

Improving Low-Density Polyethylene/Poly(ethylene terephthalate) Blends with Graft Copolymers

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ABSTRACT: Blends of low-density polyethylene (LDPE) and poly(ethylene terephthalate) (PET) were prepared with different weight compositions with a plasticorder at 240°C at a rotor speed of 64 rpm for 10 min. The physico-mechanical properties of the prepared blends were investigated with special reference to the effects of the blend ratio. Graft copolymers, that is, LDPE-grafted acrylic acid and LDPE-grafted acrylonitrile, were prepared with γ -irradiation. The copolymers were melt-mixed in various contents (i.e., 3, 5, 7, and 9 phr) with a LDPE/PET blend with a weight ratio of 75/25 and used as compatibilizers. The effect of the compatibilizer contents on the physico-mechanical properties and equilibrium swelling of the binary blend was investigated. With an increase in the

compatibilizer content up to 7 phr, the blend showed an improvement in the physico-mechanical properties and reduced equilibrium swelling in comparison with the uncompatibilized one. The addition of a compatibilizer beyond 7 phr did not improve the blend properties any further. The efficiency of the compatibilizers (7 phr) was also evaluated by studies of the phase morphology (scanning electron microscopy) and thermal properties (differential scanning calorimetry and thermogravimetric analysis). © 2008 Wiley Periodicals, Inc. *J Appl Polym Sci* 110: 1929–1937, 2008

Key words: compatibility; graft copolymers; mechanical properties; polyethylene (PE); thermal properties

INTRODUCTION

Poly(ethylene terephthalate) (PET) is a widely used engineering plastic.¹ Especially in the field of soft-drink-bottle applications, a steep rise in PET consumption has occurred in recent years.² Weight saving and breakage security are the major reasons for this success. As a result, fast-growing quantities of used, secondary PET material have become available.²

In the area of polymer blends, the challenge is to recycle mixed plastic³ while at the same time maintaining acceptable levels of properties and cost. Blends of PET with other polymers, particularly polyolefins (POs), may offer an attractive balance of mechanical and barrier properties and processability.

In this context, the study of PET/PO blends is of considerable technological importance. Compatibilization is then a necessary step to obtain blends of PET and PO with good properties from both virgin and recycled materials. The incompatibility of these two classes of polymers,⁴ polyesters and POs, gives rise to bad morphology, a lack of adhesion between

the phases, and then poor mechanical and barrier properties.

In the past 2 decades, many researchers have focused on the control of the morphology and interfaces of PET/PO blends to improve their compatibility. The compatibilizers have included an unfunctionalized hydrogenated styrene-butadiene-styrene triblock copolymer (SEBS) or ethylene-propylene-diene monomer (EPDM).^{5,6} Functionalized grades of SEBS containing maleic anhydride (MA),⁷⁻⁹ glycidyl methacrylate,⁹ and diethyl maleate¹⁰ have been used. SEBS has also been functionalized with 2-hydroxyl ethyl methacrylate to obtain a more reactive compatibilizer precursor.¹⁰ Reactive copolymers have also been tested successfully. Thus, the transesterification of PET with an ethylene/vinyl acetate copolymer gave a graft copolymer that acted as a compatibilizer.¹¹ An ethylene/vinyl alcohol copolymer was derived by simple and low-cost reactions from commercial ethylene/vinyl acetate copolymers and used to promote the compatibilization of this blend through different routes.¹² A polyethylene (PE)-MA copolymer¹³ was found to be effective in compatibilizing this blend by forming a compatibilizer *in situ*. Most works have been concerned with the use of commercial copolymers such as PO-grafted acrylic acid (AA),¹⁴ ethylene/glycidyl

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methacrylate,^{7,15–18} an ethylene/ethyl acrylate/glycidyl methacrylate terpolymer,^{7,18} an MA ethylene–MA copolymer,⁷ and an MA-modified ethylene–propylene copolymer¹⁹ or ionomer resins.²⁰ Boutevin et al.²¹ synthesized graft copolymers through the ozonization of PE followed by the grafting of vinylic or acrylic monomers and used them as compatibilizers for PET/high-density PE. A new class of copolyesters²² have been usefully employed as compatibilizing agents for PET/high-density PE blends. Patents also have shown the efficiency of EPDM-*g*-GMA^{23,24} as an impact modifier for PET/PE blends. Improved morphology, and better mechanical properties have been observed with the above mentioned components.

The goal of the work was PET modification or compatibilization of its blend with low-density polyethylene (LDPE) by the introduction of grafted copolymers such as low-density polyethylene grafted acrylic acid (LDPE-*g*-AA) or low-density polyethylene grafted acrylonitrile (LDPE-*g*-AN) as compatibilizers. Besides the importance of mechanical property improvement, an additional incentive was the social demand for the utilization of a recyclable engineering plastic.

EXPERIMENTAL

Materials

LDPE and PET were the blend components used in this study. LDPE was a heavy-duty grade [Dow Co., Midland, MI; density = 921 kg/m³ at 23°C and melt flow index = 0.26 g/10 min (190°C, 2.16 kg)]. PET was a waste soft-drink-bottle-grade material with a number-average molecular weight of 26×10^3 g/mol, an intrinsic viscosity of 0.76 dL/g, and melting point of 257°C. AA and acrylonitrile (AN) monomers were purchased from Sigma–Aldrich Co. (Bellefonte, PA) The solvents and all chemicals were used as received.

Methods

Synthesis of the graft copolymers

The direct radiation grafting method^{25,26} was chosen to synthesize the graft copolymers, LDPE-*g*-AA and LDPE-*g*-AN.

In this method, LDPE powder (mesh size = 80–90 μm) was washed with acetone and dried in a vacuum oven for 24 h at 60°C. AA monomer or AN monomer was dissolved in a suitable solvent (i.e., water or dimethylformamide, respectively) to obtain different concentrations (10–90 wt %) for each monomer.

In glass ampules, a known weight of completely dried LDPE (50 g) was mixed with the prepared

monomer solutions under nitrogen gas bubbling to avoid oxidation.

Then, the samples were irradiated with 30 kGy at a dose rate of 8.75 kGy/s with γ -irradiation (cobalt 60 cell, 220 V).

The prepared grafted copolymers (i.e., LDPE-*g*-AA and LDPE-*g*-AN) were removed and washed thoroughly in a reflux system with the appropriate solvent (i.e., water or dimethylformamide) to extract the residual monomer AA or AN and homopolymer that may have accumulated in the grafted copolymers.

Finally, the grafted samples were washed with distilled hot water, dried in a vacuum oven at 50–60°C for 24 h, and weighed to calculate the different grafting yield percentages according to the different monomer concentrations used. The degree of grafting was calculated as follows:

$$\text{Degree of grafting(\%)} = [(w_g - w_0)/w_0] \times 100$$

where w_g and w_0 represent the weights of the grafted and initial samples, respectively.

Melt processing

LDPE/PET blends of various weight compositions (i.e., 0/100, 25/75, 50/50, 75/25, and 100/0 wt %) with and without compatibilizers were prepared with a Brabender plasticorder (C. W. Bra Instrument, Inc., Hackensack, NJ). LDPE-*g*-AA and LDPE-*g*-AN were used as compatibilizers in various contents (i.e., 3, 5, 7, and 9 phr). At the start, PET was introduced into the Brabender plasticorder preheated to 240°C for 5 min, and LDPE was added thereafter. The mixer was operated at 240°C, the rotor speed being maintained at 64 rpm for 10 min. The molten mix was quickly removed from the Brabender plasticorder and mixed with different concentrations of the tested compatibilizers in a two-roll mill (outside distance = 470 mm, working distance = 300 mm, speed of slow roll = 24 rpm, and fraction ratio = 1.4 : 1). The blends were removed and subsequently compressed in an electrically heated hydraulic press at about 220°C for 10 min under 7 kN into thin sheets about 1 mm thick, and from these, test specimens were prepared.

Property evaluation

The tensile strength (σ_R), elongation at break (ϵ_R), and Young's modulus (E) were determined with a Zwick 1425 testing machine (Germany). The compressed sheets were cut into dumbbell-shaped specimens with appropriate punching dies with a width of 4 mm (DIN 53504STAB1N^EF). The specimens, with a width of 4 mm, a neck length of 15 mm, and

a thickness of 1–1.5 mm, were tested at a crosshead speed of 50 mm/min and with a load cell of 10–20 N.²⁷ The hardness of test specimens at least 6 mm thick was measured with a Shore A durometer (Härteprüfer; DIN 53505).²⁸ Equilibrium swelling of the test pieces was carried out in the solvent toluene. About 0.1–0.2 g of each specimen was weighed in a weighing bottle, which was covered with toluene for 24 h so that the state of equilibrium swelling could be reached. The swollen samples were weighed and then dried in an oven to a constant weight. The last weight was taken as the correct weight of the sample free from dissolved matter. The swelling percentage ($Q\%$) of the samples was calculated as follows:

$$Q\% = [(w - \hat{w})/\hat{w}] \times 100$$

where w and \hat{w} represent the weights of the samples after swelling and free from dissolved matter, respectively.²⁹ All these tests were performed at room temperature ($25 \pm 1^\circ\text{C}$), and the reported results were averaged from a minimum of five specimens. The surface topography of the samples was analyzed by scanning electron microscopy (SEM). The samples were sputter-coated with gold and examined in a JEOL SEM-25 scanning electron microscope (Japan). The middle part of the cross section of the specimens was chosen as representative of the overall morphology. The thermal studies were made with differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA). Analysis by DSC was performed with a Mettler model TA 3000 calorimeter (Shimadzu, Japan). The running conditions were a nominal weight of 20 mg, a heating rate of $10^\circ\text{C}/\text{min}$, and an N_2 flow rate of 10 mL/min. An appropriate temperature range was chosen according to the particular composition of the studied blend. The melting temperatures (T_m 's) of the samples were taken as the endothermic peaks of the thermograms. TGA was conducted with a Shimadzu TGA-4000 system (Shimadzu, Japan). The studies were performed in a nitrogen atmosphere at a flow rate of 10 mL/min and a heating rate of $10^\circ\text{C}/\text{min}$ from 0 to 600°C . The weight of the samples was 5 mg in all cases. All samples were previously heated to 100°C for 5 min to eliminate residual water (humidity)

RESULTS AND DISCUSSION

The importance of the interface in multiphase polymer systems has been long recognized.³⁰ Physical and chemical interactions across the phase boundaries are known to control the overall performances of immiscible polymer blends. Graft copolymerizations of AA or AN monomers with LDPE powder have been widely studied by various research-

ers.^{25,26,31,32} The copolymer products have been used extensively in the area of polymer blending as compatibilizers or in composite matrices.^{33,34}

Uncompatibilized LDPE/PET blends

The physicomechanical properties (σ_R , ϵ_R , E , and Shore A hardness) of the pure polymers and the prepared blends without the compatibilizers are listed in Table I. The data indicate that the addition of LDPE as a PO to the blends improved the brittle character of PET. As the LDPE content increased within the blends, the stiffness and brittleness of the blends decreased gradually with an associated increase in ϵ_R . However, the poor mechanical properties of uncompatibilized LDPE/PET blends indicated that these blends were still incompatible during the thermomechanical processing.

Compatibilized LDPE/PET blends

In this work, a third component (compatibilizers) capable of specific interactions with the blend constituents (75/25 wt % LDPE/PET) was added to improve compatibility. The choice of LDPE-*g*-AA and LDPE-*g*-AN as compatibilizers was based on the miscibility or reactivity of their segments with at least one of the blend components.

Effect of the monomer content on the grafting yield

The backbone of LDPE was grafted with AA or AN as a unity monomer. The effects of the monomer concentrations of both AA and AN on the grafting percentage are shown in Figure 1. Experiments were performed in the monomer concentration range of 10–90 wt % in the solvents at an irradiation dose of 30 kGy. Most of the monomers were grafted onto LDPE, providing a high grafting yield (i.e., 350% for 70 wt % AN and 300% for 50 wt % AA). However, at higher monomer concentrations, a reduction of the grafting yield was observed. This may be

TABLE I
Physicomechanical Properties of the Pure Polymers and Uncompatibilized LDPE/PET Blends

Blend ratio		Physicomechanical properties			
LDPE	PET	σ_R (N/mm ²)	ϵ_R (%)	E (N/mm ²)	Shore A hardness
0	100	39.18	62	1900	95
25	75	35.14	210	990	83
50	50	27.12	350	860	70
75	25	17.80	520	720	60
100	0	14.50	610	500	50

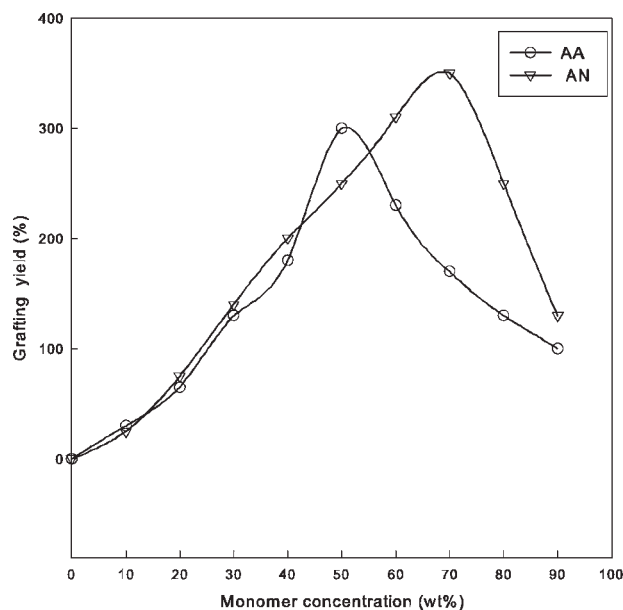


Figure 1 Effect of the AA and AN monomer concentrations on the grafting yield of the LDPE powder at an irradiation dose of 30 kGy.

attributed to the increasing trend of side reactions, such as chain transfer to monomers as the predominant termination process or homopolymer production of both AA and AN.²⁵

Physicomechanical properties

Table II and Figures 2–5 summarize the effects of LDPE-*g*-AA and LDPE-*g*-AN grafted copolymers as compatibilizers with various contents (3, 5, 7, and 9 phr) on the physicomechanical properties of LDPE/PET (75/25 wt %) blends. The tested grafted copolymers had an influence on the mechanical behavior of the blends because all the mechanical properties were improved in each case with respect to the uncompatibilized blends. The properties increased

up to 7 phr compatibilizer and then leveled off. Also, all the compatibilized blends became ductile; in particular, the ternary blend with 7 phr LDPE-*g*-AA showed a high value of ϵ_R in comparison with the blend with 7 phr LDPE-*g*-AN. Although compatibilized blends with 7 phr LDPE-*g*-AN exhibited higher values of σ_R , E , and hardness, a higher proportion of the compatibilizer (i.e., 9 phr), achieved the opposite effect. This may be attributed to the formation of a third phase in the system, which destabilized the phase morphology and lowered the mechanical properties.³⁵

The variation of the mechanical data of the ternary blends with different compatibilizers depended on the morphologies of the blends with respect to the size of the dispersed phase, interfacial adhesion, and dispersion of the minority phase in the matrix. Also, this variation depended on the chemical structure of the tested compatibilizers, which determined the main mechanisms of phase interaction, as discussed later.

Equilibrium swelling

The effect of the blend composition on the equilibrium swelling of the uncompatibilized LDPE/PET blends in the solvent toluene for 24 h is shown in Figure 6. The equilibrium swelling increased with increasing LDPE content in the composition. These results indicated that there was no adhesion between the two phases, suggesting the incompatibility of the components.

The effects of the compatibilizer type and content (i.e., 3, 5, 7, and 9 phr) on the equilibrium swelling of LDPE/PET (75/25 wt %) are illustrated in Figure 7. The results indicated that the addition of a compatibilizer to the blend decreased its equilibrium swelling value in comparison with the uncompatibilized one. This observation was perhaps due to the enhancement of the adhesion between LDPE and PET phases. With the increase in the compatibilizer

TABLE II
Physicomechanical Properties of the Compatibilized LDPE/PET (75/25 wt %) Blends

Blend ratio		Compatibilizer		Physicomechanical properties			
LDPE	PET	Type	Concentration (phr)	σ_R (N/mm ²)	ϵ_R (%)	E (N/mm ²)	Shore A hardness
75	25	—	—	17.80	520	720	60
75	25	LDPE- <i>g</i> -AA	3	19.83	525	725	62
			5	25.44	540	800	75
			7	34.98	630	925	88
			9	32.38	580	900	83
75	25	LDPE- <i>g</i> -AN	3	20.26	522	744	70
			5	28.44	530	820	78
			7	38.49	600	930	92
			9	36.26	570	920	90

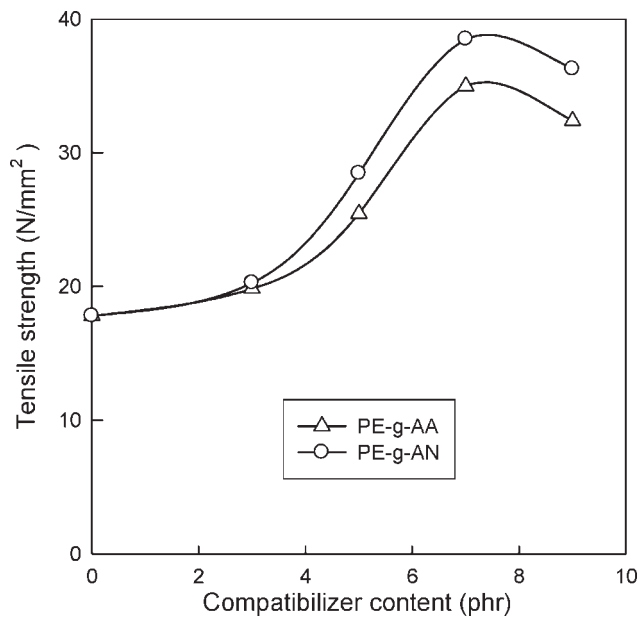


Figure 2 σ_R values of the LDPE/PET (75/25 wt %) blends versus the compatibilizer content.

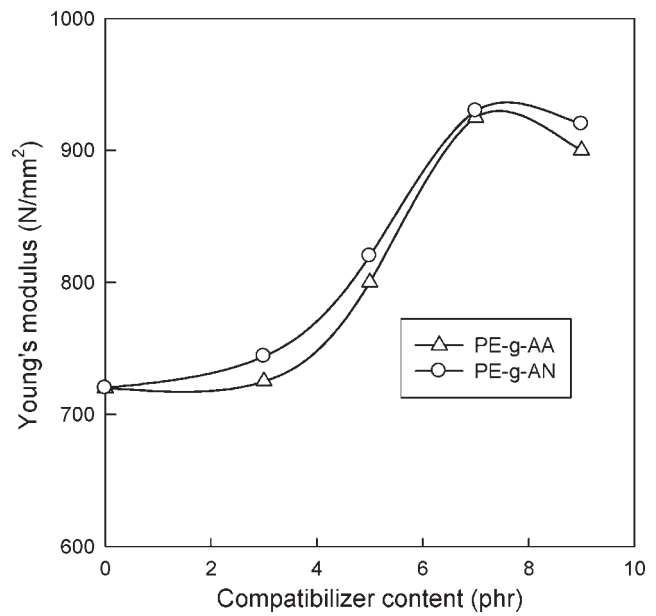


Figure 4 E values of the LDPE/PET (75/25 wt %) blends versus the compatibilizer content.

content, the equilibrium swelling decreased up to 7 phr and then leveled off for the two tested compatibilizers. Also, the compatibilized blends with LDPE-g-AN exhibited lower equilibrium swelling with respect to those with LDPE-g-AA. This was due to the self-reinforcing character of AN. Thus, this made blends with LDPE-g-AN stiffer than those with LDPE-g-AA and subsequently reduced the equilibrium swelling further. These results were in accordance with σ_R , E , and hardness values (see Table II).

In the following, because only the type of compatibilizer was varied in the ternary blends, they are characterized by the name of this component; thus, a PE-g-AA ternary blend signifies a blend of LDPE and PET with a weight ratio of 75/25 with 7 phr PE-g-AA as a compatibilizer.

Blend morphology

The fractured surface and intersection of the LDPE/PET blend with and without compatibilizers were

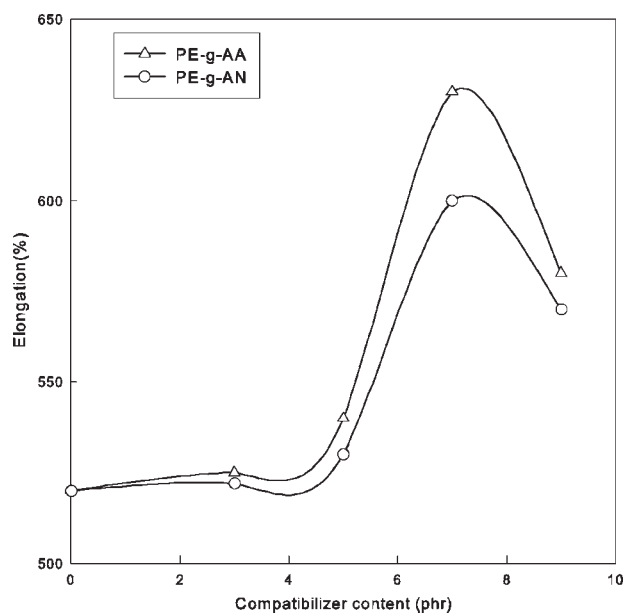


Figure 3 ϵ_R values of the LDPE/PET (75/25 wt %) blends versus the compatibilizer content.

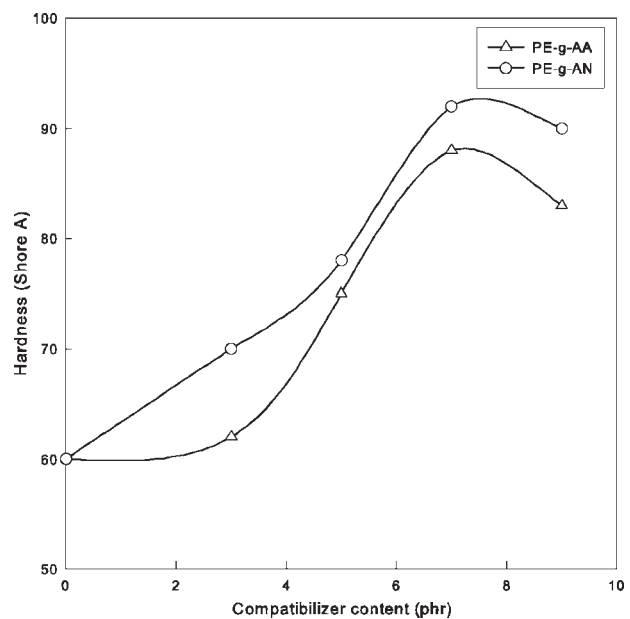


Figure 5 Hardness of the LDPE/PET (75/25 wt %) blends versus the compatibilizer content.

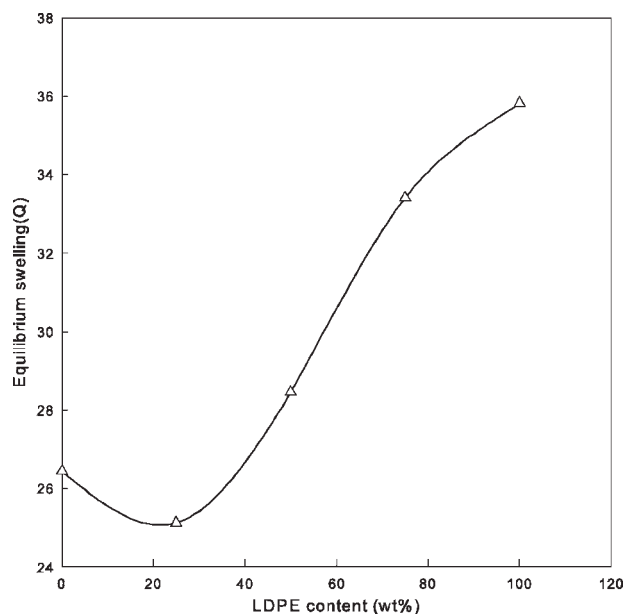


Figure 6 Effect of the LDPE content on the equilibrium swelling of the LDPE/PET blends in toluene solvent.

observed by SEM to study the effects of the compatibilizers on the morphology. The fractured surfaces of the blends without compatibilizers possessed a coarse morphology [Fig. 8(a)] with a larger domain size in comparison with the compatibilized ones. The larger particle size, with no evidence of adhesion between the matrix (LDPE) and dispersed phase (PET), confirmed the incompatibility of the two components, although the PE-g-AA ternary blend [Fig. 8(b)] showed a more regular dispersion

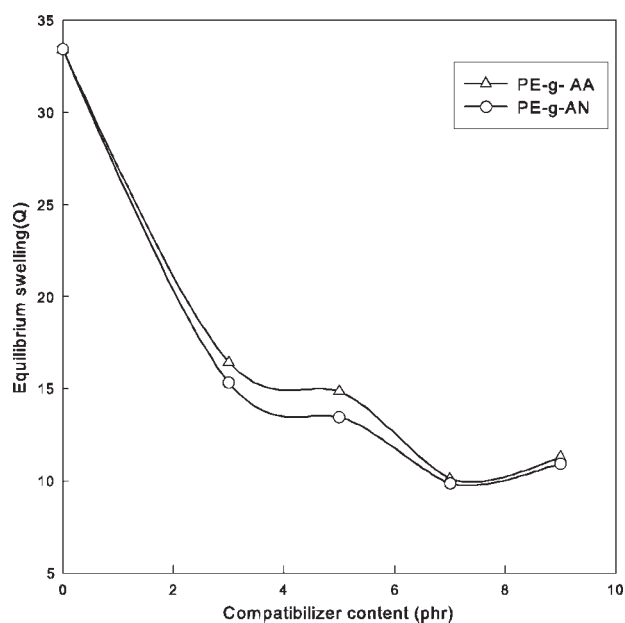


Figure 7 Variation of the equilibrium swelling of the LDPE/PET (75/25 wt %) blends in toluene solvent as a function of the compatibilizer type and content.

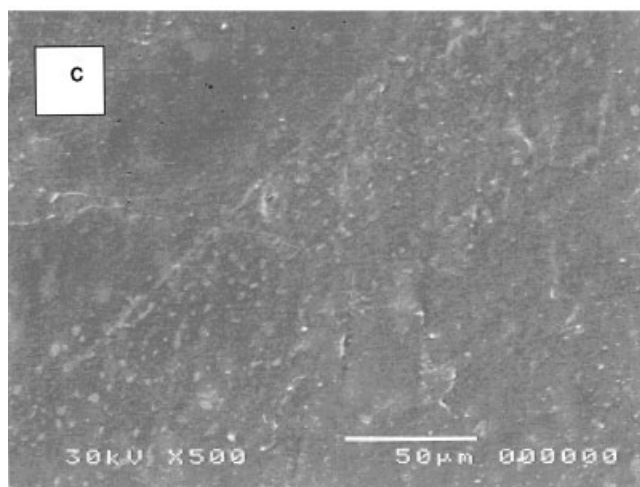
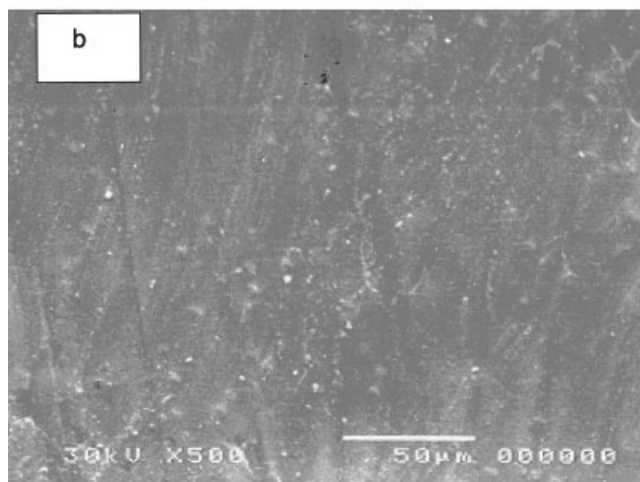
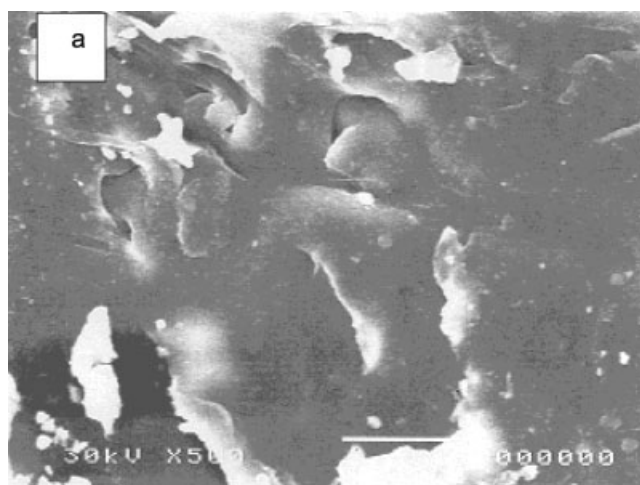


Figure 8 SEM of the LDPE/PET (75/25 wt %) surfaces with (a) 0 phr compatibilizer, (b) 7 phr PE-g-AA, and (c) 7 phr PE-g-AN.

of PET nodules inside the LDPE matrix, a smaller nodule size, and an improvement in interfacial adhesion with respect to the PE-g-AN ternary blend [Fig. 8(c)]. The surface photographs were in line with the intersection inspection [Fig. 9(d-f)].

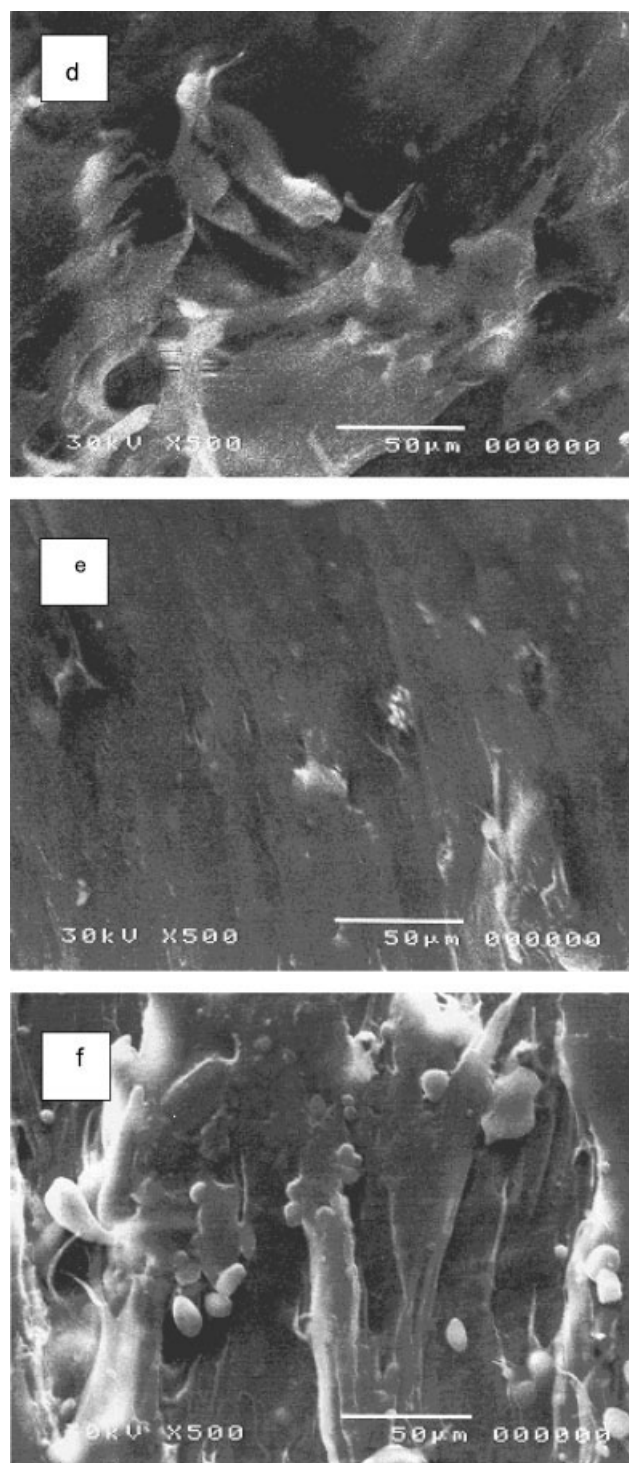


Figure 9 SEM of the LDPE/PET (75/25 wt %) intersection with (d) 0 phr compatibilizer, (e) 7 phr PE-g-AA, and (f) 7 phr PE-g-AN.

Given the different blend morphologies, one could qualitatively explain the mechanical properties. The ultimate tensile properties and especially ϵ_R are very sensitive to the blend component adhesion strength³⁶ and are routinely used to evaluate the degree of compatibilization in polymer alloys.¹⁵ The

improvement of the interfacial adhesion due to localization of the additive at the interface and the presence of small nodules of the additive inside the dispersed phase could explain the good values of ϵ_R for the PE-g-AA ternary blend (see Table II). The compatibilization performance difference between these two compatibilizers was due to their types of interactions with the PET segments. During melt mixing, the compatibilizer with a polyolefinic chain was expected to mix with LDPE. In the former case, PE-g-AA with AA functions was linked to PET chains by hydrogen bonding.^{37,38} On the other hand, in the case of the AN-containing compatibilizers, the compatibilizing efficiency of PE-g-AN was due to the dipolar interaction between nitrile groups of PE-g-AN and PET segments. These kinds of interactions were weaker than the hydrogen links. Thus, in the case of the AA-containing compatibilizers, the dispersed phase was more efficiently stabilized, and more uniform submicrometer domains were obtained.

Thermal analyses

The effects of compatibilization on the polymer blends were investigated by the determination of their thermal properties.

The DSC results for the uncompatibilized and compatibilized blends are shown in Figure 10. The melting peaks of LDPE in the blends were actually the same as those in the pure state. All the T_m 's were about 111°C, as listed in Figure 10. These results were easy to understand because LDPE was the major phase in the blends. With respect to the T_m values of the PET component, it appeared that they were all somewhat lower than those of neat PET (with $T_m = 257^\circ\text{C}$) and dependent on the blend

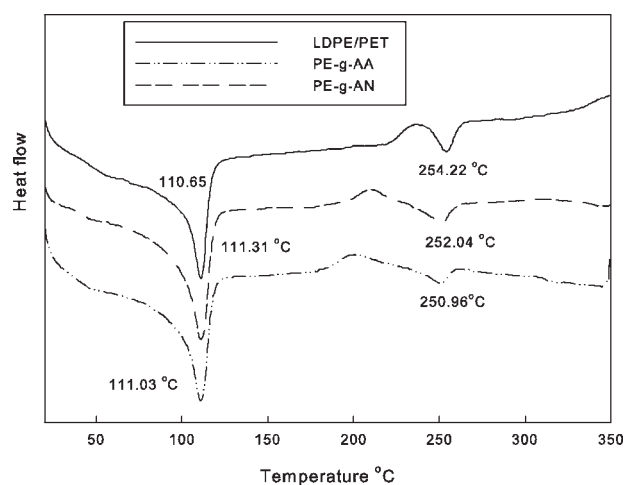


Figure 10 DSC thermogram of the uncompatibilized and compatibilized LDPE/PET (75/25 wt %) blends.

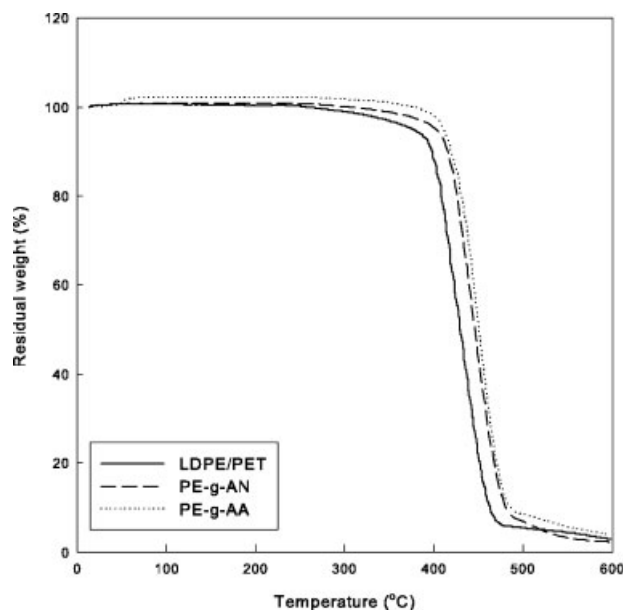


Figure 11 TGA thermograms of the uncompatibilized and ternary blends.

composition. The data in Figure 10 suggest that the T_m value of the PET component was related to the morphology of the blend: the smaller the PET domain size was, the lower its T_m value was. It was more pronounced in the PE-g-AA ternary blend. This result may have originated from the effect of interfacial tension between the two phases.^{39,40}

The TGA results for the uncompatibilized and ternary blends are shown in Figure 11 and summarized in Table III. Thermogravimetric curves (Fig. 11) showed that thermal degradation occurred in two stages. The TGA thermogram indicated the initial thermodegradation temperature (T_i) corresponding to a 5–7% weight loss; after that, degradation occurred rapidly. T_{10} , T_{30} , T_{50} , and T_{75} are the temperatures at which 10, 30, 50, and 75 wt % loss occurred, respectively, and the final decomposition temperature (T_f) corresponds to the temperature after which there was negligible weight loss. The uncompatibilized LDPE/PET blend (75/25) showed a T_i value of 296°C. T_i increased with the addition of a compatibilizer to the system. The T_i value increased from 296 to 347°C for PE-g-AN to 383°C for PE-g-AA ternary blends. Also, T_f for the compati-

bilized blends was higher than that for the uncompatibilized blends. T_f for the PE-g-AA ternary blends was as high as 498°C with the highest residual weight percentage, and this indicated that this blend was more stable than the other blends studied. The same trend was also observed for T_{10} , T_{30} , T_{50} , and T_{75} , as observed from Table III. Thus, the compatibilized blends displayed higher thermal resistance with a higher residual weight percentage than the uncompatibilized blends. This could be attributed to the fact that the addition of a compatibilizer to the system brought the stability of the dispersed phase due to compatibilization. On the other hand, the blends without compatibilizers tended to form a separate phase, thus destabilizing the phase morphology and lowering the thermal stability. This initiated degradation at a much lower temperature than expected.³⁵ In the presence of the graft copolymer compatibilizer (i.e., PE-g-AA), a finer dispersion and stable morphology [Figs. 8 and 9(b,e), respectively] were obtained, and the thermal stability increased compared with that of the PE-g-AN ternary blends. Thus, T_i and T_f supported the idea that the compatibilized blends were more resistant to thermal degradation than the uncompatibilized ones.

CONCLUSIONS

The compatibility of LDPE and PET is poor and can be enhanced by the addition of compatibilizers. In this study, LDPE/PET (75/25 wt %) polymeric blends were modified by the addition of compatibilizers; that is, LDPE-g-AA and LDPE-g-AN were prepared by the radiation-induced graft copolymerization of LDPE with AA and AN monomers. The effect of the monomer concentration (i.e., 10–90 wt %) on the grafting yield was also studied at an irradiation dose of 30 kGy. The compatibilization performance difference between these two compatibilizers lay in their route of interaction with the PET segment. Ternary blends with LDPE-g-AA (7 phr, 300% grafting yield for 50 wt % AA) showed an improvement in flexibility with a smooth surface and fine dispersion, which in turn lowered T_m and increased the thermal stability in comparison with those compatibilized with LDPE-g-AN. The addition of LDPE-g-AN (7 phr, 350% grafting yield for 70 wt

TABLE III
TGA of the Uncompatibilized and Ternary Blends

Sample code	Weight-loss temperatures (°C)						Residual weight (%)
	T_f	T_{75}	T_{50}	T_{30}	T_{10}	T_i	
LDPE/PET	475	396	415	430	443	296	6.1
LDPE-g-AN	486	413	433	445	459	347	7.9
LDPE-g-AA	498	418	438	450	468	383	9.2

% AN) increased the tensile properties with a sharp decrease in the equilibrium swelling. The addition of both compatibilizers beyond 7 phr did not improve the mechanical properties or equilibrium swelling any further.

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