Polym. Bull. (2011) 66:939–953
DOI 10.1007/s00289-010-0367-x

ORIGINAL PAPER

Thermal properties and degradation characteristics of polylactide, linear low density polyethylene, and their blends

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Received: 16 April 2010 / Revised: 8 August 2010 / Accepted: 18 August 2010 /

Abstract Melt blending of linear low density polyethylene (LLDPE) and polylactide (PLLA) was performed in an extrusion mixer with post extrusion blown film attachment with and without compatibilizer-grafted low density polyethylene maleic anhydride. The blend compositions were optimized for tensile properties as per ASTM D 882-91. Based on this, LLDPE 80 (80 wt% LLDPE & 20 wt% PLLA) and M-g-L 80/4 (80 wt% LLDPE, 20 wt% PLLA and 4 parts compatibilizer per hundred parts of resin) were found to be an optimum composition. FTIR reveals that the presence of compatibilizer shifts carbonyl peak hence some increase in interaction between LLDPE and PLLA. Morphological characteristics of the fracture surface of with and without compatibilizer blends were examined by scanning electron microscopy. It shows that use of compatibilizer enhances the dispersions of PLLA in LLDPE matrix. Thermogravimetric (TG) analysis of blends shows the M-g-L 80/4 blend has higher thermal stability among studied blends. The degradation study under different pH of soil compost gives that in alkaline condition and the presence of compatibilizer was favorable for degradation. This blend may be used for packaging application.

Keywords Poly(L-lactic acid) · Linear low density polyethylene · Blending · Thermal stability · Soil compost

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Introduction

Polymers are widely used because of their easy process ability, low density, and desirable physical, chemical, mechanical, thermal, and electrical properties. Since, mid-nineteenth century, the polymer industry has grown enormously and has developed many important products. One of the most used polymers is polyethylene, which has a high chemical resistance and low cost. However, synthetic thermoplastic polymers are known for their resistance to biological degradation [1]. Polyethylene, the polymer present in the highest proportion in modern waste, is degraded in the presence of oxygen and ultra-violet radiation [2], but its extremely slow rate of degradation is a problem after disposal. The increasing environmental problems caused by the disposal of plastic waste have renewed interest in the development of environmentally degradable and "environmentally friendly" polymers [3–5]. One solution for this problem is to develop new biodegradable polymers.

The use of biodegradable polymers has contributed to a reduction in environmental problems, but the cost of producing these materials is still high. As a result, there has been a trend toward the production of degradable natural and synthetic polymers and natural/synthetic polymer blends. One way of rendering biodegradability to otherwise inert synthetic polymers is by its partial substitution with a poly(L-lactic acid) (PLLA) type of biodegradable polymer. In this regard, poly(L-lactic acid) (PLLA) has been attracting the attention of many researchers because of its biomass-origin and recyclability based on its biodegradation, hydrolysis, and depolymerization functions.

Poly(L-lactic acid) is a linear aliphatic polyester synthesized by ring opening polymerization of lactides which are the cyclic dimmers of lactic acids and are typically derived from corn starch fermentation. PLLA has been found to be environmentally biodegradable through a two-step process [6] that begins with the high molecular weight polyester chains hydrolyzing to lower molecular weight oligomers under an appropriate temperature and moisture environment. In the second step, microorganisms convert these lower molecular weight components to carbon dioxide, water, and humus.

The toughening of polylactide, an environmentally friendly thermoplastic, has been investigated using a wide variety of toughening agents, mostly emphasizing biocompatible materials [7]. Successful methodologies include copolymerization strategies [8], plasticization with a miscible component [9], and blending with an immiscible homopolymer [10–13] or a block copolymer [7]. Blending PLLA with other polymers can substantially modify the mechanical and thermal properties, degradation rate, and permeability. PLLA/poly(ε-caprolactone) (PCL) blends have been extensively studied [14, 15]. PLLA was also blended with other non biodegradable polymers, including polyethylene, poly(ethylene oxide), poly(ethylene glycol), poly(vinyl acetate), poly(4-vinylphenol), and polyacrylates [7, 13, 16, 17]. Melt blending of polylactide and linear low-density polyethylene (LLDPE) was reported [10] in an effort to toughen polylactide. In addition, two model polylactide–polyethylene (PLLA–PE) block copolymers were investigated as compatibilizers. However, the researchers had only investigated the effect of 20 wt% LLDPE with PLA and the study focused on the compatibility of LLDPE in PLLA. In our study, melt blending of linear low density polyethylene (LLDPE) and polylactide (PLLA) was performed in an extrusion mixer with post extrusion blown film attachment with and without compatibilizer-grafted low density polyethylene maleic anhydride. Varying degrees of property modifications were achieved by blending these polymers. Many of these blends are immiscible or only partially miscible and need compatibilizers to increase their compatibility. Degradation of polymer may be achieved by two major paths viz. (a) design of a polymer from monomers which are vulnerable to micro-organisms, and (b) incorporation of biodegradable additives or groups in the polymer. This in turn can be done by two methods. The first one involves the copolymerization of biodegradable monomers with the non-degradable monomer, and the second method involves the blending of a biodegradable additive/polymer with a non-degradable polymer.

The aim of the research article was to develop polymeric blends having optimum performance properties based on poly (L-lactic acid) using linear low density polyethylene (LLDPE) that will be partially degradable under some specific environmental conditions and to investigate the effect of PLLA composition & compatibilizer content on the thermal and degradation properties of blends.

Experimental

Materials

Commercial grade of linear low density polyethylene (LLDPE) (trade name Halene-L, melt flow index (MFI) 1.0 gm/10 min with 2.16 kg standard die at 190 °C, grade 71601S), was obtained from Haldia Petrochemical Ltd, Haldia, India. Commercial-grade poly (L-lactic acid) (trade name Biomer L 9000, $\bar{M}_w = 20$ kDa, $\bar{M}_n =$ 10.1 kDa, MFI 3.0 gm/10 min with 2.16 kg standard die at 190 °C) was supplied by Biomer, Kailling, Germany. The commercial grade compatibilizer, maleic anhydride-grafted low density polyethylene (M-g-L) (trade name OPTIM E142, MFI is 4.0 gm/10 min with 2.16 kg standard die at 190 °C, density 0.925 (gm/mL), T_m 103 °C) was obtained from Pluss Polymes Pvt. Ltd, New Delhi, India. The compatibilizer is maleic anhydride grafted LDPE resin and it contains 0.9–1.3% maleic anhydride. Furthermore, in the following text, it is denoted as maleic anhydride-grafted low density polyethylene (M-g-L).

Procedures

Blend preparation

The melt blending was performed at 200 °C and screw speed of 30 rpm in a Haake PolyLab extrusion mixer with post extrusion blown film attachment. (Thermo Haake, Karlsruhe, Germany). Before blending, the polymers were manually premixed in the desired compositions (Table 1) and films were prepared for all the blends. It was not possible to prepare the film by blowing process using higher amounts of PLLA because of its brittle nature. The second set of films was prepared

Table 1 Blend designation

Blend code	Composition		
	LLDPE (wt%)	PLLA (wt%)	M-g-L (phr)
LLDPE 100	100	0	0
LLDPE 95	95	5	0
LLDPE 90	90	10	0
LLDPE 85	85	15	0
LLDPE 80	80	20	0
LLDPE 75	75	25	0
LLDPE 70	70	30	0
LLDPE 65	65	35	0
LLDPE 60	60	40	0
LLDPE 55	55	45	0
LLDPE 50	50	50	0
M-g-L 80/2	80	20	2
M-g-L 80/4	80	20	4
M-g-L 80/6	80	20	6
M-g-L 80/8	80	20	8
M-g-L 80/10	80	20	10
M-g-L 65/2	65	35	2
M-g-L 65/4	65	35	4
M-g-L 65/6	65	35	6
M-g-L 65/8	65	35	8
M-g-L 65/10	65	35	10

by adding 80/20 and 65/35 blend with compatibilizer 2–10 parts per hundred parts of resin (phr). The polylactide can degrade by hydrolysis of the ester bond, drying of polylactide pellets are commonly performed before processing. The PLLA pellets had been dried at 40 °C under vacuum for 24 h.

Testing

Tensile testing

Physical properties such as tensile strength and elongation at break were measured according to ASTM D 882-91 procedure on Zwick Universal testing machine (model Z010 Zwick/Roell, Einsingen, Germany) at room temperature (25 °C), 50% relative humidity, and a cross-head speed of 50 mm/min. Five replicates were run for each composition and the average values have been reported.

Fourier transform infrared (FTIR)-attenuated total reflectance (ATR) spectroscopy

Fourier transform infrared-attenuated total reflectance (FT-ATR) spectroscopic studies were carried out on film samples using a Perkin Elmer FT-IR spectrophotometer

(Model BX-II, Shelton, USA. www.perkinelmer.com) in the horizontal ATR mode, using a zinc-selenide crystal. A total of 16 scans were taken with a resolution of 4 cm^{-1}. The spectrum was analyzed using spectrum software (Perkin Elmer, Shelton, USA).

Morphological evaluation using scanning electron microscopy (SEM)

Scanning electron micrographs of the fracture film surfaces were taken with a scanning electron microscope (JEOL, Model JSM 5800, Tokyo, Japan). The accelerating voltage was 15 kV. The specimens were coated with 50 nm of thick gold film in an automatic sputter coater (Polaron, East Sussex, UK) to avoid charging under an electron beam prior to SEM studies.

Thermal analysis

Thermal analysis data were measured by thermogravimetry (Perkin Elmer Pyris, diamond TG/DTA) and differential scanning calorimetry (DSC-2, Perkin Elmer, Shelton, USA). All measurements were performed under nitrogen. In this study, the thermal degradation behavior of the samples was recorded with heating from room temperature to 400 °C at a rate of 10 °C/min. TGA data showing the thermal degradation onset temperatures of the samples were used as references for ensuing DSC measurements. Thermal analysis was carried out using differential scanning calorimetry (Perkin Elmer DSC-2, USA). All measurements were performed under nitrogen. DSC measurements were carried out by heating from room temperature to 300 °C at a rate of 10 °C/min and were controlled by compatible computer running the Perkin Elmer (STAR SW900) instrument software. The software collected data and provided graphical analysis tools to determine transition temperatures and peaks areas. DSC studies revealed the significant thermal properties of the samples, such as transition temperature (T_g) and melting temperature (T_m). T_g determination from the DSC data followed these steps: first, two smooth base lines were determined for before and after the transition. Second, a tangent line in the transition region was drawn. A line equally positioned between the two base lines was drawn, and this line crossed the experimental data line. The point at which that they cross is the half-height T_g. These data are important indices for observing the miscibilities of the blends.

Measurement of degradability in soil compostage with different pH values

Soil compostage consisted of 23% loamy silt, 23% organic matter (cow manure), 23% sand, and 31% distilled water (wt%). Calcium hydroxide was added to provide pH values of 7, 9, and 11. The films of LLDPE 100, LLDPE 80 and M-g-L 80/4 blends were weighed and buried, in triplicate, in soil compostage at pH 7, 9, and 11. Five same samples of each were buried in a row in their respective tray. Biodegradation was monitored every 30 days for approximately 12 months by measuring the residual mass. For this, the buried samples were recovered, washed with distilled water, and dried at room temperature before being weighed. The residual mass was calculated as the ratio between the final and the initial weights.

Results and discussion

Blend composition optimization using Mechanical Properties

The stress–strain curves of the LLDPE 100, LLDPE 80, and M-g-L 80/4 films in both machine direction (MD) and transverse direction (TD) are also shown in the Fig. 1. The maximum force required for break was decreased by 5 and 11 N in case of LLDPE 80 and M-g-L 80/4, respectively, as compare to LLDPE 100. The decrease in elongation at break and yield stress can also be attributed to the addition of brittle component PLLA and high MFI comapatibilzer. The tensile properties of the LLDPE 100 and its blends are shown in Fig. 2. Tensile strength as well as elongation at breaks of the PLLA/LLDPE blends show decreasing trend with increase PLLA content, as PLLA is brittle in nature and acts as filler when it is dispersed in LLDPE. It has been observed that up to 20% PLLA addition to the LLDPE matrix, decrease in the tensile properties was very small. This small decrease can be attributed to the interaction, chain entanglement, and stress-induced crystallization behavior of PLLA with LLDPE during post extrusion biaxial oriented blown film in melt blending. But with further increasing proportion of PLLA, the effect was nullified and PLLA polymer plays a dominant role and hence decreases the mechanical properties. The effect of compatibilizer from 2 to 10 phr was studied for polymeric blend LLDPE 80. The addition of compatibilizer decreases the tensile strength as well as elongation at break of blends. This is due to high melt flow index of the compatibilizer and its polymeric nature. It also acts as a plasticizer which increases or decreases the elongation at break. It has been observed that the without compatibilzer blend have better retention of tensile

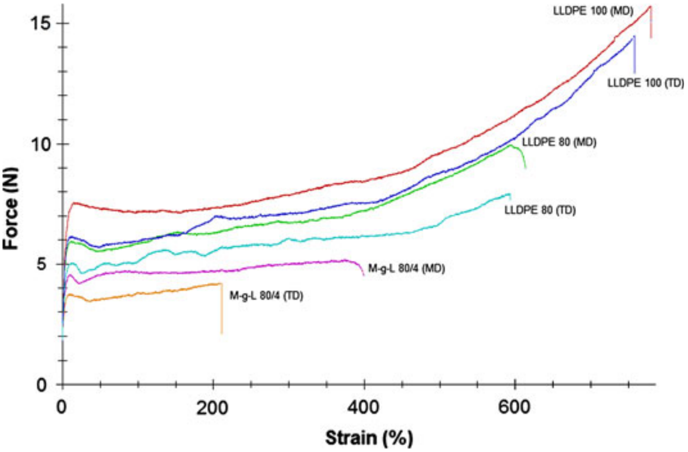

Fig. 1 Stress–strain curve of LLDPE 100, LLDPE 80 and M-g-L 80/4

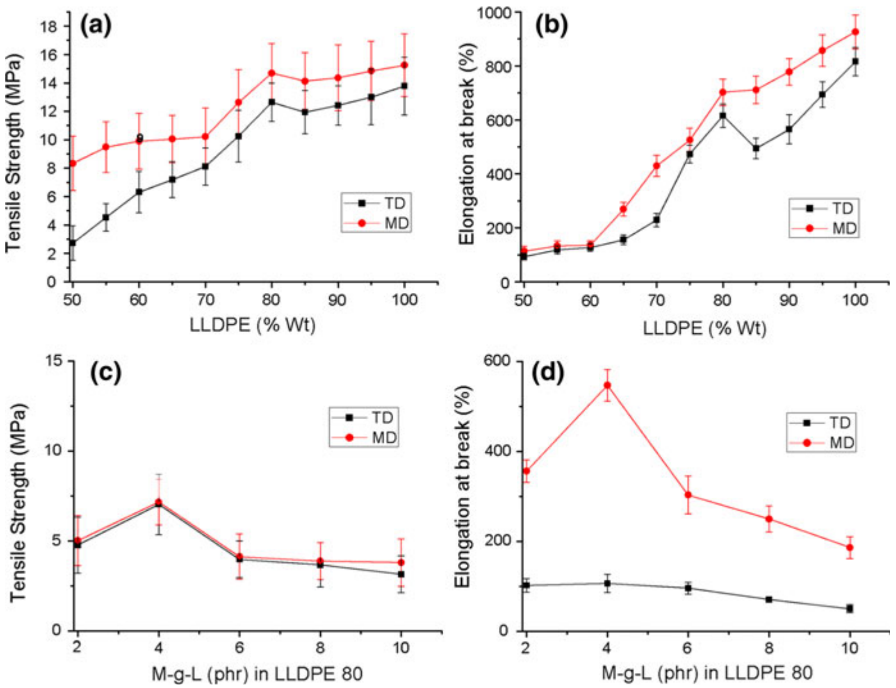

Fig. 2 Tensile strength and elongation at break of blends. **a**, **b** Change in tensile strength and elongation at break by varying LLDPE composition in blend. **c**, **d** Change in tensile strength and elongation at break by varying M-g-L composition in LLDPE 80 blend

properties than with compatibilized blend and M-g-L 80/4 showed optimum tensile properties. Therefore, we have selected LLDPE 80 for further study considering better retention of mechanical properties as well as relatively low proportion of expensive PLLA. The blends LLDPE 80 and M-g-L 80/4 with relatively better mechanical properties can be used in packaging applications. The values of tensile strength and elongation at break for packaging application for all thickness are 11.7 MPa, and 225%, respectively (ASTM D 4635).

FTIR spectroscopy of optimized blend films

FTIR spectra of LLDPE 100, LLDPE 80, M-g-L 80/4, PLLA, and M-g-L are shown in Fig. 3. The FTIR spectra for the study were shown in the region 1000–2000 cm^{-1}. The strongest peaks observed in the case of PLLA at 1760 (–C==O), 1465 (–CH–), 1362 and 1265 (–C–O–). M-g-L exhibits absorptions at 1864–1860 cm^{-1} {the asymmetric stretching of carbonyl ν_{as}(C=O)}, 1786–1784 cm^{-1} {the symmetric stretching of carbonyl ν_s(C=O)}, 1224 cm^{-1} {the asymmetric ring stretching ν(=C–O–C=)}, and 1064 and 1051 cm^{-1} {the symmetric ring stretching ν(=C–O–C=)}, characteristic of cyclic ethers [18]. In many compatible blends polymer containing carbonyl group usually undergo some interaction such as hydrogen bonding and a shift this peak is observed. In PLLA the peak centered at 1760 cm^{-1} is attributed to

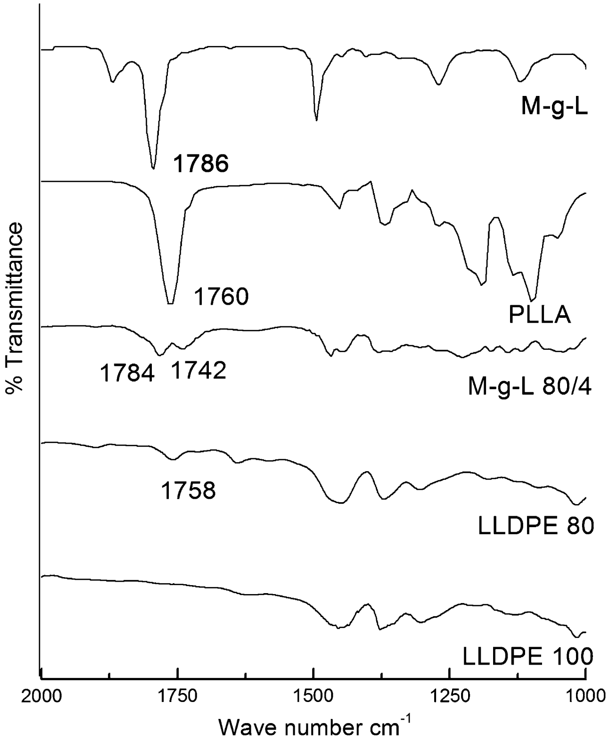

Fig. 3 FTIR spectra of LLDPE 100, LLDPE 80, M-g-L 80/4, PLLA and M-g-L

the carbonyl group and is of interest because any interactions would shift peak positioning. However, no significant shift in this peak was observed in the blend LLDPE 80. The peaks observed in the M-g-L 80/4 spectra at 1784 and 1742 cm^{-1} are assigned as carbonyl peak of M-g-L and PLLA, respectively. The 18 cm^{-1} shift in peak can be attributed to the blend M-g-L 80/4 which may be due to hydrogen bonding interaction in the two phases of PLLA and LLDPE in the presence of compatibilizer. This indicates that the two polymers are incompatible but in the presence of compatibilizer shows some extent of compatibility.

Morphological properties of optimized blend films

Figure 4 shows the scanning electron micrographs of the LLDPE 100 and its blends fracture surface. The Fig. 4a of LLDPE 100 tensile fracture surface is showing stretch fibrils. This stretch fibrils accounts for relatively high ductility as well as high toughness since considerable amount of energy consumed in the process. Figure 4b, c shows LLDPE 80 and M-g-L 80/4 blend film fracture surfaces. The addition of PLLA, a brittle material has influenced the morphology of LLDPE 80. The mix of river and fibrils like pattern gives brittle mechanism of fracture. The M-g-L 80/4 shows same type of pattern with some what less river pattern indicating that the compatibilizer has influenced the morphology of the LLDPE/PLLA blends.

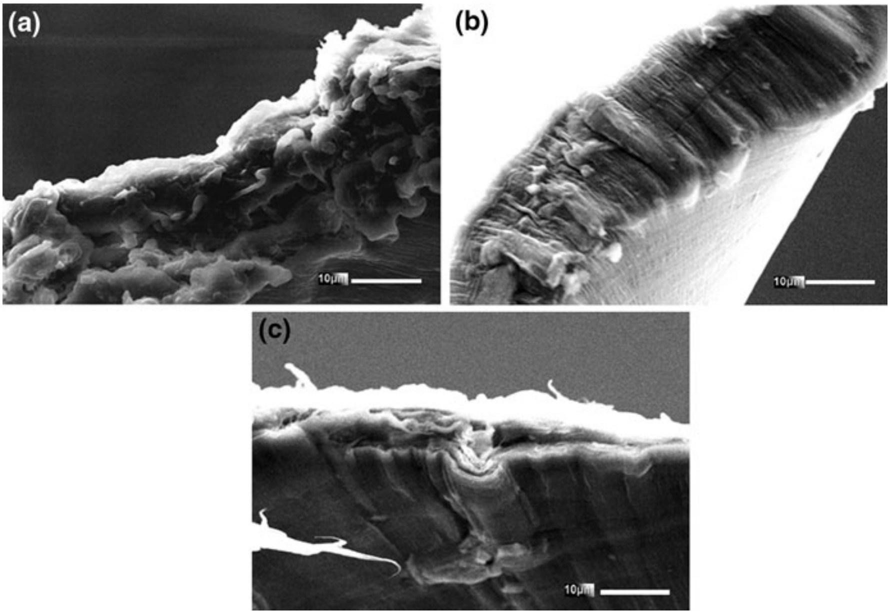

Fig. 4 Scanning electron micrographs of tensile fracture surface **a** LLDPE 100, **b** LLDPE 80, and **c** M-g-L 80/4

This fracture morphology can also be related to brittle fracture. The break pattern of LLDPE 80 and M-g-L 80/4 indicates that the brittle break gives more river pattern in LLDPE 80 as compared to M-g-L 80/4 where the break is some what ductile. The compatibilzer has influenced the break pattern, elongation may have decreased but because of compatibilization, it required more energy at break.

Thermal properties

Interpretations of DSC curves

Thermal properties are the most important macro properties of thermo plastics. They are direct responses of the results of the thermo processes. T_g and T_m are indices that correspond to the amorphous and crystalline regions of the materials, respectively. The molecules of solids are distributed as lattices. In single phase solids, molecules have relatively stable oscillations among molecules. However, when solids with two or more phases are heated, the molecular vibrations are significantly different because of intermolecular forces among the different phases. Therefore, T_g and T_m may function as indices of degree of mixing (homogenous and heterogeneous) of multiphase polymeric systems. Well-mixed or homogenous polymeric systems tend to have higher T_g's and T_m's than heterogeneous systems.

Miscible polymer pairs show a single glass transition (T_g) intermediate between those of pure polymers which makes DSC a well-known method to study the miscibility of polymer blends. The DSC traces of LLDPE 100, LLDPE 80, M-g-L

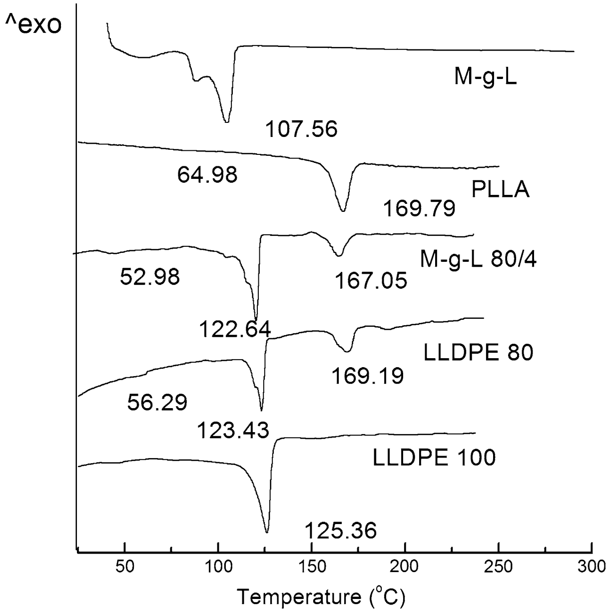

Fig. 5 DSC thermograms of pure polymers and their blends

80/4, and PLLA and M-g-L are shown in Fig. 5. PLLA, is a semi crystalline polymer and the major features of its DSC curve are the T_g at 65 °C and its melting peak (T_m) at 170 °C. LLDPE is semi crystalline and the T_m is 125.36 °C. The transitions shown at 45 and 72 °C can be attributed to chemical composition distribution of copolymer components of pure material. This transition does not appear in the blends LLDPE 80 and M-g-L 80/4. This may be due to reaction or interaction between the PLLA and LLDPE components. The compatibilizer (M-g-L) is also semi crystalline polymer and its T_m is 107 °C. The blend LLDPE 80 shows the decrease of T_g from 65 to 56.29 °C, and T_m negligible change. It may due to interaction between the two polymer interfaces. The T_m which does not change significantly may be because of stress induced crystallization. In M-g-L 80/4 blend, their is further decrease in T_g to 52.98 °C. This can be attributed to the plasticizing effect of the compatibilizer. There may be the more uniform dispersion which increases the interfacial interaction which is also supported by SEM micrograph and FTIR spectroscopy analysis.

Thermal stability analysis of blend films

The weight loss behavior during the thermal degradation of the PLLA, LLDPE 100, M-g-L, LLDPE 80, and M-g-L80/4 films has been studied. The thermally stable material shows resistance to weight loss up to processing temperatures. This directly gives the idea about the processing conditions and applications of the blends. Thermogravimetric (TG) curves of these pure and their blend films are illustrated in Fig. 6 and results are summarized in Table 2 which shows the thermal degradation

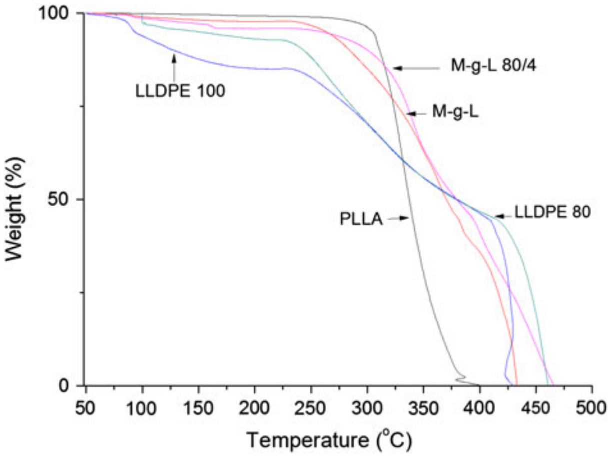

Fig. 6 Thermogravimetric curves of pure polymers and their blends

Table 2 TG analysis of pure polymers and their blends showing the thermal degradation

Sample	T_i (°C)	T_{max} (°C)	T_f (°C)	Weight loss (%)
PLLA	280	350	385	100
LLDPE 100	80	250	425	100
M-g-L	152	381	440	100
LLDPE 80				
1st stage degradation	84	153	253	18
2nd stage degradation	253	320	460	82
M-g-L 80/4	240	375	465	100

T_i initial temperature, T_{max} maximum temperature, T_f final temperature

initiation temperature (T_i), maximum temperature (T_{max}), final temperature (T_f), and percent weight loss. The initial degradation temperature (T_i), corresponding to 1% weight loss of the polymer sample. T_{max} is measured from the mid point of the TGA curve which is showing major degradation (perpendicular line drawn at x-axis between mid point of T_i and T_f.). The final degradation temperature (T_f), corresponds to 1% residual left after which no appreciable loss is possible. The thermal degradation of LLDPE 100 appears to be in three steps. This can be explained as formation of intermediate products due to crosslinking and branched chains that compete with the back bone cleavage reaction. The degradation of LLDPE is not a single step decomposition process but seems to follow a more complex degradation pattern [19]. The addition of PLLA improves the thermal stability as shown in TGA curve of LLDPE 80. There must be some interaction between the intermediate component of LLDPE and PLLA. It shows a two-step degradation process, clearly reflecting each degradation process of the PLLA and LLDPE ingredients. Each degradation temperature range was in the same temperature range with that of the PLLA or LLDPE homo films, and the weight loss ratio in each step was compatible with the composition ratio of each ingredient.

These results suggest that the degradation of PLLA and LLDPE ingredients proceeded independently; that is, they did not influence each other [4]. The M-g-L 80/4 blend shows single stage degradation which reflects that miscibility improves with compatibilizer as result the thermal stability further improves with addition of the compatibilizer.

Degradation characteristics of blends in soil compost

The degradation of polymeric blends especially under environmental conditions is the very important parameter for commercialization point of view. Any inert packaging material if converted into smaller particle size it will solve the solid waste management problem. If the one component, i.e., PLLA degrades from the blend matrix, the films changes into eroded surface having very less mechanical properties. The partial degradation study of blends also gives the idea how the new material behave in soil. Therefore, the degradation was evaluated in alkaline soil compost at pH (7, 9, and 11). The degradation at alkaline pH was selected because the polymers are more susceptible to hydrolysis in alkaline medium [5]. The degradation behavior of LLDPE 100, LLDPE 80, and M-g-L 80/4 at pH 7, 9, and 11 are shown, respectively, in Fig. 7a–c. It has been observed that PLLA showed some

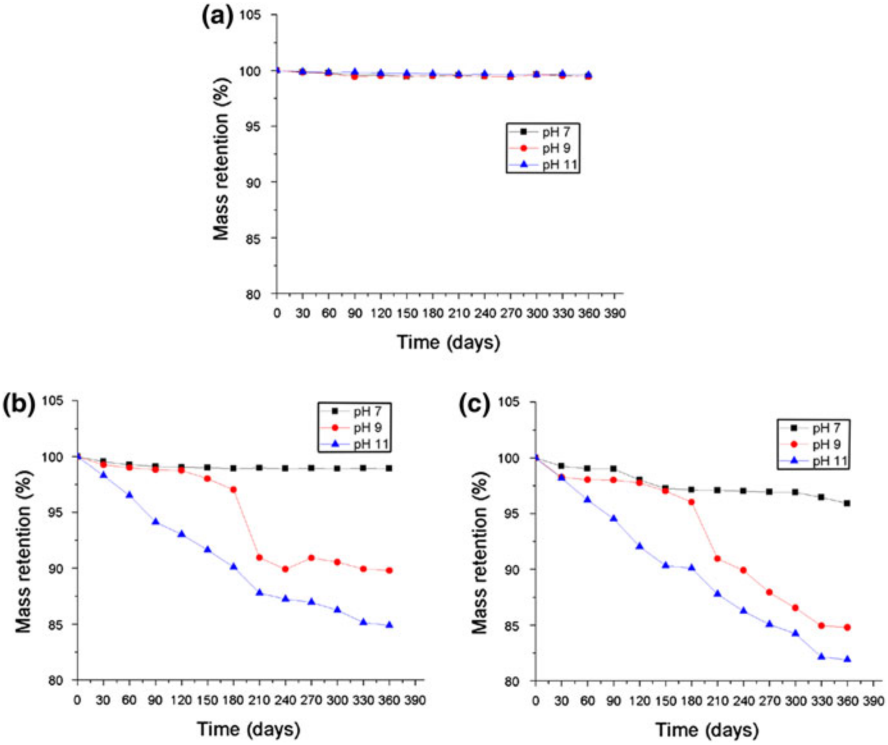

Fig. 7 Degradation in soil compost **a** Mass retention of LLDPE 100, **b** LLDPE 80, **c** M-g-L 80/4 at different pH levels: (*filled square*) pH 7, (*filled circle*) pH 9, and (*filled triangle*) pH 11

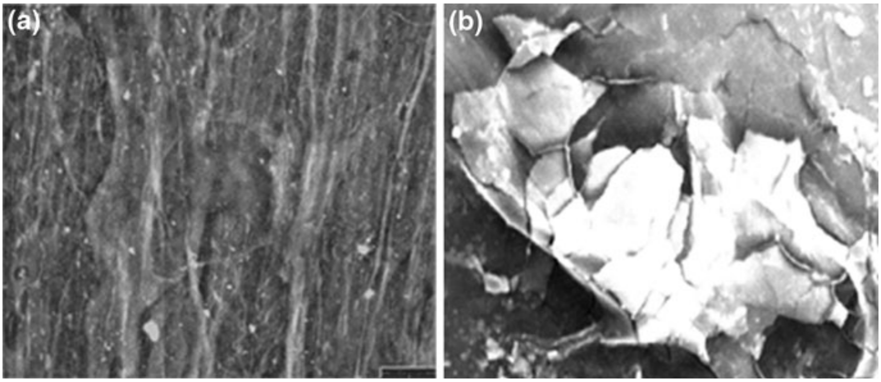

Fig. 8 Scanning electron micrographs of the M-g-L 80/4 blend: **a** before and **b** after degradation

degradation, whereas LLDPE did not. For the LLDPE 80 blend, the mass loss was maximum after aging in soil compostage for around 210 days, showed 10% degradation compared to LLDPE. The degradation was favored by alkaline soil compostage, as shown by the maximum loss of mass at pH 11. The M-g-L 80/4 blend had the highest degradation, because the presence of M-g-L increases the dispersion of PLLA in LLDPE matrix which leads to faster degradation.

Change in morphological properties after degradation

The Fig. 8a, b shows the SEM images of the M-g-L 80/4 blend before and after degradation, respectively. The before degradation image is shown for reference. There is a uniform dispersion of different phases before the degradation. The change in the uniformity after the degradation can be seen by the white spots, indicating that the surface erosion has taken place due to degradation of PLLA phase which would loosen the grip within the matrix resulting in weight loss.

Conclusions

The polymer blends LLDPE/PLLA with and without compatibilizer, using grafted low density polyethylene maleic anhydride, were prepared at melting condition in an extrusion mixer with post extrusion blown film attachment. The mechanical properties of the polymer blends depended on the component polymer ratios and the compatibilizer content. Among the investigated sample, the polymer blend LLDPE 80 and M-g-L 80/4 had the optimum tensile strength and elongation at break. The FTIR analysis indicated that the specific interaction in the blend with compatibilizer. Those specific interaction of absorption peak of C=O group in the blend. SEM micrographs of tensile fracture surfaces show the addition of compatibilzer has influenced the fracture morphology. DSC study support the some interaction between LLDPE/PLLA blends. Thermal properties show that the PLLA gives higher thermal stability compared to all blends. It has been also observed that the addition of the compatibilizer enhances the thermal stability of blend due to more uniform dispersion of PLLA in the LLDPE matrix. The M-g-L 80/4 blend shows greater thermal stability compared to LLDPE 80 blend. It has been observed that blend in alkaline medium weight loss is higher. The M-g-L 80/4 shows maximum weight loss at pH 11.

Acknowledgment The authors are thankful to the All India Council of Technical Education (AICTE), New Delhi, for part funding the project and Pluss Polymers, New Delhi for gifting the compatibilizer.

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