

# Comparative study of different maleic anhydride grafted compatibilizer precursors towards LDPE/PA6 blends: Morphology and mechanical properties

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## Abstract

The study deals with the effectiveness of maleic anhydride grafted styrene-*b*-ethylene-*co*-propylene copolymer (SEPMA) as compatibilizer precursor (CP) for blends of low density polyethylene (LDPE) with polyamide-6 (PA). The CP was produced by grafting MA onto SEP in the melt. The specific interactions between the CP and the blends components have been investigated through characterizations of the binary LDPE/CP and PA/CP blends. The compatibilizing efficiency of the MA-grafted SEP, as revealed by the thermal properties and the morphology of the compatibilized blends, has been shown to be excellent. The morphology, as well as the mechanical properties of the compatibilized with SEPMA 75/25 w/w and 25/75 w/w LDPE/PA6 blends have been compared with those of the blends compatibilized with maleic anhydride functionalized HDPE sample (1-HDPE-*g*-MA) and with a commercial maleic anhydride grafted styrene-*b*-(ethylene-*co*-1-butene)-*b*-styrene copolymer (SEBSMA1). The results show that the strong compatibilizing efficiency of SEPMA is comparable with that of SEBSMA1, while 1-HDPE-*g*-MA exhibits a slightly lower activity, particularly for the blends, in which PA is the matrix phase.

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**Keywords:** LDPE/PA6 blends; MA-functionalized compatibilizer precursors; Morphology and mechanical properties

## 1. Introduction

Recently [1–5] we have discussed the reactive compatibilization of blends of polyamide-6 (PA) with two grades of low density polyethylene (LDPE), carried out using different compatibilizer precursors (CPs). In particular, the CP effectiveness of three ethylene/acrylic acid copolymers (EAA) having different acrylic acid content and different molar mass [1], of an EAA zinc ionomer [1], had been studied and compared. The same has been done with several commercial and home-made ethylene-*g*-maleic anhydride copolymers (HDPE-*g*-MA and LDPE-*g*-MA) with different MA content [2], of an ethylene-glycidyl methacrylate

copolymer (EGMA) [3] and of some thermoplastic elastomers grafted with maleic anhydride (SEBS-*g*-MA) or with glycidyl methacrylate (SEBS-*g*-GMA) [4]. The interfacial tension measurements run by the breaking thread method have shown the interfacial tension of the compatibilized blends strongly decreased in comparison to that of the uncompatibilized blends [5]. All these CPs react during blending, with the functional groups of PA to produce CP-*g*-PA copolymers, though via different kinetics and at different yields. The results confirm that the anhydride functional groups possess considerably higher efficiency, for the reactive compatibilization of LDPE/PA blends, than that of the ethylene-acrylic acid and ethylene-glycidyl methacrylate copolymers.

Concerning the efficiency of the different MA-grafted precursors it has been demonstrated that [2], the efficiency of the PE-*g*-MA copolymers changes dramatically depending on the structure and the molar mass of the polyethylene substrate. The efficiency of the MA grafted elastomer

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(SEBS-*g*-MA) practically coincides with that of 1-HDPE-*g*-MA [4] for the compatibilization of PE/PA6 75/25 w/w blend. It is worth studying the efficiency of another type of MA-grafted polymer, namely a maleic anhydride grafted styrene-*b*-ethylene-*co*-propylene copolymer (SEP-*g*-MA) as a compatibilizer precursor (CP) for the same blends.

CPs functionalized with MA group have been employed mainly for compatibilization of polyamide/polypropylene blends [6–13]. Holsti-Miettinen and co-workers [6] investigated the SEBS-*g*-MA reactive compatibilization of PA/*i*-PP blends by mechanical, morphological, thermal and rheological analyses and showed that this CP is more effective than an ethylene-butyl acrylate copolymer grafted with 0.15 wt% MA and an ethylene-ethyl acrylate glycidyl methacrylate terpolymer. Kim et al. [7,8] and Wilkinson et al. [9] studied the *i*-PP/PA/SEBS-*g*-MA ternary blends with *i*-PP as the matrix phase. Gonza'lez-Montiel et al. [10–12] also published several papers on the preparation and the properties of PA/*i*-PP/SEBS-*g*-MA blends. Chen and Harrison [14] used six different CPs, including two grades of SEBS-*g*-MA, to compatibilize 80/20 blends of PE with an amorphous polyamide with the aim of producing PE films reinforced with polyamide fibers for balloon applications. Armat et al. [15] demonstrated that SEBS-*g*-MA is a good CP for the reactive compatibilization of 25/75 LDPE/PA blends.

The literature data on the compatibilization of polymer blends with styrene-*b*-ethylene-*co*-propylene copolymer (SEP) are scarce. SEP has been used as CP for the compatibilization of polypropylene/polystyrene blends mainly [16–18]. SEP-*g*-MA has not been studied yet as CP for the compatibilization of polymer blends.

In the present paper a styrene-*b*-(ethylene-*co*-propylene) copolymer (SEP) grafted with maleic anhydride (SEP-*g*-MA) has been used for the compatibilization of LDPE/PA6 blends. The CP has been produced by radical grafting MA onto a SEP copolymer in the melt. The morphology and the mechanical properties of the compatibilized 75/25 w/w and 25/75 w/w LDPE/PA6 blends have been compared with those of the blends compatibilized with 1-HDPE-*g*-MA and SEBS-*g*-MA. The aim has been to evaluate comparatively the compatibilizing efficiency of the different MA-functionalized CPs.

## 2. Experimental part

### 2.1. Materials

A commercial low density polyethylene (Riblene FF20, provided by Polimeri Europa) with a melt flow index (MFI) equal to 0.8 g/10 min, referred to herein as LD08, and a polyamide-6 (PA) (provided by Snia Tecnopolimeri) with a relative viscosity in sulfuric acid (95.7%) of 3.66 and contents of amine and carboxyl end groups of 34 and 35 mequiv kg<sup>-1</sup>, were used for the blends preparation. The

compatibilizer precursors (CPs) were a home made SEP-*g*-MA sample, referred to herein as SEPMA, obtained by melt free-radical functionalization of a commercial SEP (KRATON G 1701 provided by Shell Chemicals) with 28 wt% of styrene; a maleic anhydride functionalized HDPE sample of low molar mass with 1.0 wt% MA and MFI 5.0 g/10 min (Polybond 3009 provided by Uniroyal) referred to herein as 1-HDPE-*g*-MA [2] and a commercial maleic anhydride grafted styrene-*b*-(ethylene-*co*-1-butene)-*b*-styrene copolymer with 1.7 wt% MA (Kraton FG 1901X provided by Shell Chemicals) referred to herein as SEBSMA1 [4].

Before use, all the polymers were accurately dried under vacuum.

### 2.2. SEP functionalization

SEPMA was synthesized as described elsewhere [4]. The required amounts of SEP, MA and dicumyl peroxide (40 g, 6 and 0.2/h, respectively) were pre-mixed, charged into the pre-heated Brabender mixer at 160 °C for 150 s at a rotor speed of 30 rpm and for 300 s at 60 rpm. The functionalized SEP was purified by solution in THF (40 mL g<sup>-1</sup> of polymer) and precipitation into excess methanol. The solid was then dried under vacuum. The content of grafted MA (0.1 mmol g<sup>-1</sup> SEP) was determined by titration by known procedures [4,19–21]. About 1.0 g purified product was dissolved in 100 mL xylene, and the solution was refluxed for 40 min. Water (3 mL) was then added to hydrolyze the anhydride groups and, after 25 min stirring, the hot mixture was added with excess KOH solution in ethanol (0.025 N) and back titrated with HCl in ethanol (0.025 N), using phenolphthalein as an indicator. The same titration procedure was applied to commercial sample of SEBS-*g*-MA (SEBSMA1) [4], and for 1-HDPE-*g*-MA sample [2].

### 2.3. Blends preparation

Binary (75/25 LD08/SEP, LD08/SEPMA, PA/SEP and PA/SEPMA) and ternary (75/25/1-5 and 25/75/1-5 LD08/PA/SEP and LD08/PA/SEPMA) blends were prepared by charging the components into the 50 mL static mixer of a Brabender Plasticorder and blending them under nitrogen for about 6 min at 235 °C. The rotor speed was kept about 1 min at 30 rpm and was then increased to 60 rpm. For the ternary LD08/PA/SEPMA blends the concentration of SEPMA was varied within the 0–5/h range. The ternary LD08/PA/SEBSMA1 and LD08/PA/1-HDPE-*g*-MA 75/25/1-8 and 25/75/1-8 w/w/w were prepared at similar procedure.

### 2.4. Blends characterizations

Scanning electron microscopic (SEM) analysis was made on a JEOL JSM-5600 apparatus on polymer specimens fractured under liquid nitrogen and coated with gold. The

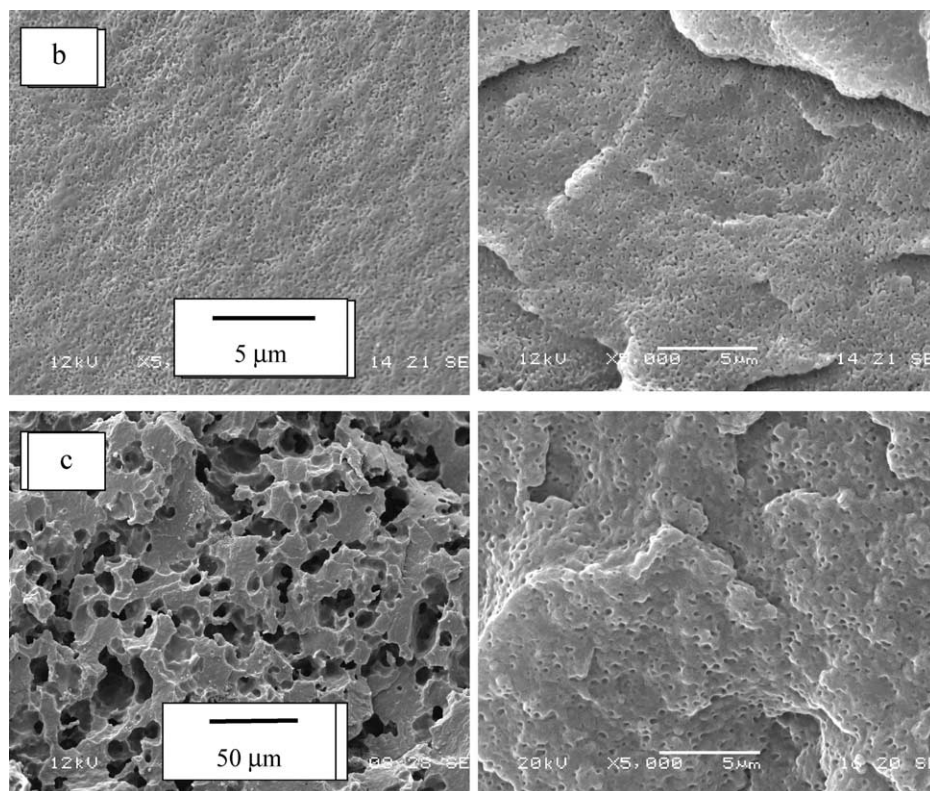


Fig. 1. SEM micrographs of the binary 75/25 LD08/CP and 75/25 PA/CP blends after etching with THF: (a) LD08/SEP; (b) LD08/SEPMA; (c) PA/SEP; (d) PA/SEPMA.

size and the size distribution of the dispersed phase droplets were determined on the micrographs of solvent etched fracture surfaces with an automatic image software, measuring the diameter of at least 500 droplet marks per sample. 85% formic acid and THF were used to etch the fracture surfaces of blends samples for scanning electron microscopy observation and to establish the nature of the matrix phase, e.g. for binary PA/CP blends. The polymer samples were usually soaked into the selected solvent at room temperature for about 48 h.

Differential scanning calorimetry (DSC) measurements were carried out under nitrogen flow, at a scanning rate of  $10\text{ }^{\circ}\text{C min}^{-1}$ , using a Pyris Perkin–Elmer apparatus calibrated with indium and tin standards.

The mechanical properties, namely the Young's modulus ( $E$ ), tensile stress (TS), and the elongation at break (EB) were measured using an Instron mod. 1122 apparatus at a crosshead speed of  $50\text{ mm min}^{-1}$ . The samples were cut out of compression molded sheets prepared in a Carver laboratory press at  $240\text{ }^{\circ}\text{C}$ . The gauge length was 30 mm, the thickness was about 1 mm, and the width was 5 mm. The results of at least seven measurements were averaged.

### 3. Results and discussion

The composition of the SEP-g-MA copolymer is

compared to that of the other MA-functionalized copolymers. The grafting yield of SEPMA is similar to that of 1-HDPE-g-MA ( $0.1\text{ mmol g}^{-1}\text{ CP}$ ) and lower than that of SEBSMA1 ( $0.173\text{ mmol g}^{-1}\text{ CP}$ ).

The possible reactions between PA and the MA functionalities of the CPs have already been discussed in previous papers [2,4]. It should be mentioned that, the anhydride groups can only react with the amine end groups or the amide inner groups of PA and lead to CP-g-PA copolymers containing pendant PA branches. In order to evaluate qualitatively the interactions between the CP and both components of the blends the morphology and the thermal properties of the binary LD08/SEP, LD08/SEPMA, PA/SEP and PA/SEPMA 75/25 w/w have been studied.

#### 3.1. Binary blends

The morphology of the 75/25 LD08/SEP, LD08/SEPMA, PA/SEP and PA/SEPMA blends after etching with tetrahydrofuran is shown in Fig. 1(a)–(d). The micrographs of the blends LD08/SEP and LD08/SEPMA (Fig. 1(a) and (b)) clearly demonstrate that both components in the blends are highly compatible. The blends LD08/SEBSMA1 and LD08/1-HDPE-g-MA are immiscible on a molecular level too, though being highly compatible [2, 4]. The micrograph of the PA/SEP blend demonstrates a biphasic morphology characterized by poor dispersion of



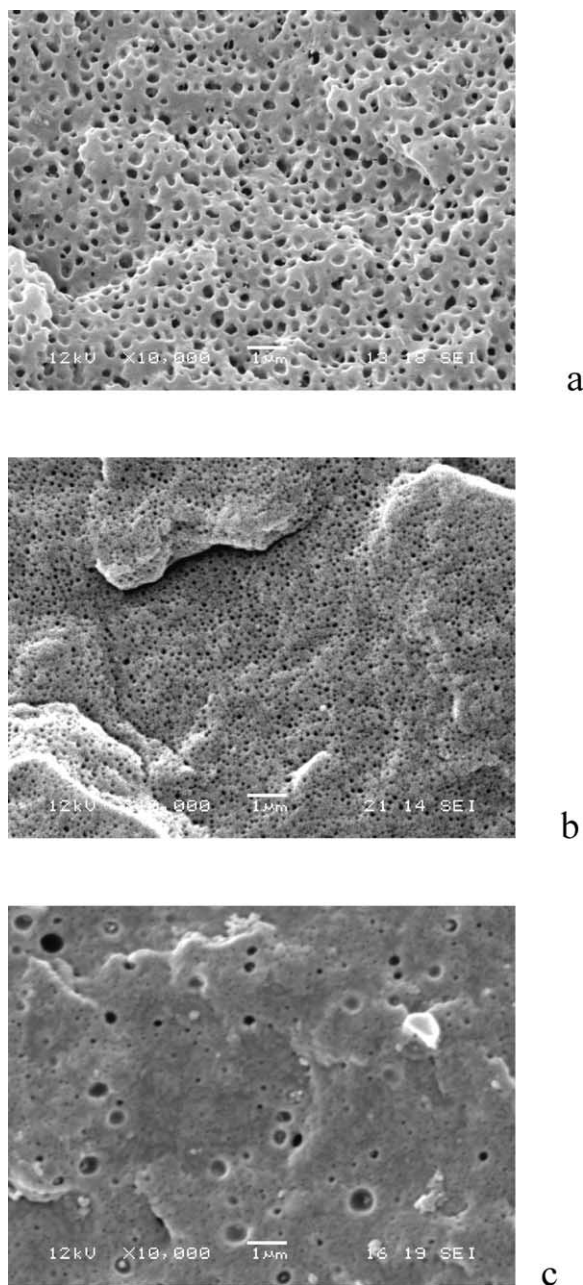


Fig. 2. SEM micrographs of the binary 75/25 PA/CP blends after etching with THF (a), (b) or xylene (c): (a) PA/SEPMA; (b) PA/SEBSMA1; (c) PA/1-HDPE-*g*-MA.

the minor phase (Fig. 1(c)). Contrary, the morphology of the blend PA/SEPMA (Fig. 1(d)) is characterized by fine dispersion of the minor phase and improved adhesion, due to the formation of PA-*g*-SEPMA copolymer through the reaction between the reactive groups of PA and CP. The morphology of PA/SEPMA blend is compared to that of PA/SEBSMA1 and PA/1-HDPE-*g*-MA blends on Fig. 2(a)–(c). As seen the dimensions of the dispersed particles in PA/SEPMA blend (about 0.3 μm) are a little bigger than those in PA/SEBSMA1 blend (about 0.1 μm). That is in agreement with the higher degree of functionalization of

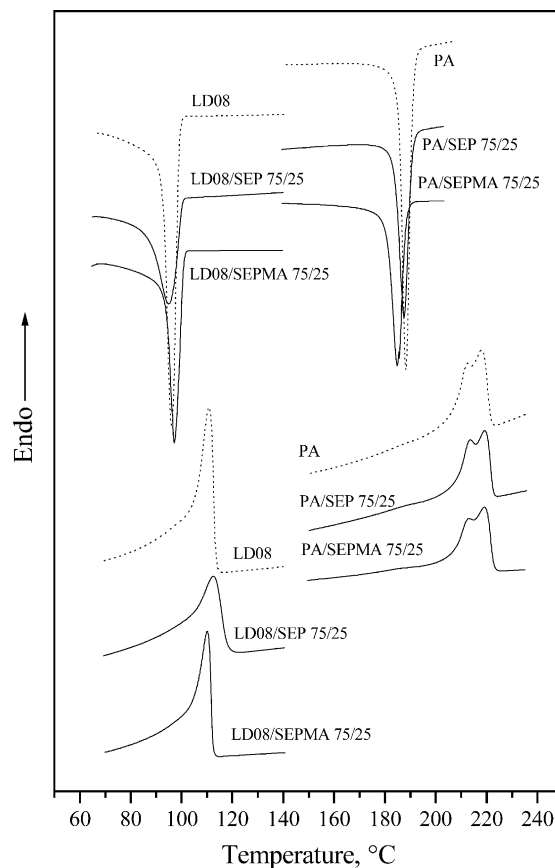


Fig. 3. Cooling and second heating DSC traces of the pure PA and LD08 and binary LD08/CP and PA/CP blends 75/25 w/w blends.

SEBSMA1. Both blends are characterized by good dispersion of the minor phase, while the dispersion of the blend PA/1-HDPE-*g*-MA is not completely homogeneous.

The thermal properties of the binary blends has been studied by DSC. Cooling and second heating DSC traces of the blends as well as those of the pure components are shown in Fig. 3. The thermal characteristics are collected in Table 1. As seen (Fig. 3, Table 1) the addition of SEPMA into LD08 has practically no effect on the temperature and the enthalpy of the melting/crystallization transitions of LD08, while the addition of SEP into LD08 leads to the broadening of the peaks and to the decrease of the enthalpies of PE phase transitions. This means that SEP is partially

Table 1  
Thermal properties of the pure materials and of the binary blends

Blend	LDPE or PA Phase			
	$T_c$ (°C)	$\Delta H_c^a$ (J g <sup>-1</sup> )	$T_m$ (°C)	$\Delta H_m^a$ (J g <sup>-1</sup> )
PA	187.6	69.8	219.4	67.7
LD08	96.6	82.3	110.9	84.0
LD08/SEP 75/25	95.0	74.3	111.5	70.2
LD08/SEPMA 75/25	97.1	80.5	110.1	83.3
PA/SEP 75/25	187.5	65.7	219.3	64.0
PA/SEPMA 75/25	183.8	54.3	219.0	54.6

<sup>a</sup> Figures normalized to the amount of the relevant phase.

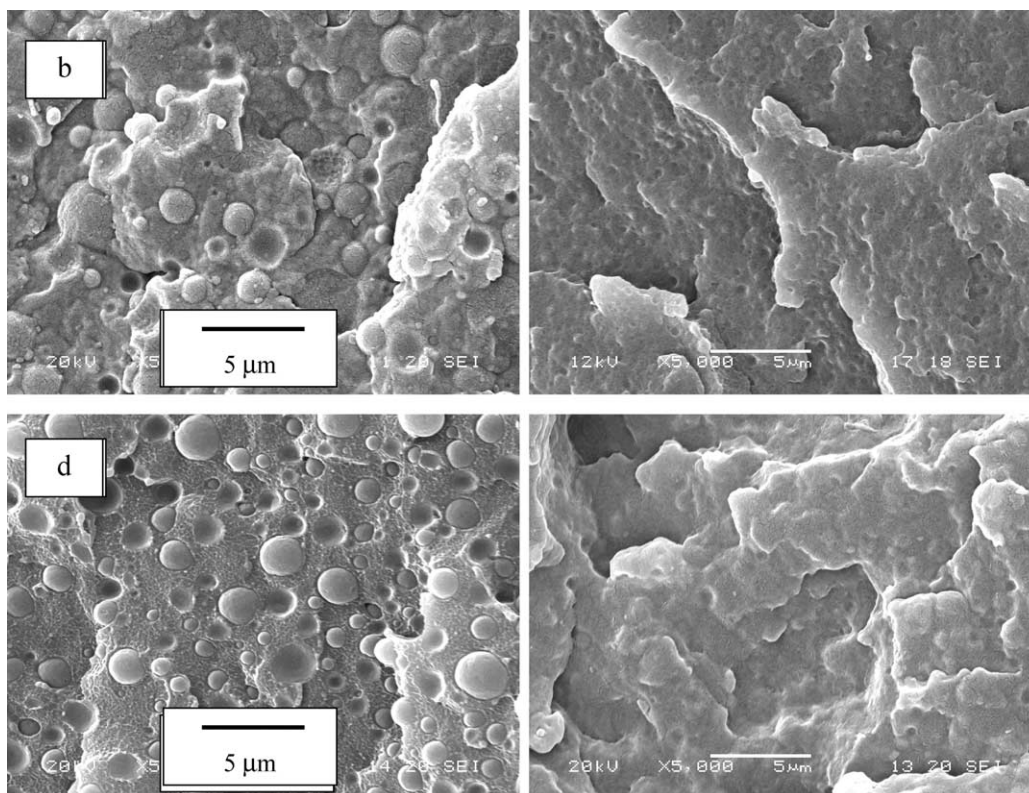


Fig. 4. SEM micrographs of the 75/25 LD08/PA (a), (b) and 25/75 LD08/PA (c), (d) blends, without (a), (c) and with (b), (d) 2/h of SEPMA.

miscible with LD08, while SEPMA is not miscible with LD08. It could be mentioned that SEBSMA1 and 1-HDPE-g-MA are not miscible with LD08 too [2,4]. Concerning the PA/CP blends, the temperatures and the enthalpies of the melting and crystallization transitions of PA are practically unaffected by the addition of SEP into the polyamide. On the contrary, the thermal behaviour of the PA phase is considerably altered when the SEPMA is blended with PA. Both the temperature and the enthalpy of the PA crystallization transition decrease; moreover the enthalpy of the PA melting transition decreases too. This is due to the strong interactions between PA and SEPMA. SEBSMA1 and 1-HDPE-g-MA demonstrate similar behaviour when blended with PA [2,4].

The results demonstrate that SEP is inactive in the compatibilization of PA phase; moreover there is a partial miscibility between SEP and LD08. Contrary, strong interactions occur between SEPMA and PA, due to the proceeding of the chemical reactions between both components. In addition, SEPMA is highly compatible with LD08 phase. So, ternary blends LD08/PA/SEPMA have been prepared and characterized.

### 3.2. Ternary blends

SEM micrographs of the fracture surfaces of the 75/25 and 25/75 LD08/PA blends without and with 2/h of SEPMA (Fig. 4), clearly demonstrate the strong reduction of the

Table 2  
Thermal properties of the ternary LD08/PA/SEPMA blends

Blend	SEPMA (per h)	LDPE phase				PA phase			
		$T_c$ (°C)	$\Delta H_c^a$ (J g <sup>-1</sup> )	$T_m$ (°C)	$\Delta H_m^a$ (J g <sup>-1</sup> )	$T_c$ (°C)	$\Delta H_c^a$ (J g <sup>-1</sup> )	$T_m$ (°C)	$\Delta H_m^a$ (J g <sup>-1</sup> )
LD08/PA	0	98.1	78.6	111.7	97.4	188.0	66.0	220.3	67.7
75/25	1	98.1	75.1	111.9	96.0	187.2	48.5	220.0	62.6
	3	99.0	76.8	110.6	95.3	185.7 (116.1)	12.4 (4.9)	218.2	63.6
	5	98.0	77.1	113.3	99.6	184.5 (119.9)	6.8 (15.7)	218.7	68.0
LD08/PA	0	98.2	77.7	109.7	95.3	188.8	69.8	219.9	68.1
25/75	1	98.6	76.6	109.9	93.8	189.9	64.8	219.1	66.9
	3	98.8	78.7	109.3	91.7	189.3	66.5	218.6	64.2
	5	98.9	74.2	109.7	99.7	190.1	63.6	218.8	67.9

<sup>a</sup> Figures normalized to the amount of the relevant phase.

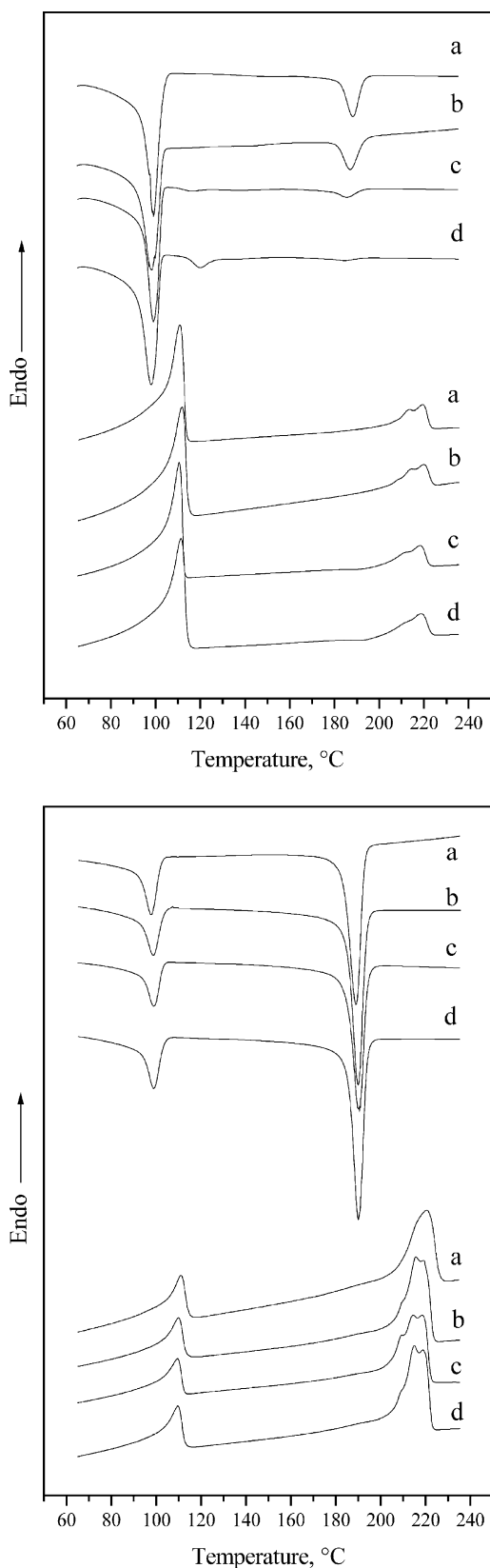


Fig. 5. (a) Cooling and second heating DSC traces of the 75/25 LD08/PA blends without (a) or with 1 (b), 3 (c) or 5/h (d) of SEPMA; (b) cooling and second heating DSC traces of the 25/75 LD08/PA blends without (a) or with 1 (b), 3 (c) or 5/h (d) of SEPMA.

dimensions of the dispersed particles in the presence of the CP. Furthermore the adhesion and the minor phase dispersion are considerably improved.

DSC measurements of the blends, compatibilized with varying amounts of SEPMA have been performed in order to evaluate the influence of the addition of the CP on the thermal characteristics of the blend components. The DSC traces of LD08/PA 75/25 blend without and with 1, 3 or 5/h of SEPMA are shown in Fig. 5(a), while those of the blend 25/75 without and with 1, 3 or 5/h of SEPMA are presented in Fig. 5(b), respectively. The thermal characteristics of the blends are collected in the Table 2. The LD08 temperatures and enthalpies of the phase transitions have been almost unaffected by the presence of the CP as could be expected. Contrary, an appearance of a fractionated crystallization in the compatibilized blends could be expected for the PA phase, wherein PA is the dispersed phase [1,4], due to the strong reduction of the dimensions of PA particles. In fact, the addition of CP causes PA crystallization at several steps at considerably lower temperatures and reduced enthalpy. The fractionated crystallization involves some transformation of the usual heterogeneous crystallization mechanism into a homogeneous one, caused by the reduction of the PA particle size [1,4]. The appearance of the fractionated crystallization in LD08/PA 75/25 blends in the presence of the SEPMA confirms the strong compatibilizing efficiency of this CP.

The results demonstrate that SEPMA possess strong compatibilizing efficiency towards LD08/PA 75/25 and 25/75 blends.

### 3.3. Compatibilizing efficiency of different MA-functionalized CPs

The morphology of LD08/PA 75/25 and 25/75 blends compatibilized with different MA-functionalized precursors (2/h) is demonstrated on Fig. 6. As seen the average droplet dimensions are smaller when LD08 is the matrix phase for all CPs (Fig. 6(a), (c) and (e)). In fact, in LD08/PA 75/25 blends the average droplet dimensions are ca. 0.3  $\mu\text{m}$  for SEPMA and SEBSMA1 and 0.4  $\mu\text{m}$  for 1-HDPE-*g*-MA, while in LD08/PA25/75 the average dimensions of minor phase are 0.6  $\mu\text{m}$  for SEPMA and SEBSMA1 and 0.9  $\mu\text{m}$  for 1-HDPE-*g*-MA. These results could be explained by a partial inclusion of the formed PA-*g*-CP copolymers into the bulk when PA is the matrix phase, thus decreasing the compatibilizing efficiency of the CPs. Contrary, when LD08 is the matrix phase the formed PA-*g*-CP copolymers migrate more easily to the interface, which is a requirement for a strong compatibilizing activity. Fig. 7 presents the emulsification curves of the blends compatibilized with the different CPs. As seen for LD08/PA 75/25 blends the three-compatibilizer precursors demonstrate almost similar efficiency. However, for LD08/PA 25/75 blends the efficiency of SEPMA and SEBSMA1 is stronger than that of 1-HDPE-*g*-MA. The results show that all three CPs has



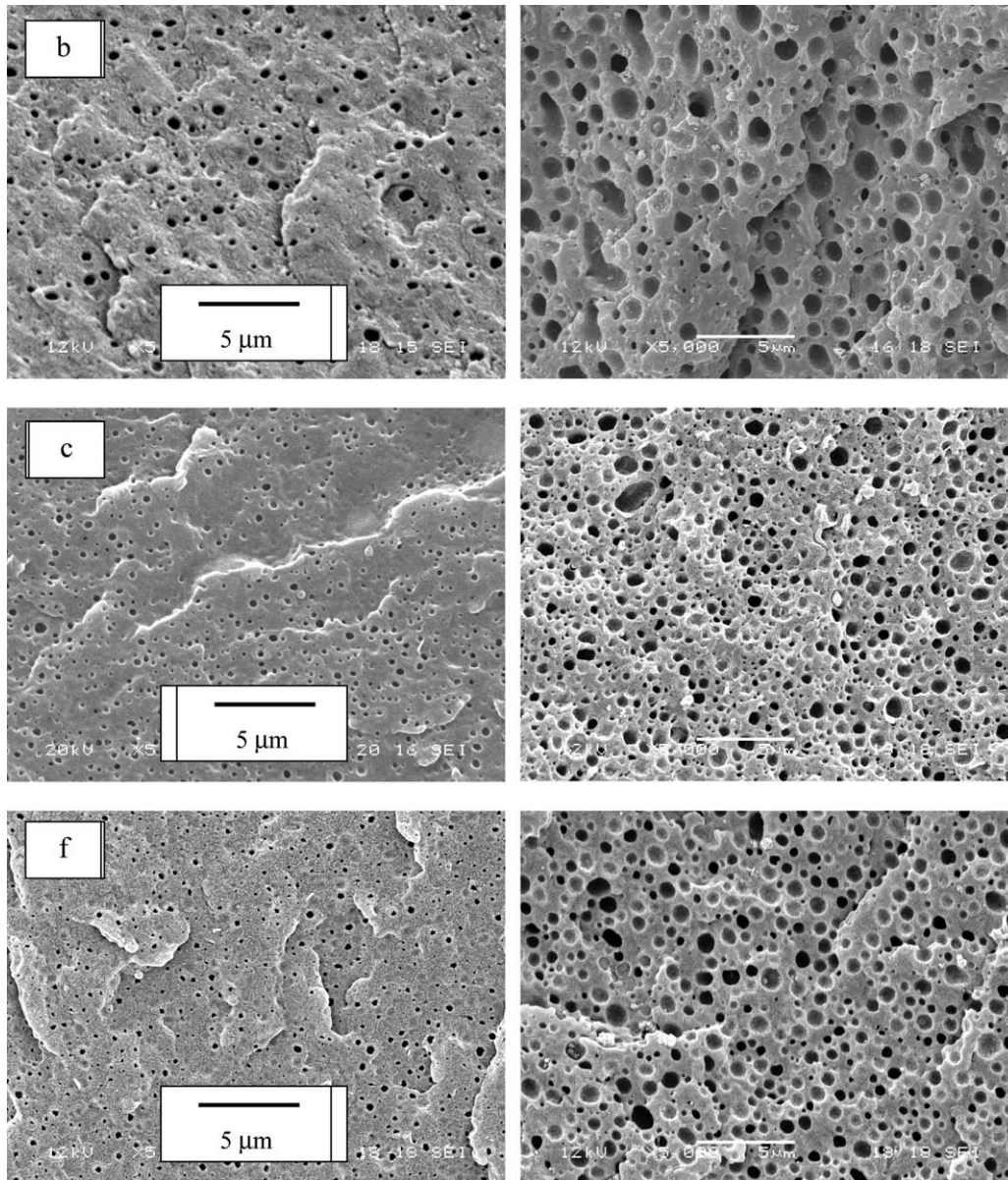


Fig. 6. SEM micrographs of the ternary 75/25/2 and 25/75/2 LD08/PA/CP blends after etching with xylene or formic acid: (a) 75/25/2 LD08/PA/1-HDPE-*g*-MA; (b) 25/75/2 LD08/PA/1-HDPE-*g*-MA; (c) 75/25/2 LD08/PA/SEBSMA1; (d) 25/75/2 LD08/PA/SEBSMA1; (e) 75/25/2 LD08/PA/SEPMA (f) 25/75/2 LD08/PA/SEPMA.

very strong comparable activity towards LD08/PA 75/25 blends; in the LD08/PA 25/75 blends the activity of 1-HDPE-*g*-MA is slightly lower than that of SEPMA and SEBSMA1. This is probably due to the higher compatibility between SEPMA and SEBSMA1 with LD08, than those between 1-HDPE-*g*-MA and LD08.

The mechanical properties of the compatibilized blends are collected in Table 3. For LD08/PA 75/25 the values of the modulus of elasticity is slightly lower, while for LD08/PA 25/75 these values are higher or almost the same for the compatibilized blends in comparison to the uncompatibilized ones. The tensile strength remains almost unchanged in the presence of all kind of CPs for the blend 75/25, while for blend 25/75 the tensile strength

significantly increases in the presence of CPs. The elongation at break increases markedly in the presence of CPs. As seen the EB values for the LD08/PA 75/25 and 25/75 blends compatibilized with SEPMA and SEBSMA1 are 3–5 times greater than those of the corresponding uncompatibilized blends. For 1-HDPE-*g*-MA the EB values are one or two times greater than those of the corresponding uncompatibilized blends. The increase in the elongation at break values in the presence of CPs could be interpreted by the enhanced interfacial adhesion in the compatibilized samples. The results from the mechanical testing confirm the strong compatibilizing efficiency of SEPMA, which is comparable with that of SEBSMA1, while 1-HDPE-*g*-MA demonstrates a slightly lower activity. It should be noted

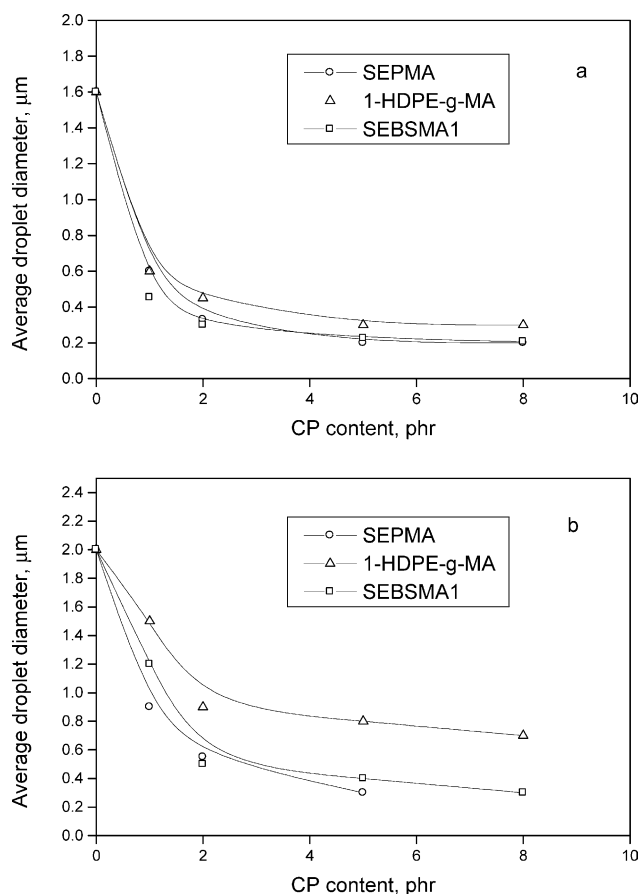


Fig. 7. Emulsification curves for the blends LD08/PA6 75/25 (a) and for 25/75 (b) using different MA-functionalized CPs.

that the increase of the tensile strength of 25/75 blend in the presence of SEPMA would be of great interest and importance for the practical application of these compatibilized materials.

#### 4. Conclusion

The results of this work demonstrate that the SEP thermoplastic elastomer, though being practically inactive as such for the compatibilization of polyethylene/polyamide

Table 3  
Mechanical properties of the compatibilized blends

Blend	<i>E</i> (MPa)	TS (MPa)	EB (%)
LD08/PA 75/25	262	12.3	45
LD08/PA6/1-HDPE-g-MA 75/25/2	241	12.5	122
LD08/PA6/SEBSMA1 75/25/2	208	11.8	135
LD08/PA6/SEPMA 75/25/2	200	11.9	257
LD08/PA6 25/75	370	25.9	113
LD08/PA6/1-HDPE-g-MA 25/75/2	411	28.1	145
LD08/PA6/SEBSMA1 25/75/2	363	28.5	323
LD08/PA6/SEPMA 25/75/2	372	28.6	305

blends, can nevertheless be used for the production of highly effective CP by the melt grafting of MA. In fact, the home-made SEP-g-MA copolymer used in this work for the reactive compatibilization of 75/25 and 25/75 LDPE/PA blends has been shown to possess a very strong compatibilizing efficiency. The comparative study of the morphology, the thermal properties and the mechanical properties of different MA-functionalized CPs proves that the strong compatibilizing efficiency of SEPMA towards LD08/PA blends is comparable with that of SEBSMA1, while 1-HDPE-g-MA demonstrates a slightly lower activity, especially when PA is the matrix phase. The results have been interpreted by the different extent of compatibility between the CPs and the LD08 phase. The slightly lower activity of the CPs for the blend LD08/PA 25/75 could be explained by a partial inclusion of the formed PA-g-CP copolymers into the bulk when PA is the matrix phase.

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