Mechanical Properties and Morphology of Polylactide, Linear Low-Density Polyethylene, and Their Blends

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ABSTRACT: Melt blending of linear low density polyethylene (LLDPE) and polylactide (PLA) 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. On the basis of this, LLDPE 80 [80 wt % LLDPE and 20 wt % poly(L-lactic acid) (PLLA)] and MA-*g*-low-density polyethylene 80/4 (80 wt % LLDPE, 20 wt % PLLA, and 4 phr compatibilizer) were found to be an optimum composition. The blends were characterized according to their mechanical, thermal, and morphological behavior. Fourier transform infrared spectroscopy revealed that the presence of compatibilizer enhanced the blend compatibility to some extent. The morphological characteristics of the blends with and without compatibilizer were examined by scanning electron microscopy. The dispersion of PLLA in the LLDPE matrix increased with the addition of compatibilizer. This blend may be used for packaging applications. © 2010 Wiley Periodicals, Inc. J Appl Polym Sci 118: 496–502, 2010

Key words: blending; blends; compatibility; FT-IR; miscibility

INTRODUCTION

The mixing of two or more polymers to produce blends by common processing steps is today a well-established approach for obtaining suitable materials for specific end uses. Specific applications of biodegradable blends have drawn marked attention in offering an attractive route for further improving environmental waste management.[1] To move toward the more sustainable society predicted for the near future, renewable resources need to be used more effectively. Environmental pollution caused by the careless disposal of bioinert plastic materials is now a serious and critical problem all over the world. In the last few years, studies concerning the total or partial substitution of synthetic plastics by biodegradable materials have been increasing steadily and have proven very useful in the search for a solution to the plastic solid waste management problem, at least to some extent.[2–4] The major limitation for the widespread use of totally biodegradable polymers is its high cost. This is due to two factors, namely, the use of costly monomers and/or the requirement of expensive additives or compatibilizers. Also, additional processing, such as mixing the ingredients with a mechanical mixing mill or an extruder, is necessary. The possibility of replacing commercial polymers, such as polyethylene, polypropylene, poly(vinyl chloride), nylons, and polyesters, with totally biodegradable polymers for clean environment is remote. The attainment of high performance of the former by the latter is yet not possible. Second, biodegradable polymers are meant for single uses; that is, these are meant to be thrown in the garbage after only one use. These polymers, therefore, should be cheap. To overcome this limitation, partially biodegradable polymers have been developed as a compromise between cost and performance. At present, the demand for disposable items, such as packing bags; milk, water, and soft drinks cartons; coffee and tea cups, and agricultural mulch films, is that they must be biodegradable and should degrade into safe byproducts after use under normal composting conditions. 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, PLLA has attracted the attention of many researchers because of its biomass origin and recyclability on the basis of its biodegradation, hydrolysis, and depolymerization functions.[5]

PLLA is a linear aliphatic polyester synthesized by the ring-opening polymerization of lactides, which are the cyclic dimers 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. PLLA offers a wide range of properties, depending on its stereochemical makeup.[7] It exists in two optical isomeric forms, PLLA and poly(D-lactic acid), and as a copolymer of the two as poly(D,L-lactic acid). Optically pure PLLA and poly(D-lactic acid) are semicrystalline polymers, whereas with the incorporation of about 15% meso-lactide, the polymer [poly(D,L-lactic acid)] becomes amorphous. PLLA is currently used in a number biomedical applications, such as sutures, internal fixation rods, fixation tacks, and drug-delivery devices, and it has also been used for tissue engineering applications.[8–10] Typically, amorphous polylactide (PLA) products are suitable for biomedical applications, uniform degradation, and resorption under physiological conditions.[11] Being biodegradable, it is used to replace petroleum-derived plastics in loose-fill packaging, compost bags, food packaging, and agricultural applications.[12–14] For most of these commodity applications, PLLA is often compared with other thermoplastics, mainly, polystyrene. The lower glass-transition temperature (T_g) of PLLA ($\sim 65°C$) compared with polystyrene ($\sim 100°C$) limits its uses for many such applications. Moreover, these applications require higher flexural properties, heat distortion temperatures, and impact strengths, where the crystallinity is a key parameter, because it increases the stiffness and heat distortion temperature of the product but reduces the impact properties.

The toughening of PLA, an environmentally friendly thermoplastic, has been investigated with a wide variety of toughening agents, usually with an emphasis on biocompatible materials.[15] Successful methodologies include copolymerization strategies,[16] plasticization with a miscible component,[17] and blending with an immiscible homopolymer[18–21] or a block copolymer.[15] Because none of the PLLA copolymers are commercially available at this time, the blending of PLLA with other polymers presents a more practical and economic measure to obtain products with properties not currently attainable. Blending PLLA with other polymers can substantially modify the mechanical and thermal properties, degradation rate, and permeability. PLLA/poly-(ε-caprolactone) blends have been extensively studied.[22,23] Various compatibilizers, such as lactic

acid and ε-caprolactone) copolymer, have been used to improve the miscibility between PLA and poly(ε-caprolactone).[24] PLLA was also blended with other nonbiodegradable polymers, including polyethylene, poly(ethylene oxide), poly(ethylene glycol), poly(vinyl acetate), poly(4-vinylphenol), and polyacrylates.[15,19,25,26] Various degrees of property modifications of PLLA have been achieved by blending with these polymers. Many of these blends are immiscible or only partially miscible and may need compatibilizers to increase their compatibility. The degradation of a polymer may be achieved by two major paths, namely, (1) the design of a polymer from monomers that are vulnerable to microorganisms and (2) the 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 nondegradable monomer, and the second method involves the blending of a biodegradable additive or polymer with a nondegradable polymer.

The aim of this research study was to develop polymeric blends with optimum performance properties based on PLLA with linear low-density polyethylene (LLDPE) and to investigate the effects of the blend composition and compatibilizer content on the physicomechanical properties of the blends.

EXPERIMENTAL

Materials

Commercial-grade LLDPE [trade name Halene-L, melt flow index (MFI) = 1.0 g/10 min with a 2.16-kg standard die at 190°C, grade 71601S] was obtained from Haldia Petrochemical, Ltd. (Haldia, India). Commercial-grade PLLA (trade name Biomer L 9000, weight-average molecular weight = 20 kDa, number-average molecular weight = 10.1 kDa, MFI = 3.0 g/10 min with a 2.16-kg standard die at 190°C) was supplied by Biomer Forst-Kasten-Str Kailling (Krailling, Germany). The commercial-grade compatibilizer, grafted low-density polyethylene maleic anhydride [MA; trade name OPTIM E142, MFI = 4.0 g/10 min with a 2.16-kg standard die at 190°C, density = 0.925 g/mL, melting temperature (T_m) = 103°C], was obtained from Pluss Polymers Pvt., Ltd. (New Delhi, India). The compatibilizer was MA-grafted LDPE resin, and it contained 0.9 to 1.3% MA. Furthermore, in the following text, it is denoted as maleic anhydride-g-low-density polyethylene (M-g-L).

Blend preparation

The melt blending was performed at a temperature of 200°C and a screw speed of 30 rpm in a Haake

TABLE I
Blend Designation

Sample	Blend code	LLDPE (wt %)	PLLA (wt %)	M-g-L (phr)
1	LLDPE 100	100	0	0
2	LLDPE 95	95	5	0
3	LLDPE 90	90	10	0
4	LLDPE 85	85	15	0
5	LLDPE 80	80	20	0
6	LLDPE 75	75	25	0
7	LLDPE 70	70	30	0
8	LLDPE 65	65	35	0
9	LLDPE 60	60	40	0
10	LLDPE 55	55	45	0
11	LLDPE 50	50	50	0
12	M-g-L 80/2	80	20	2
13	M-g-L 80/4	80	20	4
14	M-g-L 80/6	80	20	6
15	M-g-L 80/8	80	20	8
16	M-g-L 80/10	80	20	10
17	M-g-L 65/2	65	35	2
18	M-g-L 65/4	65	35	4
19	M-g-L 65/6	65	35	6
20	M-g-L 65/8	65	35	8
21	M-g-L 65/10	65	35	10

PolyLab extrusion mixer with a postextrusion blown film attachment (Thermo Haake, Karlsruhe, Germany). Before blending, the polymers were manually premixed in the desired compositions (Table I), and all of the blend films were prepared. It was not possible to prepare the films by the blowing process with higher amounts of PLLA. The second set of films was prepared by the addition of 80/20 and 65/35 blends with 2–10 phr compatibilizer. It was not possible to prepare the film by the blowing process with higher amounts of PLLA because of its brittle nature. The PLA could degrade by the hydrolysis of the ester bond; the drying of the PLA pellets was commonly performed before processing. Several melt blends were prepared with PLLA pellets that had been dried at 40°C *in vacuo* for 24 h, and we determined that there was no significant effect on the ultimate properties. Therefore, for the preparation of the melt blends, the predrying of the PLA was not performed.

Tensile testing

Physical properties, such as tensile strength at break and elongation at break, were measured according to the procedure in ASTM D 882-91 on a Zwick universal testing machine (model Z010, Zwick/Roell, Einsingen, Germany) at room temperature (25°C), 50% relative humidity, and a crosshead speed of 50 mm/min. The films of different blend samples were cut in both the transverse direction (TD) and machine direction (MD). A minimum of five repli-

cates were tested for each blend. The average values and their standard deviations are reported.

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

FTIR ATR spectroscopic studies were carried out on film samples with a PerkinElmer FTIR spectrophotometer (model BX-II, Shelton, USA) in the horizontal ATR mode with a zinc–selenide crystal. A total of 16 scans were taken with a resolution of 4 cm^{-1}. The spectra were analyzed with spectrum software (LX100627-I, PerkinElmer, Shelton, USA).

Thermal analysis

Thermal analysis was carried out with differential scanning calorimetry (DSC; PerkinElmer DSC-2). All measurements were performed under nitrogen. DSC measurements were carried out with heating from room temperature to 300°C at a rate of 10°C/min and were controlled by a compatible computer running the PerkinElmer (STAR SW900) instrument software. The software-collected data and provided graphical analysis tools were used to determine the transition temperatures and peak areas. DSC studies revealed the significant thermal properties of the samples, such as T_g and T_m.

X-ray diffraction (XRD) analysis

All of the samples were characterized by XRD with a Rigaku X-ray diffractometer (model Geiger flex, Tokyo, Japan) with Cu Kα radiation (λ = 1.5418 Å). During the experiment, the scanning speed and diffraction angle were 5°/min and 5–100° (2θ) at 40 kV and with a current of 20 mA.

Scanning electron microscopy (SEM)

Scanning electron micrographs of the films 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 μm of thick gold film before the SEM studies in an automatic sputter coater (Polaron, East Sussex, UK) to prevent charging under an electron beam.

RESULTS AND DISCUSSION

Mechanical properties

The mechanical properties of LLDPE 100 (where the number after LLDPE indicates the percentage of LLDPE in blends of LLDPE and PLLA) and its blends are shown in Table II. The tensile strength and the elongation at break values of the PLLA/ LLDPE blends showed a decreasing trend with

TABLE II
Mechanical Properties of the Blends

Sample	Blend code	Tensile strength (MPa)		Elongation at break (%)	
		TD	MD	TD	MD
1	LLDPE 100	13.78 (2.03)	15.25 (2.21)	816.00 (52)	926.00 (63)
2	LLDPE 95	13.01 (1.96)	14.85 (2.09)	694.00 (47)	857.00 (58)
3	LLDPE 90	12.42 (1.39)	14.36 (2.32)	565.00 (54)	778.00 (49)
4	LLDPE 85	11.43 (1.52)	13.92 (2.01)	494.00 (38)	711.00 (51)
5	LLDPE 80	12.65 (1.35)	14.69 (2.09)	615.60 (44)	702.00 (49)
6	LLDPE 75	10.25 (1.82)	12.62 (2.31)	473.20 (33)	525.00 (45)
7	LLDPE 70	8.12 (1.32)	10.22 (2.02)	228.80 (25)	429.20 (39)
8	LLDPE 65	7.19 (1.26)	10.05 (1.67)	155.80 (18)	269.20 (26)
9	LLDPE 60	6.32 (1.46)	9.90 (1.96)	127.20 (14)	136.00 (16)
10	LLDPE 55	4.53 (0.97)	9.48 (1.78)	118.20 (15)	133.00 (19)
11	LLDPE 50	2.72 (1.22)	8.34 (1.91)	93.00 (12)	113.60 (18)
12	M-g-L 80/2	4.77 (1.56)	5.02 (1.39)	102.00 (15)	356.00 (25)
13	M-g-L 80/4	7.02 (1.68)	8.55 (1.27)	206.40 (20)	446.80 (35)
14	M-g-L 80/6	3.98 (1.02)	4.13 (1.26)	95.80 (13)	303.00 (42)
15	M-g-L 80/8	3.67 (1.23)	3.88 (1.02)	70.60 (5)	249.60 (29)
16	M-g-L 80/10	3.15 (1.02)	3.80 (1.32)	50.20 (9)	186.00 (24)
17	M-g-L 65/2	4.20 (1.33)	6.56 (1.69)	10.60 (4)	249.00 (32)
18	M-g-L 65/4	6.98 (1.47)	7.12 (1.45)	16.00 (3)	349.00 (43)
19	M-g-L 65/6	4.93 (1.95)	5.25 (1.25)	11.20 (4)	251.60 (32)
20	M-g-L 65/8	4.45 (1.52)	5.99 (1.02)	12.00 (6)	239.00 (29)
21	M-g-L 65/10	3.88 (1.26)	5.28 (1.24)	12.80 (4)	224.00 (26)

Standard deviations are shown in parenthesis.

increasing PLLA content, as PLLA was brittle in nature and acted as filler when it was dispersed in LLDPE. With up to a 20% PLLA addition to the LLDPE matrix, the decrease in the tensile properties was very small. It may have been due to the interaction, chain entanglement, and stress-induced crystallization behavior of PLLA with LLDPE during postextrusion biaxial oriented blown film in melt blending. LLDPE 80 may have had maximum interfacial entanglements that were trapped upon the melt crystallization of LLDPE and PLLA. However, with further increasing proportions of PLLA, the effect was nullified, and the PLLA polymer played a dominant role and, hence, decreased the mechanical properties. Therefore, we selected LLDPE 80 for further study because of the better retention of mechanical properties and the relatively low proportion of expensive PLLA. The effect of compatibilizer from 2 to 10 phr was studied for the polymeric blend LLDPE 80. The addition of compatibilizer decreased the tensile strength and the elongation at break of the blends. This was due to high MFI of the compatibilizer and its polymeric nature. It also acted as a plasticizer and increased or decreased the elongation at break. In this case, it was attributed to a higher dose of compatibilizer. M-g-L 80/4 showed the optimum tensile properties. The blends LLDPE 80 and M-g-L 80/4 with relatively better mechanical properties were selected for further studies, as the blend material could be used in packaging applications. The values of the tensile strength and elongation at

break for packaging applications for all thicknesses were 11.7 MPa and 225% in MD and 8.8 MPa and 350% in TD, respectively (ASTM D 4635).

FTIR spectroscopy

The FTIR spectra of LLDPE, M-g-L, and blends are shown in Figure 1. The FTIR spectra for the films in the study were obtained in the region 500–4000 cm^{-1}.
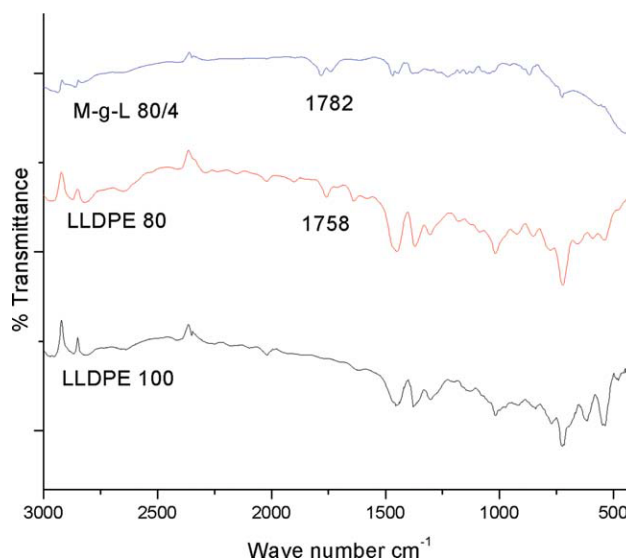

Figure 1 FTIR spectra of LLDPE 100, LLDPE 80, and M-g-L 80/4. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]
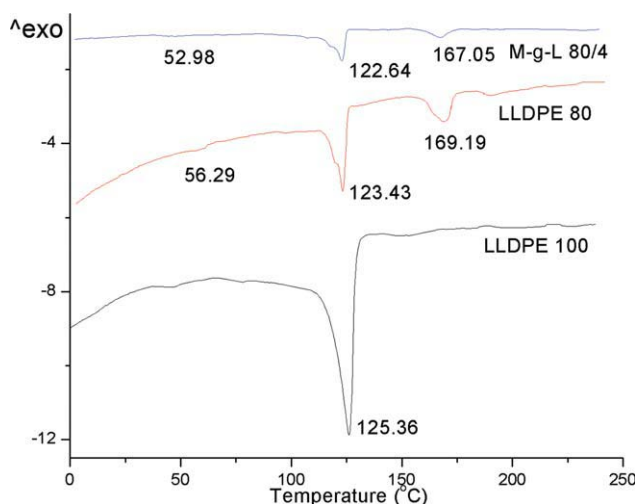
Figure 2 DSC thermograms of the LLDPE 100, LLDPE 80, and M-*g*-L 80/4 blends. [Color figure can be viewed in the online issue, which is available at www.interscience. wiley.com.]

The strongest peaks were observed in the case of PLLA at 1753 cm^{-1} (—C==—O), 1465 cm^{-1} (—CH—), and 1362 and 1265 cm^{-1} (—C—O—). The FTIR spectra of M-*g*-L are shown in Figure 1. M-*g*-L exhibited 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.[27] In many compatible blends, polymers containing carbonyl groups usually undergo some interaction, such as hydrogen bonding, and a shift this peak is observed. In PLLA, the peak centered at 1753 cm^{-1} was attributed to the carbonyl group and was of interest because any interactions would shift the peak positioning. However, no shift in this peak was observed in the blend LLDPE 80. However, about a 20–27 cm^{-1} shift was observed in the blend M-*g*-L 80/4, which was caused by hydrogen-bonding interaction in the two phases in the presence of compatibilizer. This indicated that the two polymers were incompatible but, in the presence of compatibilizer, had some extent of compatibility.

Thermal analysis

The thermal properties are the most important macroproperties of thermoplastics. They are direct responses of the results of the thermoprocesses. 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 the degree of mixing (homogeneous and heterogeneous) of multiphase polymeric systems.

The DSC traces of LLDPE 100 and the blends are shown in Figure 2, and the thermal property data are shown in Table III. PLLA was a semicrystalline polymer, and the major features of its DSC curve were the T_g at 65°C and its T_m at 170°C. LLDPE was semicrystalline, and the T_m was 125.36°C. M-*g*-L was also a semicrystalline polymer, and its T_m was 107°C. The LLDPE 80 T_g decreased from 65 to 56.29°C, and T_m showed a negligible change. This may have been due to interaction between the two polymer interfaces. T_m, which did not change significantly, may have been due to stress-induced crystallization. In the M-*g*-L 80/4 blend, there was a further decrease in T_g to 52.98°C. This was attributed to the plasticizing effect of the compatibilizer. There may have been a more uniform dispersion, which increased the interfacial interaction; this was also supported by the SEM micrographs and FTIR spectroscopy analysis.

XRD

XRD patters of films of the pure PLLA, LLDPE, M-*g*-L, and blends are shown in Figure 3. The diffractogram of PLLA showed a broad halo commencing from 14° in 2θ up to 17° in 2θ with a lower magnitude. This showed that PLLA was amorphous in character, and there was no trace of a crystalline region.

The XRD data showed the crystalline structures LLDPE and PLLA. The XRD diffraction peaks for LLDPE were at 2θ = 8.98, 21.0, and 28.16°. These peaks were still evident after the addition of 20% PLLA, but the intensity was greater in the case of the LLDPE 80 blend, which was, hence, more crystalline. This could be attributed to the interaction, chain entanglement, and crystallization behavior of PLLA with LLDPE during the postextrusion blown film in blending, which was also evident in the tensile properties.

TABLE III
Thermal Properties of the Blends

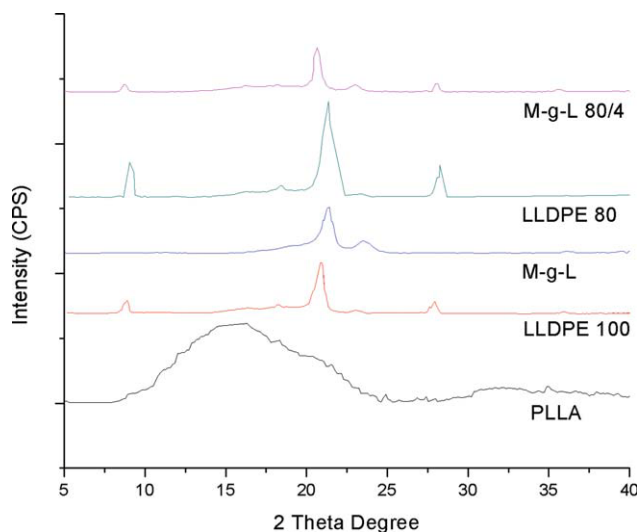| Blend	T_g (°C)	T_m (°C)
PLLA	65	170
LLDPE	—	125.36
M-*g*-L	—	107
LLDPE 80	56.29	123.23, 169.19
M-*g*-L 80/4	52.98	122.64, 167.05

Figure 3 XRD patterns for PLLA, LLDPE 100, M-*g*-L, LLDPE 80, and M-*g*-L 80/4. [Color figure can be viewed in the online issue, which is available at www.interscience. wiley.com.]

Morphological properties

Figure 4 shows the scanning electron micrographs of the LLDPE 100 and its blends. As shown in Figure 4(a), the LLDPE100 surface was smooth. Figure 4(b) shows the LLDPE 80 blend. The PLLA was dispersed in the LLDPE matrix in a nonuniform manner. PLLA formed large narrow and nonuniform channels; this suggested that LLDPE formed a continuous phase and PLLA formed a separated coarse phase. The boundary between the PLLA and LLDPE was very clear and sharp; this indicated a strong incompatibility and weak interfacial adhesions. Once the compatibilizer was added to the LLDPE 80 blend, the PLLA was uniformly dispersed in the LLDPE matrix, as shown in Figure 4(c). This indicated that the compatibilizer influenced the morphology of the LLDPE/PLLA blends. The particle size of the PLLA phase decreased with the addition of compatibilizer, as reported in the literature.

CONCLUSIONS

The LLDPE/PLLA polymer blends with and without compatibilizer with grafted low-density polyethylene MA were prepared at melting conditions in an extrusion mixer with a postextrusion blown film attachment. The mechanical properties of the polymer blends depended on the component polymer ratios and the compatibilizer content. Among the investigated samples, the polymer blend LLDPE 80 and M-*g*-L 80/4 had the optimum tensile strengths and elongations at break. The FTIR analysis indicated the specific interaction in the blend with compatibilizer. Those specific interactions

were the absorption peak of the C=O group in the blend. DSC and XRD studies supported some interaction between the LLDPE/PLLA blends. The
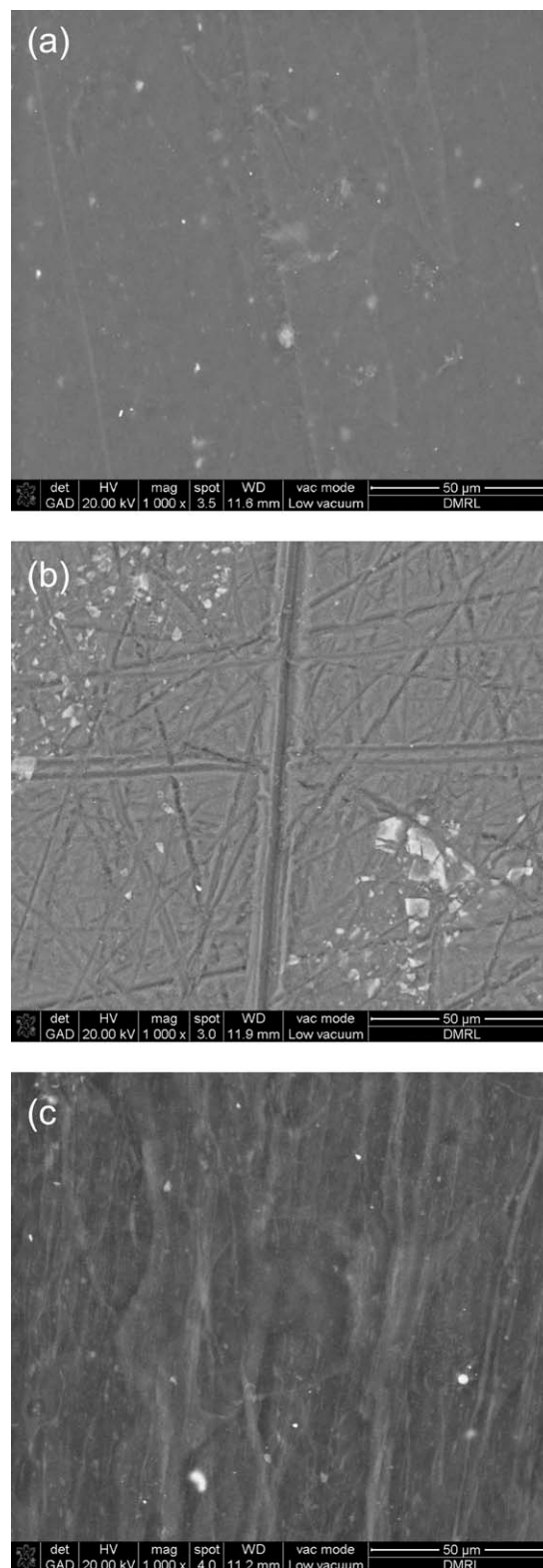

Figure 4 SEM of the blends: (a) LLDPE 100 at 1000× magnification, (b) LLDPE 80 at 1000× magnification, and (c) M-*g*-L 80/4 at 1000× magnification.

morphological studies indicated that the compatibilizer influenced the morphology of the LLDPE/PLLA blends.

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