

Effect of EPDM as a compatibilizer on mechanical properties and morphology of PP/LDPE blends

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Nina Vranjes Penava¹, Vesna Rek¹ and Ivona Fiamengo Houra²

Abstract

Blends of polypropylene (PP) and low-density polyethylene (LDPE) with and without ethylene-propylene-diene (EPDM) terpolymer as a compatibilizer were studied. Mechanical properties were chosen to estimate the compatibilization efficiency of EPDM. The interactions between phases were valued through glass transition shifts in dynamic mechanical spectra, and morphology of the blends was obtained using scanning electron microscopy. Interfacial adhesion was improved by EPDM addition. Addition of EPDM to PP/LDPE blends improved mechanical properties, especially Izod impact strength in LDPE-rich blends and with higher EPDM content.

Keywords

Blends, compatibilization, morphology, mechanical properties, dynamic mechanical analysis

Introduction

Development of new material with a broader application is possible by blending polymers, giving more enhanced properties than individual polymer. Producing new materials by blending two homopolymers is economically acceptable also from an ecological view. In the field of recycling postconsumer waste, economical costs linked to the separating steps could be decreased and, hence, the recycling of plastic waste becomes more profitable.¹ The

¹ Faculty of Chemical Engineering and Technology, University of Zagreb, Zagreb, Croatia

² Brodarski institut, Avenija V Holjevca, Zagreb, Croatia

Corresponding author:

Nina Vranjes Penava, Faculty of Chemical Engineering and Technology, University of Zagreb, Marulicev trg 19, Zagreb 10 000, Croatia.

Email: nvrnjes@fkit.hr

major polymeric components of plastic waste streams are polyethylene, polypropylene (PP), polystyrene, polyvinyl chlorides, and polyethylene terephthalate.² Polyolefins representing most of postconsumer plastic wastes, which are difficult to separate, and therefore, the valorization of these materials is possible, rather by reprocessing of commingled wastes.³ Because most of the polymers are incompatible and immiscible,^{4,5} the polymer mixtures will exhibit poor mechanical, thermal, and morphological characteristics.

PP and polyethylene blends have been studied for many years. There have been many discussions about their miscibility. Gunderson and Chilcote,⁶ Nolley et al.,⁷ and Teh⁸ reported on LDPE/PP incompatibility. Dong et al.⁹ and Avalos et al.¹⁰ found limited miscibility of PP/LDPE blends, evidence of small portion of PP being dissolved in the LDPE as well as LDPE effect on PP crystallization.

To improve the compatibility between the PP and LDPE, a third component as compatibilizer^{3,11–14} or other additives as fillers^{15,16} can be added.

Chang-Sik et al.¹³ compatibilized LDPE/PP blends with ethylene-propylene-diene terpolymer (EPDM) and EPDM grafted with maleic anhydride (EPDM-g-MAH). EPDM increased the blend compatibility greatly, more than EPDM-g-MAH. Fortelný et al.³ and Si and Chen¹⁴ showed that the addition of 5 wt% of EPDM in LDPE/PP blends improved their impact strength as a consequence of EPDM localization at LDPE/PP interface. Bertin and Robin¹ studied the virgin and recycled LDPE/PP blends. With the addition of EPDM, ethylene-propylene monomer (EPM) or PE-g-(2-methyl-1,3-butadiene) graft copolymer, elongation at break and impact strength were improved in all blends. Li et al.¹⁷ studied the miscibility and isothermal crystallization of PP in polyethylene melts. By hot-stage optical microscopy, they observed that PP crystallized as open armed diffuse spherulite in PP and linear low-density polyethylene (LLDPE) blends, whereas in PP/LDPE and PP and high-density polyethylene (HDPE) blends, PP crystallized in phase-separated droplets. In PP/LLDPE blends, the crystallization rate of PP decreased, whereas in PP/LDPE and PP/HDPE blends, it was similar to that of pure PP.

Our study was focused on improvement of compatibility of PP/LDPE blends with the addition of EPDM as compatibilizer. The effect was evaluated through an investigation of morphological structure, mechanical characteristics, and dynamic mechanical properties. The effect was examined with two different EPDM amounts added to various ratios of PP and LDPE components.

Experimental details

Materials

The following materials were used to prepare the PP/LDPE blends with and without EPDM as a compatibilizer:

PP, HC206TF, supplied by Borealis, MFR 5 g/10 min (2.16 kg/230°C); LDPE, Okiten 245A, supplied by Dioki, MFR 2.3 g/10 min (2.16 kg/190°C), density 0.924g/cm³; EPDM terpolymer, Nordel IPNDR 4520, supplied by Dupont Dow Elastomers, containing ethylene 49–51 wt%, propylene 43.7–47 wt%, and ethylene norbornene 4–5.30 wt%. Mooney viscosity of EPDM was 16–24 at 125°C.

Preparation of the blends

A sample of neat polymers and blends of PP/LDPE with and without a compatibilizer were prepared by Haake Record 90 twin screw extruder with intensive mixing profile, Haake TW 100, with the zone temperatures of 180/190/195/205°C and at 60 rpm. After the extrusion, the dumbbell test specimens were prepared by injection molding with Zwick injectometer at 230°C, the injection rate of 200 mm/s, and the mold temperature of 40°C. Two different amounts (5 and 7 g) of EPDM were added to PP/LDPE blends. For example, a blend containing 80 g of PP, 20 g of LDPE, and 5 g of EPDM has the following notation: PP/LDPE/EPDM 80/20/5.

Mechanical properties

The measurements of tensile properties, tensile strength at break, and elongation at break of dumbbell-shaped samples were carried out by an Instron 1185 tester according to standards ISO 527-1: 1993 and ISO 527-2: 1993, at a crosshead speed of 50 mm/min. The Izod impact strength was measured according to ISO 180:1993. For each analysis, five specimens were tested and the average values were reported.

Dynamic Mechanical Analyzer 987 (DMA), TA Instruments, was used to determine the viscoelastic properties of the samples. The measurement was carried out at a constant frequency of 1 Hz, temperature range of -150°C to $+150^{\circ}\text{C}$, amplitude of 0.2 mm, and heating rate of $5^{\circ}\text{C}/\text{min}$.

Scanning Electron Microscopy (SEM)

The samples of PP/LDPE and PP/LDPE/EPDM blends were fractured in liquid nitrogen and sputtered with a thin platinum layer to prevent charging. The fracture surfaces were observed using the SEM (VEGA TS5136LS, Tescan) using a secondary electron detector.

Results and discussion

Mechanical properties

The results of mechanical properties are presented in Figure 1. Addition of LDPE to PP reduced the tensile strength value, so the tensile strength values of PP/LDPE blends were between values of pure homopolymers (Figure 1 (a)). Addition of EPDM (5 phr) in PP/LDPE blends slightly reduced a tensile strength value, whereas a higher EPDM amount (7 phr) did not have any influence on tensile strength, except for PP/LDPE/EPDM 80/20/7 blend (Figure 1 (a)). This suggests that the tensile strength of compatibilized blends is determined by not only the interfacial adhesion but also the strength of the matrix that is highly affected by the amount of the compatibilizer.¹⁸

Elongation at break for pure PP and pure LDPE and their blends is presented in Figure 1 (b). For PP/LDPE 80/20 and 20/80 blends, elongation at break values was between the values of pure homopolymers. PP/LDPE 60/40 and 40/60 blends had lower values of elongation at break compared with pure homopolymers. The result of very low

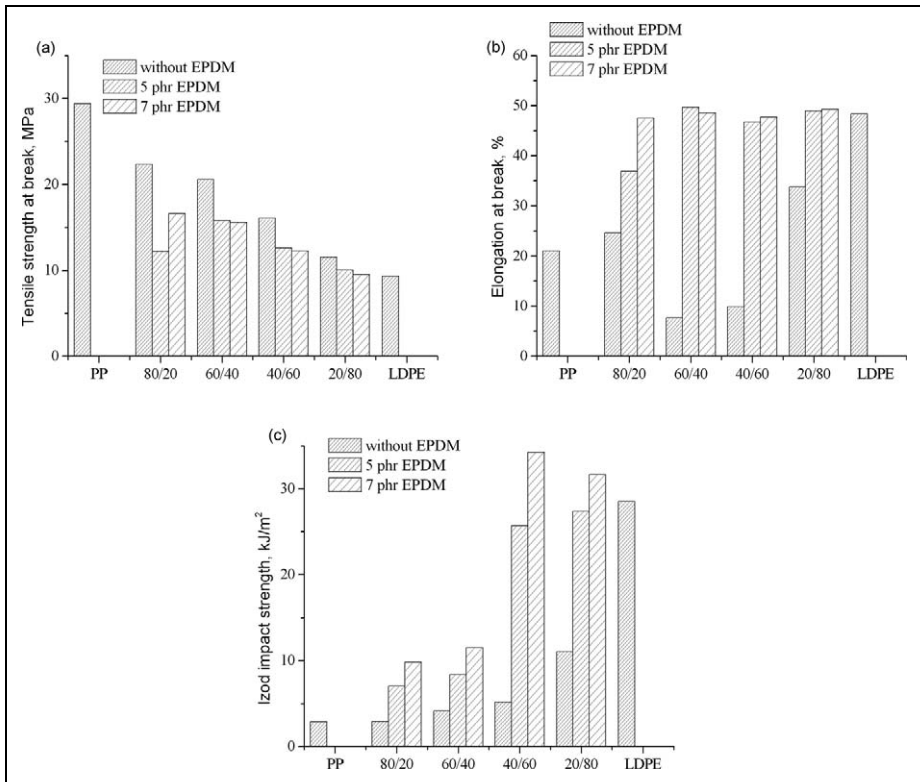


Figure 1. Mechanical properties of PP/LDPE blends. (a) Tensile strength at break, (b) elongation at break, (c) Izod impact strength. PP: polypropylene; LDPE: low-density polyethylene.

elongation at break was probably the consequence of weak interfacial adhesion and high interfacial tension that indicated incompatibility of the systems. The explanation of poor properties of PP/LDPE 60/40 and 40/60 blends may be explained in the following way: it was established that at temperatures above the crystallization melting points of homopolymers, the PE and PP chains are found to segregate into distinct domains, and the PE phase shows more significant clustering.¹⁹ The clustering of PE and PP is due to the unfavorable cross-correlations between PE and PP chains in the blend.²⁰ This unfavorable clustering increases as the amount of individual polymers increases in the blend. Jose et al.²¹ explained that the maximum incompatibility may be for the blends containing 40–60 wt% of HDPE. This may be the plausible explanation for the minimum mechanical properties observed for the blend containing 40–60 wt% of PE. Addition of EPDM had very high impact on elongation at break (Figure 1 (b)). In all PP/LDPE blends, the EPDM addition improved that value, especially for 60/40 and 40/60 blends, compared with noncompatibilized blends. The higher amount of EPDM did not significantly change elongation at break, except for 80/20/7 blend, which improved their elongation at break value for 10.64%.

It is visible from Figure 1 (c) that the pure LDPE had a higher Izod impact strength compared with pure PP. Addition of LDPE to PP slightly increased the value. Compatibilizer EPDM improved Izod impact strength in all PP/LDPE blends. Improvement was more expressed in PP/LDPE blends with LDPE matrix and a higher EPDM amount (Figure 1 (c)). That improvement is a consequence of better compatibility of the system and finer homogeneity, which was proved by SEM (Figure 2). In general, the impact strengths of PP and PE are improved with the addition of random or block ethylene-propylene rubber (EPR), whereas the tensile strengths are decreased.²

Several explanations of compatibilizing effect on mechanical properties can be resumed: Bartlett et al.²² studied the mechanical properties of PP/HDPE blends compatibilized by ethylene-propylene elastomer. They showed that the property relationship observed depends strongly on the process used to fabricate the blends as shown by comparisons of specimens made by injection and compression molding. Strength and modulus may show additive behavior or have positive or negative deviations, depending on the process conditions. Addition of an appropriate ethylene-propylene elastomer greatly improves the ductility of these blends but with corresponding decrease in strength and modulus. Choudhary et al.²³ showed that decrease in tensile strength and improvement in Izod impact strength in PP/HDPE/EPDM blends may be related to the fact that the overall morphology²⁴ as well as mechanism and the mode of fracture were greatly modified by the presence of such an additive. Tchomakov et al.²⁵ concluded that although elastomer addition improves impact strength, it will necessary decrease the tensile and flexural modulus in PP/HDPE/EPDM blends. The tailoring of impact-modified blends is always a trade-off between rigidity and ductility.

From the tensile measurement and dynamic mechanical analysis, the brittleness, B , was calculated:

$$B = 1/(\varepsilon_b E') \quad (1)$$

where E' , storage modulus, corresponds to 25°C from testing at 1.0 Hz and a value of ε_b , elongation at break, taken also at that temperature.²⁶

The results of B are presented in Table 1. Pure PP showed less brittleness than pure LDPE, and the values of B of binary PP/LDPE 80/20 and 20/80 blends were between the B of pure homopolymers. B values of PP/LDPE 60/40 and 40/60 blends were much higher compared with pure homopolymers and already mentioned binary blends. This is in accordance with poor elongation at break values of PP/LDPE 60/40 and 40/60 blends, as explained earlier. Addition of EPDM decreased the B values in all PP/LDPE blends. The higher EPDM addition did not have significant effect on the B values of the blends. More pronounced effect on B value by the EPDM addition was obtained in PP/LDPE 60/40 and 40/60 blends, which is in accordance with the same effect on elongation values. Addition of EPDM decreased brittleness and, at the same time, increased the Izod impact strength.

Dynamic mechanical analysis

Phase structure and stiffness of PP/LDPE blends with and without EPDM obtained using dynamic mechanical analyzer (DMA) are presented in Figures 3 and 4 and Table 2. The

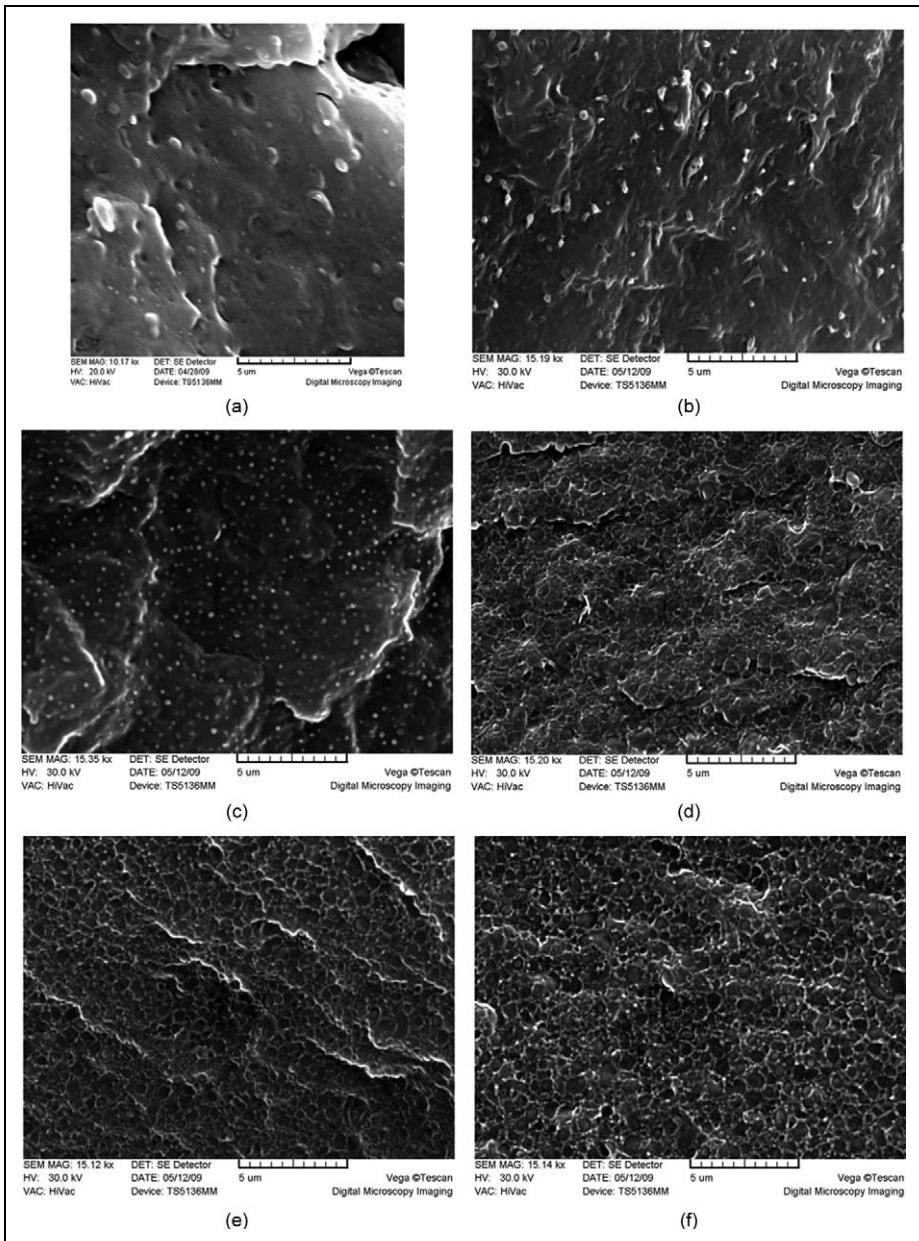


Figure 2. Scanning electron microscope (SEM) images of PP/LDPE/EPDM blends: (a) 80/20/0, (b) 80/20/5, (c) 80/20/7, (d) 20/80/0, (e) 20/80/5 and (f) 20/80/7. PP: polypropylene; LDPE: low-density polyethylene; EPDM: ethylene-propylene-diene.

Table 1. Values of brittleness B expressed as $10^{10}B/(\%Pa)$ for PP/LDPE and PP/LDPE/EPDM blends.

PP/LDPE/EPDM	B (% Pa)	ϵ_b /%	E'/Pa
100/0/0	0.129	21.05	3.67E + 09
0/100/0	0.308	48.40	0.67E + 09
80/20/0	0.216	24.64	1.88E + 09
80/20/5	0.152	36.88	1.78E + 09
80/20/7	0.140	47.52	1.51E + 09
60/40/0	0.887	7.67	1.47E + 09
60/40/5	0.148	49.69	1.36E + 09
60/40/7	0.156	48.59	1.32E + 09
40/60/0	0.893	9.91	1.13E + 09
40/60/5	0.209	46.73	1.02E + 09
40/60/7	0.209	47.71	1.00E + 09
20/80/0	0.299	33.77	0.99E + 09
20/80/5	0.229	48.99	0.89E + 09
20/80/7	0.250	49.27	0.81E + 09

PP: polypropylene; LDPE: low-density polyethylene; EPDM: ethylene-propylene-diene.

glass transitions of each component were determined from loss modulus (E'') in DMA spectra, and interactions between phases were valued through glass transition shifts (Table 2). Addition of LDPE to PP decreased a storage modulus as an amount of LDPE increased (Figure 3). In the case of PP/LDPE blends at $-100^\circ C$, the E' is higher than at $25^\circ C$ because the testing temperature is below the T_g of the PP, which is in glassy state and results in a higher modulus. EPDM decreased the storage modulus in all PP/LDPE blends as amount of LDPE and EPDM increased. At $-100^\circ C$, the storage modulus of all PP/LDPE/EPDM blends had higher values compared with the storage modulus at $25^\circ C$ (Figure 3) because at negative temperature, LDPE and EPDM are in a glassy state. Thus, it had a higher modulus, whereas at $25^\circ C$, the LDPE and EPDM are in a rubbery state.²⁷

According to McCrum et al.,²⁸ PP exhibited three relaxations peaks, α , β , and γ relaxation maximum. β relaxation maximum is believed to correspond to the glass transition temperature ($28.72^\circ C$) (Table 2), and α -relaxation, which looks like a shoulder, is related to a slip mechanism of polymer chains in the crystallites. The γ -peak is due to the motions of small-chain groups like methyl and methylene and is present at negative temperature. The neat LDPE has two relaxation maximums, one at a negative temperature ($-123.7^\circ C$) (Table 2), γ relaxation, believed to be associated with segmental motion of as few as three or four methylene groups in the carbon-carbon backbone in the amorphous phase and considered the primary glass transition of PE, and another at a positive temperature, α relaxation, associated with the branching of the polyethylene backbone.²⁹⁻³¹ The neat EPDM has glass transition temperature (T_g) at $-40.08^\circ C$ (Table 2). The PP/LDPE system with and without EPDM is very complex because of several relaxations obtained in the DMA spectra. Because of this complexity, the focus was on glass transition of each phase in noncompatibilized and compatibilized blends.

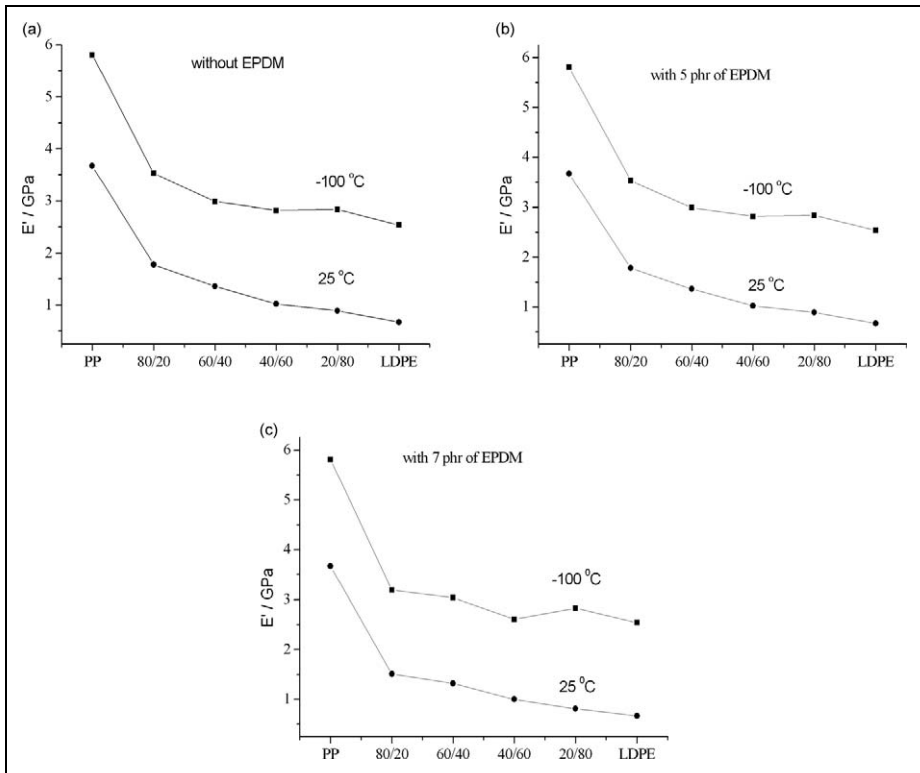


Figure 3. Storage modulus (E') vs. temperatures (T) of (a) PP/LDPE without EPDM, (b) PP/LDPE with 5 phr of EPDM and (c) PP/LDPE with 7 phr of EPDM. PP: polypropylene; LDPE: low-density polyethylene; EPDM: ethylene-propylene-diene.

Addition of LDPE to PP in PP/LDPE blends increased a T_g of LDPE, compared with pure LDPE. In the same blends, T_g of PP phase was decreased compared with pure PP. DMA spectra showed the two glass transitions in PP/LDPE blends (Figure 4 (a-d)), one of PP phase and one of LDPE phase, which indicated their incompatibility. It is also evident that the shift of T_g in PP phase and the shift of T_g in LDPE phase toward one another (Figure 4 (a-d)) indicated some kind of interaction between the phases, showing that the two components had a certain degree of compatibility.

Dong et al.⁹ reported that PP had a limited miscibility with highly branched polyethylene, LDPE. They observed using transmission electron microscope phase separation but with evidence of a small portion of PP being dissolved in the LDPE. Avalos et al.¹⁰ also studied PP/LDPE blends and showed that a small addition of LDPE (10 wt.%) caused the depression of spherulite growth rate of PP and increased the chain-folding energy in PP crystallization. They interpreted that as a partial miscibility of PP and LDPE in the melt.

Addition of EPDM (5 phr) in all PP/LDPE blends decreased the T_g values of the PP phase (Figure 4 (a-d), Table 2) compared with noncompatibilized blends. The increase in

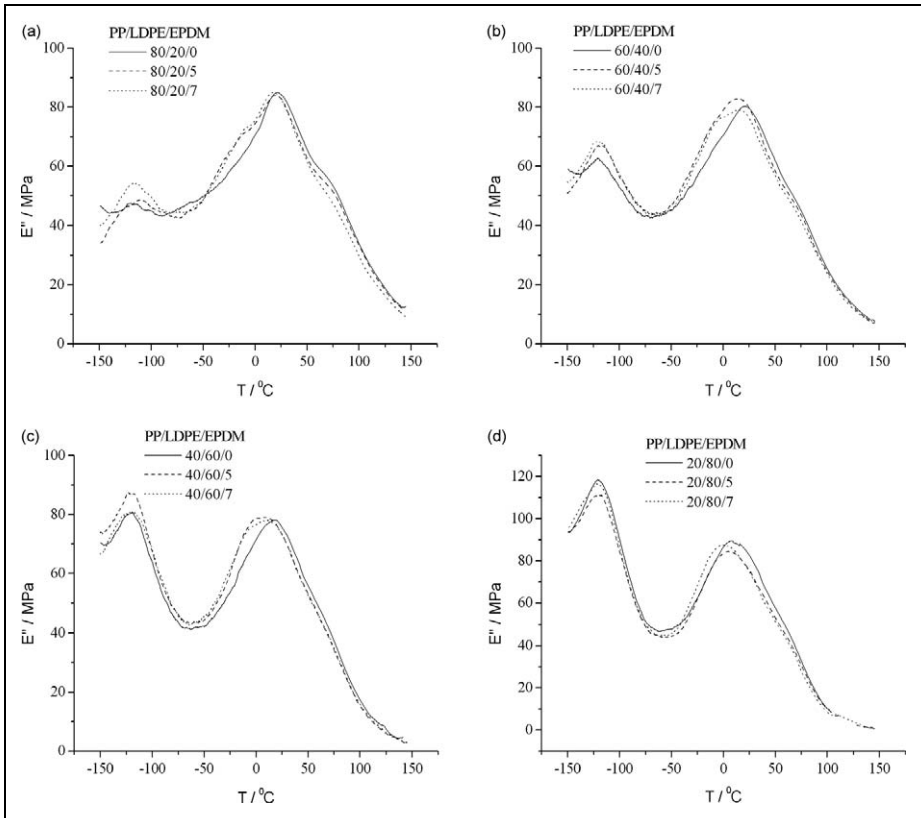


Figure 4. Loss modulus (E'') vs. temperatures (T) of PP/LDPE blends with and without EPDM from DMA spectra's: (a) 80/20, (b) 60/40, (c) 40/60 and (d) 20/80. PP: polypropylene; LDPE: low-density polyethylene; EPDM: ethylene-propylene-diene.

T_g values of LDPE phase upon an addition of EPDM (5 phr) was observed in PP/LDPE 80/20 blend and 60/40 blend, whereas the addition of EPDM in blends with LDPE matrix (40/60 and 20/80) did not affect the T_g of LDPE phase (Table 2). Compared with noncompatibilized blends, compatibilized blends had T_g s of LDPE and PP phases, which shifted toward one another. This indicated an improvement of compatibility, as well as an effect of EPDM as compatibilizer for PP/LDPE blends. Xiao et al.³² studied the miscibility of EPDM/PP blends and proved that the phenomenon of the peak shift of EPDM demonstrated that there was interpenetration between the noncrystalline portion of PP and the interface of EPDM. This indicated that the two components had a certain degree of miscibility. Addition of higher EPDM content (7 phr) (Figure 4 (a-d)) decreased the T_g of PP phase compared with pure PP component, as well as T_g of PP phase in PP/LDPE blends. The T_g of LDPE phase increased compared with pure LDPE. In PP/LDPE blends with PP matrix, the addition of higher EPDM amount increased T_g s of LDPE phase compared with noncompatibilized blends, whereas in the 40/60 blend,

Table 2. Glass transition values of each component in PP/LDPE and PP/LDPE/EPDM blends from loss modulus (E'') in DMA spectra.

PP/LDPE/EPDM	$T_g/^\circ\text{C}$ (PP phase)	$T_g/^\circ\text{C}$ (EPDM phase)	$T_g/^\circ\text{C}$ (PE phase) blends
100/0/0	28.72	/	/
80/20/0	20.21	/	-118.6
80/20/5	19.97	/	-110.6
80/20/7	17.26	/	-116.6
60/40/0	20.25	/	-121.6
60/40/5	13.27	/	-118.5
60/40/7	14.27	/	-120.5
40/60/0	16.99	/	-120.6
40/60/5	4.20	/	-120.5
40/60/7	9.14	/	-120.6
20/80/0	8.27	/	-120.5
20/80/5	5.02	/	-120.6
20/80/7	-1.91	/	-122.6
0/100/0	/	/	-123.7
0/0/100	/	-40.08	/

PP: polypropylene; LDPE: low-density polyethylene; EPDM: ethylene-propylene-diene.

the EPDM did not affect T_g of LDPE phase. In the 20/80 blend, 7 phr of EPDM decreased T_g of LDPE phase, which was closer to the T_g of pure LDPE. In almost all PP/LDPE blends with higher EPDM content, the T_g s of PP and LDPE phases approached one another, which indicated a better compatibility compared with noncompatibilized blends. This is in accordance with obtained morphology and mechanical properties. Krivoguz et al.³³ showed that the PP/LDPE blends grafted with itaconic acid (IA) had the approaching values of T_g , which allowed us to believe that in PP/LDPE-g-IA systems, interactions between PP and LDPE lead to partial mutual dissolution.

Morphology of the blends

Figure 2 (a-f) shows scanning electron micrographs of PP/LDPE blends with and without EPDM. PP/LDPE 80/20 blend (Figure 2 (a)) with PP matrix revealed coarse, two-phase morphology, with LDPE particles incorporated in PP matrix. The voids presented in PP matrix are a result of the pullout effect of LDPE particles. The coarse morphology of the blends and the detachment of dispersed particles confirmed bad adhesion at the interface between the homopolymers³⁴ and pointed to incompatibility, which is likely to stem from the high interfacial tension occurring between components during the melt mixing process. Addition of EPDM (5 phr) (Figure 2 (b)) in PP/LDPE 80/20 blend resulted in finer morphology. The LDPE particles were attached in PP matrix and the voids from LDPE particles were reduced. PP/LDPE/EPDM 80/20/7 blend (Figure 2 (c)) showed fine dispersion of LDPE particles in the PP matrix, without voids, as a result of improved interfacial adhesion. Addition of EPDM reduced the

particle size, especially in 80/20/7 blend. Reduction is also a consequence of improved interfacial adhesion and decreased interfacial tension. Better homogeneity of PP/LDPE 80/20 blend was achieved with EPDM addition.

SEM micrograph of PP/LDPE 20/80 blend (Figure 2 d) revealed a two-phase morphology with LDPE matrix comprising net and droplet PP spread inside the LDPE matrix. Those droplets, composed of PP, appear during the breaking of specimens in liquid nitrogen, whereas the breaking alone of LDPE provided filaments.^{1,6} Additions of EPDM (Figure 2 e and f) resulted in fine morphology and better homogeneity.

Lovinger and Williams³⁵ found that for blends of less than 50% PP, interpenetrating networks of the two polymers were observed, whereas for blends containing more than 50% PP, the morphology was typified by polyethylene islands dispersed in a PP matrix. Spherulitic morphology was observed for all blends. The size of the PP spherulite was drastically reduced with the presence of PE. When the well-mixed PE/PP blends were cooled from temperatures above the melting point of PP to a crystallization temperature well above the melting point of PE, polyethylene-dispersed melt droplets were incorporated in the intraspherulite regions during the growth of PP spherulite.³⁶ In general, the PE occlusions increased in size with an increase in PE content of the blends.²

Conclusion

The addition of EPDM to PP/LDPE blends improved the mechanical properties, especially the Izod impact strength in LDPE-rich blends and those with higher EPDM content. Brittleness was decreased by EPDM addition. Storage modules were decreased with LDPE addition to PP as well as with EPDM addition to PP/LDPE blends. Phase interactions of PP/LDPE with and without EPDM were observed by glass transition shifts. Interfacial adhesion was improved by EPDM addition. The compatibilizing efficiency of EPDM on PP/LDPE blends was confirmed on improvement of elongation, Izod impact strength and brittleness, and morphological and phase structure but did not show compatibilizing efficiency on the improvement of tensile strength, except for PP/LDPE 80/20 blend.

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