

# Synthesis and Application of MDPE-g-GMA as Reactive Compatibilizer in Blends of MDPE/PET and MDPE/PA6

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Received 18 May 2011; accepted 11 July 2011

DOI 10.1002/app.35235

Published online 25 October 2011 in Wiley Online Library (wileyonlinelibrary.com).

**ABSTRACT:** In the present study, glycidyl methacrylate (GMA) grafted medium density polyethylene (MDPE-g-GMA) was synthesized in the molten state and applied as a reactive compatibilizer in MDPE/polyamide6 (PA6) and in MDPE/poly(ethylene terephthalate) (PET) blends. Graft copolymerization of GMA onto MDPE was performed in presence and absence of styrene, with different concentrations of dicumyl peroxide (DCP) as a radical initiator. In the presence of styrene, the MDPE-g-GMA with 6% GMA was obtained by addition of only 0.1 phr of DCP. Furthermore, the maximum grafting was reached when 0.6 and 0.7 phr concentration of DCP for styrene containing and styrene free samples were used, respectively. Torque-time measurement showed faster grafting reaction rate in the presence of styrene. Four MDPE-g-GMA samples were selected as com-

patibilizers in the blends. Furthermore, the effects of melt flow index and grafting content of compatibilizers on mechanical properties and morphology of the blends were investigated through tensile tests and SEM analysis. Tensile test results indicated that the presence of compatibilizers in the blends led to 250 and 133% increase in elongation at break for PA6 and PET blends, respectively. Moreover, the best tensile results for blends were obtained using MDPE-g-GMA with high flow ability. The average particle size of the dispersed phase decreased by 350% for PA6 and 300% for PET blends compared with nonreactive blends. © 2011 Wiley Periodicals, Inc. *J Appl Polym Sci* 124: 2048–2054, 2012

**Key words:** glycidyl methacrylate; compatibilization; blending; functionalization of polymers; graft copolymers

## INTRODUCTION

Blending of polyolefins with polar polymers offers an interesting route to achieve new materials with promising properties. The blends of polyolefins with polar polymers, e.g., polyamide6 (PA6) and poly(ethylene terephthalate) (PET), have been extensively studied in the last years due to their practical advantages. Polyolefins are relatively inexpensive, highly flexible, easy for processing, and insensitive to moisture. In addition, PA6 and PET are rigid, thermally more stable, and possess excellent barrier properties to oxygen and organic solvents. Thus, dispersion of PA6 or PET in a polyolefin may enhance the oxygen resistance and hydrocarbon permeation of polyolefin, or act as a reinforcing agent.

However, polyolefins and polar polymers are thermodynamically immiscible. Therefore, the addition of a graft or block copolymer as a compatibilizing agent is necessary to reduce interfacial tension and achieve interfacial adhesion. The compatibilizer copolymer can be suitably generated *in situ* in the

presence of polyolefins bearing functional groups. Grafting of Acid, ester, or anhydride groups onto polyolefins is capable of giving rise to reactions with the polyamide end-groups during melt mixing. In the last two decades, a sizeable amount of effort has been made for chemical modification of polyolefins to give additional properties by means of grafting functional monomers onto the backbone of these polymers.<sup>1–3</sup> Reactive monomers, such as maleic anhydride (MAH), maleic acid, dibutyl maleate, acrylic acid, and its esters, have been usually used in the chemical modifications, and there is a large patent literature covering this area.<sup>4–6</sup> Extensive works have been done on the compatibilization of polyamide/polyolefin blends through maleic anhydride functionalized polyolefins, such as polypropylene-g-MAH,<sup>7</sup> ethylene/propylene-g-MAH,<sup>8</sup> and polyethylene-g-MAH.<sup>9</sup> The anhydride units of MAH can react readily with the amine end groups of the polyamide to form block or graft copolymer acting as *in situ* compatibilizer. In recent years, GMA has received lots of attention for functionalization of polyolefins. GMA is a bifunctional monomer containing an unsaturated group for free radical grafting onto polyolefins and an epoxy group capable of reacting with numerous functional groups such as hydroxyl, carboxyl and amine. Several authors have examined the radical grafting of GMA onto polyolefins in recent years.<sup>10–16</sup> Cartier and Hu<sup>14,15</sup> investigated the

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Contract grant sponsors: Isfahan University of Technology (IUT), National Petrochemical Company, Research and Technology (NPC-RT).

styrene-assisted free radical grafting of GMA onto polyethylene and polypropylene in the melt and reported that use of styrene as comonomer greatly promotes GMAs grafting yield and grafting rate. It must be borne in mind that the first step in the grafting reaction is the formation of primary radicals as a result of the thermal decomposition of a free radical initiator (usually peroxide). The abstraction of a hydrogen atom from the polymer substrate by the primary radical leads to the formation of a macroradical. The macroradical may then follow one of the two competing routes: it either reacts with the monomer, which is desirable, or undergoes structural change, which is often undesirable. The monomer may also homopolymerize in the presence of primary radicals.

The melt radical grafting of virgin as well as recycled high density polyethylene (HDPE) with GMA has been carried out for reactive mixing with recycled PET.<sup>17</sup> GMA functionalized polyolefins have been used as *in situ* compatibilizers in blends of polyolefins with polyamides<sup>18,19</sup> or polyesters.<sup>20–23</sup>

GMA is used as a suitable reactive monomer for compatibilization of polyamide/polyethylene, and PET/polyethylene blends as the epoxy group of GMA can react with both amino and carboxyl-end groups present in polyamide and PET.<sup>18</sup> In some cases, the (equilibrium) reactivity of epoxy functionalized polyolefins has been reported to be higher than that found for maleic anhydride and acrylic acid-grafted polyolefins.<sup>24</sup> Moreover, there is no water formation during ring opening reaction of epoxy with COOH and  $\text{—NH}_2$ , which prevents the hydrolysis of  $\text{—NH—CO—}$  along the backbone of the polyamide. Also, reactive blending processes have been investigated for model systems of PET and polyolefins by several authors.<sup>17,24–27</sup>

In this article, the melt free radical grafting of GMA onto MDPE is investigated with the aim of performing reactive blending of PA6 and PET with MDPE using MDPE-g-GMA as a compatibilizer. In particular, the influence of radical initiator concentration and addition of styrene as comonomer on the grafting degree are examined. Then, the effect of synthesized compatibilizer on the morphology and mechanical properties of blends are analyzed before and after the reactive blending.

## EXPERIMENTAL PROCEDURE AND MATERIALS

### Materials and equipment

Granule and free additive MDPE powder from Tabriz Petrochemical Company (Iran) with density of 0.938 g/mL and MFI (190°C, 2.16kg) = 4 g/10 min was used. GMA (purity, 96%), styrene (purity,

99.5%), and dicumyl peroxide (DCP) were purchased from Merck and used as received. Trichloroacetic acid (TCA) (Merck), potassium hydroxide (Merck), acetone (Mojalai Company, Iran), xylene (Mojalai Company, Iran), and methanol (Mojalai Company, Iran) were used for chemical titration. Bottle grade PET (BG-821; end group content = 32 mmol/kg) was supplied by Tondgoian Petrochemical Company (Iran). B40L grade of PA6 (produced by BASF) with end group content of 52 mmol/kg was used as another polar polymer. The free radical grafting experiments were carried out in a Brabender internal mixer.

### Melt functionalization

DCP as radical initiator was dissolved in the GMA or GMA/styrene mixture. The MDPE powder was premixed with these solutions and then charged into the Brabender chamber at 170°C and mixed for 15 min at a rotor speed of 50 rpm. To investigate the effect of reagent concentration on the grafting, the amount of radical initiator was varied.

### Sample purification and measurement of grafting content

To determine the GMA grafting degree, the reaction products were purified to remove unreacted GMA and other secondary products (homopolymerized GMA and/or styrene copolymerized with GMA). Crude product (2 g) was dissolved in hot xylene (85 mL) by stirring for one hour and precipitated with acetone (200 mL); before precipitation the hot solutions of polymer were filtered through a grid to determine the possible formation of gel. The precipitate was thus purified in a soxhelt extractor using acetone as solvent for 24 h. Purified species were dried in vacuum at 80°C overnight.

A nonaqueous back-titration method was used to determine the amount of grafted GMA.<sup>13</sup> A sample of the purified product (about 0.5 g) was dissolved in hot xylene (80 mL), followed by addition of 2 mL of TCA (0.3M xylene solution). The mixture was kept at 105 to 110°C for 90 min to achieve the complete reaction of TCA with grafted GMA. The solution was cooled and became foggy. Unsolvable grafted polyethylene was removed and washed using a filter paper. The filtrate (TCA residue) was titrated with 0.1M KOH solution in methanol by using ethanolic phenolphthalein as indicator. Grafting degree was calculated using eqs. (1) and (2).

$$(V_{\text{KOH-s}} - V_{\text{KOH-b}}) * N_{\text{KOH}} = V_{\text{TCA}} * N_{\text{TCA}} \quad (1)$$

$$\text{GMA}\% = \frac{(V_{\text{TCA0}} - V_{\text{TCA}}) * N_{\text{TCA}} * 142.16 \frac{\text{g}}{\text{mol}}}{W_{\text{sample}}} \quad (2)$$

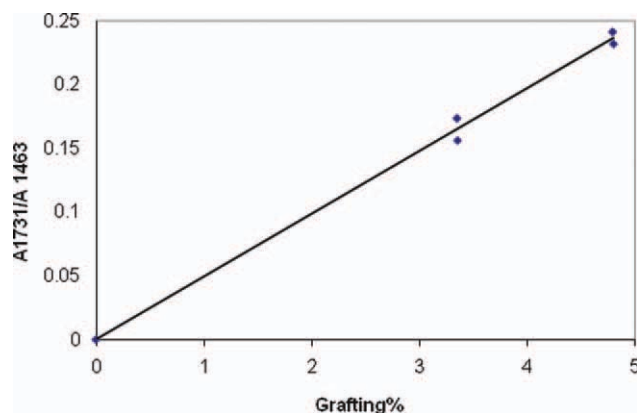


Figure 1 Calibration curve of MDPE-g-GMA.

Where,  $V_{\text{KOH} - s}$  is volume of potassium hydroxide for sample titration;  $V_{\text{KOH} - b}$  is volume of potassium hydroxide used for titrating the filtrate of polyethylene solution;  $N_{\text{KOH}}$  is normality of potassium hydroxide;  $V_{\text{TCA}}$  is the TCA volume calculated by potassium hydroxide titration;  $N_{\text{TCA}}$  is normality of TCA solution;  $V_{\text{TCA}0}$  is initial volume of TCA solution (2 mL); and GMA% is grafting degree of GMA on the PE.

#### FT-IR calibration curve of MDPE-g-GMA

A calibration curve was developed to correlate the relative amount of grafted GMA as determined from FT-IR measurements with that obtained by the titration method. Measurements of grafting degree were performed with a JASCO 680 plus FT-IR spectrometer on compression molded films of purified functionalized products. The bands of carbonyl group of GMA ( $1731 \text{ cm}^{-1}$ ) and of the C–H stretching group of MDPE ( $1463 \text{ cm}^{-1}$ ) were used to determine the extent of grafting. The smooth polymer films were prepared by compression molding. The thickness of the films was  $60 \mu\text{m}$ . The effect of thickness variation on both peaks and the peak ratio approximately

TABLE I  
Compatibilizers Properties

| Compatibilizer code | MFI (5 kg, 190°C) | GMA grafting (%) |
|---------------------|-------------------|------------------|
| C1                  | 1.4               | 6.17             |
| C2                  | 3.7               | 6.20             |
| C3                  | 4.8               | 4.62             |
| C4                  | 4.4               | 6.11             |

remains constant. Figure 1 shows the calibration curve of MDPE-g-GMA.

#### Blend preparation

In this part, four MDPE-g-GMAs with different MFI and graft percentages were used as compatibilizers. Table I shows the MFI and grafting percentages of these compatibilizers.

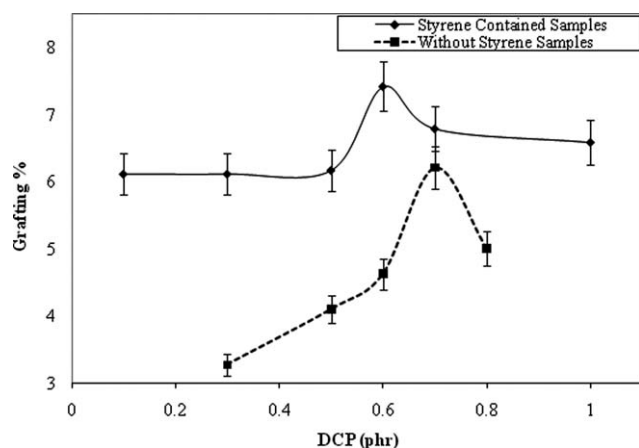
PA6, MDPE, and the compatibilizers were kept in a vacuum oven at  $105^\circ\text{C}$  overnight; also PET was dried at  $150^\circ\text{C}$  in a vacuum oven for 8 h. Blends of PA6/MDPE and PA6/MDPE-g-GMA/MDPE were prepared in a Brabender mixer at  $235^\circ\text{C}$  and 60 rpm for 4 min. The blends were rapidly removed from the mixing chamber, cooled in air, and stored at room temperature. Also, PET/MDPE and PET/MDPE-g-GMA/MDPE were prepared in the same mixer at  $260^\circ\text{C}$  and 60 rpm for 4 min. Blends compositions are presented in Table II. The Brabender internal mixer is capable of measuring mixing torque. Final mixing torque for functionalized and unfunctionalized blends is measured by the mixer software (Mixerprogram for windows, version 2.0.1) and shown in Table II.

#### Microscopy

The morphological analysis of fractured surfaces of the blends was carried out using a scanning electron microscope. The specimens were fractured in liquid nitrogen. The identification of dispersed polymer phase in the blends was carried out by etching

TABLE II  
Blends Compositions and the Final Mixing Torque

| Compatibilizer code | Compatibilizer (wt %) | PET (wt %) | PA6 (wt %) | MDPE (wt %) | Final mixing torque (after 4 min mixing, NM) |
|---------------------|-----------------------|------------|------------|-------------|--|
| –                   | 0                     | –          | 25         | 75          | 8.4  |
| C1                  | 10                    | –          | 25         | 65          | 12.8   |
| C2                  | 10                    | –          | 25         | 65          | 13.2   |
| C3                  | 10                    | –          | 25         | 65          | 12.5   |
| C4                  | 10                    | –          | 25         | 65          | 11.8   |
| –                   | 0                     | 25         | –          | 75          | 4.7  |
| C1                  | 10                    | 25         | –          | 65          | 7.4  |
| C2                  | 10                    | 25         | –          | 65          | 7.5  |
| C3                  | 10                    | 25         | –          | 65          | 7.5  |
| C4                  | 10                    | 25         | –          | 65          | 7.2  |



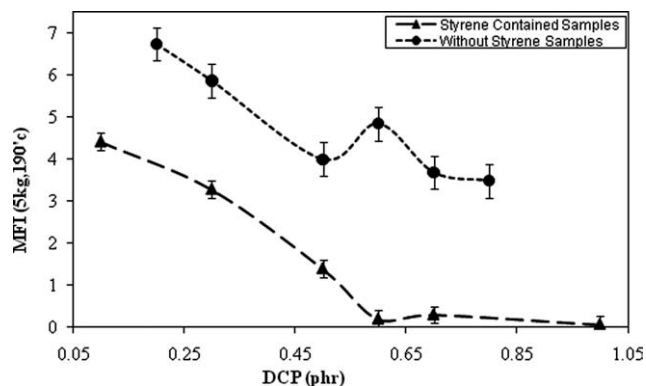
**Figure 2** Grafting content versus peroxide concentration; solid line in presence and dashed line in absence of styrene comonomer; GMA = 8 phr.

treatments with selective solvents, namely, formic acid for PA6. All fractured surfaces were coated with a thin gold film using a gold sputter. Dispersed PET particles were distinguishable in SEM images and etching was not necessary. The size of dispersed phase was measured using Solid Works program.

## RESULTS AND DISCUSSION

### GMA grafting onto MDPE

Figure 2 shows that grafting content of all styrene containing samples is higher than that of the styrene free samples. Radical initiator concentration effect on the melt grafting of GMA is shown in Figure 2. The amount of grafted GMA has increased by increasing the amount of the peroxide up to maximum (at a concentration of about 0.7 phr in styrene free samples and 0.6 phr in styrene containing samples) and then its content has decreased. The decrease is due to the homopolymerization reaction of GMA and crosslinking of polyethylene chains. Formation of



**Figure 3** Melt flow index of synthesized MDPE-g-GMA versus peroxide concentration in presence and absence of styrene comonomer; GMA = 8 phr.

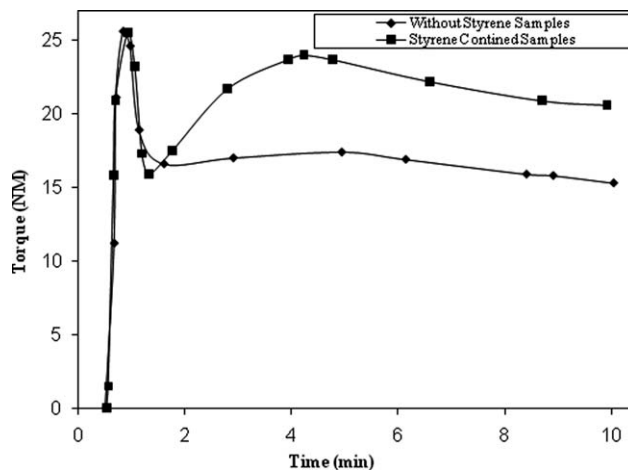
crosslinked MDPE has led to the decrease of GMA conversion. An analogous trend has been observed for the GMA radical grafting of LDPE, with maximum yield at about 0.6 wt % of *bis*[1-(*tert*-butylperoxy)-1-methylethyl]benzene (BTP).<sup>28</sup> Comparison of the two sets of experiments in Figure 2 shows that styrene free samples are more sensitive to DCP concentration than the other ones, and it seems that mechanism of reaction is different in the presence of styrene. Due to the low reactivity of unsaturation of GMA toward polyolefin macroradicals, styrene has been commonly used for its higher reactivity forming styryl-macroradicals, which in turn can easily react with GMA monomer.<sup>14</sup>

Figure 3 shows the MFI of two series of samples versus initiator concentration. It can be seen that the MFI decreases with increasing of peroxide concentration in presence and absence of styrene. This is because an increase in side reactions such as crosslinking causes a dramatic decrease in MFI. Moreover, owing to the low stability of the macroradicals, crosslinking reactions are expected for PE.<sup>14</sup>

Figure 4 shows the torque-time curves of grafting MDPE in presence and absence of styrene comonomer. In styrene containing samples, the torque-time curve includes two peaks, melting peak followed by reaction peak. Grafting reaction is more intensive and faster in styrene containing samples, and reaction peak is reached sooner compared with styrene free samples.

### Blends of PA6 or PET with MDPE and MDPE-g-GMA

Ternary blends at fixed composition ratios with different compatibilizers (C1, C2, C3, and C4) were prepared to investigate the effect of grafted polyethylene on the phase interaction phenomena. The morphology



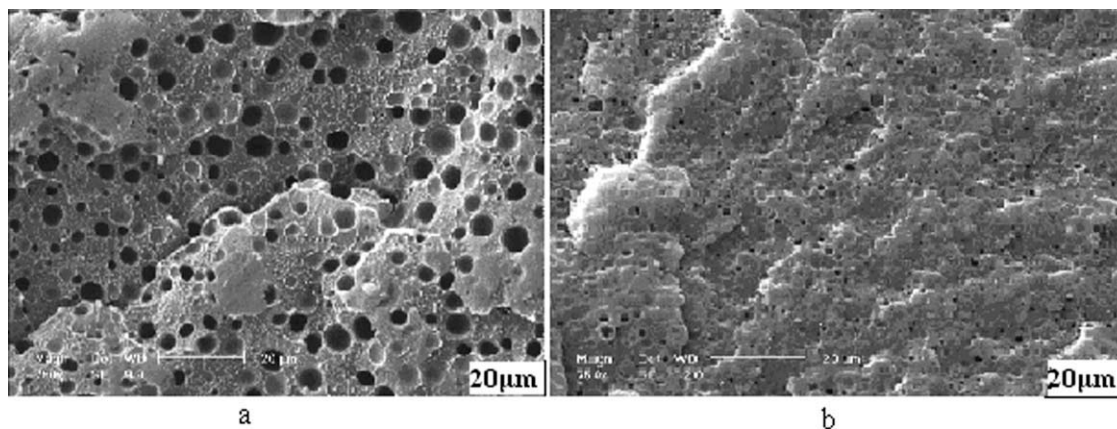
**Figure 4** Torque-time curve for grafting of GMA onto MDPE in presence and absence of styrene comonomer; DCP = 0.6 phr; GMA = 8 phr.

**TABLE III**  
Tensile Properties of PA6/MDPE-g-GMA/MDPE Blends

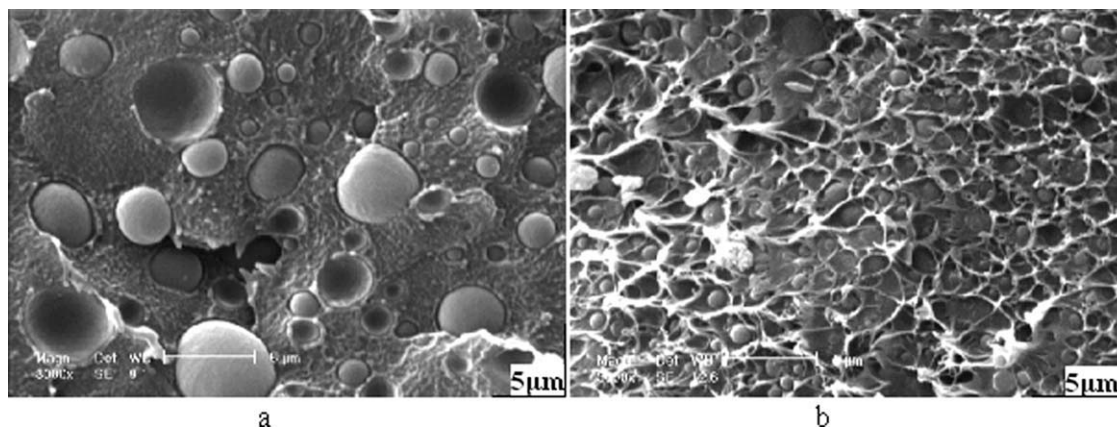
| Compatibilizer code | Compatibilizer (wt %) | PA6 (wt %) | MDPE (wt %) | Elongation at break (%) | Elongation at break improvement (%) | Stress at peak (MPa) | Stress at peak improvement (%) |
|---------------------|-----------------------|------------|-------------|-------------------------|-------------------------------------|----------------------|--------------------------------|
| –                   | 0                     | 25         | 75          | 12.8 ± 3.1              | –                                   | 20.2 ± 0.15          | –                              |
| C1                  | 10                    | 25         | 65          | 30.7 ± 8                | 140                                 | 20.8 ± 0.95          | 2.7                            |
| C2                  | 10                    | 25         | 65          | 30.8 ± 7                | 141                                 | 22.1 ± 0.75          | 9.5                            |
| C3                  | 10                    | 25         | 65          | 44.7 ± 10               | 249                                 | 21.9 ± 0.3           | 8.5                            |
| C4                  | 10                    | 25         | 65          | 42.8 ± 4                | 234                                 | 21 ± 0.9             | 4.1                            |

**TABLE IV**  
Tensile Properties of PET/MDPE-g-GMA/MDPE Blends

| Compatibilizer code | Compatibilizer (wt %) | PET (wt %) | PET (wt %) | Elongation at break (%) | Elongation at break improvement (%) | Stress at peak (MPa) | Stress at peak improvement (%) |
|---------------------|-----------------------|------------|------------|-------------------------|-------------------------------------|----------------------|--------------------------------|
| –                   | 0                     | 25         | 75         | 16.2 ± 1.4              | –                                   | 17.2 ± 0.4           | –                              |
| C1                  | 10                    | 25         | 65         | 17.3 ± 2                | 6.5                                 | 21 ± 0.15            | 23                             |
| C2                  | 10                    | 25         | 65         | 21.8 ± 7                | 33.5                                | 19.6 ± 1             | 14.5                           |
| C3                  | 10                    | 25         | 65         | 38 ± 4                  | 133                                 | 19.5 ± 0.7           | 13.5                           |
| C4                  | 10                    | 25         | 65         | 35 ± 7                  | 115                                 | 20.6 ± 0.4           | 20                             |



**Figure 5** SEM micrographs of fracture surfaces of (a) PA6/MDPE (25/75) and (b) PA6/C4/MDPE (25/10/65).



**Figure 6** SEM micrographs of fracture surfaces of (a) PET/MDPE (25/75) and (b) PET/C4/MDPE (25/10/65).

TABLE V  
Average Particle Size of Dispersed Phase in Blends

| Type of blends     | Blend composition (%) |    |            | Average particle size ( $\mu\text{m}$ ) |      |
|--------------------|-----------------------|----|------------|---|------|
|                    | MDPE                  | C4 | PA6 or PET | PET                                     | PA6  |
| Nonreactive blends | 75                    | –  | 25         | 2.22                                    | 2.99 |
| Reactive blends    | 65                    | 10 | 25         | 0.73                                    | 0.85 |

and mechanical properties of compatibilized (reactive) blends were compared with uncompatibilized (nonreactive) blends.

Table III shows the blend composition and tensile test results for samples with PA6 as dispersed phase, and Table IV shows PET containing blends. The results of PA6 blends show that the elongation at break has increased in range of 140 to 249% after reactive blending. Also, this property has improved in range of 6.5 to 133% in PET containing blends. Tensile strength as another mechanical property was improved with all coded compatibilizers. Reaction of grafted GMA epoxy groups with amine group of PA6 and carboxyl end group of PET is the main reason for this compatibilization. On the other hand, in all the blends, C3 and C4 were much more efficient than C1 and C2. It seems that lower MFI and higher gel content of C1 and C2 is the reason for this behavior. A remarkable increase of the torque was observed after melt processing for PET/MDPE-g-GMA/MDPE and PA6/MDPE-g-GMA/MDPE (Table II) with respect to the unfunctionalized blends. A similar effect has been reported for blends of polyamides or polyesters with various functionalized polyolefins.<sup>19,21,23,28</sup> Since the mixing torque is an indication of melt viscosity, the increase of torque may be accounted for by the formation of high molecular weight species from the chemical reaction between epoxy groups of MDPE-g-GMA with carboxyl and amine end-groups of PA6 and PET.

Comparison of PA6 and PET blends clearly shows that compatibilizers are more effective for PA6 blends than PET blends. This higher efficiency is due to the high end group content of PA6 rather than PET (52 mmol/kg for PA6 and 32 mmol/kg for PET).

The morphological characteristics of blends of PA6 or PET with MDPE using C4 as functional compatibilizers have also been studied in this work. The surface of fractured blends was examined by SEM, and the results are shown in Figures 5 and 6.

The nonreactive blends (part a of Figs. 5 and 6) display separated phase morphology with no adhesion at the interface and poor dispersion of minor phase. However, in the reactive blends (part b of Figs. 5 and 6) the particle size of dispersed phase has decreased. Decrement of particle size is due to the reaction of GMA epoxy group with end groups of PA6 or PET, which has decreased surface tension

of the dispersed phase.<sup>29</sup> The average particle size of dispersed phase (PA6 and PET) was measured and reported in Table V.

## CONCLUSIONS

This study showed that styrene is an effective comonomer in melt grafting of GMA onto medium density polyethylene. In presence of styrene, high grafting content was achieved using low initiator contents, and the grafting reaction was also faster compared with styrene free samples. Fast grafting reaction may be an advantage for industrial production of the compatibilizers. Presence of all produced compatibilizers improved mechanical and morphological properties of final blends. Furthermore, content of carboxyl end group of PET and amine end group of PA6 were effective parameters in final blend properties. Higher end group contents mean more reactions between them and epoxy group of grafted GMA, finally leading to better compatibilization. The results of elongation at break showed that high MFI compatibilizers (C3 and C4) were more effective than low MFI ones (C1 and C2). The result may be accounted for by faster distribution and better mixing of high MFI species in the blends.

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