

# Blends of Poly(ethylene terephthalate) and Low Density Polyethylene Containing Aluminium: A Material Obtained from Packaging Recycling

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**ABSTRACT:** Composites of polyethylene and aluminium (PEAL) may be obtained from the recycling of postconsumed Tetra Pak aseptic packaging. The components of the composite are low density polyethylene (LDPE), aluminium and an ethylene-methacrylic acid random copolymer (EMAA). The presence of metallic filler and a functionalized copolymer, which may act as a compatibilizer, suggests that blending PEAL with other thermoplastic would be a way to obtain reinforced and compatibilized blends from recycled materials. Blends of PEAL and recycled poly(ethylene terephthalate) (PET) were prepared in the compositions of 30, 50, and 70 wt % of PET in a twin-screw extruder. Blends of PET/LDPE and PET/

EMAA were also prepared for comparison. The morphological analysis showed that the PET/PEAL blends present an excellent interfacial adhesion, similar to the PET/EMAA blend. The improvement of adhesion in comparison with the PET/LDPE blend is a result of the interaction between polar groups of PET and EMAA. PET/PEAL blends presented lower elongation at break and impact strength than the other blends whereas Young modulus was higher. © 2007 Wiley Periodicals, Inc. *J Appl Polym Sci* 106: 2524–2535, 2007

**Key words:** aseptic packaging; recycling; blends; ionomers; PEAL; PET

## INTRODUCTION

Most of the immiscible blends present poor mechanical properties because of the low adhesion between the components. When the blend is submitted to mechanical effort, tension transfer from one phase to another occurs at the interface. If the interfacial adhesion is low, tension concentration may generate fractures.<sup>1–3</sup> However, if the better interface is achieved through interfacial modification, the immiscible blends properties may be improved. The addition of a third component, a compatibilizer, can lead to a finer dispersion of phase and more stable morphology by reducing interfacial energy and increasing adhesion between the phases.<sup>4,5</sup>

Block and grafting copolymers are the most used compatibilizers.<sup>6</sup> These polymers migrate to the interface promoting a specific interaction or a chemical reaction between the blend components. Although

there is no consensus about the mechanism involved in blends compatibilization through random copolymers,<sup>7</sup> several studies showed that these copolymers also can improve interfacial adhesion between immiscible phases.<sup>8–12</sup>

Ionomers constitute a subclass of random copolymers. These polymers contain ionic groups at the main chain or the lateral groups and are prepared through ionization of acid groups followed by total or partial neutralization with metallic cations such zinc and sodium.<sup>13</sup> The properties of ionomers are dependent on the type of main chain polymer, the content of ionic groups, the counterion, and the degree of neutralization.<sup>14</sup> Because of their capacity of establishing strong interactions with several chemical groups, ionomers present excellent potential to be used as compatibilizers.<sup>15</sup>

Many works have investigated this application for these polymers. Ionomers of ethylene-containing methacrylate groups have been used in the compatibilization of blends of polyamide 6 with polyethylene<sup>16</sup> or polypropylene<sup>17</sup> as well as the blends of the copolymer of ethylene and vinyl alcohol (EVOH) with aromatic copolyesters<sup>18</sup> and high density polyethylene (HDPE).<sup>19</sup>

Blends of poly(ethylene terephthalate) (PET) with linear low density polyethylene (LLDPE)<sup>20</sup> or HDPE<sup>21</sup> present significant enhancement in the mechanical properties when compatibilized with the

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**TABLE I**  
**Properties of the Virgin and Recycled Polymers Used for Blend Preparation**

	PEAL	PET	LDPE	EMAA
Origin	Recycled from aseptic packaging	Recycled from soda packaging	Virgin	Virgin
Supplier	Tetra Pak/Mercoplás	ReciPet	Braskem	BP Chemicals
Grade	–	–	BC-818	Novex M 21 N 430
Density (g/cm <sup>3</sup> )	–	–	0.918 <sup>a</sup>	0.922 <sup>a</sup>
Crystallinity (%)	47 <sup>b</sup>	23 <sup>c</sup>	44 <sup>b</sup>	39 <sup>b</sup>
Melting flow index (g/10 min)	3.9 <sup>b</sup>	>20 <sup>c</sup>	7.5 <sup>b</sup>	7.5 <sup>b</sup>
Aluminium content (%)	15 <sup>b</sup>	0	0	0
Methacrylic acid content (%)	<1 <sup>b</sup>	0	0	1.2 <sup>a</sup>

<sup>a</sup> From supplier datasheet.

<sup>b</sup> From ref. 31.

<sup>c</sup> From ref. 24.

ionomer of polyethylene/methacrylic acid with lithium as counterion.

Besides being an interesting compatibilizer, ionomers can be mixed with other thermoplastic resulting in blends with good mechanical properties because of the excellent interfacial adhesion. Because of its low glass transition temperature, polyethylene ionomers may have performance equivalent to elastomers as impact modifier at room temperature. Some patents refer to the modification of polyamide with polyethylene ionomer containing carboxylate groups for improving the impact resistance.<sup>22,23</sup>

Blending postconsumer polymers can be an alternative to aggregate better properties and therefore broad the application potential of recycled plastics. For achieving this objective, the use of effective compatibilizer is very important. Some studies involve blends of recycled PET with polyolefins also recycled<sup>24–28</sup> and blends of postconsumed polypropylene with LDPE<sup>29</sup> and with high impact polystyrene.<sup>30</sup>

Since plastic packaging has becoming the major component of plastic waste stream because of the short time of usage, polymers components of the most used packaging for food were chosen for this work: the Tetra Brik aseptic packaging produced by Tetra Pak and PET bottles for soda.

The Tetra Brik multilayer packaging is constituted of three materials organized in six layers: paper (75%), LDPE (20%), and aluminium (5%). Adhesion between aluminium and plastic layer is achieved through the introduction of a layer of ethylene-methacrylic acid copolymer (EMAA).

The Tetra Pak recycling proposal for Tetra Brik packaging involves the separation of the paperboard from the polyethylene and aluminium film.<sup>31,32</sup> The remaining mixture composed of films of polyethylene, EMAA, and aluminium is washed in water, pressed, dried, agglutinated, and extruded

to generate pellets of a composite denominated PEAL.

The properties of PEAL were studied in previous works.<sup>33–35</sup> Mixture of this composite of recycled origin, which contains metallic filler and a compatibilizer, with other thermoplastics offers the possibility of producing low price compatibilized and reinforced blends.

## EXPERIMENTAL

### Materials

The composite PEAL, obtained from the recycling of postconsumed aseptic packaging, consists of LDPE, a small amount of EMMA, and 15% of aluminium. For comparison, blends of recycled PET with pure virgin LDPE and EMMA, both used in the composition of the multilayer packaging, were also prepared. The properties of recycled and virgin materials are presented in Table I.

### Processing

The composite PEAL, received as granules were dried at 90°C for 2 h and processed in a Wortex single screw vented extruder (Campinas, Brazil) with five temperatures zones. The screw profile is typical for polyolefins and contains a Maddock dispersive mixture element. The screw *L/D* ratio is 30 : 1 and the diameter is 32 mm. The temperature profile used in the five zones was 140, 150, 175, 160, 165°C, and the screw speed was 150 rpm.

Blends PET/PEAL, PET/EMAA, and PET/LDPE in compositions of 30, 50, and 70 wt % of PET were prepared in a five temperature zones twin screw extruder MPC/V30 (Dursley, England). The corotational and interpenetrating screws have *L/D* ratio of

**TABLE II**  
Injection Parameters for PET/LDPE, PET/PEAL and PET/EMAA Blends

	Zone 1	Zone 2	Zone 3	Zone 4	Zone 5
Temperature profile (°C)	255	260	265	270	275
Injection speed (cm <sup>3</sup> /s)			50		
Injection pressure (bar)			1400		
Back pressure (bar)			700		
Cooling time (s)			28		
Mould temperature (°C)			20 ± 5		

8 : 1 and diameter of 38 mm. The screw speed was 100 rpm and the temperature profile used was 240, 250, 265, 265, and 270°C. Before extrusion the materials were mixed and dried in a conventional oven for 6 h.

The blends were injection molded in Arburg 221K injection machine (Lossburg, Germany) for obtaining the specimens for morphological analysis, tensile, and impact test. The injection conditions for the blends are presented in Table II.

### Characterization

Morphology of the blends was investigated by field emission scanning electron microscopy (FE-SEM; Tokyo, Japan). Injection-molded specimens were cryogenically fractured parallel and perpendicular to the injection flow direction as illustrated in Figure 1. The fractured surfaces were coated with carbon and gold in a Bal-Tec Med 020 sputtering instrument (Principality of Liechtenstein) and analyzed using secondary electron image (SEI) in a JEOL JSM-6340F microscope (Middleton, WI) working at an acceleration voltage of 3 kV. The average domain sizes of the dispersed phase were estimated by image analysis using Image-Pro Plus 4.0 (Media Cybernetics, Sil-

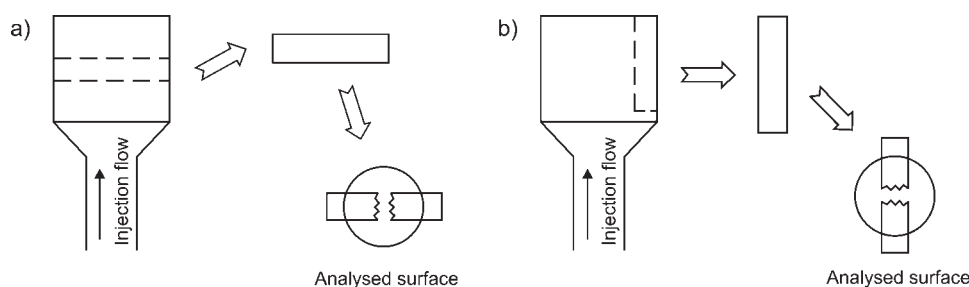
ver Spring, MD). About 200–500 domains were considered for each calculated value, and Saltikov<sup>36</sup> correction was applied.

Selective solubility test was performed for the identification of blends phases. PET phase was extracted by keeping the samples for 60 min in concentrated chlorosulfonic acid at room temperature [(23 ± 2)°C]. After washing with distillate water, drying, and coating, the samples were examined in the FE-SEM microscope.

Aluminum distribution in the extrudate and injection-molded samples was investigated using a LEO 435 VP Zeiss microscope (Oberkochen, Germany). Uncoated surfaces analyzed were obtained from cutting perpendicular to extrusion direction and parallel to injection flow direction with a conventional blade at room temperature [(23 ± 2)°C]. Image was generated from backscattering electron image (BEI) and acceleration voltage used was 15 kV.

Tensile properties were characterized using EMIC DL2000 universal testing machine (São José dos Pinhais, Brazil) with a load of 5000 N and test speed of 50 mm/min. The injection-molded specimens were conditioned for 72 h at (23 ± 2)°C and 44% relative humidity before testing. The specimen dimensions, 165-mm length and 41.6-mm<sup>2</sup> cross area, as well as the test conditions were chosen according to standard ASTM D-638.<sup>37</sup> At least eight specimens of the same sample were tested and eventual discrepant results were eliminated by applying a *Q* test.<sup>38</sup>

Notched Izod impact tests of injection-molded specimens with dimensions 63.5 mm × 10 mm × 3.2 mm were performed with EMIC pendulum-type testing machine (São José dos Pinhais, Brazil) according to ASTM D-256.<sup>39</sup> The load was 2.7 J and temperature (26 ± 3)°C. At least eight specimens of the same sample were tested and eventual discrepant results were eliminated by applying a *Q* test.<sup>38</sup> The topography of fracture under impact was analyzed through reflective optical microscopy in a stereoscope Zeiss Stemi SV11 (Oberkochen, Germany).



**Figure 1** Position and direction of fracture of specimens for morphological analysis: (a) parallel and (b) perpendicular to the injection flow.

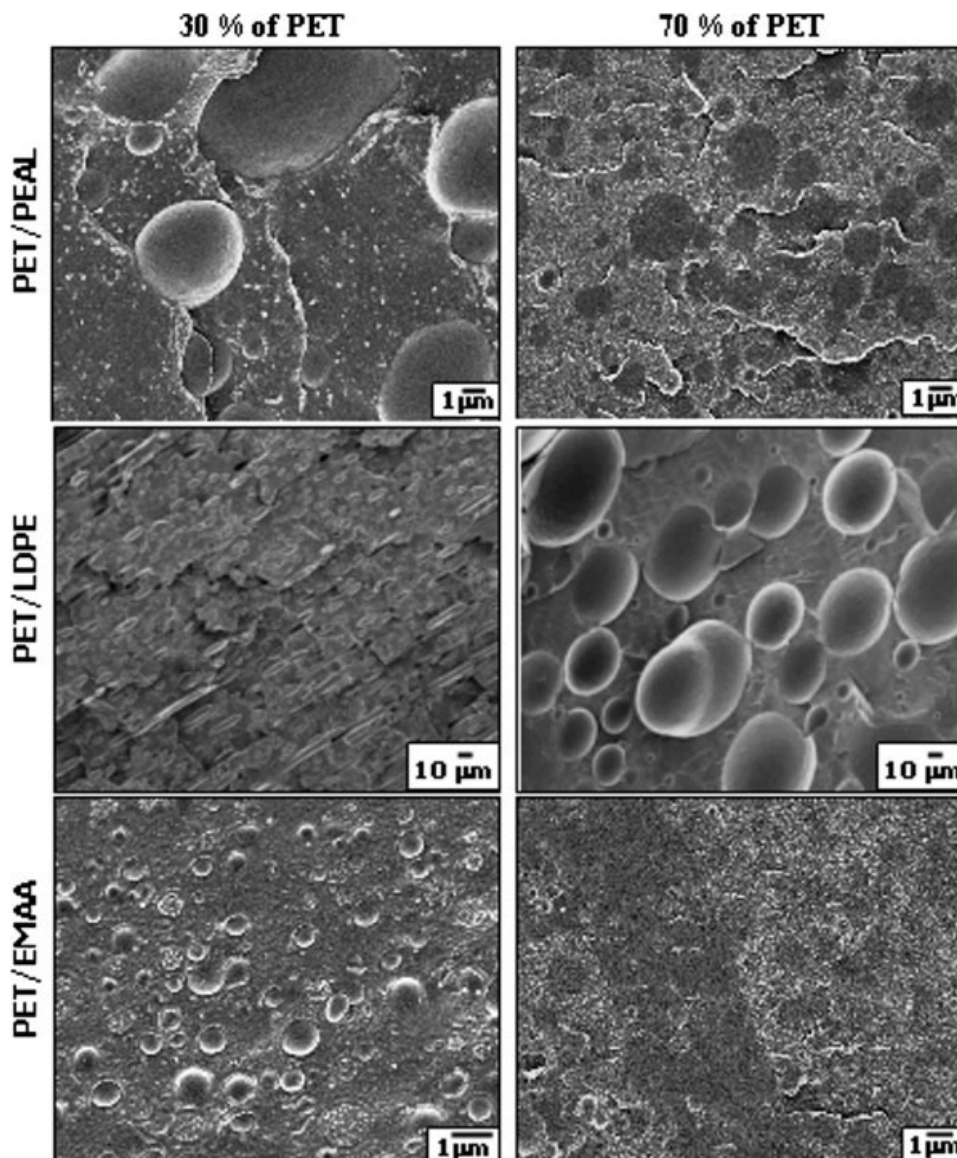


Figure 2 Parallel fractured surface of PET/PEAL, PET/LDPE, and PET/EMAA blends containing 30 and 70% of PET.

## RESULTS AND DISCUSSION

### Morphology

The morphology of the parallel fractured injection-molded blends containing 30 and 70% of PET is shown in Figure 2. All blends present spherical dispersed phase morphology. However, the parallel fracture of the blend PET/LDPE with 30% of PET shows that the domains are oriented in the flow direction. This orientation is caused by the high shear rate and is also related to the difference of viscosity between PET and LDPE. Figures 3 and 4 present the morphology of blends containing 50% of PET. Blends PET/PEAL also presented dispersed phase morphology (Fig. 3); however, the correspondent blends of PET/LDPE and PET/EMAA present a

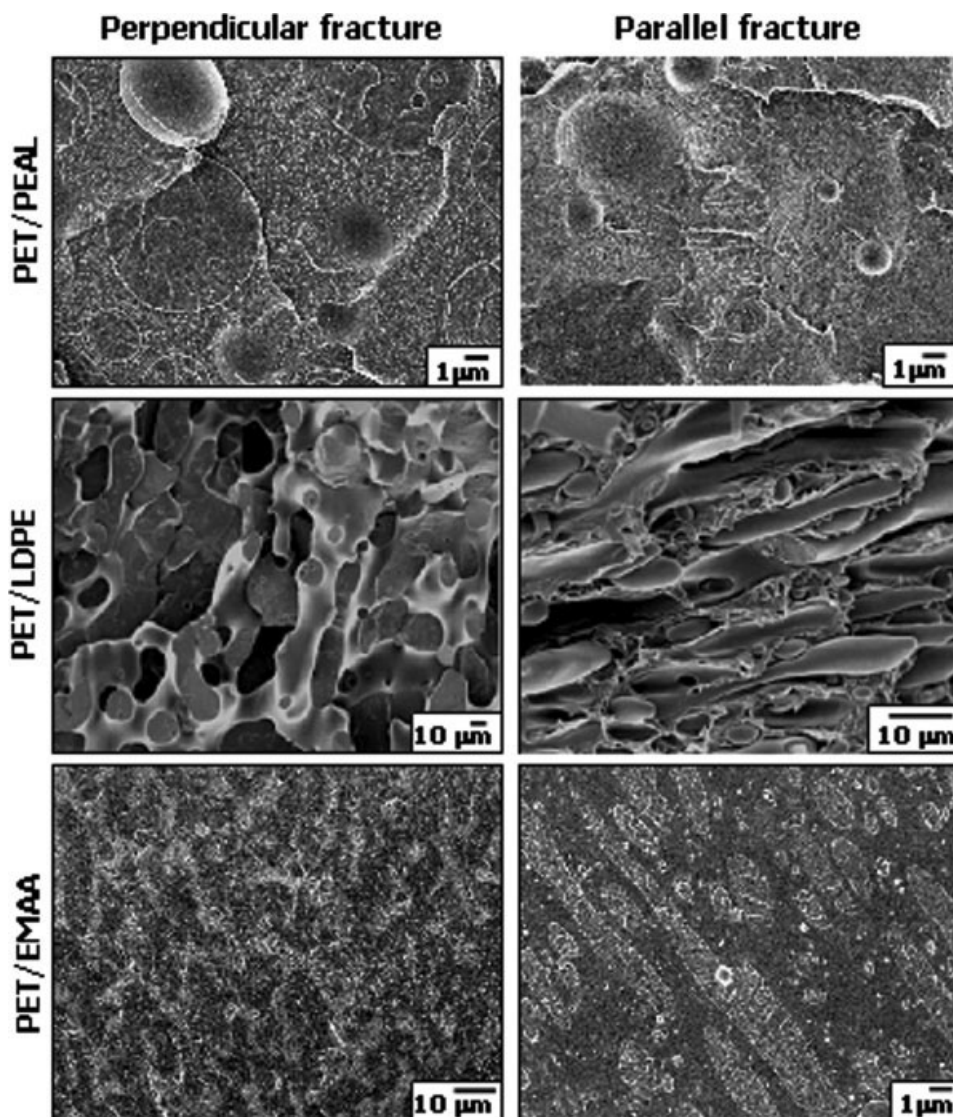
cocontinuous morphology, confirmed by microscopy analysis after the extraction of PET phase (Fig. 4).

Selective solubility test indicates that PET constitutes the dispersed phase in all blends containing 30% of PET, and the matrix in the 70% PET blends. In the 50/50 PET/PEAL blends, PET is the matrix.

The type of polyethylene used to prepare the blend as well as its composition strongly influences the dispersed phase dimensions. The average diameter of the dispersed phases is presented in Table III. Since blends with LDPE and EMMA containing 50% of PET have cocontinuous morphology, only compositions of 30 and 70% of PET are compared.

PET/PEAL blends present domain sizes intermediate to those of the correspondent PET/LDPE and





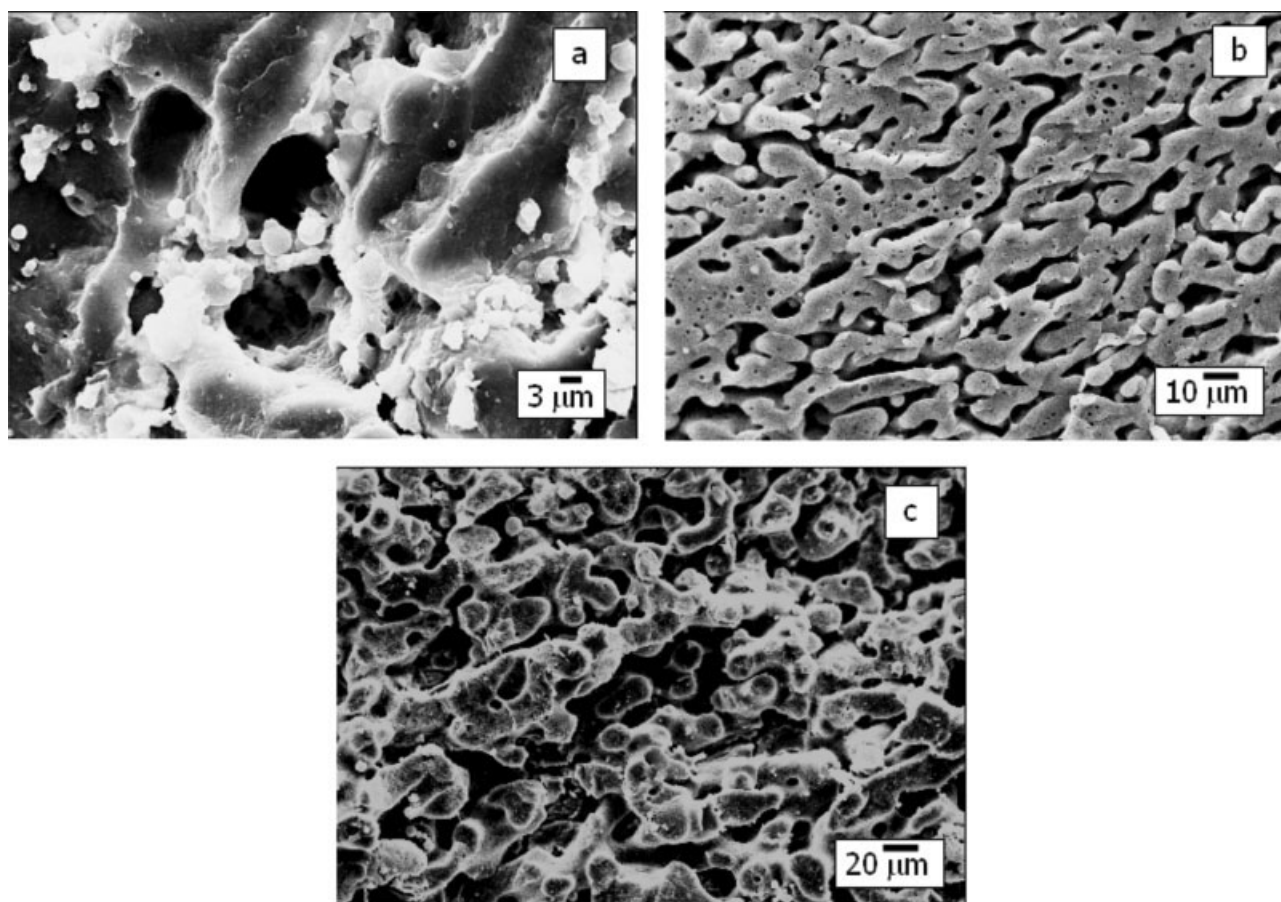
**Figure 3** Perpendicular and parallel fractured surface of PET/PEAL, PET/LDPE, and PET/EMAA blends containing 50% of PET.

PET/EMAA blends. Blends of PET/LDPE, which are noncompatibilized, present a high interfacial tension and, consequently, have a strong tendency to coalesce, which results in larger dispersed phase size. Figure 5 shows a detail of the morphology of these blends where the coalescence of domains can be observed.

On the other hand, in the PET/EMAA blends, the strong interaction expected between the ester group of PET and the carboxylic group of EMAA is responsible for a significant decrease in the interface tension leading to much smaller domains. In the case of PET/PEAL blend, the presence of copolymer causes the reduction of interfacial tension between PET and LDPE and stabilization of the dispersed phase against coalescence. Consequently, the domains are smaller compared to PET/LDPE blends.

Besides the reduction on the dispersed phase dimensions, another difference observed in the morphology of blends containing copolymer is the path of the cryogenical fracture through the material. PET/LDPE blends present rough fracture surface with intact spheres, indicating that the fracture occurs in the matrix. However, in the other blends, the fracture surface is smooth and many domains were fractured along with the matrix, indicating very strong interfacial adhesion, as strong as the cohesive force of the matrix.

Details of the interface can be observed in the magnified micrographies of blends containing 70% of PET, illustrated in Figure 6. The blend with LDPE is characterized by domains clearly segregated from the matrix and by wide voids on domains boundary; whereas in the other blends the dispersed phase is



**Figure 4** Perpendicular fractured surface, after PET phase extraction, of (a) PET/PEAL, (b) PET/LDPE, and (c) PET/EMAA blends containing 50% of PET.

strongly adhered to the matrix and the fracture propagates throughout both phases.

The formation of voids in the noncompatibilized blend can be explained by different volume shrinkages during the thermal transition of the polymers in the fast cooling. Compatibilized blends of PET with polyolefins normally present significant de-

crease in the size of spherical domains when compared with the noncompatibilized blends. However, the dispersed phase is yet detached from the matrix during the cryogenical fracture. Examples of this behavior can be found for PET/polyethylene<sup>25,40</sup> and PET/polypropylene<sup>24,41</sup> blends.

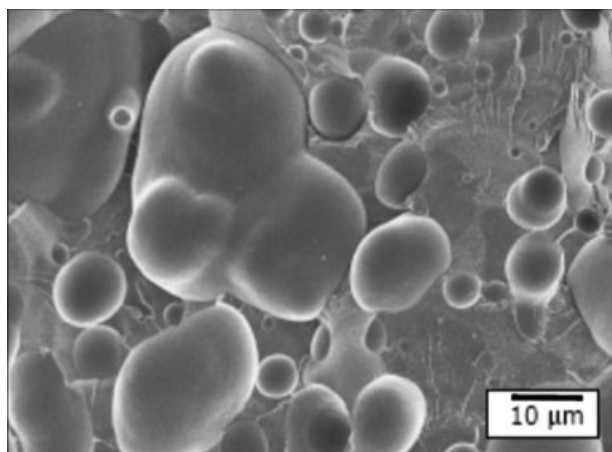
The improvement of interfacial adhesion in the polyolefin/EMAA system when compared with the PET/LDPE is due to the strong H-bonding interaction of the copolymer with PET, as proposed in Figure 7. It is also possible that, during processing, polar groups of PET undergo exchange reaction, resulting in the formation of a grafting copolymer.<sup>42</sup>

The nature of the interaction of EMMA with LDPE is related to the similarity of the hydrocarbon chains. Previous work<sup>33</sup> has shown by FTIR that the main chain of EMMA has branching characteristic corresponding to those of LLDPE. However, this structural difference still allows the occurrence of interactions between the two polymers. Studies performed with excimer fluorescence showed that blends of LDPE and LLDPE present a certain degree of interpenetration of the segments of one polymer in the other.<sup>43</sup>

**TABLE III**  
Average Numeric Diameter ( $d_n$ ), Average Minimum Diameter ( $d_{min}$ ) and Average Maximum Diameter ( $d_{max}$ ) of the Dispersed Phase in PET/LDPE, PET/PEAL and PET/EMAA Injection-Molded Blends

PET content (%)	Injection-molded blends		
	$d_{min}$ ( $\mu\text{m}$ )	$d_{max}$ ( $\mu\text{m}$ )	$d_n$ ( $\mu\text{m}$ )
PET/LDPE			
30	0.5	9.5	2.5
70	2.5	62.5	17.3
PET/PEAL			
30	0.5	4.5	1.3
70	2.5	13.5	5.8
PET/EMAA			
30	0.1	1.5	0.5
70	0.6	2.6	1.2





**Figure 5** Coalescence of dispersed phase in PET/LDPE blends with 30% of PET.

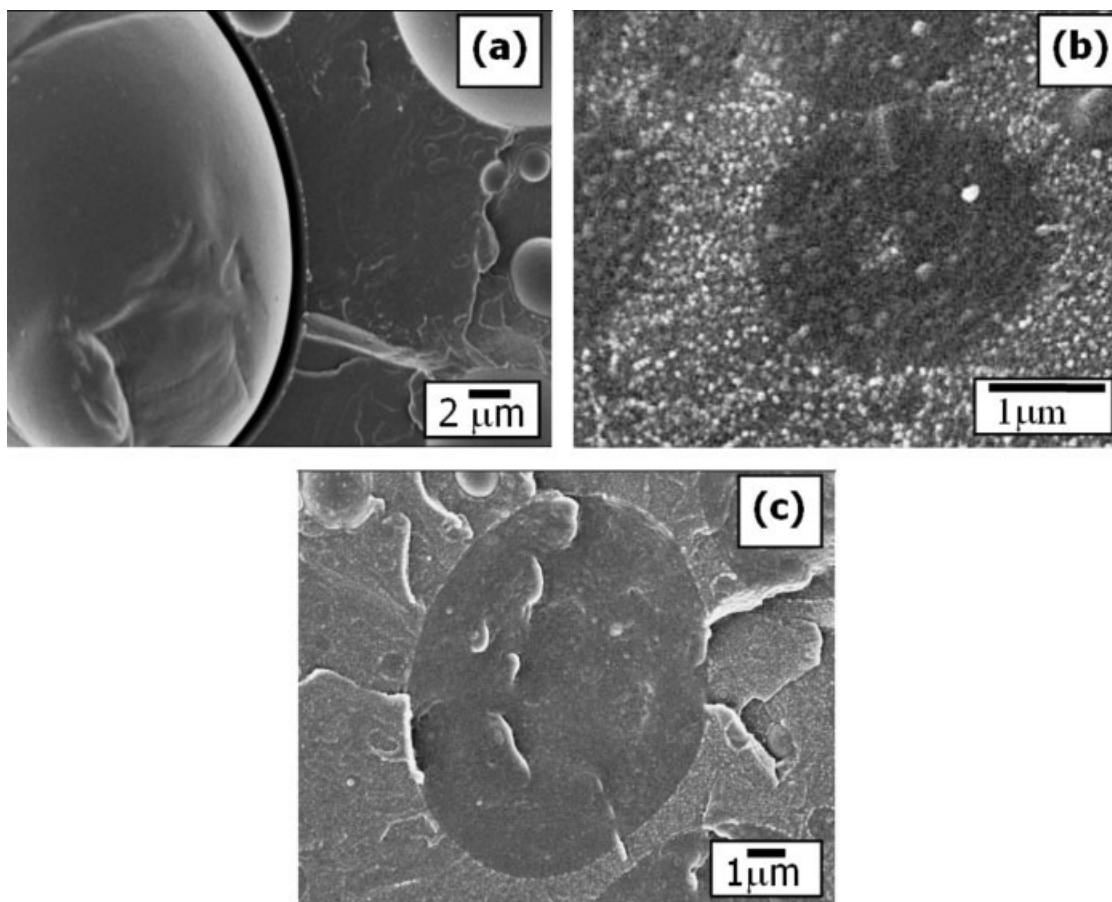
Very few studies in the literature deal with the use of random copolymer as interface modifiers, besides, there is no clear consensus regarding the mechanisms of blend compatibilization through random copolymers. However, it has been shown that these copolymers preferentially locates at polymer–

polymer interface and only critical concentration or molecular weight would lead to the formation of a segregate third phase.<sup>7,8</sup>

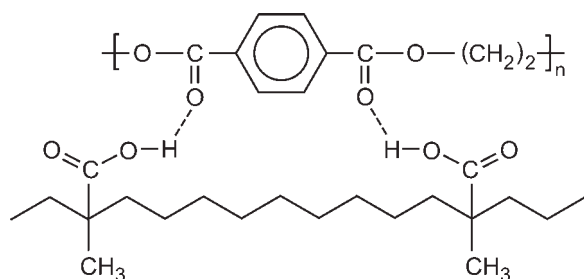
In the case of PET/PEAL blends, which contain a random copolymer, no evidence of a third phase was found. Therefore, the amount of copolymer in PEAL composition is suitable for modifying the PET/LDPE interface, promoting an increase in interfacial adhesion, and the reduction of the dispersed phase size compared with the noncompatibilized PET/LDPE blends, as is shown by morphological analysis.

Besides the polymeric phase distribution, another subject of morphological analysis of PET/PEAL blends is the distribution and location of the aluminium particles. Figure 8 presents SEM micrographs of the cut surface of extruded and injection-molded specimens. BEI mode was used for achieving better image contrast of the metal filler.

The aluminium foil of the laminate was chopped during extrusion and the pieces were assumed a cylindrical shape. In the extruded samples, the particles seem to be disposed in a circular orientation driven by the screw rotation whereas in the injec-



**Figure 6** Details of matrix/dispersed phase interface in the (a) PET/LDPE, (b) PET/EMAA, and (c) PET/PEAL with 70% of PET. Matrix is PET in all the blends.



**Figure 7** Scheme of hydrogen interaction between PET and EMAA copolymer.

tion-molded blends the filler is oriented in the injection flow direction.

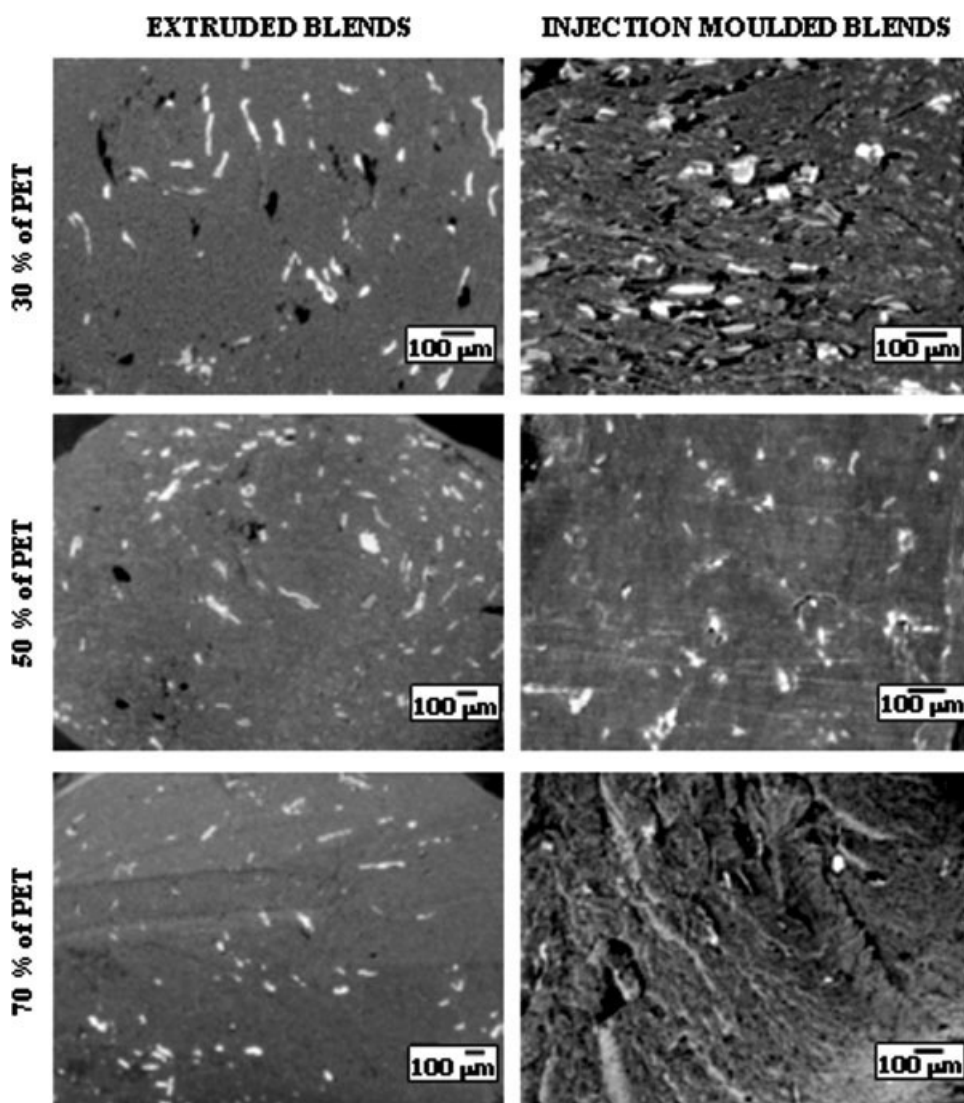
The dependence of concentration of aluminium particles at the surface with blend composition is clearer for the injection molded, especially comparing blends with 30 and 70% of PET. The aver-

age size of aluminium particles is in the order of 100 μm, and therefore, much larger than the average dimensions of the polymeric dispersed phase (Table III).

**Mechanical properties**

Table IV presents the mechanical properties of the PET/PEAL, PET/LDPE, and PET/EMAA blends. The average stress–strain curves are shown in Figure 9, and the results of tensile tests are presented in Figure 10.

Comparing the different polyethylenes, it can be observed that the modulus of PEAL is higher than the two other polymers and the copolymer EMAA presents lower modulus than that of LDPE. Elongation at break is also different for each type of polyethylene, being EMAA the one with the higher



**Figure 8** Aluminium particles at the cutting surface of extruded and injection-molded PET/PEAL blends.



**TABLE IV**  
**Mechanical Properties of PET/LDPE, PET/PEAL, and PET/EMAA Blends**

PET content (%)	PET/LDPE	PET/PEAL	PET/EMAA
Young modulus (MPa)			
0	96 ± 2	164 ± 4	80 ± 12
30	215 ± 2	278 ± 5	186 ± 6
50	397 ± 4	485 ± 4	390 ± 6
70	563 ± 25	681 ± 9	660 ± 9
100	1158 ± 24	1158 ± 24	1158 ± 24
Elongation at break (%)			
0	92 ± 2	47 ± 4	156 ± 3
30	55 ± 9	10 ± 1	154 ± 17
50	9 ± 1	11 ± 1	164 ± 29
70	4 ± 1	6 ± 1	125 ± 42
100	8 ± 1	8 ± 1	8 ± 1
Impact energy (J/m)			
0	Not broken	315 ± 32	Not broken
30	139 ± 14	27 ± 3	Not broken
50	25 ± 1	7 ± 1	155 ± 13
70	6 ± 1	9 ± 2	43 ± 3
100	32 ± 2	32 ± 2	32 ± 2

values, followed by LDPE and PEAL, in this order. Although all the blends have presented a decrease in modulus values compared with the pure PET, this reduction was less intense for the PEAL blends.

Values of elongation at break of blends of EMAA practically remained invariable in compositions up to 50% of PET. The other blends presented a decrease in elongation at break with the increase of PET content. This decrease is even more accentuated than that predicted by additivity rule (Fig. 10).

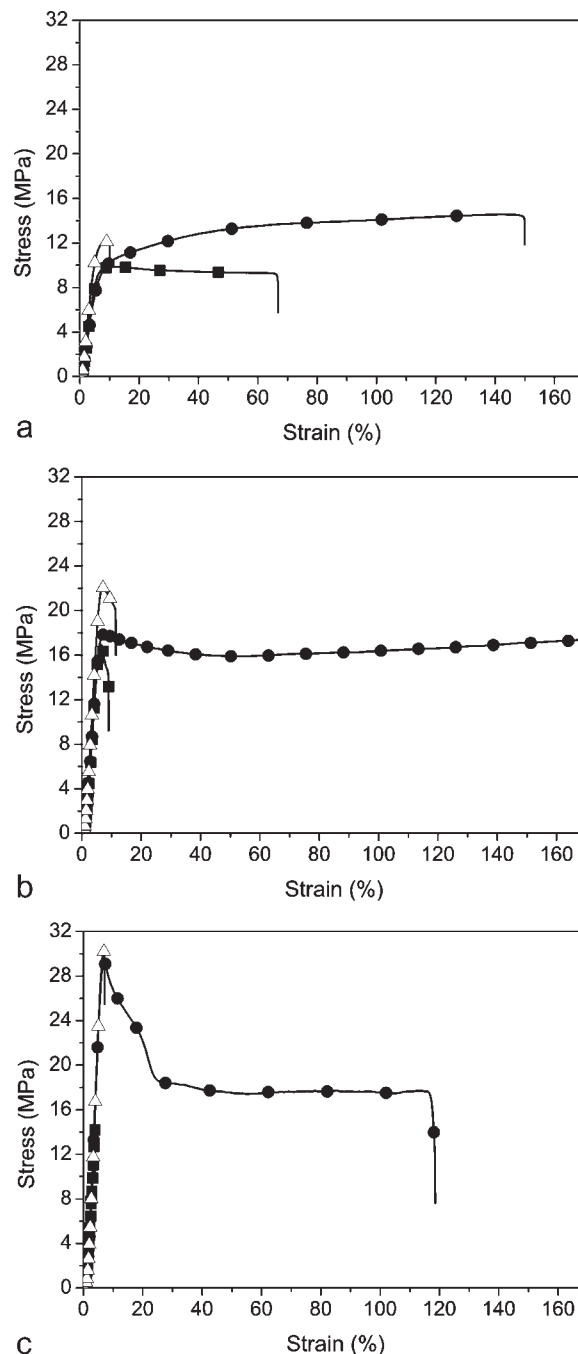
Elongation at break is a very sensitive indicator of interface adhesion in immiscible blends.<sup>44</sup> The occurrence of a positive deviation in relation to additivity is a result of the strong interaction between the phases. In the case of PET/LDPE blends, this type of interaction is absent and in the blends PET/PEAL, although a good interaction of the components exists, the effect of aluminium particles in the elongation is negative, and then the composite presents elongation smaller than the pure polyethylene.

Figure 11 presents the results for notched Izod impact test of the different blends. It is possible to observe the blends with ionomer present impact resistance superior to that of the pure PET. This is a very positive result considering that in relation to PET the same system presented a considerable enhancement in the elongation at break (Fig. 10). Alike pure EMAA and pure LDPE, the blend of PET/EMAA containing 30% of PET could not be fractured under the chosen experimental conditions.

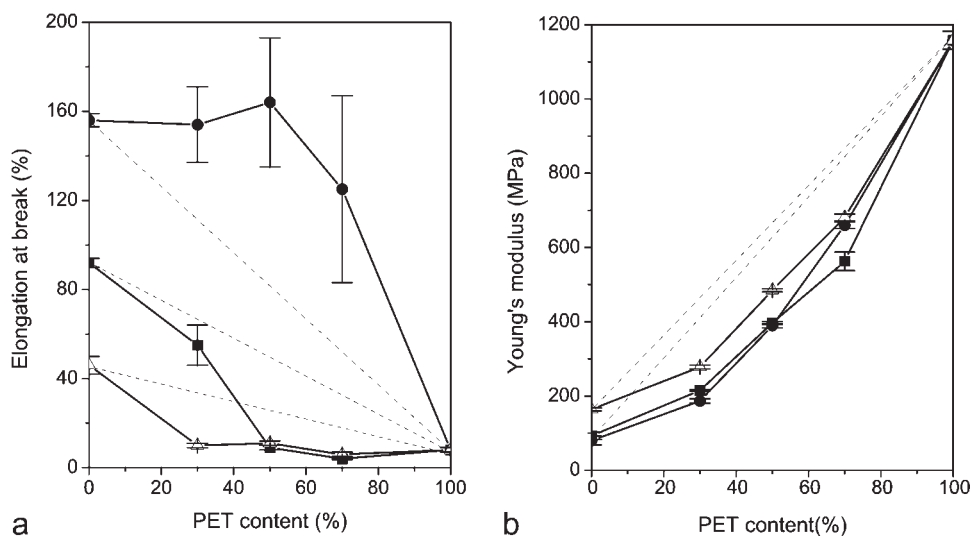
Blends of PEAL present a significant decrease in the impact resistance in relation to blends of LDPE. This effect is caused by the presence of aluminium particles, reducing the flexibility of the composite

and introducing more interfaces, which may concentrate on tension to initiate the fracture.

The structure of the blend fractured surface provides information regarding the deformation mechanism and the stability of crack propagation upon loading. Figure 12 presents the impact fractured surface of unmodified PET revealing the typical characteristics of a brittle failure; a relatively flat, smooth surface without any sign of deformation. The modifications



**Figure 9** Stress-strain curves of (-△-) PET/PEAL, (-■-) PET/LDPE, and (-●-) PET/EMAA blends containing (a) 30, (b) 50, and (c) 70% of PET.



**Figure 10** (a) Young's modulus and (b) elongation at break in function of composition for (-Δ-) PET/PEAL, (-■-) PET/LDPE, and (-●-) PET/EMAA blends. Dashed line corresponds to values predicted by additivity rule.

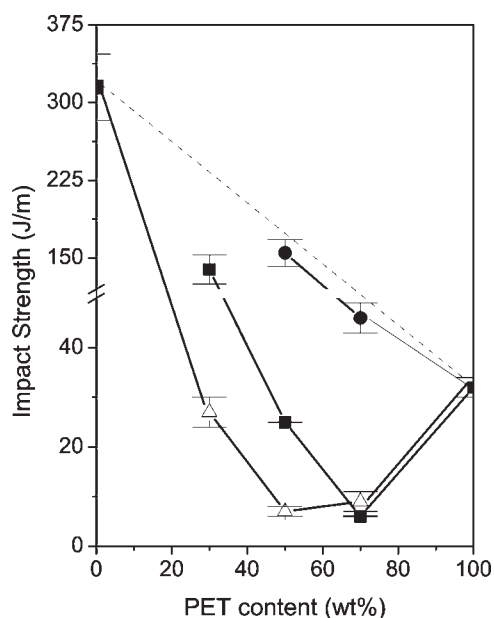
in the fracture characteristics when PET is blended with the different polyethylenes can be observed in Figure 13.

Blends of PET and EMAA present fracture features different of those of pure PET. In blends containing 70% of PET, the crack propagation is much slower as evidenced by the markings near the notched root, meaning that the copolymer dispersed as small spherical domains strongly attached to the

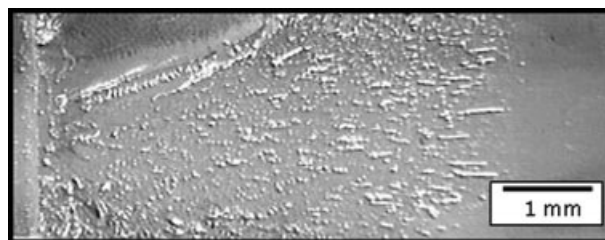
matrix can act as an impact modifier. This topographic characteristic is typical of a material with ductile fracture mechanism, which means that the presence of copolymer changed the fracture mechanism of PET. In blends with 50% of PET, because of the cocontinuous morphology, the effect of copolymer on the fracture characteristics was less prominent.

## CONCLUSIONS

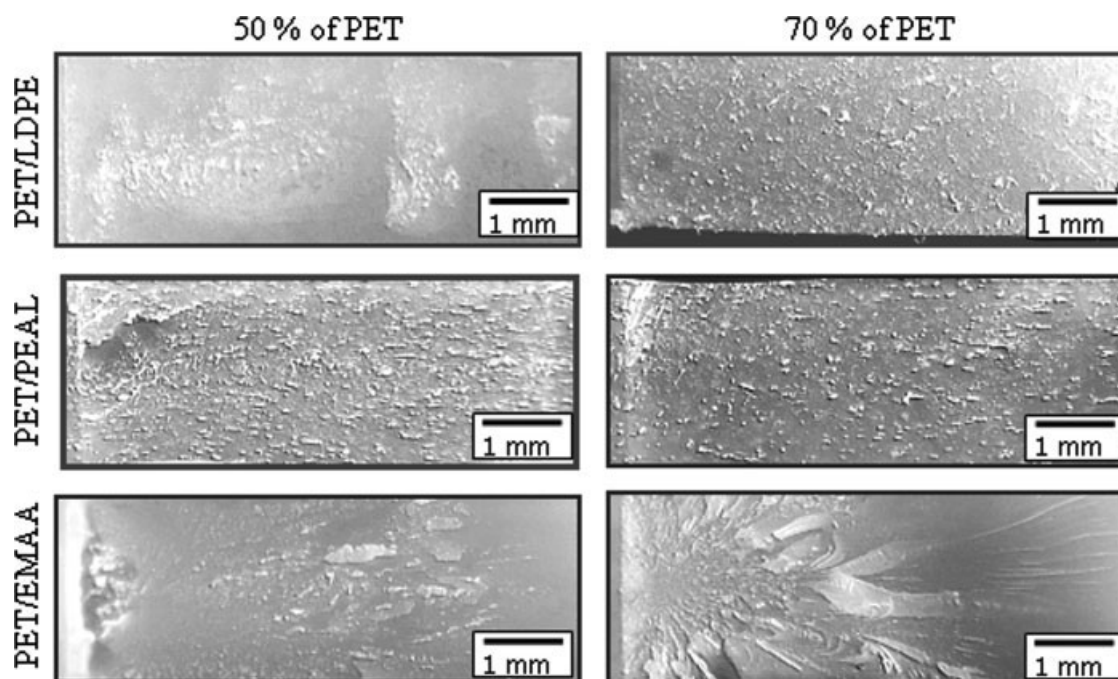
The evaluation of the potentiality to use the composite PEAL, recycled from aseptic packaging, for the obtainment of self-compatible and self-reinforced blends with thermoplastics was investigated. It was observed that the EMAA ionomer present in the composite changes completely the morphology of the blend with PET when compared with the non-compatible PET/LDPE blend. The main morphological characteristics observed in the PEAL blends, which are evidences for compatibilization, are the strong interfacial adhesion, the better dispersion of the dispersed phase and the good stability against



**Figure 11** Notched Izod impact strength of (-Δ-) PET/PEAL, (-■-) PET/LDPE, and (-●-) PET/EMAA blends. Dashed line corresponds to values predicted by additivity rule.



**Figure 12** Topography of notched Izod impact fracture of PET. Notch is on left.



**Figure 13** Topography of notched Izod impact fracture of PET/PEAL, PET/LDPE and PET/EMAA blends containing 50 and 70% of PET. Notch is on left.

coalescence in postprocessing. Such morphology features, comparable to those of the ionomer/PET blends, result from the strong interactions between the acrylate groups of EMAA and the ester groups of PET.

In respect to the mechanical properties, blends PET/PEAL present elongation at break and impact resistance similar to the PET/LDPE blends and Young's modulus values superior to PET/LDPE and PET/EMAA blends. These results reflect the predominant influence of aluminium particles in the mechanical properties over the polymeric phase morphology. In this way, the potential applications for PEAL blends must require properties that can be improved by the presence of aluminium, as for instance, modulus and thermal and electrical conductivity.

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