking tendency is apparent and is more prominent in the case of DCPD containing EPDM where state of cure has been found to be lowest. This necking behaviour is associated with high tensile at higher elongation at break.[14] The EPDM containing ENB, in the blend has comparatively lower necking and 1,4 HD containing EPDM has intermediate necking tendency.

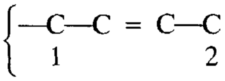
After ageing tensile properties are shown in the Table II (a, b, c). There is drop in tensile strength and elongation at break for the blends of higher EPDM content but at higher level of EVA the tensile strength increases. Modulus however increases for all the blend ratio with more increase in the case of high EPDM/EVA blends. As observed 1,4 HD containing EPDM blends suffer more change in these properties. Minimum change is observed in the case of ENB containing EPDM.

TABLE II(c)

Physical properties both before and after ageing of EPDM(DCPD)/EVA blends

Compound Nos.	C_1	C_2	C_3	C_4	C_5	C_6	C_7	C_8	C_9
Modulus 300% (kg/cm^2)	6	11	18	29	33	40	44	48	60
Tensile strength (kg/cm^2)	21	30	48	58	69	80	109	128	164
Elongation at break(%)	600	700	850	900	960	1000	1020	1050	1050
Hardness (IRHD)	23	35	45	55	63	70	76	82	90
Change in properties after ageing (120°C for 50 hrs)									
Modulus	+30	+26	+22	+18	+20	+22	+16	+14	+14
Tensile strength	−20	−22	−18	−12	−8	+4	+8	+6	+4
Elongation at break	−35	−32	−30	−26	−25	−24	−21	−20	−21

The more degradation for 1,4 HD containing EPDM blends may be due to the coupling through the 2 position as shown[17]



$$\left\{ \underset{1}{\text{—C}}\text{—C} = \underset{2}{\text{C—C}} \right.$$

leaving double bond in between the main chain and crosslink which is prone towards oxidative degradation. In the case of ENB containing EPDM blends this double site either stays outside the crosslink juncture by crosslinking via isomerisation through equation (4) or addition of peroxy radical to double bond accompanied by subsequent polymerization as discussed in earlier section.

Effect of diene on the fracture mechanism

The failure mode changes with the blend ratio and the diene type as shown in Figures 14–18 as SEM fractogram. At higher EPDM/EVA blend ratio (Figure 14) the failure appears to be ductile with cavitation and surface irregularities have occurred from the crack initiating flows.[18,23] As the EVA content increases in the blend (Figure 15) the fracture mode changes to layer like structure with flow mark associated with minor cavitation. At the higher EVA content (Figure 16) the failure is supposed to be brittle one with distinct plastic flow with thick fibrils and foldings at certain places. At the same blend ratio (EPDM:EVA ≈ 60:40) the failure of DCPD containing EPDM blends is more brittle in nature than 1,4 HD and ENB containing EPDM as shown in Figures 15, 17 and 18. Plastic flow and cavitation is more in the case of DCPD containing EPDM. Plastic flow in ENB containing EPDM (Figure 15) is less than that of 1.4 HD containing EPDM (Figure 17) and compared to other two EPDMs this failure is more towards the ductile side with less elongation at break and no necking. More flow and foldings at the crack end in the case of DCPD containing EPDM/EVA blend may be due to larger extent of necking on extension.

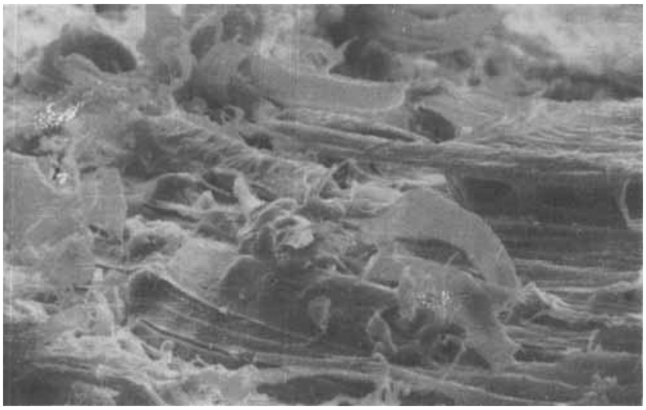


FIGURE 14 SEM fractogram of the blend B_2 (500×).

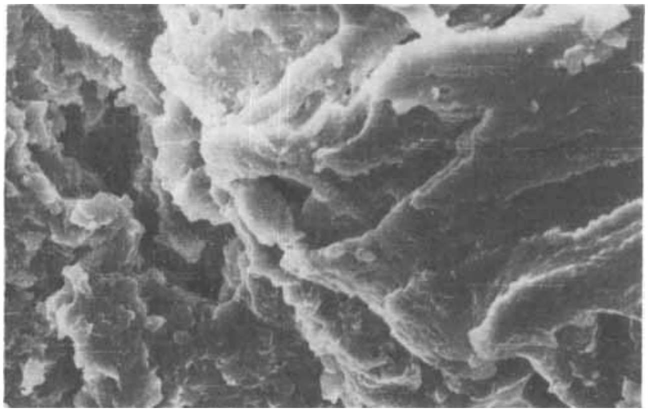


FIGURE 15 SEM fractogram of the blend B_4 (500×).

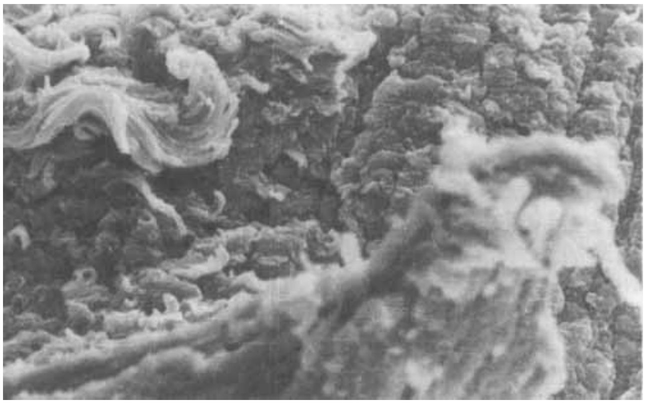


FIGURE 16 SEM fractogram of the blend B_7 (500×).

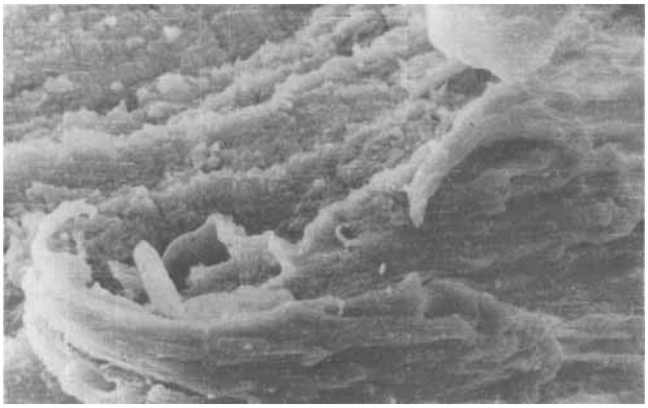


FIGURE 17 SEM fractogram of the blend A_4 (500 ×).

FIGURE 18 SEM fractogram of the blend C_4 (500 ×).

CONCLUSION

The diene type of EPDM has tremendous effects on the EPDM/EVA blends. Entire cure mechanism, the stability and the failure mechanism change depending on diene monomer in EPDM. ENB diene has an edge over DCPD and 1,4 HD so far as the properties are concerned. Curing efficiency of DCPD in the lowest and probability of remaining EVA as plastic phase is more resulting higher amount of necking. It appears some co-vulcanization is occurring in the case of ENB containing EPDM, in blends with EVA thus restricting the plastic flow of EVA at low elongation at break. 1,4 HD containing EPDM is less thermally stable than ENB containing EPDM/EVA blends. Increase in EVA improves the crystallinity and reduce the swelling.

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