

Thermogravimetric and dynamic mechanical analysis of LLDPE/EMA blends

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Abstract The thermal stability of linear low density polyethylene (LLDPE)/ethylene methyl acrylate (EMA) blends was studied using thermogravimetry. The blend ratio as well as the presence of compatibilizer has significant effect on thermal stability of the blends. The compatibilization of the blends using LLDPE-g-MA has increased the degradation temperature. Phase morphology was found to be one of the most decisive factors that affected the thermal stability of both uncompatibilized and compatibilized blends. Dynamic mechanical behavior of the blend was studied by dynamic mechanical analysis. The storage modulus of the blends decreased with increase in EMA content. When compatibilized with LLDPE-g-MA the storage modulus of the blend increases. LLDPE-g-MA is an effective compatibilizer as it increases the thermal stability and modulus of the blend.

Keywords LLDPE/EMA blend · Morphology · Thermal behavior · Compatibilization · Dynamic mechanical analysis

Introduction

Thermal degradation and dynamic mechanical behavior of polymers and polymer blends are very important to predicting their suitability in the specific field of applications. Knowledge of degradation and mode of decomposition under the influence of heat is highly recommendable in the processing and fabrication procedures because the

threshold temperature for decomposition determines the upper limit of the fabrication temperature. Polyolefin's are known for potential applications where the properties of thermal stability and fire resistance are necessary to be controlled [1–3]. Blending of two or more compatible polymers has been reported to have great impact on the thermal stability of polymers [4–10]. Compatibility between two components in a binary blend is one of the most decisive factors, which determines the thermal stability of polymer blends [11, 12], and therefore, compatibilization has profound effect on the thermal stability of polymer blends.

Thermogravimetry (TG) can be used as a way to measure the thermal stability of polymer due to the simplicity of the weight loss method [13]. The normal (TG) and derivative (DTG) thermogravimetric curves provide information about the nature and number of stages of thermal break down, weight loss of the material in each stage, threshold temperature, stability and extent of degradation of polymeric materials [14]. The thermal degradation of polymer blends was investigated by various researchers using thermogravimetric method [15–18]. Lopez et al. [19] studied the effect of thermoplastic on cure reaction and thermal stability of epoxy-amine thermoset and thermoplastic-based polymer blends, and reported that modifier did not affect the thermal and thermo-oxidative stability of the system. Komalan et al. [20] investigated the effect of compatibilizer EPM-g-MA on nylon copolymer and EPDM blends and found that the thermal stability of these blends was improved upon compatibilization. Jana et al. [21] studied the effect of different compatibilizer on LDPE/PDMS blends. They reported that compatibilized blends were more thermally stable than uncompatibilized blend, and EMA provides the maximum compatibilization as well as thermal stability to the LDPE/PDMS blend.

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Dynamic mechanical analysis (DMA) is a sensitive technique that measures the mechanical response of materials by monitoring property changes with respect to temperature and/or frequency of oscillation. Studies of dynamic mechanical properties are important because polymeric material can undergo various types of dynamic stressing during services. Several researchers [22–27] have investigated the effect of blend ratio and compatibilization on dynamic mechanical properties. Karger-Kocsis and Kiss [28] have investigated morphology and dynamic mechanical properties of EPDM/PP blends and PP block polymers. In these blends as the concentration of EPDM increases, storage modulus of the blend decreases. Kumar et al. [29] have studied the dynamic mechanical properties of EPDM-g-VOS/LLDPE blends with special reference to the effect of blend ratio. They found that increasing the proportion of LLDPE decreases the T_g value of blend, and there were increase in storage modulus and loss modulus due to increase in crystallinity. Moly et al. [30] have studied the effect of compatibilization on the dynamic mechanical properties of LLDPE/EVA blends and found that compatibilization increased the storage modulus of the system which is due to the fine dispersion of EVA domains in the LLDPE matrix providing an increased interfacial interaction.

The authors have recently studied and reported the effect of blend composition and compatibilization on the morphology, rheology, physico-mechanical, and thermal properties of LLDPE/EMA blends [31, 32]. In this communication effect of blend ratio and compatibilization on the thermal and dynamic mechanical behavior of LLDPE/EMA blends were investigated. An attempt was taken to establish a correlation between the thermal degradation and dynamic mechanical properties of the blends with their phase morphology.

Experimental

Materials

LLDPE (LLT12) having a density of 0.926 g cm^{-3} and MFI (melt flow index) $3.7 \times 10^{-1} \text{ g min}^{-1}$ was obtained from Haldia Petrochemicals Ltd., India. Commercial grade of EMA, Elvaloy 1330 with 30 mass% of methyl acrylate and a melt flow index (MFI) of $3.0 \times 10^{-1} \text{ g min}^{-1}$ of DuPont, USA was supplied by NICCO Corporation Ltd., 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 453 K and 60 rpm for 8 min. The modified materials were then purified to remove any unreacted monomer and cut into small pieces for use as compatibilizers.

Blend preparation

The blends having different composition were designated as E_x ($x = 0, 20, 30, 40, 50, 60, 70, 100$), where x represents the mass percentage of EMA in the blend. The compatibilized 60/40 LLDPE/EMA blends with 0, 1, 3, 5, and 7 mass% LLDPE-g-MA compatibilizer are represented as E_{40} , $1CE_{40}$, $3CE_{40}$, $5CE_{40}$, and $7CE_{40}$, respectively. The blending was carried out in a HAKKE Rheomix OS (Germany) using a cam-type rotor. The temperature of mixing was set at 413 K, and the rotor speed at 60 rpm. LLDPE was melted first for 2 min and then EMA was added and mixed for another 6 min. The hot mass was then taken out from the mixer and sheeted out in a two roll mill set at 2 mm nip gap. The sheeted material was then compression molded in an electrically heated hydraulic press (Moore Presses, George E. Moore and Sons Birmingham Ltd., UK) at 423 K for 5 min under a pressure of 5 MPa to obtain a film of thickness $0.025 \pm 0.003 \text{ cm}$. The mold was allowed to cool under pressure till ambient temperature is attained before removing the rectangular sheet from the mold.

Morphological study

Morphological studies of the blends were carried out on a scanning electron microscope (SEM), JSM 5800 microscope (JEOL, Japan), operating at 15 kV. The cryogenically fractured surface of the sheeted blend was etched by a suitable solvent (chloroform) to selectively extract only the EMA phase. The extraction process was carried out at 323 K for 24 h. The samples were then kept in a vacuum oven at 323 K for 12 h to remove the solvent. The dried samples were coated with gold for subsequent study.

Thermogravimetric analysis

The thermogravimetric analysis was carried out on a TA Instrument's (USA), model Q 50 in N_2 atmosphere at a heating rate of 283 K min^{-1} over a temperature range from 300 to 873 K.

Dynamic mechanical analysis

Dynamic mechanical measurements were carried out on a dynamic mechanical analyzer (METRAVIB DMA50). The experiment was performed in tension mode at a frequency of 1.256 Hz. The temperature range of the

testing was 123–398 K with a programmed heating rate of 10 K min⁻¹.

Results and discussion

Thermal stability of neat polymers and blends

Thermal degradation temperature of LLDPE, EMA, and their blends with and without compatibilizer was determined by thermogravimetric analysis. The TG and DTG curves of LLDPE and EMA in nitrogen atmosphere are given in Fig. 1. As can be seen from Fig. 1, the degradation of LLDPE starts around 706 K and completed almost at 784 K. The peak of the DTG curves gives the temperature corresponding to maximum degradation (T_{max}). In DTG curves of LLDPE, the peak appears at 763.92 K. The thermal decomposition of polyethylene has been studied by various researchers [33–36]. The thermal decomposition mechanism of LLDPE involving random chain scission leads to the formation of cross-linking and branched chain that compete with the backbone cleavage reactions [37].

The TG and DTG curves of EMA (Fig. 1) show apparently one stage degradation but if the curve is fitted using multi-Gaussian deconvolution technique it results in two overlapping Gaussian curves. This shows that it is actually a two-stepped process. Pure EMA starts to degrade in nitrogen atmosphere at about 682 K and is completely degraded at about 768 K. The random scission of EMA (around 682–768 K) would be initiated by homolytic scission of a methoxycarbonyl side group followed by β scission rather than by main chain scission [38, 39]. The methoxycarbonyl side group makes the β C–C scission easier due to its electronic and steric effects in EMA molecules [40]. Thus the loss of methoxycarbonyl side

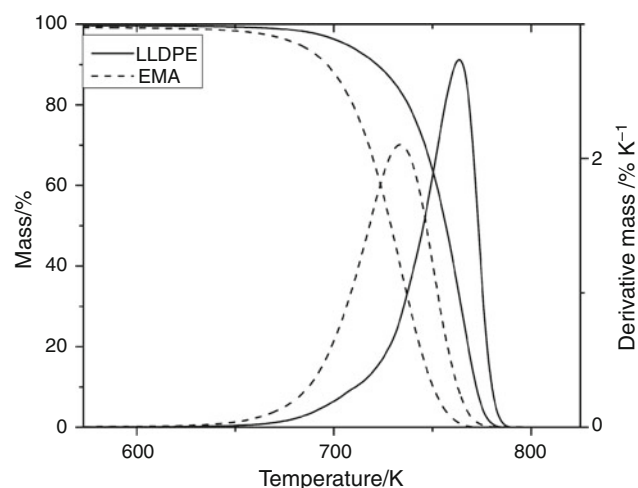


Fig. 1 TG/DTG curves of LLDPE and EMA

group is the initial degradation step. Scission in ethylene chain is the major degradation step.

The effect of blend ratio on the TG and DTG of blends is presented in Figs. 2 and 3, respectively. The important results obtained from the figures are summarized in Tables 1 and 2. It is seen that addition of EMA into LLDPE decreases the thermal stability of the blends only marginally. Addition of 20 and 30 mass% of EMA into LLDPE (i.e., E_{20} and E_{30}) decreases the IDT (initial decomposition temperature) by 1.80 and 5.90 K, respectively. On the other hand, addition of 50 mass% EMA shows a remarkable decrease (16.36 K) in IDT . In addition, when we look at T_{10} , T_{20} , etc., it is seen that there is no considerable difference between E_0 and E_{20} (and E_{30}). This is a clear evidence of the dependence of thermal stability on the phase morphology of the blends. The phase morphology of the blends can be evaluated from SEM micrographs given in Fig. 4. It should be noted that E_{20} and E_{30} blends possess dispersed phase morphology in which thermally more stable LLDPE forms the continuous phase whereas less stable EMA forms the dispersed phase. In this case 20 and 30 mass% of the dispersed phase has no considerable deteriorating effect on the thermal stability of the blends, since the matrix phase suppresses the degradation of dispersed phase. However, it is seen that E_{50} possesses a co-continuous phase structure in which both LLDPE and EMA form continuous phase. This situation makes EMA phase to undergo degradation in a much faster rate. In the case of E_{40} blends, where EMA forms the onset of co-continuity possesses intermediate thermal stability between E_{30} and E_{50} blends. On the other hand, in the case of E_{60} and E_{70} blends, thermally less stable EMA is the major phase, and therefore, it is more prone to thermal degradation. However, compared to E_{100} (pristine EMA), E_{60} and E_{70} are thermally more stable due to the existence of co-continuity of both the phases even though LLDPE is the minor phase (Fig. 4). This means that despite EMA and LLDPE are immiscible and incompatible, the presence of LLDPE in EMA improves the thermal stability of the blends.

The relative thermal stabilities of polymers and their blends can be assessed from their integral procedural decomposition temperature ($IPDT$) proposed by Doyle [41]. Table 2 displays the effect of blend ratio on T_{max} (the temperature at which maximum degradation occurs) and $IPDT$ of LLDPE/EMA blends. $IPDT$ has been employed to evaluate the relative thermal stability of blends under procedural conditions. $IPDT$ of the blends was measured using the equation given below as proposed by Doyle.

$$IPDT = A * K * (T_f - T_i) - T_i \quad (1)$$

where T_i and T_f are the initial and final experimental temperatures, A^* is the ratio of area under the curve and the

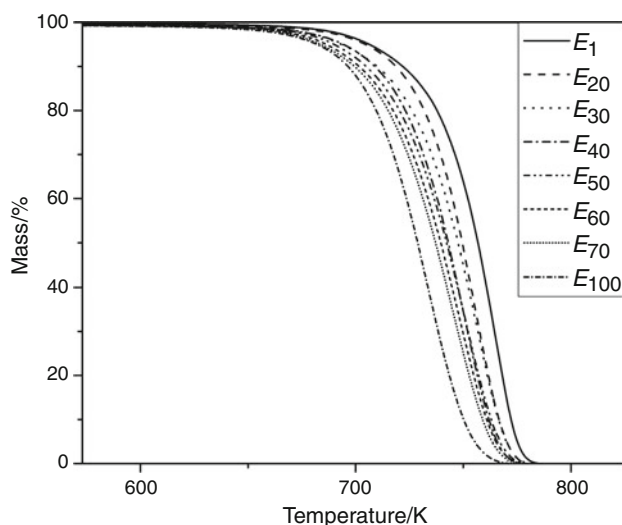


Fig. 2 Effect of blend ratio on the thermograms (TG) of neat LLDPE, EMA, and their blends

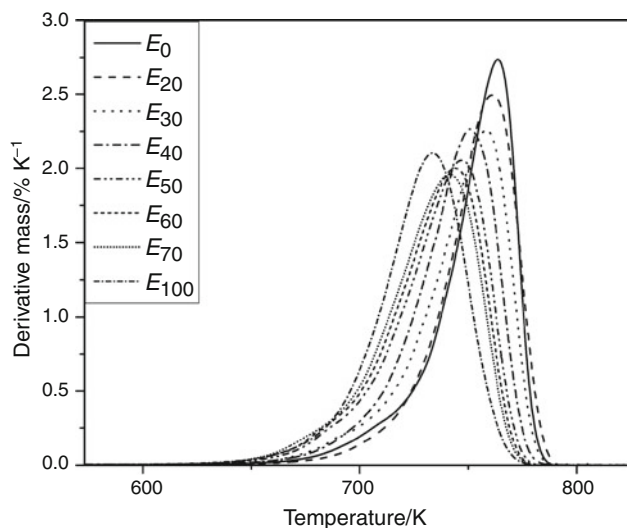


Fig. 3 Effect of blend ratio on the derivative thermograms (DTG) of neat LLDPE, EMA, and their blends

Table 1 Effect of blend ratio on the temperatures corresponding to different percent mass losses in LLDPE/EMA blends

Blends	IDT/K	T_{10} /K	T_{20} /K	T_{30} /K	T_{40} /K	T_{50} /K
E_0	706.30	722.05	737.90	746.28	752.15	756.82
E_{20}	704.51	718.84	731.97	739.47	745.10	749.60
E_{30}	700.46	709.94	726.24	735.45	742.17	747.68
E_{40}	694.30	708.95	723.22	731.56	737.93	743.10
E_{50}	689.94	704.94	720.53	729.62	736.50	742.30
E_{60}	686.23	701.65	717.45	726.83	733.67	739.59
E_{70}	683.57	699.57	715.24	724.41	732.25	737.25
E_{100}	682	696.55	709.67	717.64	723.64	728.89

Table 2 Effect of blend ratio on the T_{max} and $IPDT$ of uncompatibilized LLDPE/EMA blends

Blends	T_{max} /K	$IPDT$ /K
E_0	763.92	750.51
E_{20}	761.58	748.76
E_{30}	758.92	746.87
E_{40}	750.49	740.87
E_{50}	746.92	736.02
E_{60}	744.26	734.09
E_{70}	742.26	732.84
E_{100}	733.60	723.37

total area of the thermogram, and K^* is the coefficient of A^* . It is seen that T_{max} and $IPDT$ of LLDPE are maximum (763.92 and 750.51 K, respectively) and whereas those of EMA are minimum (733.60 and 723.37 K, respectively) indicating that LLDPE is thermally more stable than EMA. A gradual decrease in $IPDT$ with the addition of EMA into LLDPE is observed. Note that there is no considerable difference between the $IPDT$ of E_0 and E_{20} (and E_{30}) blends. On the other hand, the $IPDT$ of E_{70} is appreciably greater than that of E_{100} . In summary, the thermal degradation studies of uncompatibilized LLDPE/EMA blends revealed that the type of morphology has profound effect on the thermal stability of the blends.

Thermal stability of compatibilized blends

Compatibilizer has substantial influence on thermal properties of polymer blend. Figures 5 and 6 show the effect of compatibilization on TG and DTG of E_{40} blends. It is seen that the peak corresponding to major mass loss is shifted to higher temperature with addition of maleic anhydride grafted linear low density polyethylene (LLDPE-g-MA) on LLDPE/EMA blends. This indicates that thermal stability of the blends increases on compatibilization with LLDPE-g-MA. Tables 3 and 4 show the degradation temperature at different mass loss, T_{max} , and $IPDT$ for the compatibilized blends. The addition of even 1% of LLDPE-g-MA resulted in the improvement of initial decomposition temperature (IDT) and T_{max} . The addition of 3 mass% of the compatibilizer to E_{40} blend has got maximum degradation temperature. Further addition of the compatibilizer (5 and 7 mass%) also showed improvement in degradation temperature compared to unmodified E_{40} . Thus it is clear from the table that all blends with LLDPE-g-MA are thermally more stable compared to those containing without compatibilizer.

During the melt blending, the compatibilizer molecules were forced to locate at the interface of LLDPE and EMA. Therefore, the polar functional groups in LLDPE-g-MA

Fig. 4 SEM micrograph of solvent (chloroform) extracted LLDPE/EMA blend **a** E_{20} **b** E_{30} **c** E_{40} **d** E_{50} **e** E_{60} **f** E_{70}

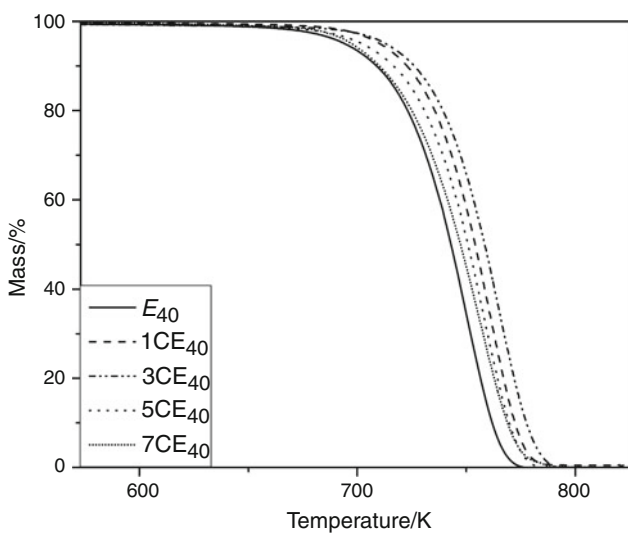
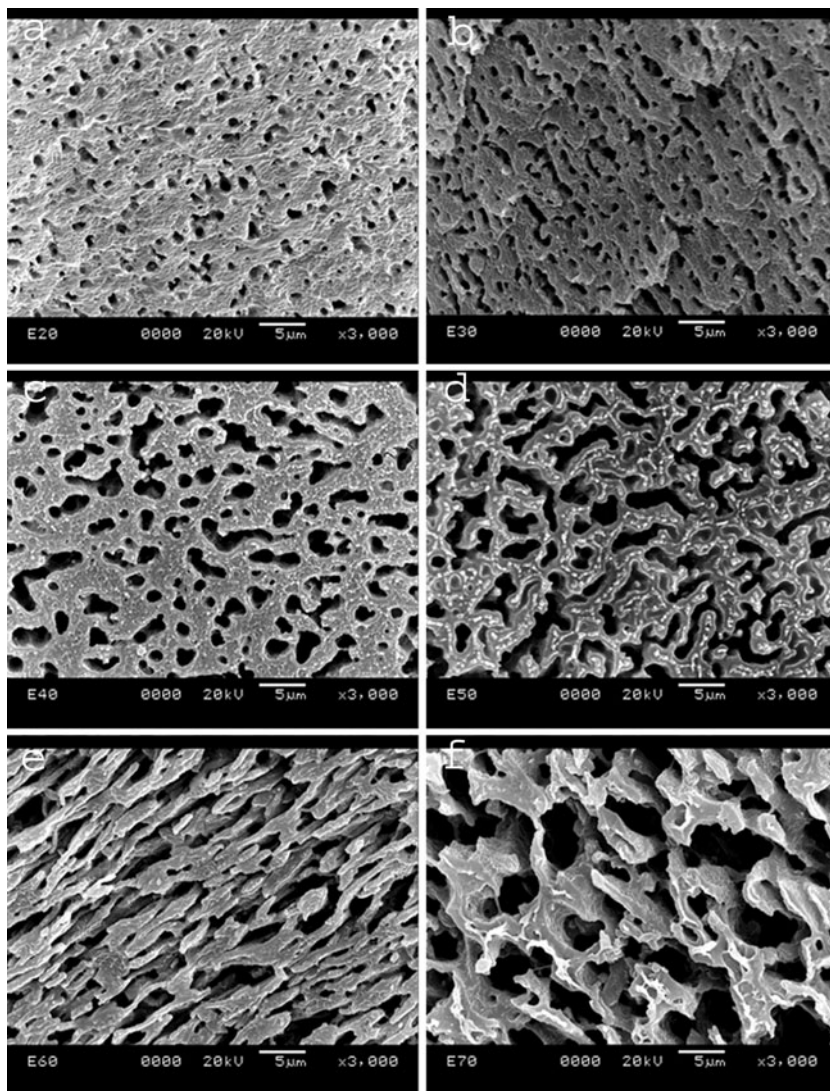


Fig. 5 Effect of compatibilization on the thermograms (TG) of E_{40} blends

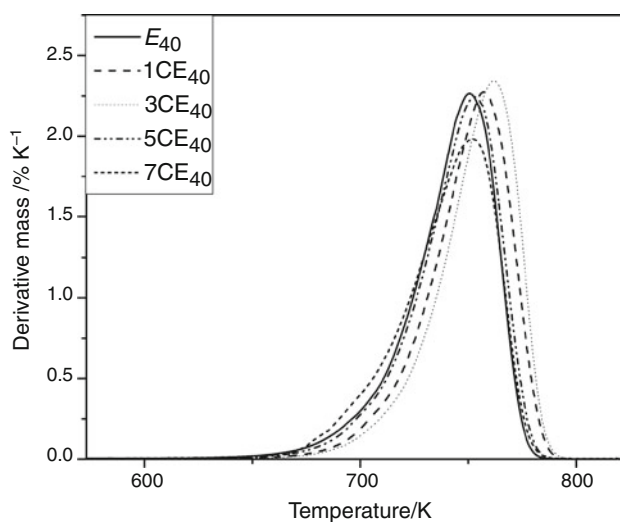


Fig. 6 Effect of compatibilization on the derivative thermograms (DTG) of E_{40} blends

Table 3 Effect of compatibilization on the temperatures corresponding to different percent mass losses in LLDPE/EMA (60/40) blends

Blends	IDT/K	T_{10}/K	T_{20}/K	T_{30}/K	T_{40}/K	T_{50}/K
E_{40}	694.30	708.95	723.22	731.56	737.93	743.10
1CE ₄₀	710.25	722.96	735.73	743.30	749.30	754.21
3CE ₄₀	712.98	726.27	739.93	747.40	753.48	758.49
5CE ₄₀	702.10	716.75	731.02	739.36	745.70	750.86
7CE ₄₀	697.34	710.25	725.03	734.38	741.48	747.36

Table 4 Effect of compatibilization on the T_{max} and IPDT of LLDPE/EMA (60/40) blends

Blends	T_{max}/K	IPDT/K
E_{40}	750.49	740.87
1CE ₄₀	757.16	750.50
3CE ₄₀	761.96	752.58
5CE ₄₀	753.34	744.74
7CE ₄₀	751.92	742.16

could interact with polar functional groups in EMA; while the LLDPE backbones are capable of compatibilizing with the LLDPE blend component. This leads to a reduction in interfacial tension and an improvement of interfacial adhesion between the LLDPE and EMA phases. The compatibilizing efficiency of LLDPE-g-MA is well evident from the finely dispersed uniform morphology compared to the uncompatibilized blend. The scanning electron micrographs of the compatibilized blends are shown in Fig. 7. From the micrograph it is clear that the domain size of the dispersed EMA decreased considerably in the compatibilized system. This in turn will contribute toward the improvement of thermal stability. Therefore, the enhanced thermal stability of E_{40} blends upon compatibilizer addition can be attributed to the compatibilizing efficiency of LLDPE-g-MA.

Dynamic mechanical analysis

DMA is sensitive to molecular motion, and transitions have been chosen as a tool for characterizing blend compatibilization. Figure 8 shows the variation of storage modulus (E') with temperature for different blend composition of LLDPE and EMA. It can be seen that storage modulus of the blends decreases with increase in EMA content, indicating the decrease in material rigidity, which may be related to the change in the molecular architecture caused by the addition of EMA [42].

The variation of storage modulus as a function of temperature for LLDPE/EMA (60/40) blend with different concentration of LLDPE-g-MA is shown in Fig. 9. The

storage modulus values of compatibilized blends are higher than that of the uncompatibilized blend at the same temperature. The increase in modulus upon the addition of compatibilizer is due to the increase in the interfacial interaction between EMA and LLDPE by the dipole/dipole interaction between the polar EMA part and polar MA part of LLDPE-g-MA. The addition of LLDPE-g-MA makes the blend technologically compatible to some extent even though molecular level miscibility cannot be achieved. From Fig. 9 it is clear that the maximum interfacial interaction (maximum modulus) is provided by a 3 mass% compatibilizer loading, and beyond that level of loading modulus value decreases because of micelle formation of the compatibilizer. The micelle could act as a lubricant in the blend system and decreases the material rigidity. From the SEM images also (Fig. 7) it is clear that upon addition of 3 mass% compatibilizer, the morphology shows a fine and uniform distribution of the dispersed EMA phase, which provides maximum interaction between the phases.

Degree of entanglement density

Degree of entanglement of the polymer blends can be obtained from swelling measurements, stress-strain curve, and also from dynamic mechanical analysis. One can use the storage modulus data for determining the entanglement density using the Eq. 4:

$$N = E'/6RT \quad (4)$$

where E' is the storage modulus obtained from the plateau region of E' versus temperature curve, R is the universal gas constant, and T is the absolute temperature. The values for degree entanglement between LLDPE and EMA are given in the Table 5. The compatibilized blends show higher degree of entanglement as compared to the uncompatibilized one. Addition of compatibilizer increases the entanglement between the homopolymers, and a better adhesion is achieved as a result of decrease in the interfacial tension. From the morphology studies it is clear that immiscible polymers exhibit a coarse dispersion. By the addition of the compatibilizer the particle size decreased leading to a better adhesion. Oommen et al. [43] reported that the entanglement density increases as a result of compatibilization. It is interesting to note that the entanglement density increases up to 3 mass% of compatibilizer concentration followed by a decrease at higher loading. This indicates clearly that the compatibilizer leaves the interface after critical micelle concentration (CMC). This is in agreement with the results obtained from morphology and thermogravimetric analysis.

Fig. 7 SEM micrograph of solvent (chloroform) extracted LLDPE/EMA blend **a** without compatibilizer **b** 1CE₄₀ **c** 3CE₄₀ **d** 5CE₄₀ **e** 7CE₄₀

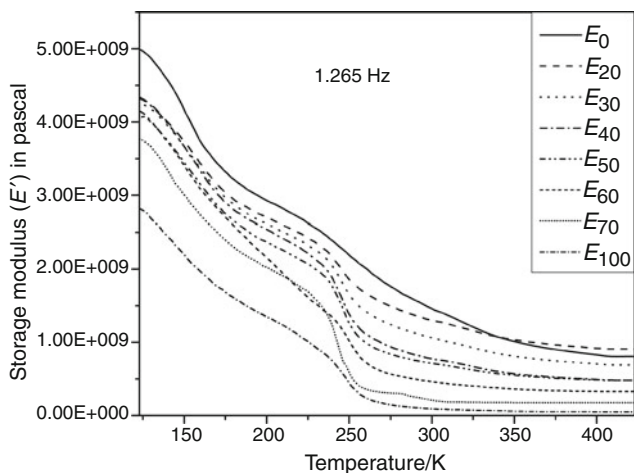
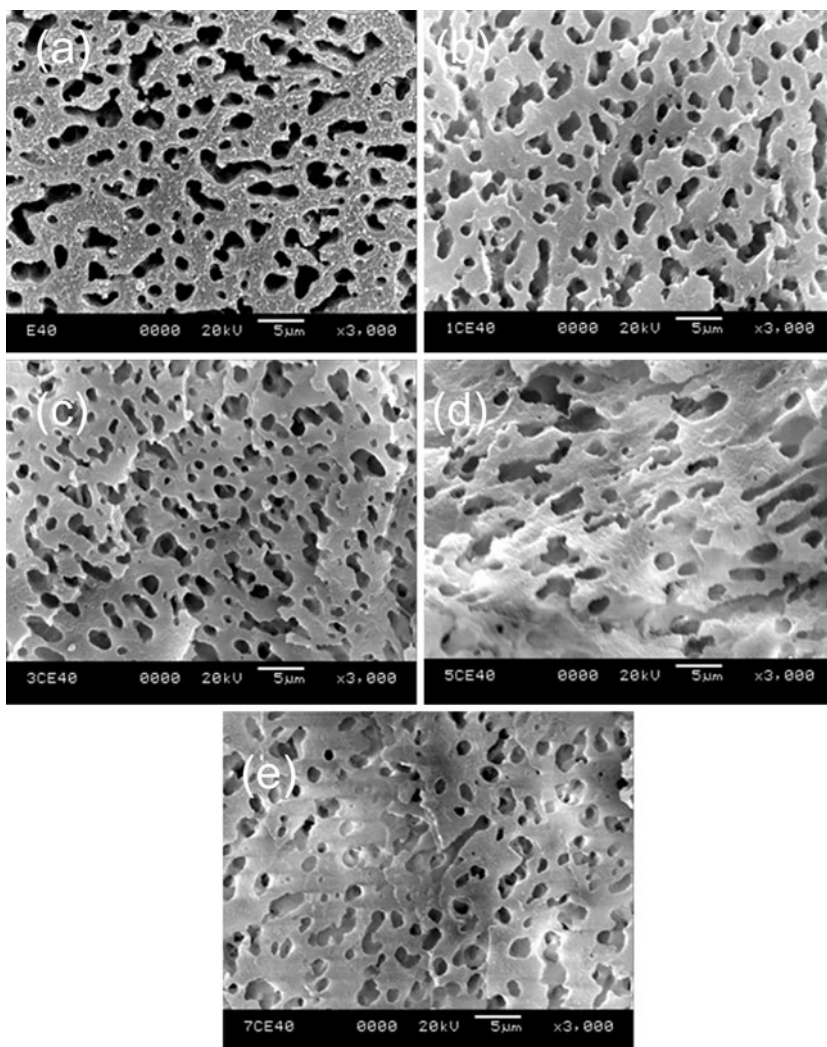


Fig. 8 Temperature versus storage modulus curves for LLDPE/EMA blends at different composition

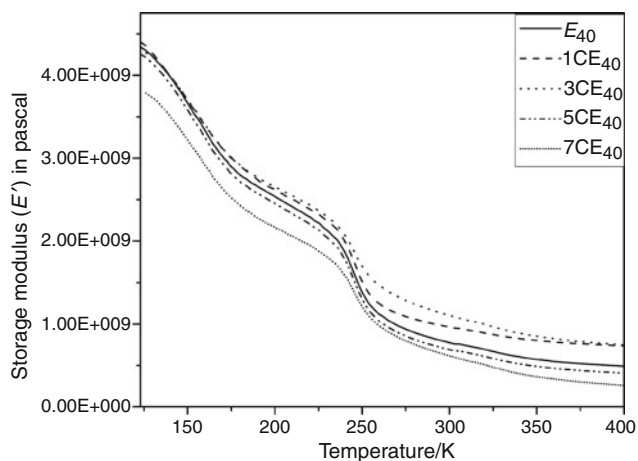


Fig. 9 Effect of compatibilizer concentration on temperature versus storage modulus curves of LLDPE/EMA (60/40) blends

Table 5 Entanglement density of 60/40 LLDPE/EMA blends

Temperature/K	Compatibilizer/mass%	Entanglement density/ moles m ⁻³ × 10 ³
243	0	144.51
	1	155.04
	3	165.12
	5	137.56
	7	124.90

Conclusions

Thermogravimetric data showed that blend ratio has significant impact on the thermal stability of the polymers. LLDPE is thermally more stable than EMA. Addition of EMA on LLDPE up to 30 mass% did not alter the thermal stability of the blends significantly. On the other hand, addition of 30 mass% of LLDPE into EMA improved the thermal stability of the blends. Phase morphology was found to be one of the most decisive factors that affected the thermal stability since the thermal stability depends on the stability of the matrix phase.

LLDPE-g-MA has been found to be an effective compatibilizer in LLDPE/EMA system as it improves thermal stability of the systems by providing improvement in the interfacial interaction between LLDPE and EMA. The compatibilizer increased the decomposition temperature of these systems especially at a critical value of its concentration in the blends.

Dynamic mechanical studies showed that addition of EMA into LLDPE decreases the storage modulus of the blends. On compatibilization, modulus value increases due to the improvement of interfacial interaction between LLDPE and EMA.

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