

# Covulcanization of LLDPE/EMA Blends Using Dicumyl Peroxide

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**ABSTRACT:** The effect of dicumyl peroxide (DCP) content on the gel fraction, mechanical, dynamic mechanical, and thermal properties of linear low-density polyethylene (LLDPE)/ethylene-*co*-methyl acrylate (EMA) blends were studied. Gel content of the blends increases with increasing DCP content, and EMA is more prone to crosslinking than LLDPE. Wide-angle X-ray diffraction (WAXD) and differential scanning calorimetry (DSC) were used to study the effect of DCP crosslinking on percent crystallinity and crystalline structure of the blends and individual components. At lower level of DCP loading, crosslinking process does not have significant effect on the crystalline structure of the LLDPE, which was confirmed from the percent crystallinity and lattice distance value. However, at higher DCP content, percent crystallinity decreases significantly.

At lower EMA concentration (<50%), percent crystallinity and lattice distance remain unchanged up to 2 wt % of DCP. For EMA contents of more than 50 wt %, increasing DCP content reduces the crystallinity of the blends and increases the lattice distance. The highest level of mechanical and dynamic mechanical properties was observed for 60/40 LLDPE/EMA blends at 2 wt % DCP. Addition of LLDPE-*g*-MA (3 wt %) as a compatibilizer enhances the properties of the vulcanizates. Blends crosslinked with DCP up to 0.3 wt % can easily be reprocessed. © 2011 Wiley Periodicals, Inc. *J Appl Polym Sci* 123: 502–509, 2012

**Key words:** mechanical properties; vulcanization; differential scanning calorimetry; reprocessability; LLDPE/EMA blends

## INTRODUCTION

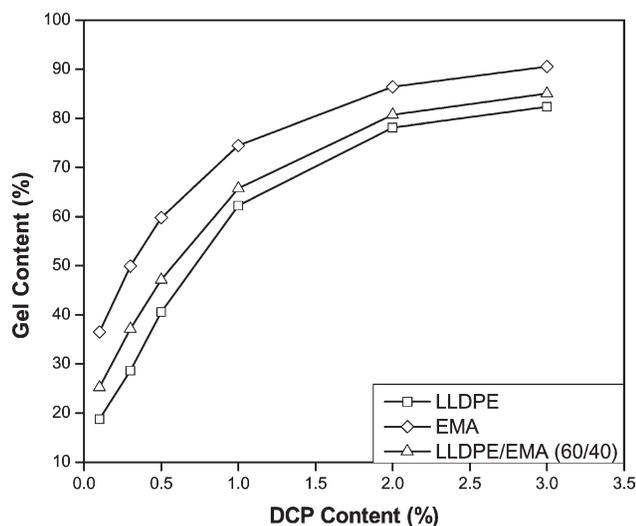
The blending of existing polymers is an economically attractive route to develop new materials that combine the desirable properties of more than one polymer.<sup>1–4</sup> In recent decades, numerous blend systems have been developed and commercialized. Polyolefins, especially polyethylene-based blends, have been studied much more because of its good mechanical properties, good electrical insulating property, good film-forming properties, and good compatibility to many other polymers. However, their use is restricted in certain application because of its low melting point, low stress cracking resistance, and solubility or swelling in hydrocarbons.<sup>5–10</sup> To overcome these problems, considerable work has been carried out based on compatibilization and crosslinking of polymer blends.<sup>11–14</sup>

Compatibilization reduces the interfacial tension between the blend components resulting in a finer and more stable morphology, better adhesion between the components, and consequently, better properties of the final product.<sup>15</sup> The compatibilization can be achieved either by addition of presynthe-

sized graft or block copolymer (physical compatibilization) or through the *in situ* generation during processing (reactive compatibilization). Various researchers have studied the effect of addition of block or graft copolymer on immiscible polymer blends.<sup>16–19</sup> Chiu and Hsiao<sup>20</sup> showed that the addition of the maleic anhydride-grafted polyethylene-octene elastomer (POE-*g*-MA) in PET/PP blends reduced the size of the dispersed phase and improved the processability and toughness of the blends. Borah and Chaki<sup>10</sup> also observed similar kind of behavior for linear low-density polyethylene (LLDPE)/ethylene-*co*-methyl acrylate (EMA) blends when compatibilized by LLDPE-*g*-MA. The effect of compatibilization on mechanical and dynamic mechanical properties of LLDPE/EVA (ethylene-*co*-vinyl acetate) blends had been studied by Moly et al.<sup>8</sup> It was reported that compatibilization improved the modulus of the blends, which is due to the fine dispersion of EVA domains in the LLDPE matrix providing an increased interfacial interaction.

The crosslinking of polymer leads to an improvement in the thermal stability. Moreover, it can increase the tensile strength of polymers and thus, at a reasonable yield of crosslinking, improve their mechanical properties.<sup>21</sup> The crosslinking process involves the formation of chemical bonds (crosslinks) between adjacent molecular chains to form a three-dimensional network. There exist several methods to

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**Figure 1** Effect of DCP content on the gel content of crosslinked LLDPE, EMA, and LLDPE/EMA (60/40) blends.

produce crosslinked polymers, such as chemical methods, using mainly peroxides<sup>22</sup> or silanes,<sup>23,24</sup> and high-energy irradiation (electronic beam) techniques.<sup>25–27</sup> A detailed description of the various crosslinking processes has been given in a comprehensive review by Lazar et al.<sup>28</sup>

Thermochemical crosslinking involving organic peroxides is most widely used for its controlled decomposition rate, minimal side products, and economical process.<sup>29</sup> Peroxide is incorporated into the polymer, at the time of mixing, below the activation temperature of the peroxide. The crosslinkable polymer will later be molded and shaped and then cured under pressure and temperature. Suitable peroxide has to be selected to give fast crosslinking without pre-curing in the mixing chamber; hence, dicumyl peroxide (DCP) is widely used for this purpose.

In recent years, crosslinked LLDPE has been widely explored for a number of industrial applications requiring withstanding high-temperature environments. Examples of such applications include wire and cable coating, heat shrinkable materials, hot water tubing, and steam-resistant food packaging.<sup>30–33</sup>

The use of ethylene methyl acrylate copolymer in blends and composites is most important from the technological point of view. Because of low-temperature flexibility, somewhat rubbery nature, low permeability, and good impact strength, it is becoming interesting as a stretched film for packaging technology.<sup>34</sup> It has drawn interest as a cable insulating material because of the good resistance to stress cracking and because the polymer may be easily crosslinked.

The authors have recently studied and reported the effect of blend composition and compatibilization on the morphology, rheology, dynamic mechan-

ical, mechanical, and thermal properties of LLDPE/EMA blends. It was reported that blend ratio and compatibilization have significant effect on morphology of the LLDPE/EMA blends. The 60/40 LLDPE/EMA blend with 3 wt % LLDPE-g-MA (compatibilizer) was found to have provided good balance between stiffness and toughness of the blend.<sup>7,10</sup> In this article, the main focus is to study the effect of DCP content in LLDPE/EMA blends.

## EXPERIMENTAL

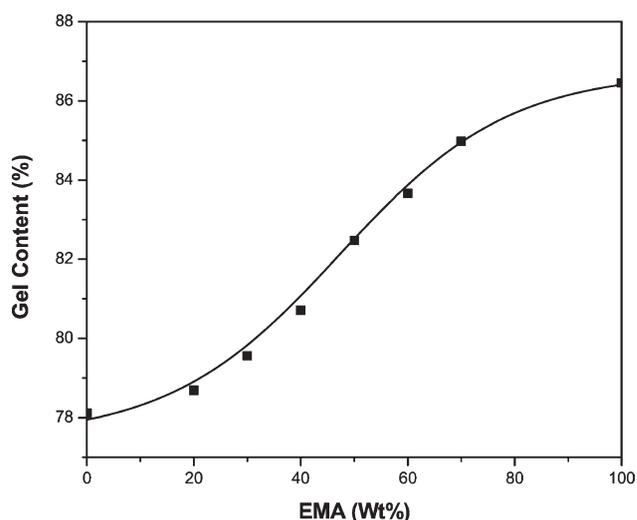
### Materials

LLDPE (LLT12), having a density of 0.926 g/cm<sup>3</sup> and melt flow index (MFI) 3.7 g/10 min, was obtained from Haldia Petrochemicals (India). Commercial grade of EMA, Elvaloy 1330 with 30 wt % of methyl acrylate and an MFI of 3.0 g/10 min of DuPont (Mechelen, Belgium), was supplied by Nicco Corp. (India).

The compatibilizer used for this study is maleic anhydride-grafted linear low-density polyethylene (LLDPE-g-MA). LLDPE-g-MA was prepared by melt blending LLDPE (100 g) with maleic anhydride (5 g) and dicumyl peroxide (DCP-40% activity; 0.5 g). The melt mixing was carried out in an internal mixer at 180°C and 60 rpm for 8 min.

### Blend preparation

The blends having different compositions are designated as *mEx/y*, where “*x*” represents the weight percentage of EMA, “*y*” represents the weight percentage of DCP, and “*m*” represents the weight percentage of LLDPE-g-MA in the blend. The blending was carried out in a Haake Rheomix OS (Germany) using a cam-type rotor. The temperature of mixing



**Figure 2** Effect of EMA content on the gel content of crosslinked LLDPE/EMA blends at constant DCP (2 wt %) content.

TABLE I  
Effect of DCP Content on the Mechanical Properties of LLDPE/EMA (60/40) Blends

Sample code	Tensile modulus (MPa)	Tensile strength (MPa)	Elongation at break (%)	Tensile impact strength (J/m)	Percent tension set
E40	72.9	8.3	226	1638.8	39.6
E40/0.1	79.4	9.2	346	1888.9	34.4
E40/0.3	85.6	11.4	494	2122.8	29.3
E40/0.5	92.2	13.9	474	3233.3	25.6
E40/1	96.2	17.0	461	3736.1	21.3
E40/2	96.6	18.9	450	4736.0	17.6
E40/3	66.7	17.6	209	4424.7	16.0
3CE40/2	97.8	20.3	474	4858.3	16.5

was set at 140°C and the rotor speed at 60 rpm. LLDPE was melted first for 2 min; EMA was then added and mixed for 6 min. Then DCP was added to the mixer and mixed for another 3 min. For compatibilized blends, after melting LLDPE for 2 min, the compatibilizer was added and mixed for 1 min and then EMA was added and mixing was continued for 6 min. This was followed by addition of DCP and mixed for 3 min. The hot mass was then taken out immediately from the mixer and sheeted out in a two roll mill set at 2-mm nip gap. The sheeted material was cured in a compression mold (Moore Presses, George E. Moore and Sons Birmingham, United Kingdom) at 170°C for 12 min under a pressure of 5 MPa. The mold was allowed to cool under pressure till ambient temperature is attained before removing the rectangular sheet from the mold.

### Gel fraction analysis

The gel content of the crosslinked samples was determined gravimetrically by using a 16-h soxhlet extraction with xylene as the solvent at 140°C. After the extraction, the sample was vacuum dried to a constant weight. The gel fraction was calculated as the percentage ratio of the final weight of the polymer to its initial weight.

### Mechanical properties

The tensile testing of the blends was carried out in a Hioks-Hounsfield Universal Testing Machine (Test

Equipment, Surrey, England) according to ASTM D 412-98a test method using dumbbell shaped specimen at a cross-head speed of 500 mm/min at room temperature (25 ± 2°C). Impact strength was determined by using a tensile impact tester, CEAST-type 6545/0000 using a load of 7 kg per the DIN53448.

### Differential scanning calorimetry

Differential scanning calorimetry (DSC) measurements were performed using a DSC Q 100 V 8.1 Build 251 thermal analyzer at a heating rate of 10°C/min under nitrogen atmosphere. The temperature range was -50°C–+150°C.

The percent crystallinity of the blend was calculated from the heat of fusion, which is the area of the melting peak, using the relation

$$\text{Crystallinity (X\%)} = \left( \frac{\Delta H_f^{\text{obs}}}{\Delta H_f^0} \right) \times 100 \quad (1)$$

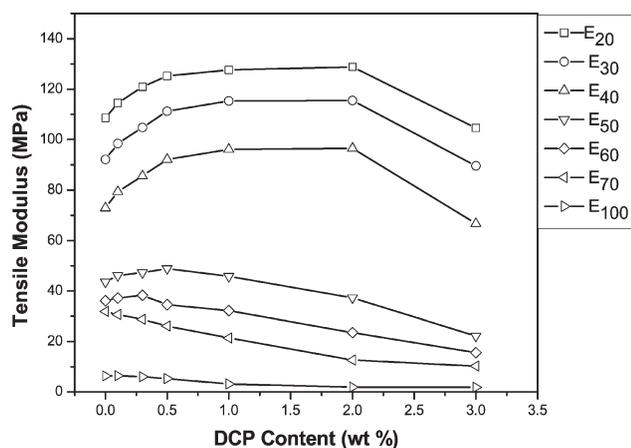
where  $\Delta H_f^0$  is the heat of fusion of a perfectly crystalline PE sample (288 J/g)<sup>35,36</sup>, and  $\Delta H_f^{\text{obs}}$  is the observed heat of fusion of the samples.

### Dynamic mechanical analysis

Dynamic mechanical analyses of the samples were carried out on a DMA 2820 (TA Instrument). The experiment was performed in tension mode at a frequency of 1.0 Hz and strain of 0.1%. The temperature range of the testing was -80°C–+100°C with a programmed heating rate of 2°C/min.

TABLE II  
Melting and Crystallization Behavior of LLDPE Phase in Crosslinked LLDPE/EMA Blends

Sample	$T_m$ (°C)	$T_c$ (°C)	Enthalpy of fusion ( $\Delta H$ ) (J/g)	Enthalpy of crystallization ( $\Delta H$ ) (J/g)	Percent crystallinity
E40	127.80	115.14	65.23	-68.41	22.7
E40/0.1	127.56	115.25	65.22	-68.12	22.7
E40/0.3	128.19	115.13	63.91	-65.34	22.2
E40/0.5	127.68	114.58	63.27	-66.10	21.9
E40/1	126.57	114.03	62.64	-64.51	21.8
E40/2	125.46	113.36	61.55	-62.46	21.4
E40/3	122.36	110.91	50.20	-53.39	17.4



**Figure 3** Effect of DCP content on the tensile modulus of LLDPE/EMA blends.

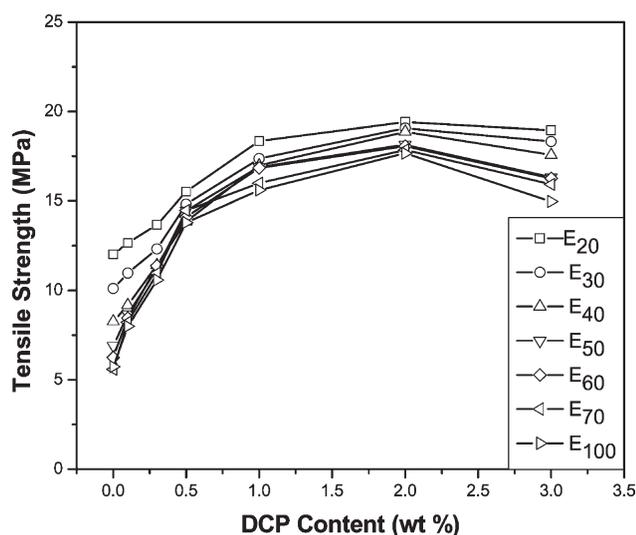
### X-ray diffraction

The X-ray diffraction (XRD) patterns of the samples were recorded with a Philips PW-1710 X-ray diffractometer (Eindhoven, The Netherlands) with crystal monochromated  $\text{CuK}\alpha$  radiation in the angular range of  $10\text{--}40^\circ$  ( $2\theta$ ) with a 40-kV operating voltage and a 20-mA current. The areas under the crystalline and amorphous portions were determined in arbitrary units, and the percentage of crystallinity ( $\chi_c$ ) was measured with the following relation:

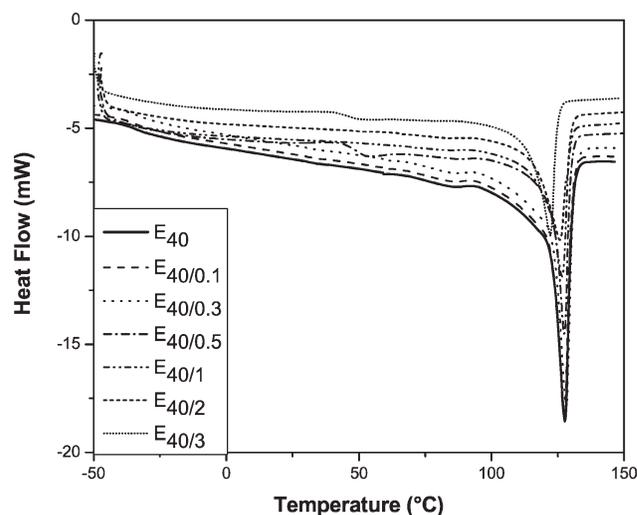
$$X_c = \frac{I_c}{I_a + I_c} \quad (2)$$

where  $I_a$  and  $I_c$  are the integrated intensities corresponding to the amorphous and crystalline phases, respectively.

The interplanar distance ( $d$ ) was calculated as follows:



**Figure 4** Effect of DCP content on the tensile strength of LLDPE/EMA blends.



**Figure 5** Effect of DCP content on the heating endotherms of LLDPE/EMA (60/40) blends.

$$d = \frac{\lambda}{2\sin\theta} \quad (3)$$

where  $\lambda$  is the wavelength of the X-ray radiation (1.79 Å).

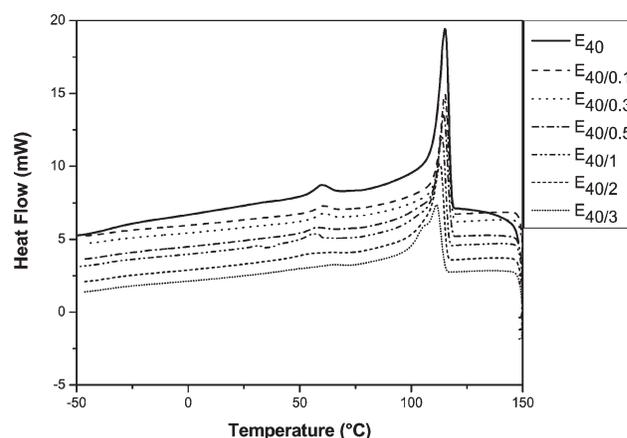
### Reprocessability studies

The reprocessing characteristics were measured by repeated mixing and molding the samples at the same processing conditions of their preparation, followed by measuring their mechanical properties. The experiments were carried out for five processing cycle for each sample.

## RESULTS AND DISCUSSION

### Gel fraction analysis

The gel content (%), which is considered as a rough estimation for crosslink density, was determined for



**Figure 6** Effect of DCP content on the cooling exotherms of LLDPE/EMA (60/40) blends.

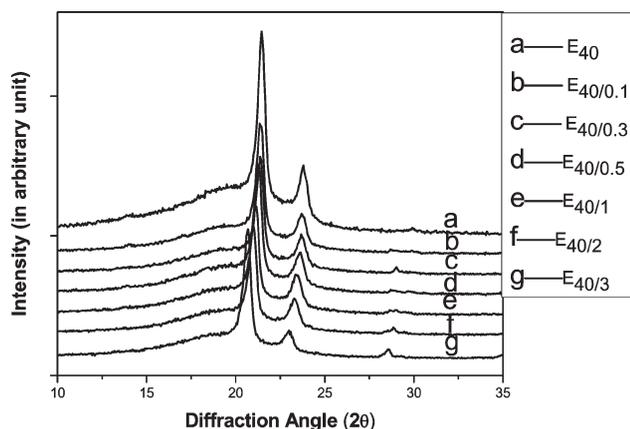
**TABLE III**  
Melting and Crystallization Behavior of EMA Phase in Crosslinked LDPE/EMA Blends

Sample	$T_c$ (°C)	Enthalpy of fusion ( $\Delta H$ ) (J/g)	Enthalpy of crystallization ( $\Delta H$ ) (J/g)	Percent crystallinity
E40	60.19	15.37	-17.89	5.4
E40/0.1	60.16	14.51	-17.56	5.0
E40/0.3	60.71	14.03	-16.63	4.9
E40/0.5	58.39	13.13	-16.02	4.6
E40/1	57.05	11.72	-12.96	4.1
E40/2	57.05	6.08	-8.11	2.1
E40/3	56.23	5.62	-7.76	1.9

the LLDPE, EMA, and LLDPE/EMA blends cross-linked by DCP. Before the gel content measurement, the solubility of pristine LLDPE and EMA and control LLDPE/EMA blend is checked in xylene at 140°C for 16 h. It is found that xylene is a good solvent for both LLDPE and EMA at 140°C.

Figure 1 shows the gel content against DCP content for crosslinked LLDPE, EMA, and LLDPE/EMA (60/40) blends. From Figure 1, it can be seen that there is a continuous increase of the gel content with DCP content up to 2 wt % after which the curve gradually levels off, i.e., an exponential variation of gel content was observed with increasing DCP content. A comparison between the gel content of LLDPE, EMA, and LLDPE/EMA blends shows that EMA having more tertiary carbon atoms in its structure is more prone to crosslinking, and hence, it has higher gel content as compared with LLDPE at the same peroxide content.<sup>37</sup> On the other hand, LLDPE/EMA blend shows an intermediate behavior.

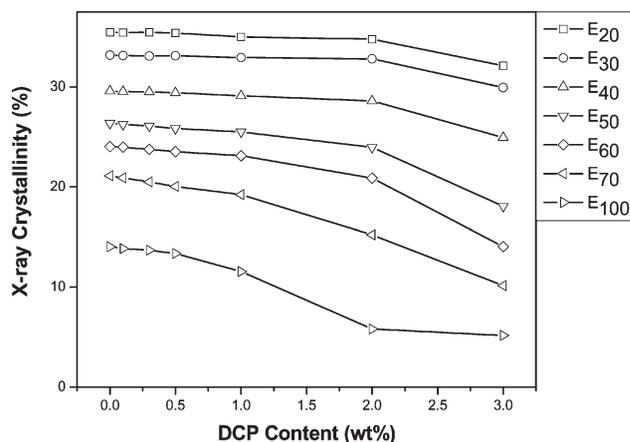
Figure 2 shows the effect of blend ratio on gel content at a constant DCP content. It can be seen that gel content increases with increase in EMA content in the blend. The increase in the EMA proportion in the blends provides number of tertiary carbon, which in turn increased its efficiency toward crosslinking at a particular DCP concentration.



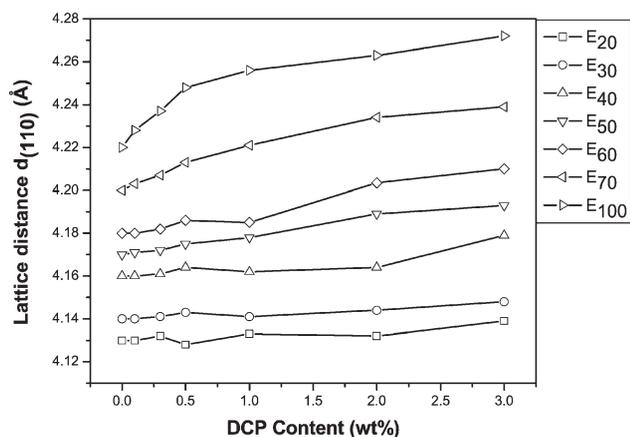
**Figure 7** X-ray scattering pattern of uncrosslinked and crosslinked LLDPE/EMA blends.

### Mechanical properties

The tensile strength, modulus, elongation at break, impact strength, and permanent set data for LLDPE/EMA (60/40) blends at different concentration of DCP are summarized in Table I. It is seen that modulus of the blend increases with increase in peroxide content up to 2 wt %; beyond that level, modulus decreases significantly. The results is likely to be ascribed to the balance of the increase in the chemical crosslinking (gel content) of EMA phase and the decrease in the degree of crystallinity of LLDPE phase with increasing DCP content. At lower loading of DCP content, decrease of crystallinity due to crosslinking is insignificant (Table II) and is dominated by the increase in gel fraction of the blend. As a result, modulus initially increases with increasing peroxide content. On the other hand, at higher DCP content, crystallinity decreases significantly and hence modulus decreases. The elongation at break initially increases and then decreases sharply with the peroxide content as compared with that of the control sample. The tensile strength is also increased with the peroxide loading. The initial increase in elongation at break and increase in tensile strength may be due to increase in the interfacial interaction, which eliminates the possibility of formation and propagation of crack at interfaces during stretching.



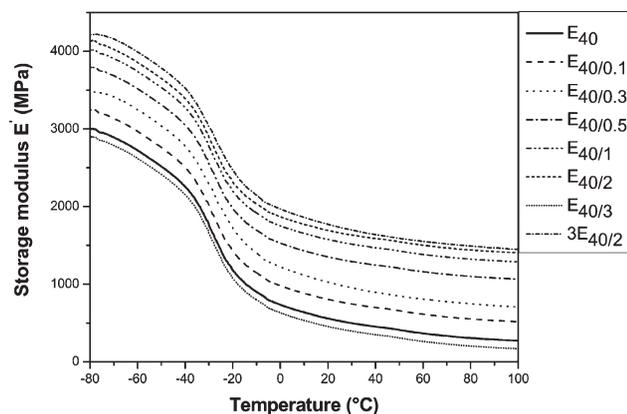
**Figure 8** Effect of DCP content on crystallinity of LLDPE/EMA blends.



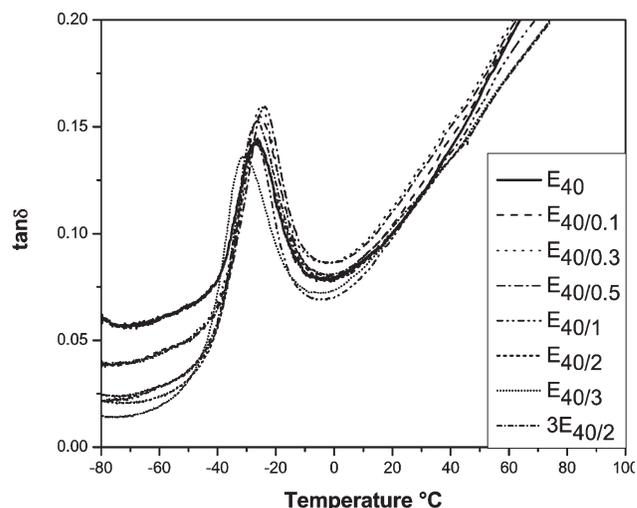
**Figure 9** Effect of DCP content on lattice distance of LLDPE/EMA blends.

The tensile impact strength increases and permanent set (%) decreases with increasing peroxide content in the blend. This is due to the increase of gel fraction with increasing peroxide content that increases the elastic properties of the blend. The maximum mechanical property is observed for 2 wt % of peroxide. Addition of 3 wt % of compatibilizer (LLDPE-g-MA) along with 2 wt % of DCP leads to further increase in mechanical properties of the blends.

Figures 3 and 4 show the effect of DCP content on the mechanical properties of the blend at various compositions of LLDPE and EMA. It can be observed that tensile modulus of the blend increases with increasing peroxide content up to 2 wt % (Fig. 3) for blends having lower content of EMA (E20, E30, and E40), where loss of crystallinity due to crosslinking is insignificant (Fig. 8) and is dominated by the increase in gel fraction of the blend. However, for blends having higher concentration of EMA, tensile modulus decreases gradually with increasing peroxide content in the blend. This is due to the decrease of crystallinity of the blend with increasing peroxide content for blends having higher content of EMA.



**Figure 10** Storage modulus curves for crosslinked LLDPE/EMA blends.



**Figure 11** Tan  $\delta$  curves for crosslinked LLDPE/EMA blends.

On the other hand, tensile strength of the blend increases with increasing peroxide content in the blend for all composition (Fig. 4). However, increase of tensile strength is more prominent for blends having higher weight percentage of EMA, since the blends consisting of EMA with higher crosslink density (with higher DCP contents) have higher resistance to tensile deformation than those of EMA with lower DCP contents.

### Differential scanning calorimetry

Figures 5 and 6 show the DSC thermograms of crosslinked LLDPE/EMA (60/40) blends. The results obtained from the DSC scans are summarized in Tables II and III. From the data, it is clear that crosslinking decreases crystallinity. The crosslinking provides some hindrance to the ordered arrangement of the polymer chains resulting in lower crystalline value in crosslinked polymer.<sup>38,39</sup> However, from Table III, it is clear that percent crystallinity of the LLDPE phase is not so much affected at lower loading level of DCP. The initial crystallinity of LLDPE phase (22.65%), for control blend, is slightly reduced

**TABLE IV**  
DMA Results:  $\beta$ -Transition Temperature and Tan  $\delta_{\max}$  Values

Sample code	$\beta$ -transition temperature (°C)	Tan $\delta_{\max}$
E40	-26.40	0.144
E40/0.1	-25.92	0.156
E40/0.3	-24.84	0.160
E40/0.5	-23.76	0.160
E40/1	-26.99	0.142
E40/2	-27.48	0.143
E40/3	-31.34	0.136
3E40/2	-25.81	0.153

TABLE V  
Reprocessability Studies of Crosslinked LLDPE/EMA Blends

Sample code	Original		First cycle		Second cycle		Third cycle		Fourth cycle		Fifth cycle	
	TS (MPa)	EB (%)	TS (MPa)	EB (%)	TS (MPa)	EB (%)	TS (MPa)	EB (%)	TS (MPa)	EB (%)	TS (MPa)	EB (%)
E40	8.3	216	8.2	206	8.2	202	8.2	198	8.2	190	8.1	176
E40/0.1	9.2	346	9.2	329	9.2	326	9.1	311	9.1	285	9.0	267
E40/0.3	11.4	494	11.4	482	11.4	476	11.4	463	11.3	456	11.3	458
E40/0.5	13.9	474	10.2	327	9.4	285	8.2	229	7.7	189	7.1	168
E50/0.3	11.2	596	9.4	550	8.0	489	7.16	464	6.86	439	6.25	419

TS: Tensile Strength; EB: Elongation at break.

to 21.37% at 2 wt % loading of DCP. Beyond that level, crystallinity decreases drastically. On the other hand, for EMA phase, crystallinity decreases significantly after 1-wt % loading of DCP (Table III).

### WAXD studies

Figure 7 shows the XRD diffraction pattern of uncrosslinked and crosslinked LLDPE/EMA (60/40) blends. From Figure 7, it can be seen that with increasing DCP concentration, (110) and (200) crystalline peaks shifted toward lower  $2\theta$  values suggesting an increase of the lattice distances of the orthorhombic unit cell of polyethylene. The effect of DCP content on crystallinity and lattice distance of the blends with various compositions of LLDPE and EMA was also studied, and the observations are given in Figures 8 and 9. It is seen that percent crystallinity and crystalline structure of the blends remain unchanged (more or less same), up to 2 wt % of DCP, for blends having lower wt % of EMA (E20, E30, and E40). However, for blends having higher concentration of EMA (E50, E60, E70, and E100), increasing DCP content reduces the crystallinity and increases the lattice distance. Moly et al. also observed similar behavior for LLDPE/EVA blends.<sup>40</sup>

From DSC and XRD studies, it can be concluded that with increasing DCP content, decrease of crystallinity is more significant in EMA as compared with LLDPE. This indicates that DCP crosslinking is more effective in EMA than in LLDPE.

### Dynamic mechanical analysis

Figures 10 and 11 show the storage modulus and  $\tan \delta$  versus temperature curves for LLDPE/EMA (60/40) blends for different DCP content. The results obtained from the  $\tan \delta$  versus temperature curves are summarized in Table IV. It is seen that initially with increasing DCP content, storage modulus as well as  $\tan \delta_{\max}$  value increases and  $\beta$ -transition peak of  $\tan \delta$  curves shifted to higher temperature, reaches a maximum, and then decreases. This could be due to the fact that at low-DCP content, crystal-

line phases of LLDPE does not affected significantly (Table II) rather crosslinking takes place mostly in the amorphous phase. This restricts the molecular mobility of the polymer chains and hence storage modulus increases and  $\beta$ -transition peak of  $\tan \delta$  curves shifted to higher temperature. On the other hand, percent crystallinity of LLDPE crystalline phases decreases significantly at higher DCP content (Table II). As a result, storage modulus decreases and  $\beta$ -transition peak of  $\tan \delta$  curves shifted to lower temperature, see Figure 11.

### Reprocessability studies

To establish the thermoplastic elastomeric behavior, reprocessability studies at five processing cycles were carried out. Reprocessability studies were performed by repeated mixing and molding the samples and evaluating their mechanical properties. The results are given in Table V. It can be seen that even in the fifth processing cycle, mechanical property changes marginally for blends having 0.1- and 0.3-wt % DCP content. Beyond that level, DCP samples are not good for additional processing. They are weak and show melt fracture and deteriorations of properties.

### CONCLUSIONS

Blends of LLDPE and EMA crosslinked by DCP were investigated for crosslink density, mechanical, thermal, wide-angle X-ray diffraction (WAXD), dynamic mechanical properties, and reprocessabilities. Measurement of the gel content indicated an increase in the degree of crosslinking by addition of DCP content, and EMA is more prone to crosslinking than LLDPE. DSC and WAXD studies reveal that at lower DCP content, crystallinity of the LLDPE phase does not significantly affected by crosslinking process. However, percent crystallinity of LLDPE phase decreases significantly at higher DCP content. As a result, mechanical properties of the blends increase, and  $\beta$ -transition peak for  $\tan \delta$  shifted toward higher temperature at low-DCP

content; an opposite trend is observed at high-DCP concentration. The highest level of mechanical properties is observed for 2 wt % DCP. Addition of 3 wt % of LLDPE-*g*-MA as a compatibilizer to the cross-linked blend further increases the mechanical properties. From reprocessability studies, we found that blends with 0.1- and 0.3-wt % DCP can easily be reprocessed.

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