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Compatibility Study of Blends of Acrylic Rubber (ACM), Poly(ethyleneterephalate) (PET), and Liquid Crystalline Polymer (LCP)

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The ternary blends of acrylate rubber (ACM), poly(ethyleneterephalate) (PET), and liquid crystalline polymer (LCP) were prepared by varying the amount of LCP, but fixing the ratio of ACM and PET using melt mixing procedure. The compatibility behavior of these blends was investigated with infrared spectroscopy (IR), differential scanning calorimetry (DSC), and dynamic mechanical analyzer (DMA). The IR results revealed the significant interaction between the blend components. Glass transition temperature (T_g) and melting temperature (T_m) of the blends were affected depending on the LCP weight percent in the ACM/PET, respectively. This further suggests the strong interfacial interactions between the blend components. In the presence of ACM, the nucleating effect of LCP was more pronounced for the PET. The thermogravimetric (TGA) study shows the improved thermal stability of the blends.

Keywords acrylic rubber, poly(ethyleneterephalate), liquid crystalline polymer, crystallinity, compatibility, glass transition temperature

Introduction

Polymer blends have recently attracted both scientific and industrial interest. They have been found to be the main route for preparation of better, cost effective plastics having the desired technological properties especially for engineering purposes. Thermotropic liquid crystalline polymers (LCPs) with rigid molecular backbones exhibit unique physical, mechanical, rheological and thermal properties (1–5). The use of LCPs as the minor phase of polymer blends has attracted considerable attention in the past two decades. This is because LCPs can improve the processability and mechanical properties of thermoplastics (6–8). LCPs tend to deform into elongated fine fibrils under the appropriate processing conditions. The LCP fibrils reinforce thermoplastic matrix effectively, producing the so-called *in situ* polymer composites. Reinforcing effect arises from improved compatibility between the matrix and dispersed LCP phase (9–12). However, the mechanical properties of LCP/thermoplastic blends do not meet expectations,

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mainly because of poor compatibility between the dispersed LCP phase and thermoplastic matrix. To increase their compatibility, block or graft copolymers can be introduced in the LCP/thermoplastic blends to promote the interfacial interactions. Compatibilization can also be promoted by molecular interchange reactions between blend components, such as transesterification in ester-containing polymer pairs. Kotliar (13) reviewed the interchange reactions involving the condensation polyesters at high temperatures, in which the chains are terminated by hydroxyl or carboxyl groups. Lee and coworkers investigated the miscibility and interfacial adhesion of the PET/LCP blends (14, 15). The LCP employed was a semi-flexible poly(ethyleneterephthalate-hydroxybenzoic acid) p(ET/HBA). In this case, a degree of compatibilization manifests itself in the form of good bonding between the components.

Highly aromatic ester based main chain LCPs, such as p(HBA/HNA), are useful due to their high strength and modulus, and also processability. A limiting factor in the use of these Ar-LCPs with commercially available thermoplastic polymers is their incompatibility, as expressed by poor interfacial adhesion (16, 17). This can be overcome by using a compatibilizer or incorporating a reactive third component. In view of the above interest, we report our observation on the blends of acrylate rubber, poly(ethyleneterephthalate), and aromatic liquid crystalline polymer, where the carboxyl end groups of PET and LCP are expected to react with the reactive epoxy groups of ACM to produce necessary graft copolymer at the interface during the melt blending operation. The influence of the interfacial reaction on the thermal and dynamic mechanical properties is highlighted.

Experimental

Materials

Acrylic rubber (ACM) NIPOL AR 51 was obtained from Nippon Zeon Co. Ltd., Tokyo, Japan. It was reported to have an epoxy cure site and made from ethylacrylate monomer. Poly(ethyleneterephthalate) (PET) was RELPET-G 5801 supplied by Reliance Industries, India. Thermotropic liquid crystalline polymer (TLCP) was Vectra A 950 from Ticona (USA). The LCP has the comonomer composition of 75 mol% of hydroxybenzoic acid (HBA) and 25 mol% of hydroxynaphthoic acid (HNA). Table 1 shows the structure and the properties of the polymers used.

Preparation of Samples

Blends were prepared by melt mixing at 300°C using a sigma internal mixer with a rotor speed of 80 rpm, the amount of time of polymer melt in the mixing chamber was in the order of 2–3 min. (ACM/PET)/LCP blends with weight ratio 100/0, 75/25, 50/50, 25/75, and 0/100 were prepared. In all the blends, the ACM/PET ratio (60/40) was kept constant. The blends were molded at 300°C, at a pressure of 20 MP in a compression molding press for 10 min, unless otherwise stated.

IR Spectroscopy

Fourier transform infrared spectroscopy (FTIR) experiments were done on ACM, PET, LCP and their blends, using a NEXUS 870 FTIR (Thermo Nicolet) in a low humidity atmosphere at a room temperature from 400–4000 cm^{-1} . A total of 32 scans were averaged with a resolution of 4 cm^{-1} .

Table 1
Structure and the properties of polymers

Polymer	Structure	T _m (°C)
Acrylic rubber (ACM)	$\left(\text{CH}_2 - \underset{\begin{array}{c} \\ \text{C}=\text{O} \\ \\ \text{OCH}_2\text{CH}_3 \end{array}}{\text{CH}} \right)_n \left(\text{CH} - \underset{\begin{array}{c} \\ \text{R} - \text{CH} - \text{CH}_2 \\ \quad \\ \quad \quad \text{O} \end{array}}{\text{CH}} \right)_m$	—
Polyethyleneterephthalate (PET)	$\text{HO} - \left[\text{CH}_2 - \text{O} - \text{C}(=\text{O}) - \text{C}_6\text{H}_4 - \text{C}(=\text{O}) - \text{O} \right]_n - \text{H}$	247
Liquid crystalline polymer (LCP)	$\text{H} - \left[\text{O} - \text{C}_6\text{H}_4 - \text{C}(=\text{O}) \right]_m \left[\text{O} - \text{C}_6\text{H}_4 - \text{C}_6\text{H}_4 - \text{C}(=\text{O}) \right]_n - \text{O} - \text{H}$	278

Differential Scanning Calorimetry

DSC measurements were carried out using a Perkin-Elmer PYRIS Diamond DSC instrument. The samples (10 mg), sealed under aluminum pans, were scanned in the temperature range of 50–300°C. The heating and cooling rate was 10°C min⁻¹ under the nitrogen atmosphere with a flow rate of 40 ml min⁻¹. The percent crystallinity of PET component is calculated assuming a heat of fusion of PET crystals of 140 Jg⁻¹ (18). The results reported here were the average of three samples. T_m and ΔH_c were reproducible within ± 1°C and ± 5%, respectively.

Dynamic Mechanical Analysis

Dynamic mechanical properties of the blends were analyzed using a TA Instrument DMA 2980 model in a bending single cantilever mode. The samples were subjected to a sinusoidal displacement of 0.1% strain at a frequency of 1 Hz from -50°C to +250°C and a heating rate of 10°C min⁻¹. The storage modulus (E'), loss modulus (E''), and loss tangent (tan δ) were measured for each sample in this temperature range.

Scanning Electron Microscopy

Morphology of the blends was studied using a Scanning Electron Microscope (SEM) (JSM-5800 of JEOL Co.), after an auto sputter coating of the sample surface with gold at a 0° tilt angle.

Wide Angle X-ray Diffraction Measurements

X-ray diffraction was performed with a PW 1840 X-ray Diffractometer with a copper target (Cu-Kα) at a scanning rate of 0.050 2θ/sec., chart speed 10 mm/2θ, range 5000 c/s, and a slit of 0.2 mm, applying 40 kV, 20 mA, to assess the change of crystallinity of the blends as a function of blend ratio.

Thermogravimetric Analysis

Thermal stability of the blends was studied using a DuPont TGA-2100 thermogravimetric analyzer in the presence of air from 50–700°C, with a heating rate of 10°C min⁻¹.

Results and Discussion

IR Analysis

The transesterification reactions between the three esters are not unambiguous since all the reactants and the products belong to the same class of compounds. The carbonyl stretching frequencies of ACM (at 1727 cm⁻¹), PET (at 1722 cm⁻¹), and LCP (at 1733 cm⁻¹) are very close to each other and overlap to form a single band in the same region (Figure 1). Hence, the detection of C=O stretching frequency of the mixed ester produced by trans-esterification reaction is also impossible due to the overlapping the bands in the same region. Preparation of reactive blends of PET with LCP is reported by many authors (19–21). They have reported that the PET and LCP readily undergo trans-esterification reactions, and it is well documented that PET can also be transesterified with polyacrylates (22, 23), thus the ternary blends studied here are able to undergo transesterification to form compatible ternary blends.

The carboxy (–COOH) end groups of PET and LCP can take part in various Lewis acid-base and nucleophilic addition type reactions, with various reactive polymers having suitable functional groups at their chain. The acrylate rubber used in this study contains epoxy groups as the reactive curing site and hence, it can react with PET and LCP to produce the graft polymer. The IR spectra of (ACM/PET)/LCP (50/50 w/w), reacted at 300°C for 5 and 10 min are shown in Figure 2. It is clear from the figure that the epoxy peak intensity at 908 cm⁻¹ decreases progressively with the reaction time. This suggests the reduction of the concentration of epoxy groups of ACM as the reaction proceeds. From the above observations, the probable reaction mechanism, which can be predicted, is shown in Scheme 1. The other peaks at 727 and 789 cm⁻¹ are due to the

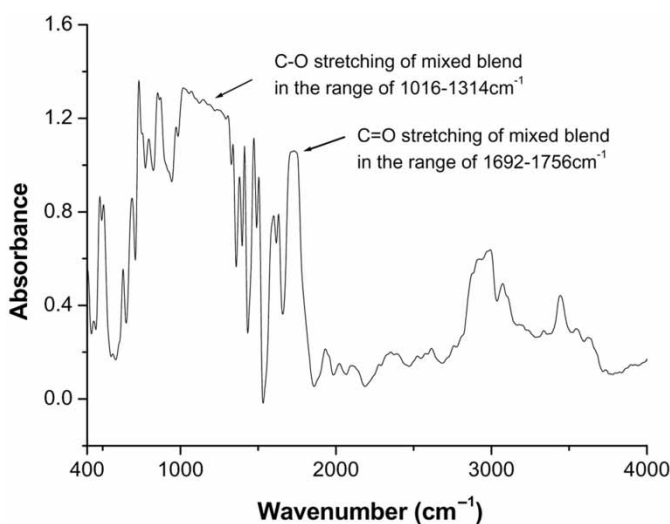


Figure 1. IR spectra of 50/50 weight ratio of (ACM/PET)/LCP blend.

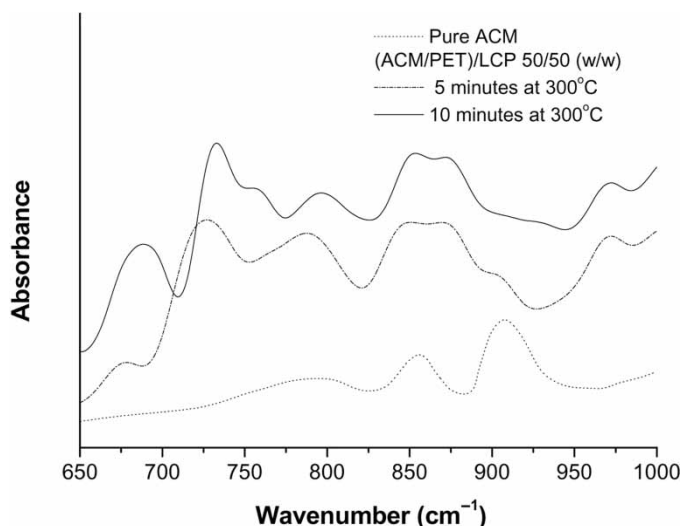
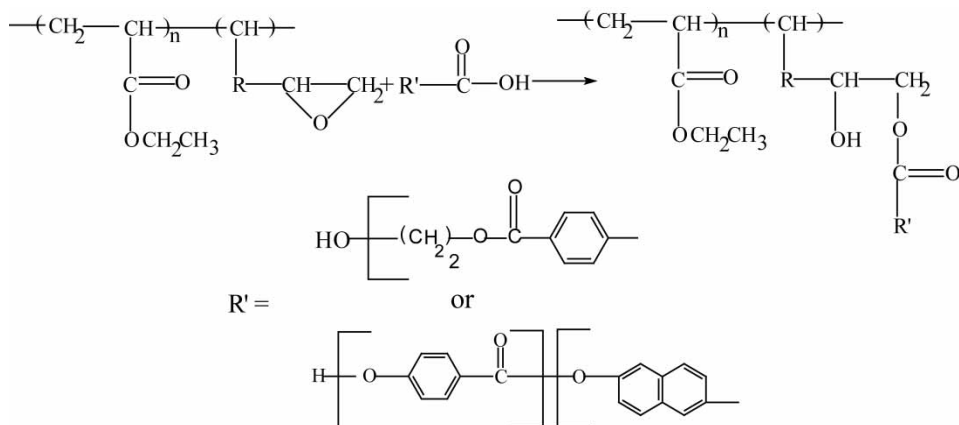


Figure 2. IR spectra of 50/50 weight ratio of (ACM/PET)/LCP blend reacted for different times.

coupled vibration of carbonyl and terephthalate ring out of plane and C–H bending of terephthalate unit, respectively, shifted towards higher frequency side with increasing reaction time. In addition, the IR spectra of 50 wt% LCP blend reacted for 10 min shows a new absorbance at about 758 cm^{-1} ; these may be a consequence of the reactions between the epoxy groups and end groups of LCP and PET or transesterification reactions.

Differential Scanning Calorimetry

DSC heating and cooling curves of the LCP are shown in Figure 3. The heating curve of the LCP reveals a transition at 102°C , corresponds to the glass transition of the LCP “presumably corresponds to the material changing from a glassy state to a mobile nematic state, rather than a rubbery phase”. Wissburn and Yoon (24) also reported a



Scheme 1. Reaction mechanism between the epoxy groups of ACM and carboxyl end groups of PET and LCP.

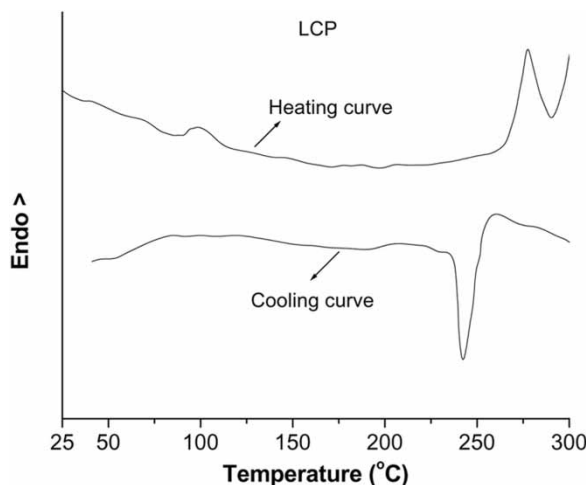


Figure 3. DSC heating and cooling curves of pure LCP.

transition around 100°C. The other transition appears at 278°C and corresponds to the solid crystal-to-nematic melt transition (K → N) (T_m) of the LCP. The cooling curve of the LCP shows crystallization temperature at 241°C.

The results of the DSC heating and cooling scans of blends are shown in Figures 4 and 5, respectively, and the corresponding parameters, cold crystallization temperature (T_{cc}), melting temperature (T_m), crystallization temperature (T_c), enthalpy of crystallization (ΔH_c) and percent crystallinity (X_c) are represented in Table 2. In order to give the same thermal history, all the samples were heated up to 300°C and held at this temperature for 5 min followed by cooling to room temperature. It has been well recognized that blending with LCP can significantly increase the PET crystallization rate. The T_{cc} of PET in PET/ACM blend is observed at 181°C, which is shifted to 190°C with the

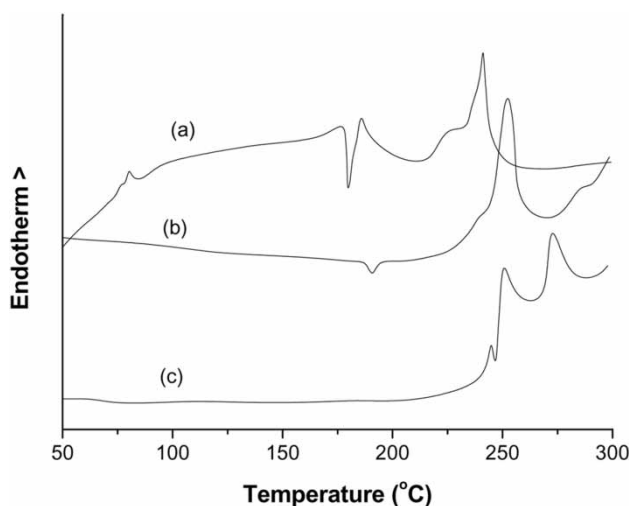


Figure 4. DSC heating curves of (ACM/PET)/LCP blends: (a) 100/0; (b) 75/25; (c) 50/50.

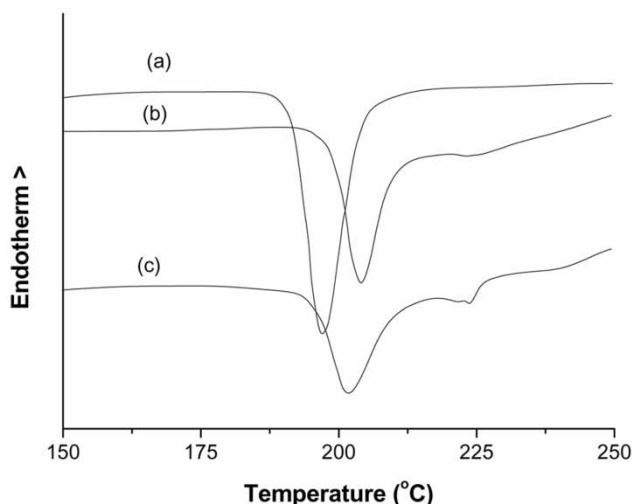


Figure 5. DSC cooling curves of (ACM/PET)/LCP blends: (a) 100/0; (b) 75/25; (c) 50/50.

addition of 25 wt% of LCP and further addition of LCP, the PET did not show any cold crystallization during the DSC heating process, which means that the PET crystallinity has already reached its highest achievable crystallinity after cooling to low temperature and no additional crystallization occurs during the DSC heating process. From Figure 4, a typical double melting behavior characteristic for the PET matrix can be observed, illustrating that the recrystallization process of thin, less perfect crystals formed during the isothermal compression molding at 300°C. The ACM/PET blend shows a melting endotherm of PET phase, T_m , at 241°C, whereas for LCP, it occurs at about 280°C. With the addition of LCP to ACM/PET, there is scarcely a variation of 10°C in the T_m of PET when compared to PET in ACM/PET. Such a type of melting behavior can be attributed to the difference in crystal size of the PET (increasing crystallinity) since the melting curves are sensitive to the crystalline orientation and size distribution. It has been noted that ACM/PET blends have a broad distribution of small and more defective crystals of PET, while the LCP containing blends have a narrow distribution of large crystals with higher perfection. This is reflected in the DSC heating cycles. Figure 5 shows the DSC traces of blends when cooled from the melt state. The crystallization peaks in the 25 wt% of LCP containing blends at 203.8°C corresponds to the PET, and

Table 2
Thermal properties of ACM/PET/LCP blends

(ACM/PET)/LCP	Heating (Endotherm)		Cooling (Exotherm)		
	T_{cc}	T_m	T_c	ΔH_c	X_c (%)
100/0	181	241	197	12.6	9
75/25	190	252	204	16.3	12
50/50	—	250	202	14.4	10

above this temperature small exotherm corresponds to the crystallization of LCP. Evidently, with the addition of LCP, the crystallization of the PET occurred at higher temperatures, suggesting that there is a nucleation effect of the LCP component on PET crystallization. This seems to be a reversal of results from other studies on blends of PET with LCP (25, 26), where the depression of PET crystallization temperature upon cooling in the presence of LCP component was found. This suggests that the LCP component in the presence of ACM imposes a more efficient nucleation on the crystallization of PET. The enthalpy of crystallization of the PET component (ΔH_c), normalized to PET content, shows a nonlinear ΔH_c , during a slow cool, indicative of a transesterification of the PET ester groups (22).

The crystallinity (X_c) of the PET component, as determined from the enthalpies of crystallization of PET, normalized for fractional content, significantly increased with the addition of LCP. This indicates that in presence of acrylate rubber, the LCP enhances the formation and nucleation of PET crystals. This is another indication of strong interface interactions between the components, which modify crystal perfection and purity.

Dynamic Mechanical Analysis

The loss tangent ($\tan \delta$) and storage modulus (E') of ACM/PET, LCP and their blends as a function of temperature and dependence of T_g 's on LCP weight percent are shown from Figures 6 through 8. The glass transition temperature (T_g) is selected as the peak position of the $\tan \delta$ when plotted against temperature. In Figure 6a, the ACM/PET blend exhibits transitions at 10.9°C and 101°C, which can be attributed to be glass transition temperature of ACM and PET, respectively. The T_g value of PET with the DSC is found at 80.1°C. This difference in T_g values between the two methods is caused by the difference in sensitivity of the techniques towards glass transitions and also mentioned in literature (27). The T_g of pure LCP is detected at 132°C. In ternary blend, the higher and lower $\tan \delta$ peak corresponding to the T_g of PET and LCP completely overlapped and shows a single peak in between the T_g 's of pure PET and LCP (Figure 6b) and also the T_g of ACM and PET gradually shifted to higher temperature side with the increasing LCP content (Figure 7). This indicates a strong interaction between the phases of ACM, PET and LCP, probably through the transesterification reaction between the components at 300°C. It is plausible that the polymer blend suitably reacts to form block copolymer and finally random copolymer (22). Formation of such products at the interface may enhance the miscibility of the blend components thereby affecting the glass transition temperature of the respective components. Moreover, from Figure 6, it is observed that the LCP containing blends shows a narrowing of the glass transition range measured from the half peak width after melt blending. This indicates that the relaxation time distribution narrowed. This is a characteristic feature of compatible blends because compatible segments lead to a narrowing of the transition (28). The height of $\tan \delta$ peak at 10 and 120°C is found to decrease with increase in the LCP content. The relative decrease in the height of $\tan \delta$ peak related to the increase in the extent of crystallinity in the polymers, since the transition behavior is associated with the local mobility of the polymer chains in the amorphous regions of the polymer. Upon increasing the temperature, the PET/ACM blend shows a crystallization transition of PET phase at about 175°C, whereas pure LCP shows it at about 220°C, which is also reflected in the increase of storage modulus of the samples around 175°C and 220°C, respectively, for the PET and LCP (Figure 8). As expected, the 25 wt% of LCP containing

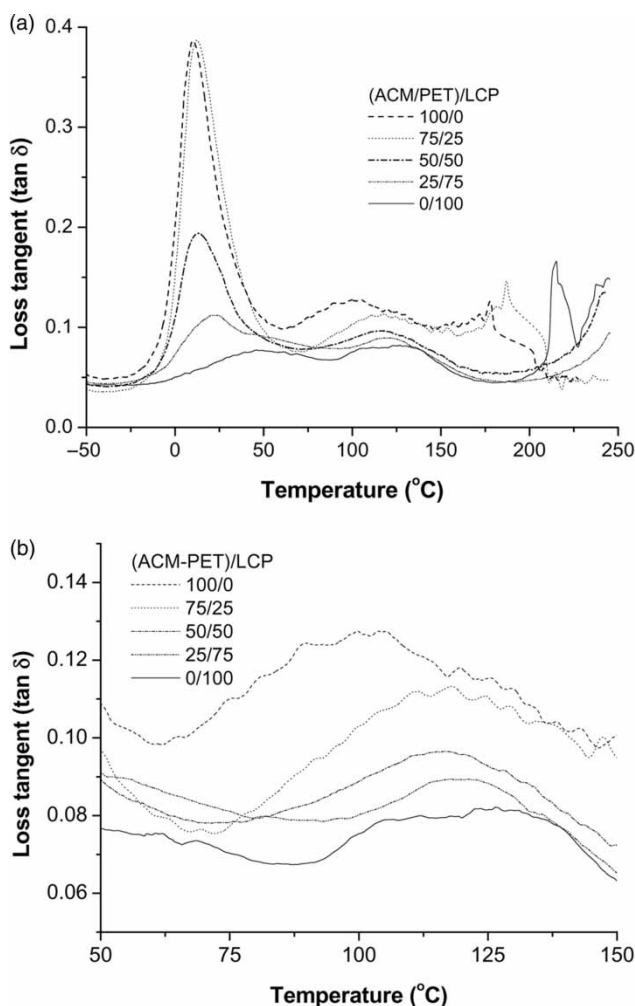


Figure 6. Loss tangent ($\tan \delta$) as a function of temperature at 1 Hz frequency in the temperature range of (a) -50 to $+250^\circ\text{C}$, (b) 50 to 150°C .

blend shows the PET crystallization temperature at a higher temperature side. Then again, the blends with the higher percentage of LCP could not show the PET crystallization transition. These results are in agreement with the studies using DSC. The storage modulus of the blends against LCP weight percent at three temperatures is shown in Figure 9. At three temperatures (i.e., 30, 100 and 200°C), the storage modulus of blends is found to increase with an increase in the LCP content. At 75 wt% of LCP, the blend shows higher E' than the pure LCP. Such a type of improvement in E' is attributed to be due to the change in morphology and also an increase of interaction between the blend components. A similar observation has also been made by Weiss et al., (29) for different pairs of LCP thermoplastics. The dynamic fracture surface of the ternary blends, studied using a scanning electron microscope (SEM), are shown in Figure 10. Figure 10a is a micrograph of 25 wt% of LCP containing blend shows the fibril nature of the LCP domains in the predominant ACM/PET matrix face. These fibrils are very short and stubby. However, at a higher

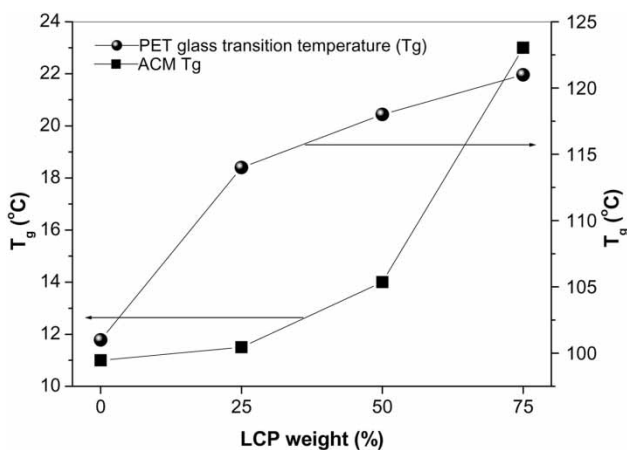


Figure 7. Dependence of T_g 's of ACM and PET as a function of LCP weight percent.

level of LCP (75 wt%), the micrograph (Figure 10b) shows a higher number of long and thin fibrils of the LCP domains, and it also shows a strong adhesion at the interface between LCP fibrils and ACM/PET. This suggests that the compatibilized blends leads to a strong adhesion at the interface by forming graft block copolymers. Such copolymer molecules obviously exist in the interfacial region, and contribute significantly to a reduction in the interfacial tension and enhance the adhesion between the phases. This is also reflected in the increase in the storage modulus of the blends.

Wide Angle X-ray Diffraction

Figure 11 shows wide-angle X-ray diffraction patterns of ACM/PET, LCP, and their blends. The diffractograms of ACM/PET consisted of six peaks, while those of LCP consisted of two broad peaks. The diffraction pattern of the blends whose major phase

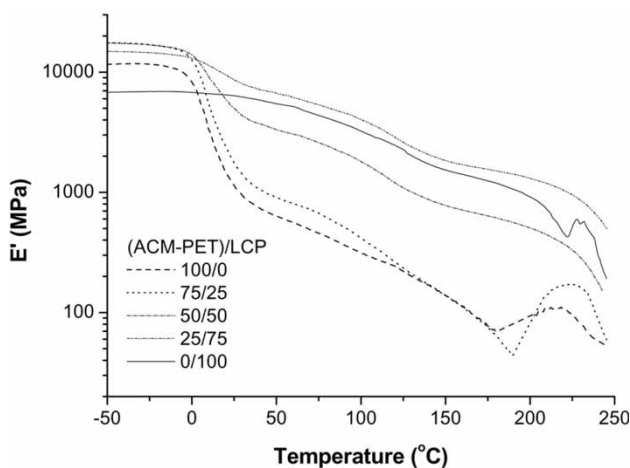


Figure 8. Variation of storage modulus (E') as a function of temperature at 1 Hz frequency.

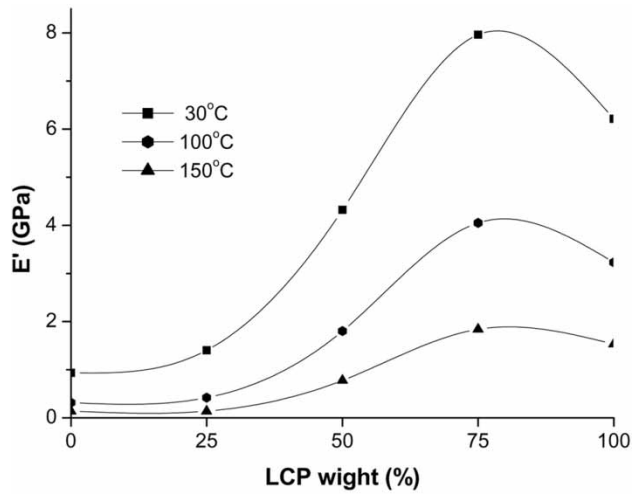


Figure 9. Storage modulus of ACM/PET/LCP blends against LCP weight percent (data obtained from Figure 6).

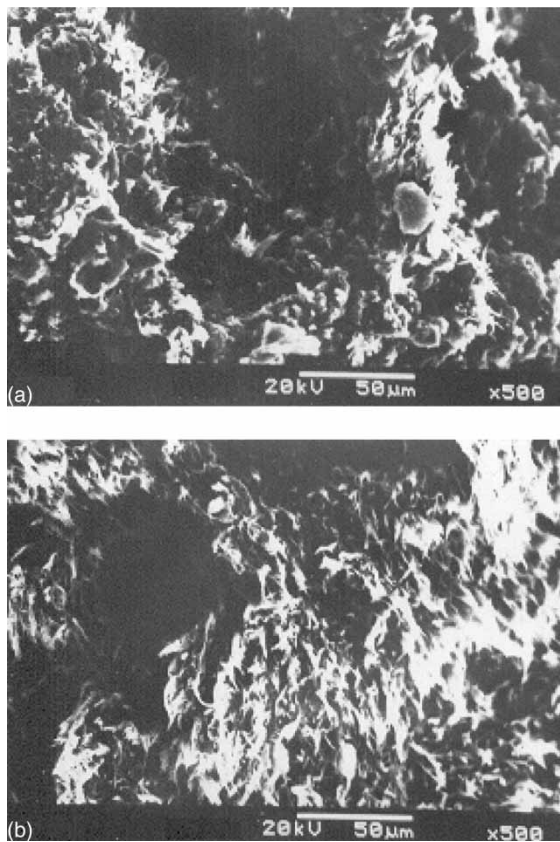


Figure 10. SEM picture of (ACM/PET)/LCP blends: (a) 75/25; (b) 25/75.

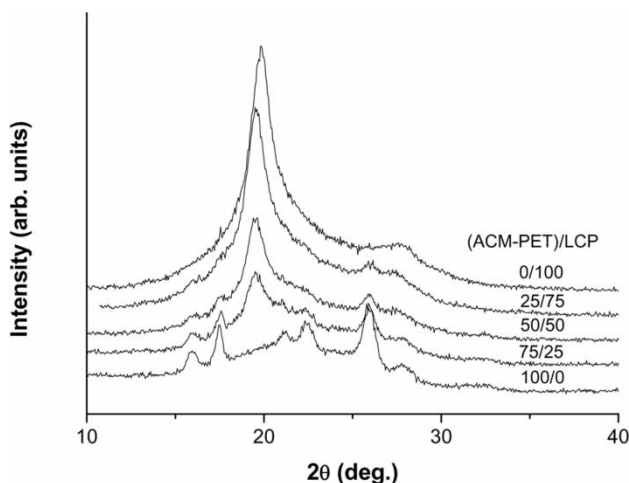


Figure 11. Wide angle X-ray diffractograms of ACM/PET/LCP blends.

is PET exhibits all the reflection of the PET component. At higher levels of LCP (50 wt% and 75 wt%) the peaks of PET at $2\theta \approx 17.5$, 21.2 , and 22.4° are overlapped with the broad peak of LCP. In ACM/PET/LCP blends the broad peak at about $2\theta \approx 19^\circ$ increased in intensity with increasing LCP content because LCP might acts as a reinforcing agent in the blends (30). The ACM/PET/LCP blends crystallinity mainly depends on the PET and LCP phases. The degree of relative percentage of crystallinity of the blends was given by the X-ray diffraction method from the following equation (31):

$$X (\%) = C/A + C \times 100$$

where A is area under the amorphous halo and C is area under the crystalline phase. The 75 wt% of LCP blend shows 26% crystallinity and followed by 25 and 50 wt% of LCP blends shows 23 and 20% crystallinity, respectively, whereas, for the ACM/PET blend the percent crystallinity is 15. This further suggests that the LCP acts as a nucleating agent for the PET phase in the blend. However, it is difficult to calculate the crystallinity of the PET phase alone in the blend using XRD method because of the overlap of respective peaks of PET and LCP.

Thermogravimetric Analysis

In order to see the effect of LCP on the thermal stability of ACM/PET, the thermogravimetric analysis was conducted. The TGA curves of the blends of various compositions, obtained at a heating rate of $10^\circ\text{C min}^{-1}$ in air are shown in Figure 12. In order to avoid any ambiguity, Wrights (32) approach has been used to define the degradation temperature. The degradation temperature has been defined as the temperature at which the polymer lost 1% of its weight during the non-isothermal heating scan. From the thermograms it is observed that the ACM/PET shows one-step degradation at 329°C , whereas ACM/PET/LCP blends shows a two-step degradation, the second step corresponds to the degradation of LCP. The ACM/PET undergoes extensive decomposition between 329 – 372°C . This means that the ACM/PET is stable till 329°C , beyond which

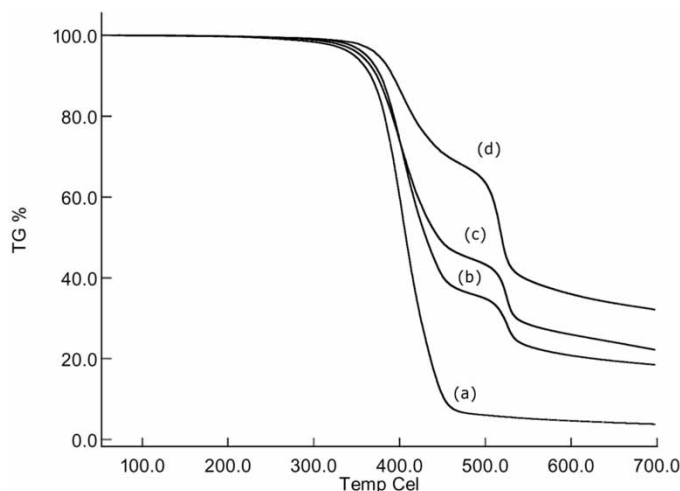


Figure 12. TGA curves of (ACM/PET)/LCP blends: (a) 100/0; (b) 75/25; (c) 50/50; (d) 25/75.

degradation begins and then intensifies. The thermal stability of the ACM/PET increased remarkably by the addition of LCP. The degradation temperatures of ACM/PET is observed at 346, 340, and 363°C for 25, 50, and 75 wt% of LCP, respectively. This type of improvement in thermal stability of ACM/PET by the addition of LCP is ascribed to be due to increasing crystallinity with the increment of LCP content. It is known that crystalline polymers are thermally more stable than their amorphous counterpart due to the energy input required to overcome both strong intermolecular and intramolecular forces. The marginal decrease in thermal stability at 50 wt% of LCP content is due to a drop in the percentage of crystallinity. In order to verify whether there is any weight loss of blends during the transesterification reaction at 300°C, the 50 wt% of LCP containing blend was isothermally heated at 300°C for 20 min. However, the thermogram (Figure 13) did not suggest a major degradation of the

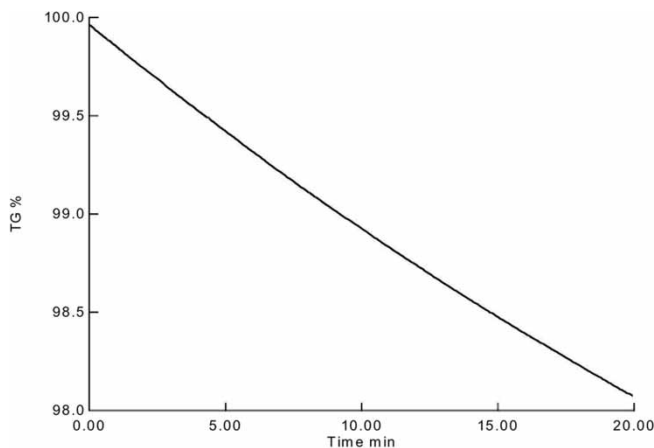


Figure 13. TGA curve of 50 wt% of LCP blend heated isothermally at 300°C for 20 min.

sample, it showed a minor weight loss (1.9%). From this, it can be concluded that the degradation of the sample is negligible during the transesterification reaction.

Conclusion

The compatibility behavior of ACM, PET, and LCP blends was investigated using infrared spectroscopy, differential scanning calorimetry, and dynamic mechanical analyzer. The IR spectroscopy analysis shows reduction in the intensity of the peak corresponding to epoxy groups of ACM with increasing heating time at 300°C, this suggests that chemical reaction between the epoxy and the end groups of PET and LCP. It is also reasonable to say that there is a transesterification reaction between the blend components at 300°C in view of the fact that there is similar functionality in all polymers used in this study. However, the IR spectroscopy failed to prove the transesterification reaction between the blend components. On the other hand, these interactions are further substantiated by the DSC and DMA results by a change in the melting temperatures and glass transition temperatures depending on the LCP content. In the presence of ACM, the nucleating effect of LCP was more pronounced for the PET phase.

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