

# Polyethylenes/PET Blend Compatibilization with Maleic Anhydride Modified Polyethylenes Obtained by a UV Preirradiation Process

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**ABSTRACT:** The compatibilization of HDPE/LDPE/LLDPE/PET blend during reactive extrusion, using compatibilizing agents, such as modified high, low, and lineal low density polyethylenes with maleic anhydride, was carried out. The agents were prepared in our laboratory by using a UV preirradiation process, containing different grafting and crosslinking degrees. The materials were compared with same maleic anhydride modified polyethylenes prepared by the traditional peroxide method in our laboratory and with a commercial maleic anhydride modified lineal

low density polyethylene. The mechanical and thermal properties, as well as their morphology, were evaluated in the compatibilized blends and changes in crystallization phases recorded. The elongation at break and impact strengths increased with compatibilization level and morphology was markedly more homogenous. © 2007 Wiley Periodicals, Inc. *J Appl Polym Sci* 104: 560–567, 2007

**Key words:** compatibilization; blends; maleic anhydride; UV irradiation

## INTRODUCTION

The big growth of the plastic industry is a consequence of the increasing substitution of the traditional materials like metals, glass, paper, and wood, by the synthetic polymers. This effect can be seen in different industrial sectors and perhaps the most representative is the packaging one, where we can observe how the paper bags has been massively substituted by film plastic bags. Most of the bottles for carbonated drinks were made, a few years ago, with glass, but nowadays these containers are fabricated with plastics.<sup>1,2</sup>

Such sector of packaging is responsible for most of the municipal solid waste (MSW) in the world, and the deposition and driving of such materials are a big problem for the environment. The percentage of plastics in the MSW is higher in the industrialized regions, comparing with third world countries,<sup>2,3</sup> but these are not free of plastic pollution. The composition of this plastics solid waste changes from one country to another, but without doubt, the polyethylenes and PET are the constituents covering near the 60% of such residues.<sup>2,4–11</sup> It was reported that in Mexico, the three types of common polyethylenes (LDPE, LLDPE, and HDPE) and PET are always present in the urban plastic solid waste in an amount of

around 70 wt %.<sup>4</sup> For this reason, it is interesting to study the compatibilization of blends of these polymers, opening the possibility of recycling these mixtures without the tedious separation process, which makes this activity economically attractive. However, studies of compatibilized blends with the three types of polyethylenes with PET are scarcely reported, like the Klementina and Riddick.<sup>8</sup>

The study of binary blends of such two polymers, polyethylene and PET has been of main importance in the last years, because of the increase in the use of plastic bottles for carbonated drinks. The main interest of such studies is related to the possibility of obtaining toughened PET by dispersion of a polyolefin phase,<sup>12</sup> mainly polyethylene. Recently, the availability of a considerable amount of postconsumer polyethylene and PET induced new studies concerning the development of convenient recycling routes.<sup>12–15</sup> The latter as a consequence of poor mechanical properties obtained after reprocessing plastic waste, by simple melt blending,<sup>14,15</sup> because of the incompatibility of these two polymers, giving rise to irregular morphology, gross phase segregation, and lack of adhesion between the phases.

For such reason, it is necessary to improve the phase interaction by addition of a compatibilizer, which usually consists of a graft or block copolymer, where one side of the copolymer is miscible with polyethylene and the other with PET. Some styrene block copolymers (SEBS, SBS) has been available for such purpose.<sup>12,16,17</sup> On the other hand, graft copolymers consist of a modi-

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fied polymer, miscible with polyethylene and containing a functional group, able to interact with PET terminal groups; such interaction could be, either through carbonyl groups and/or an *in situ* reaction, creating a grafted PE–PET copolymer. For this case the modification of polyethylene with maleic anhydride, glycidyl methacrylate or acrylic acid are the most commonly used.<sup>8,14,18–52</sup> All these compatibilizer materials have a function of decreasing the interfacial tension and increasing the adhesion and wettability, producing a stable morphology.<sup>12–17,53–61</sup>

In this work, the use of modified polyethylenes with grafted maleic anhydride, prepared in our laboratory by a UV preirradiation process, were evaluated in the compatibilization of the quaternary blends of polyethylenes (high, low, and linear low density) with PET, prepared by extrusion. For this purpose the thermal, mechanical, and morphological properties of compatibilized blends were examined and compared with both, blends prepared in our laboratory by using the traditional peroxide process and commercial LLDPE-g-MA.

## EXPERIMENTAL

### Materials

The materials used in this work were commercial resins, LDPE 20020, HDPE 60003 from PEMEX (Coatzacoalcos, México), LLDPE 2045A from DOW Chemical (Monterrey, Mexico), and PET IMPET 100 from CELANESE (México). It was also acquired a Dupont modified LLDPE known as Bynel (Monterrey, Mexico), as a commercial compatibilizer.

### Compatibilizing agents

About 12 compatibilizing agents were prepared, which basically consist of modified polyethylenes with MAH. Four of them were using LDPE, four containing HDPE, and the remaining with LLDPE. For each set of four agents, one of them was prepared by addition of a peroxide initiator and the other three were obtained by irradiation at 12, 24, and 48 h of UV exposition periods in our laboratory (details were reported elsewhere<sup>62</sup>). Melt index, grafting level, and gel content were evaluated for each compatibilizer prepared (including the commercial one).

### Blends preparation

The physical mixture of resin pellets of acquired materials, including a 5% w/w of the corresponding compatibilizing agent, was fed to the extruder for an appropriate molten mixing step. The mixture of polyethylenes and PET consisted of HDPE/LDPE/LLDPE/PET with the combination of 50/17/17/16 (% weight) respectively, prepared according with the levels reported at

present, and pretending to imitate the urban plastic waste for Mexico.<sup>4</sup>

The blends were prepared in a twin screw extruder W and F ZSK-30, with 250°C of temperature in five heating zones, at 400 rpm of screw speed. Finally, the pellets from extrusion were used to obtain specimens for mechanical properties by injection molding in a Battenfeld Machine with 75 tons capacity.

### Materials characterization

#### Melt index

MFI from compatibilization agents were evaluated in a Melt Index Kayeness instrument, model 7053, according with ASTM D 1238 (Temperature of 190°C and 21,600 g of load). This evaluation was carried out at such conditions, to visualize the agents flow differences.

#### Grafting and gel content

The unreacted MAH was removed by extracting 1 g of compatibilizing agent under 250 mL of hot xylene during 8 h, according to the described apparatus in ASTM D 2765. The xylene insoluble portion corresponds to the gel percentage, while the soluble part was precipitated and washed with acetone and finally dried at room temperature. The grafting percentage was determined by the acid number, according to a literature reported procedure.<sup>62</sup>

#### Thermal analysis

The thermal behavior of blends was obtained by using a Differential Scanning Calorimeter (TA instruments DSC-2920). Heating and cooling scans were carried out on 10 mg of material under a nitrogen flow, in a temperature range of 20–290°C, at a standard heating rate of 5°C/min. The materials were first melted at 290°C in the first run, to erase thermal history, then cooled down until 20°C and then reheated to 290°C in the second run. The bulk crystallinity was obtained, using the fusion enthalpies of HDPE and PET for calculations. Such values were determined from the second heating scan. The crystallinity percentage of the polymer in the blends was obtained using the following equation:<sup>62</sup>

$$(1 - \lambda)(\%) = \Delta H_F(100/\Delta H_{F_{eq}})$$

where  $\Delta H_F$  = enthalpy of fusion in the second scan,  $\Delta H_{F_{eq}}$  = theoretical enthalpy of fusion, reported for 100% crystalline polymers (288.8 J/g for polyethylene<sup>63,64</sup> and 136 J/g for PET<sup>65</sup>).

#### Mechanical properties

The specimens were tested following ASTM standard methods. After Injection molding, they were condi-

**TABLE I**  
Compatibilization Agents

Compatibilizers	MAH grafted (%)	MFI	Gel (%)
HDPE-MAH-PXD	1.01	34.5	0.40
HDPE-MAH-UV1	0.38	22.4	0.65
HDPE-MAH-UV2	0.70	21.5	0.84
HDPE-MAH-UV3	0.87	19.0	1.80
LDPE-MAH-PXD	1.88	15.3	0.48
LDPE-MAH-UV1	0.47	8.7	0.80
LDPE-MAH-UV2	0.85	5.5	1.27
LDPE-MAH-UV3	1.18	3.6	5.90
LLDPE-MAH-PXD	0.56	38.1	0.28
LLDPE-MAH-UV1	0.19	34.2	0.46
LLDPE-MAH-UV2	0.29	32.2	0.50
LLDPE-MAH-UV3	0.38	30.4	0.66
Bynel (BYN)	0.3	22.7	0.15

PXD, Peroxide treated; UV, Irradiated with UV light.

tioned for 40 h, according to the ASTM D618, before the mechanical evaluation. Tensile properties were evaluated using a mechanical testing machine UNITED model STM-10 and using specimens Type IV, with 3.2 mm of thickness and testing speed of 51 mm/s, according to ASTM D638. Izod impact strength was determined by using notched specimens, with 3.2 mm of thickness and a pendulum apparatus, CSI Model CS-137, with impact capacity of 2 J; ASTM D256 was followed. The reported values, for all properties, were the average of five evaluations.

### SEM microscopy

The blends morphology was examined by using a TOP-CON SM-510 scanning electron microscope (SEM). The samples were fractured under liquid nitrogen and then vapor coated with Au/Pd for microscopy, to finally being observed at 5000 magnifications and microphotographs were taken.

## RESULTS AND DISCUSSION

### Compatibilizing agents

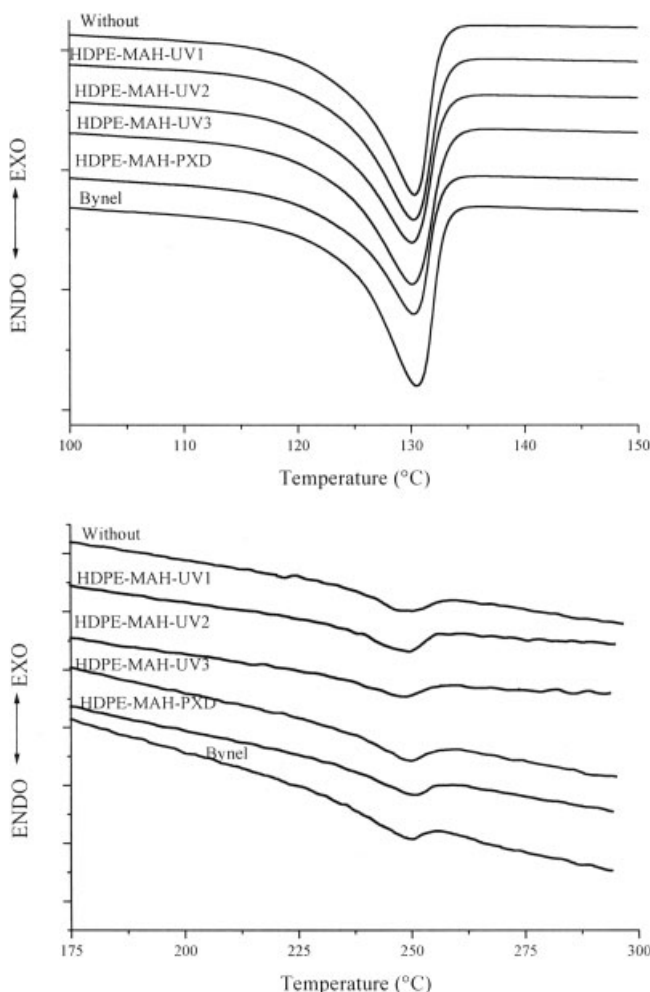
The agents used as compatibilizers were characterized before their use; the results are shown in Table I. It can be seen that the level of MAH grafting is always higher for peroxide treated materials, with respect to UV treated ones; they also have the lowest gel values and corroborated by the highest fluid index. However, the UV treated materials, reach convenient grafting levels, considering that the commercial compatibilizer (Bynel) have one of the lower values. The longer the UV treatment of the polyethylenes, the higher grafting level obtained. The grafting level obtained for both methods always follow the sequence: LDPE > HDPE > LLDPE, as mentioned in a previous paper.<sup>62</sup>

### Thermal analysis of blends

Figure 1 show the thermograms obtained from the blends indicating two DSC endotherms, corresponding to fusion, the first peak near 130°C, from polyethylenes, and a second one near 250°C, from PET. The endotherm corresponding to polyethylenes does not show any other peak related to LDPE (111.1°C), which has a lower fusion temperature than LLDPE (124.7°C) and HDPE (134.5°C). The previous has been reported before.<sup>63,64</sup>

Some reports mention that LDPE does not cocrystallize with other polyethylenes when is evaluated in binary blends,<sup>66,67</sup> although the effect is more notorious for LDPE/LLDPE than LDPE/HDPE blends,<sup>67,68</sup> even with 25% of LDPE content (very similar content to the ones used in this work). On the other hand, for the LLDPE and HDPE cocrystallization was observed at all proportions.<sup>66,67</sup>

The ternary blend of the polyethylenes, at proportions used in this work, behaves accordingly with the previous reports of no cocrystallization of LDPE; which



**Figure 1** Fusion temperature of HDPE/LDPE/LLDPE/PET (50/17/17/16) blends with different compatibilization agents.

**TABLE II**  
**Temperatures and Enthalpies of Fusion, and Crystallinity Degree of Quaternary Blends with and without Compatibilizers**

Compatibilizers	HDPE/LDPE/LLDPE			PET		
	$T_m$ (°C)	$\Delta H_m$ (J/g)	(1 - $\lambda$ ) (%)	$T_m$ (°C)	$\Delta H_m$ (J/g)	(1 - $\lambda$ ) (%)
Without	130.3	132.2	54.5	249.2	5.3	25.6
HDPE-MAH-PXD	130.2	131.6	53.7	250.4	5.6	27.1
HDPE-MAH-UV1	130.3	135.5	55.3	248.3	6.8	32.9
HDPE-MAH-UV2	130.1	134.5	54.9	247.9	6.4	30.9
HDPE-MAH-UV3	130.3	133.9	54.7	247.7	6.8	32.9
LDPE-MAH-PXD	130.5	121.2	49.5	250.0	5.4	26.1
LDPE-MAH-UV1	130.9	126.9	51.8	247.3	5.5	26.6
LDPE-MAH-UV2	130.6	123.1	50.2	248.1	5.2	25.2
LDPE-MAH-UV3	130.5	124.4	50.8	248.4	5.7	27.6
LLDPE-MAH-PXD	130.8	124.2	50.7	250.8	5.3	25.6
LLDPE-MAH-UV1	130.5	128.1	52.3	246.6	6.1	29.5
LLDPE-MAH-UV2	130.3	126.9	51.8	247.9	5.8	28.0
LLDPE-MAH-UV3	130.8	125.4	51.2	247.4	5.8	28.0
Bynel	130.4	126.1	51.5	249.5	5.2	25.2

decreases with the presence of HDPE and LLDPE. In this case, the HDPE is acting as a compatibilizer between the two low density polyethylenes.<sup>67,69</sup> The presence of PET does not affect the polyethylenes behavior, since the polyester is in a solid state when PE's solidification initiates; therefore the PET, in this case can be considered as filler.

Details of the thermal analysis results are shown in Table II, where along with thermograms of Figure 1, indicate that fusion temperatures of polyethylenes does not show significant changes when blended, suggesting that size and morphology of crystals are not affected by the presence of polyester and the compatibilizer agent. For the PET endotherm, the temperatures presented a slight change toward low temperatures, with the presence of UV preirradiation modified polyethylenes, which contain more crosslinking; however, this was not observed for peroxide modified polyethylenes.

The presence of higher crosslinking in the polyethylene could produce restrictions in the PET chains movement, as well as less perfect crystals, especially when such crosslinking has anhydride groups, which can interact with the carboxyl and/or hydroxyl end groups from the polyester, reducing molecular motion and producing the mentioned less perfect crystals in the PET. The commercial LLDPE-g-MAH (Bynel), have a similar trend in thermal properties than peroxide modified polyethylenes (low crosslinking).

The most significant effect on thermal properties, for the compatibilized blends, was observed in the crystallinity of polyethylenes, where these presented a reduction in their values; especially for those materials containing modified low density polyethylenes compatibilizers. This is possibly due to the intrinsic low crystalline structure they have, which in turn affects the values in the blends, as clearly seen in enthalpy values of poly-

ethylenes. Moreover, the changes are also related with the higher maleic anhydride content in the modified polyethylenes, as a consequence of two effects: the grafting by itself<sup>62</sup> and the reduced molecular motion obtained by the higher interactions with the polyester. For the blends containing the HDPE-g-MAH agent, the crystallinity is higher due to the intrinsic polyethylene crystalline phase.

### Mechanical properties

The mechanical behavior of compatibilized blends is shown in Table III, indicating the numerical values with their corresponding deviation; the values correspond to the average of five evaluations. The tensile strength behavior is specifically showed in the first column, where the simple presence of modified polyethylenes in the blends, improves the tensile strength at break. This is a consequence of the increase in interfacial adhesion between phases and is suggesting that interactions from MAH modified polyethylene and OH groups from PET are enough to obtain the positive changes in this tensile property; enhanced by some hydrolyzed MAH species that would be able to create interactions with the ester groups of PET through H bridging.

The modified polyethylenes by the peroxide process, produced less increment in the tensile strength (only 16%) in the blend than the one obtained by the blend added with the UV preirradiated stabilizer (increment of 35%), despite the major content of anhydride of the former. In this case, the crosslinking of the modified polyethylenes by the UV preirradiation process, had a main role in the tensile strength development, since the presence of crosslinking is known to affect positively this property, as well as the tensile modulus. On the other hand, there is a possibility that crosslinked

**TABLE III**  
**Mechanical Properties of Quaternary Blends with and without Compatibilizers**

Compatibilizers	Tensile strength (Mpa)	Tensile modulus (Mpa)	Elongation (%)	Impact strength (J/m)
Without	14.76 ± 0.25	744 ± 0.15	75 ± 3.1	67 ± 1.3
HDPE-MAH-PXD	17.18 ± 0.31	782 ± 0.13	110 ± 5.5	115 ± 1.7
HDPE-MAH-UV1	20.93 ± 0.38	796 ± 0.16	85 ± 5.2	106 ± 1.4
HDPE-MAH-2UV	20.47 ± 0.26	782 ± 0.11	108 ± 2.8	107 ± 0.9
HDPE-MAH-3UV	20.78 ± 0.30	828 ± 0.14	176 ± 4.3	109 ± 0.5
LDPE-MAH-PXD	16.66 ± 0.23	741 ± 0.11	180 ± 4.5	127 ± 1.6
LDPE-MAH-UV1	19.38 ± 0.39	739 ± 0.09	95 ± 3.3	136 ± 1.3
LDPE-MAH-UV2	19.98 ± 0.41	758 ± 0.12	125 ± 3.9	154 ± 1.9
LDPE-MAH-UV3	20.28 ± 0.32	769 ± 0.15	210 ± 3.5	176 ± 1.1
LLDPE-MAH-PXD	17.55 ± 0.29	749 ± 0.07	230 ± 4.6	135 ± 0.8
LLDPE-MAH-UV1	19.23 ± 0.37	664 ± 0.16	111 ± 5.1	130 ± 1.7
LLDPE-MAH-UV2	19.45 ± 0.33	734 ± 0.10	131 ± 6.8	136 ± 0.6
LLDPE-MAH-UV3	19.88 ± 0.45	764 ± 0.13	195 ± 3.7	152 ± 1.9
Bynel	15.88 ± 0.39	744 ± 0.11	190 ± 3.8	96 ± 1.0

structures with grafting anhydride behave as a compatibilizing phase between the polyethylene and the polyester, improving further the interfacial adhesion.

The fact that concentration of maleic anhydride in the compatibilizer agent does not affect significantly the mechanical property is probably due to the saturation of most OH groups from PET, so the major content of anhydride does not increase the interactions between polymers, and as a consequence, the tensile strength remains without changes.

The type of modified polyethylene has also a little effect in this property, as can be observed when comparing the materials containing modified HDPE and LDPE. The small difference between them has to be with the intrinsic higher tensile strength of HDPE.

The tensile modulus of blends also has a little increase due to major crosslinking presence in the compatibilizer agent (Column 2). The commercial modified polyethylene has a similar tensile strength and modulus behavior, to the ones obtained by the peroxide process.

Elongation at break and impact strength properties (Columns 3 and 4) were significantly improved and both are related, partially to each other, because when a blend presents more elongation and ductibility or in other words better toughness, the impact strength also increases, even though the improvement in adhesion, which has also some effect in such properties. Furthermore the factors which significantly have control over the elongation at break behavior of blends, for peroxide modified polyethylene, are the anhydride content, crosslinking and type of modified polyethylene. For those compatibilized blends with UV preirradiated modified polyethylenes, the elongation behavior is also due to anhydride and gel content; however, in this case it is not observed any important effect from the type of modified polyethylene in the blend.

The impact strength behavior of blends is also affected by the anhydride and crosslinking content, but

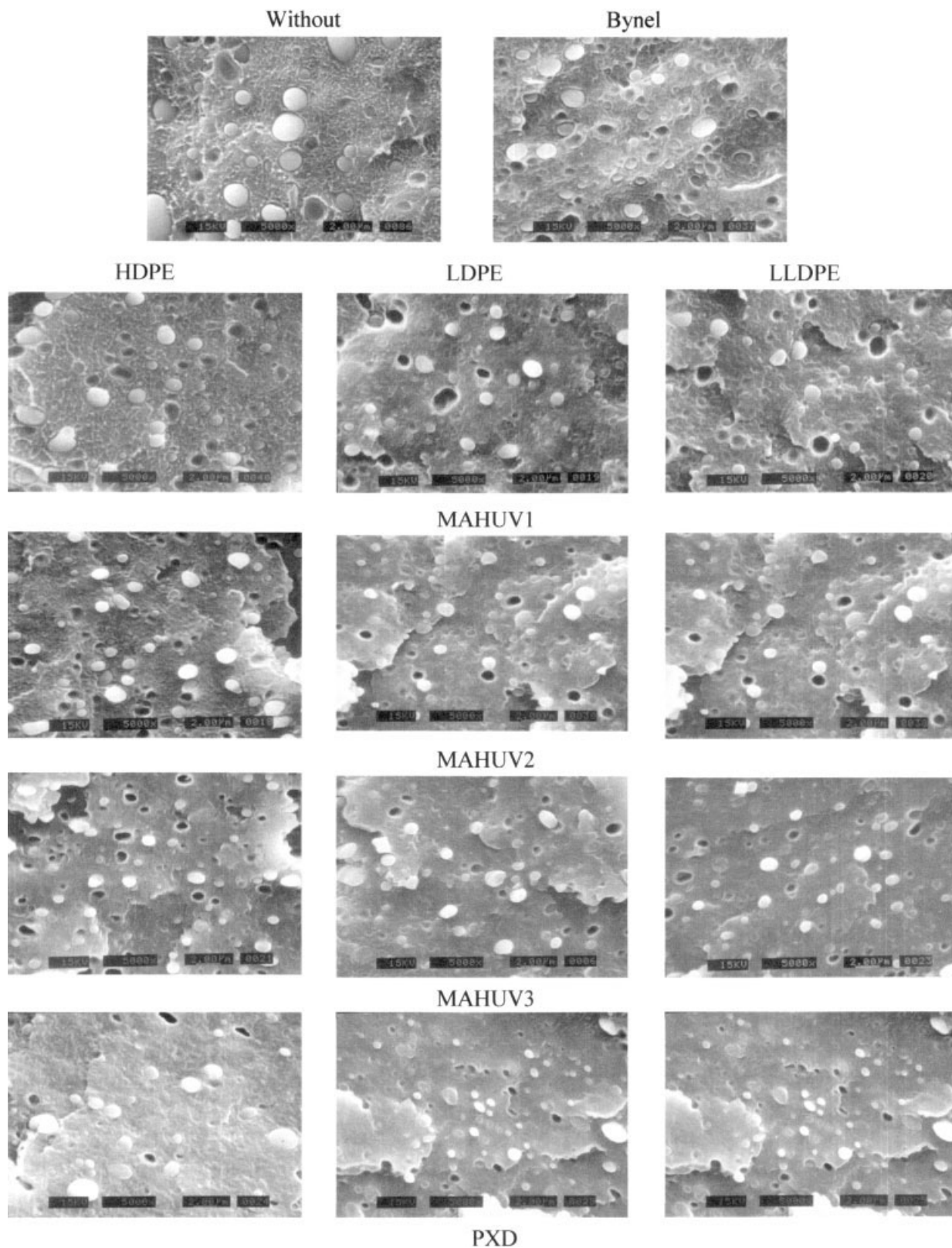
the effect of modified polyethylene type (compatibilizer agent) is mainly notorious for those obtained by the UV preirradiation process. The modified HDPE by both, peroxide and UV preirradiation processes, improves to some extent the impact strength of blends, but there are no significant differences among them. This behavior is probably due to the lower toughness the HDPE intrinsically has, according to its structural nature, comparing with the low density polyethylenes. Moreover, during the determination of deformability at high speed impact strength, is more difficult to observe changes produced by the type of compatibilizer agent, when following the behavior of blends, in opposition to what can be observed during evaluation of deformability at a low speed technique, like elongation at break.

It is also clear to notice that MAH grafted LDPE and LLDPE have higher impact strength values than the MAH treated HDPE; the reason seems to be again the structural intrinsic difference between polyethylenes, since HDPE has a well known reduced toughness comparing with the lower density materials, which in turn affects their crystallinity even in the blends.

### SEM microscopy

The microphotographies obtained by SEM of compatibilized blends are shown in Figure 2. The micrographs of fractured surfaces of noncompatibilized samples and some compatibilized ones, like those containing modified polyethylenes with the first level of UV irradiation (MAH-UV1) and the Bynel, show segregation between the PET inclusions into the continuous phase of polyethylenes. For these blends, it can also be observed the presence of voids around the PET particles, suggesting the presence of weak interactions as a consequence of a lack of adhesion between the phases of polyester and polyethylenes.

Moreover, it is noticeable the particle size differences, where for noncompatibilized blend are bigger than



**Figure 2** SEM microphotographs of HDPE/LDPE/LLDPE/PET (50/17/17/16) blends with different compatibilization agents.

those in compatibilized ones specifically for the ones containing modified low density polyethylenes (MAH-UV2, MAH-UV3, and PXD); therefore the addition of

compatibilizing agents helps to decrease the surface tension and produce smaller particles, as well as improving the adhesion, since for these compatibilized blends,

the voids around the PET particles are not observed, producing a more homogeneous morphology. Such behavior, regarding morphology, has a direct impact in the improvement of the mechanical properties, specifically in the development of the elongation at break. The blend containing the commercial modified polyethylene (Bynel) has a similar behavior to those blends that contain compatibilized agents produced with UV light.

## CONCLUSIONS

The use of any kind of polyethylene, modified with maleic anhydride groups is useful for the compatibilization of Polyethylenes with PET blends. The improvement in mechanical behavior of the compatibilized blends is due to the increment in interfacial adhesion and to some extent, to the reduction of interfacial tension. The combination of natural toughness and functional groups content in the compatibilizer agent is very important, because the agent effectiveness behavior depends on both parameters.

HDPE had more MAH grafted than LLDPE, but the latter had better impact strength for its toughness. The MAH grafted in polyethylenes had a major impact in interfacial adhesion, due to interactions with terminal OH groups from PET. From all mechanical properties, the elongation at break gave better sensibility; this technique allowed to observe the effect of anhydride content, the type of modified polyethylene and the level of their crosslinking; which in turn are the main factors controlling the compatibilization of the blends Polyethylenes/PET. The latter is supported by the observation in electron microscopy.

Finally, the results show a similar behavior between both modified polyethylenes, by UV irradiation and peroxide processes. By the other hand, the behavior of four components blends (three polyethylenes and PET) was also similar to reported blends of two components (PE/PET).

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