

# Surface Analysis of Aminated Polypropylene Films as an Adhesion Promoter to Polycarbonate Film

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**ABSTRACT:** The surface composition of aminated polypropylene films (PP-g-NH<sub>2</sub>) and their adhesion to polycarbonate (PC) film were evaluated. The detection of amine groups on PP-g-NH<sub>2</sub> surfaces was obtained by X-ray photoelectron spectroscopy (XPS). Contact angle measurements showed a decreased in the polarity on PP-g-NH<sub>2</sub> surface. The adhesion between laminated films of PP-g-NH<sub>2</sub> and PC was evaluated by T-peel test and optical microscopy. PC deposited on the PP-g-NH<sub>2</sub> surfaces was confirmed by FTIR-ATR and SEM analysis of delaminated

films, which is an indicative of an interaction between reactive sites of each polymer. The adhesion performance between PC and PP was improved by using amine modified polypropylene (PP-g-NH<sub>2</sub>). © 2010 Wiley Periodicals, Inc. *J Appl Polym Sci* 119: 336–342, 2011

**Key words:** adhesion; surface modification; aliphatic diamine; polycarbonate; polypropylene-grafted primary amine

## INTRODUCTION

Chemical modification of bulk polymer is one of the many methods used to physically modify polymer surface. Suitable techniques to chemically modify the polymer surface include flame treatment, corona discharge treatment, plasma modification, and surface graft polymerization. However, these techniques prohibit a design of chemically well-defined surfaces.<sup>1–5</sup> Modification of polyolefins (POs) by grafting of maleic anhydride, acrylic acid, or methacrylic acid monomers has been used to obtain reactive polymers with acidic functionality.<sup>6–8</sup> These reactive sites have been used as compatibilizing agents between polyolefins and polar polymers such as polyamides<sup>9,10</sup> and polyesters.<sup>11,12</sup> However, there is a growing interest in preparing polymers containing amine groups, specifically in polyolefins. Primary amines are very attractive groups to be grafted to

PP, but monomers containing primary amines are rarely used to make functionalized PP with primary amine (PP-g-NH<sub>2</sub>).<sup>13</sup> An alternative method is reacting nucleophilic bifunctional compounds with carboxylic acid or maleic anhydride groups grafted into polymer.<sup>14–20</sup> The reaction between these groups produces amide or imide bonds, leaving a polymer with pendant amine group.<sup>21</sup> Few technical studies have reported the use of PP-g-NH<sub>2</sub> as adhesion promoter agent to polymer films. Weinkauff<sup>17</sup> developed a PP-g-NH<sub>2</sub> that was used as tie-layer to polyketone film. The adhesion between layers was performed by primary amine of aminated PP and functional groups of polyketone film to form a pyrrole bonding. Lu and Macosko<sup>20</sup> showed that PP-g-NH<sub>2</sub> is an excellent adhesion promoter to polyurethane (PU) layers. They demonstrated that adhesion between PP and PU improved when PP was functionalized with secondary amine.<sup>20</sup>

Another interesting application of PP-g-NH<sub>2</sub> could be as an adhesion promoter to PC film. Polypropylene has become an important material with excellent balanced physical and mechanical properties. However, PP is limited in several applications due to its (i) low surface energy, (ii) difficulty in dyeing, (iii) lack of reactive sites, (iv) low impact strength and (v) high sensitivity to oxidation. Bisphenol-A

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polycarbonate (PC) is widely used in many different kinds of applications because of its unique combination of properties. This engineering thermoplastic is basically amorphous and, therefore, transparent. It has an excellent balance of toughness, clarity and high heat deflection temperature. However, some disadvantages of PC in the packaging industry are: (i) poor barrier to oxygen, (ii) poor solvent resistance and (iii) high moisture absorption. Several properties of PC can be improved by blending it with polymers such as PP. On the contrary, PP and PC films do not adhere to one another, which decreased the performance of multilayered films.

In our previous study,<sup>21</sup> we demonstrated that adhesion between PC and polypropylene-grafted-maleic anhydride (PP-g-MA) can be improved by grafting primary amine groups to PP-g-MA. The primary amine groups were grafted into PP-g-MA for chemical reaction between aliphatic diamines and maleic anhydride grafted groups (MA). The reaction was carried out in internal mixer chamber using 1,4-diaminobutane, 1,6-diaminohexane, and 1,12-diaminododecano. Long chain extension and cross linking between PP-g-NH<sub>2</sub> molecules were observed, as a consequence of the ability of one diamine molecule to react with two maleic anhydride units. As a continuation, this study describes how the surface properties of PP-g-NH<sub>2</sub> changes with addition of diamine. Identification of chemical groups on PP-g-NH<sub>2</sub> surface and its interaction with polycarbonate reactive sites was explored. Influence of bonding time and temperature on improving adhesion between PC and PP-g-NH<sub>2</sub> films were also studied.

## EXPERIMENTAL

### Materials

The reactive blending between PP-g-MA and three diamines (1,4-diaminobutane, 1,6-diaminohexane, and 1,12-diaminododecane) has been previously reported.<sup>21</sup> The modification of PP-g-MA (with 0.2 wt % of MA) was carried out in an internal mixer at 180°C and 60 rpm for 6 min (Brabender Plasticorder PL2200), obtaining two types of PP-g-NH<sub>2</sub>-series. In each series, the diamines were added to PP-g-MA using different addition protocol. At series PP-g-NH<sub>2</sub>-X (where X is the number of carbon atoms of diamine), the diamine was added directly to the melted PP-g-MA and this procedure was referred as direct-procedure addition protocol. The second addition protocol, referred as the packet-procedure, consisted in adding the desired amount of diamine in a sealed polypropylene bag (capacity 2 mL), and then added to the mixer chamber when the polymer was melted. The identification code used of these samples was PP-g-NH<sub>2</sub>-Xp (where X is the number of

carbon atoms of diamine and p represents packet polypropylene bag). This procedure reduced the loss of diamine by temperature processing. Polycarbonate (PC) Lexan 144 was obtained from General Electric.

### Film preparation

Films of PP, PP-g-MA, PP-g-NH<sub>2</sub>-X, and PP-g-NH<sub>2</sub>-Xp (X = 4, 6 or 12) were prepared by compression molding at 180°C for 5 min. The thickness of all films was ~ 0.15 mm and film samples were cut into strips 10-mm wide. Also, cast films of these materials were obtained by dissolving 0.2 g PP-g-NH<sub>2</sub>-X and PP-g-NH<sub>2</sub>-Xp in 10 mL of xylene at 120°C, and casting them on a glass plate and evaporating the solvent in an air forced oven at 120°C for 30 min. PC film was obtained in a single-screw extruder Killion KTS-100 equipped with a flat die at 240°C.

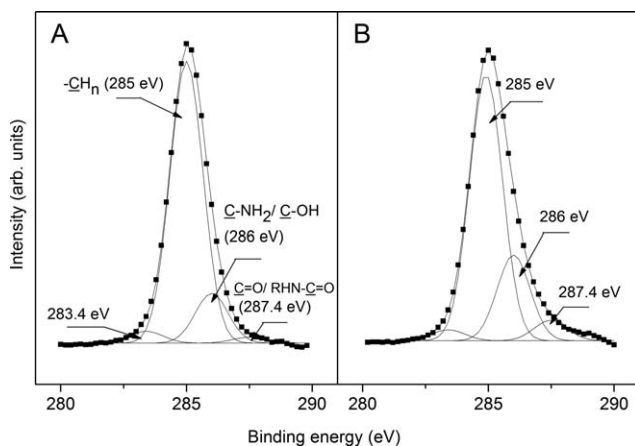
### Surface characterization

The X-ray photoelectron spectroscopy (XPS) analysis was carried out on a modified laser ablation system, Riber LDM-32, using a CAMECA Mac3 analyzer. The base pressure in the analysis chamber was in the low 10<sup>-10</sup> torr range, and about 10<sup>-9</sup> torr in the sample loading chamber. The X-ray AlK<sub>α</sub> line at 1486.6 eV was used for excitation. The binding energies were calibrated with reference to Cu 2p<sub>3/2</sub> at 932.67 eV and Ag 3d<sub>5/2</sub> at 368.26 eV. The resolution attained with this set-up is 0.8 eV measured on the C 1s signal of a graphite target. Spectra were collected by acquiring data every 0.2 eV and the energy resolution was 0.8 eV. The core-level spectra for C 1s and O 1s were obtained. Background subtraction was done using the Tougaard method.<sup>22</sup>

Contact angles of distilled water on films (prepared by compression molding) and cast films of PP-g-NH<sub>2</sub>, were measured. A water drop was placed on samples of each film using a syringe. Contact angles were measured using the sessile drop method on samples of 1 cm<sup>2</sup> in a Rame-Hart contact angle goniometer at room temperature. An average of at least five measurements was performed to determine contact angle.

### T-peel test

T-peel test was carried out to bilayer films of PP-g-MA, PP-g-NH<sub>2</sub>-X, and PP-g-NH<sub>2</sub>-Xp with PC film. Specimens were prepared in a hot press by laminating PC and each modified PP, applying a pressure of 1.9 MPa at different bonding temperatures (220 °C and 260°C) and bonding times (30, 60, and 90 s). A 10-mm-wide strip of PP homopolymer was placed



**Figure 1** C 1s core-level spectra of (A) PP-g-NH<sub>2</sub>-4 and (B) PP-g-NH<sub>2</sub>-12.

between the layers to serve as a delaminating initiator. Laminate samples were then cut into 1.0 × 10 cm<sup>2</sup> strips. Peel strengths were measured at room temperature, using a tensile tester with a crosshead speed 10 cm/min. The peel strength (N/cm) was calculated by dividing average force at initial peak load by strip width.

### Characterization of laminated films

The surfaces of the peeled films were coated with a thin layer of carbon and analyzed using a scanning electron microscope (SEM) attached to an EDX unit, JEOL 840, at 15 kV.

The laminated samples fractured at liquid N<sub>2</sub> temperature were observed by optical microscopy (Olympus BX60) under transmitted light.

Chemical composition of the delaminated films surfaces was investigated by attenuated total reflection Fourier transform (ATR-FTIR) infrared spectroscopy using a Nicolet 710 spectrometer with an ATR accessory element with 45 degree entrance face.

## RESULTS AND DISCUSSION

In our previous work,<sup>21</sup> PP-g-NH<sub>2</sub> was obtained in melt state using three low molecular weight diamines. A mixed grafting of amide-imide groups were detected in all PP-g-NH<sub>2</sub> using FTIR and a deconvolution analysis. Adhesion for a laminated film of PC and PP-g-NH<sub>2</sub> was improved due to amine groups into PP-g-NH<sub>2</sub>. However, surface properties from PP-g-NH<sub>2</sub> film and its influence on adhesion to PC film were not discuss in detail.

### Surface characterization of PP-g-NH<sub>2</sub> films

Figure 1 presents XPS spectrum and deconvolution of carbon C1s peak of PP-g-NH<sub>2</sub>-4 and PP-g-NH<sub>2</sub>-12.

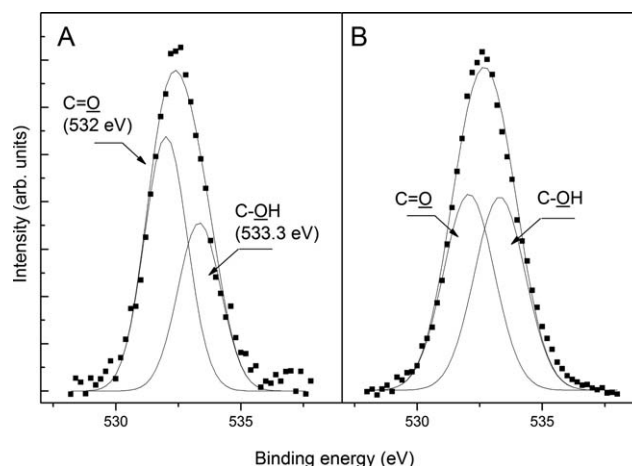
12. Three C1s environments were observed<sup>1,5,23,24</sup>: hydrocarbon (CH<sub>n</sub> ~ 285.0 eV), carbon singly bonded to oxygen (C=O ~ 286 eV), carbon doubly bonded to oxygen (C=O ~ 287.4 eV). The nonhomogeneous charge peak (283.5 eV) is an artificial peak that comes from the nonhomogeneous charging on the polymer surface caused due to the insulating nature of the material.<sup>25</sup> However, peaks at 286 eV and 283.5 eV could also be associated to carbon bonded to amine C-NH<sub>2</sub> and amide RHN-C=O.<sup>23</sup>

Figure 2 shows XPS deconvolution of O1s peak. Two O1s environments were observed<sup>1,5,23,24</sup>: oxygen doubly bonded to carbon (C=O ~ 532 eV) and oxygen singly bonded to carbon (C-O ~ 533.3 eV). In O1s spectrum of sample PP-g-NH<sub>2</sub>-4 [Fig. 2(A)], the peak at 532 eV is more intense than peak at 533.3 eV, however spectrum of PP-g-NH<sub>2</sub>-12 [Fig. 2(B)], shows an equal peak intensity to both carbon bonded oxygen. This result suggests that intensity of C1s peak at 286 eV is more related to an increase of functional groups C-NH<sub>2</sub> than C-OH, otherwise C-OH peak in O1s spectra would also increased.

### Contact angle measurements

Contact angle is sensitive to the chemical nature of polymer surfaces. The energy of a surface is directly related to its wettability, which is measured by the contact angle. In contact angle measurement, a liquid drop is placed onto a solid surface. Whether it sits on the surface in the form of a droplet or spreads out over the surface depends on the interfacial free energies of the two substances. It was reported that the surface chemistry and surface morphology affect the contact angle of the polymer surface.<sup>26</sup>

Water contact angle on film (prepared by compression molding) and cast film of all PP-g-NH<sub>2</sub> samples were measured. Table I shows contact



**Figure 2** O 1s core-level spectra of (A) PP-g-NH<sub>2</sub>-4 and (B) PP-g-NH<sub>2</sub>-12.



**TABLE I**  
Water Contact Angles (deg) for PP-g-NH<sub>2</sub> Films Prepared by Compression Molding of Samples Prepared Adding Diamine by Direct (PP-g-NH<sub>2</sub>-X) and Packet (PP-g-NH<sub>2</sub>-X<sub>p</sub>) Protocol

	$\theta$	
	PP-g-NH <sub>2</sub> -X	PP-g-NH <sub>2</sub> -X <sub>p</sub>
PP-g-MA	84.3 ± 3.8	
PP-g-NH <sub>2</sub> -4	88.5 ± 3.3	90.3 ± 0.6
PP-g-NH <sub>2</sub> -6	98 ± 5.7	91.3 ± 4.7
PP-g-NH <sub>2</sub> -12	92.3 ± 0.6	86 ± 4.2

angles of PP-g-NH<sub>2</sub>-X and PP-g-NH<sub>2</sub>-X<sub>p</sub> prepared by compression molding. Water contact angle of PP-g-MA (84°) was lower than PP (98°), as has been reported.<sup>3,27</sup> However, PP-g-NH<sub>2</sub>-X and PP-g-NH<sub>2</sub>-X<sub>p</sub> showed an increase in water contact angle, compared to neat PP-g-MA. Such results were probably due to the ionic nature of amine groups. Considering that amine groups have a lower polarity than MA groups,<sup>28</sup> the water contact angle evaluated on PP-g-NH<sub>2</sub> film surface would be larger than for PP-g-MA film surface. Similar results had been reported on PP films modified by nitrogen and acrylic acid plasma treatment.<sup>29</sup>

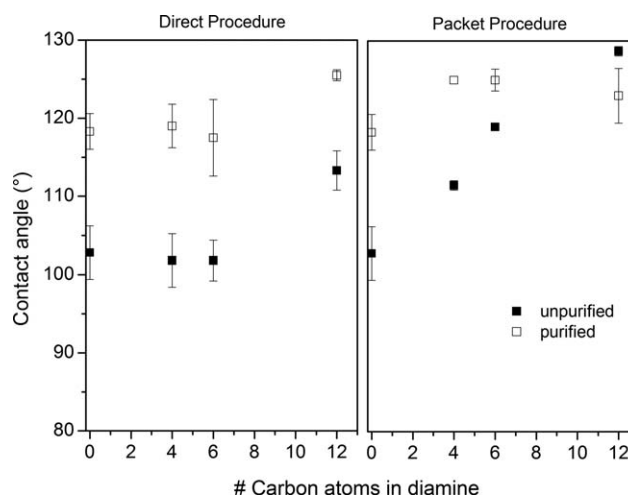
Water contact angle of cast films of PP-g-NH<sub>2</sub>-X and PP-g-NH<sub>2</sub>-X<sub>p</sub> were evaluated to determinate the effect of unreacted diamine on surface polarity. For this, an amount of each PP-g-NH<sub>2</sub> was dissolved in boiling xylene and then precipitated in acetone at room temperature three times to remove residual MA and unreacted diamine. The purified PP-g-NH<sub>2</sub> was dried under vacuum at 80°C during 6 h. The same procedure was used to obtained films from purified and unpurified PP-g-MA. The results are shown in Figure 3. The measured water contact angle for unpurified PP-g-MA cast films was 102° ± 3° and increased to 118° ± 2° for purified material. Films of unpurified PP-g-NH<sub>2</sub>-4 and PP-g-NH<sub>2</sub>-6 showed a contact angle around 102°, similar to unpurified PP-g-MA cast film. After the purification process, these films showed a contact angle higher than their similar unpurified cast films (Fig. 3, direct procedure), as was also observed for purified PP-g-MA cast films. In contrast, water contact angle for unpurified PP-g-NH<sub>2</sub>-12 cast films was of 113° ± 2°, increased to 125° ± 1° for the purified material. For the films PP-g-NH<sub>2</sub>-X<sub>p</sub>, values for the water contact angle showed the same behavior than unpurified and purified PP-g-NH<sub>2</sub> samples; however the contact angle was higher for these surfaces. In a previous work,<sup>21</sup> we showed that the amine contents for unpurified PP-g-NH<sub>2</sub>-X were 13 ± 0.3 µequiv/g, 26 ± 0.6 µequiv/g and 70 ± 0.6 µequiv/g when X = 4, 6, and 12 carbon atoms, respectively. After purifica-

tion process, the amine content was 18 ± 2 µequiv/g in all samples. For unpurified PP-g-NH<sub>2</sub>-X<sub>p</sub>, the amine content was higher due to the packet protocol used to incorporate diamine to the melt PP-g-MA (37 ± 0.7 µequiv/g, 42 ± 4.4 µequiv/g and 62 ± 4.9 µequiv/g for X<sub>p</sub> = 4, 6, and 12 carbon atoms, respectively). After purification process, PP-g-NH<sub>2</sub>-X<sub>p</sub> showed an amine content similar to PP-g-NH<sub>2</sub>-X, around 18 ± 2 µequiv/g, except for PP-g-NH<sub>2</sub>-12<sub>p</sub> (amine content = 26 ± 0.1 µequiv/g).<sup>21</sup> Apparently, if the bulk of PP-g-NH<sub>2</sub>-X and -X<sub>p</sub> had almost the same amine content after purification process, only 1,12-diaminododecane grafted groups promoted a change more significant on PP surface, as was shown by the contact angle values.

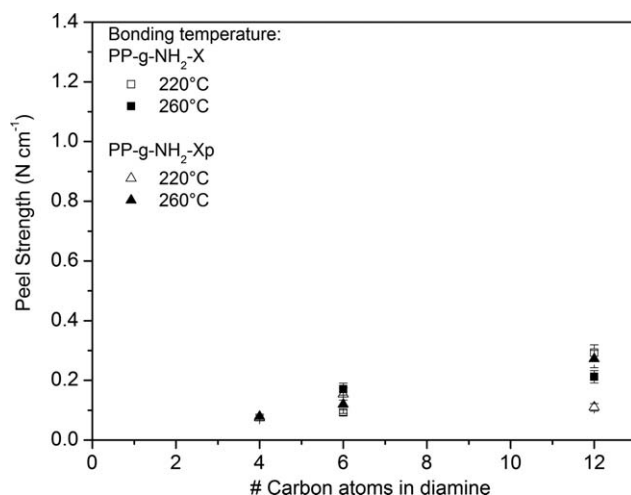
Relative differences of contact angle values between molded films and casting films were observed (PP-g-MA cast film = 84° and PP-g-MA molded film = 118°). It is possible that a rough surface on casting films promoted changes on water contact measurement, as has been reported on superhydrophobic surface of PP,<sup>30</sup> however this will be confirm in a future work. Finally, the significant changes in the polarity of PP-g-NH<sub>2</sub>-X and PP-g-NH<sub>2</sub>-X<sub>p</sub>, presented in Table I and Figure 3, indicated the incorporation of new functional groups, which would enhance the adhesion performance between PC and PP.

### Adhesion to polycarbonate film

The peel test results showed the availability of free-amine grafted groups of PP-g-NH<sub>2</sub> to react with reactive sites of PC during the lamination process. The bonding temperature and contact time between PP-g-NH<sub>2</sub> and PC were the main variables studied in the adhesion experiments. As a control sample, PP-

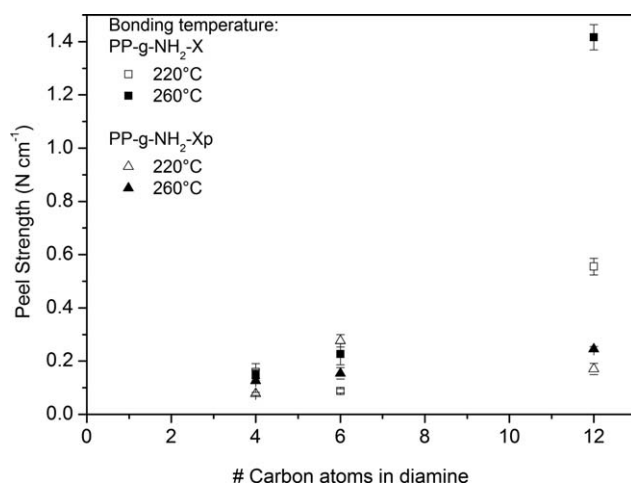


**Figure 3** Water contact angles on PP-g-NH<sub>2</sub>-X and PP-g-NH<sub>2</sub>-X<sub>p</sub> prepared by solvent casting for unpurified and purified samples.



**Figure 4** Peel strength of PC/PP-g-NH<sub>2</sub>, bonded during 30 s, as a function of number carbon atoms in diamine.

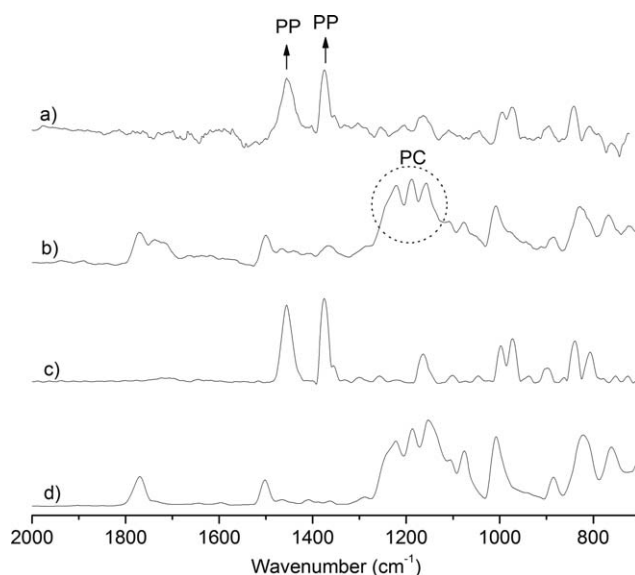
g-MA/PC film bonded at 220°C and 260°C during 30 and 60 s were evaluated. Figure 4, shows the results of peel strength for all PP-g-NH<sub>2</sub> /PC laminated for 30 s. The peel strength for PP-g-NH<sub>2</sub>-4 /PC and PP-g-NH<sub>2</sub>-4p /PC films remained essentially unchanged, even though the bonding temperature was increased from 220°C to 260°C. In the case of PP-g-NH<sub>2</sub>-6 /PC and PP-g-NH<sub>2</sub>-6p /PC films, a slightly increased adhesion was observed. In contrast PP-g-NH<sub>2</sub>-12 /PC and PP-g-NH<sub>2</sub>-12p /PC showed higher values of peel strength. This change in adhesion in laminated films could be mainly due to two reasons: bonding temperature and the molecular weight. If the bonding temperature increases, polymer chains have a better mobility and reactive sites can generate a chemical bonding between polymers.<sup>9</sup> Such interaction is also related to molecular weight of polymer: the molecular weight increases, the mobility of polymer is reduced. In our study, the



**Figure 5** Peel strength of PC/PP-g-NH<sub>2</sub>, bonded during 60 s, as a function of number carbon atoms in diamine.

weight average molecular weight of both polymers ( $M_w$ ) of PP-g-NH<sub>2</sub>-4 (227,000 g/mol) and PP-g-NH<sub>2</sub>-6 (180,000 g/mol), was higher than PP-g-NH<sub>2</sub>-12 (161,000 g/mol), which decreased the chain mobility of the former during lamination process.<sup>21</sup> For this reason, PP-g-NH<sub>2</sub>-12 and PP-g-NH<sub>2</sub>-12p showed better adhesion than the rest of PP-g-NH<sub>2</sub>.

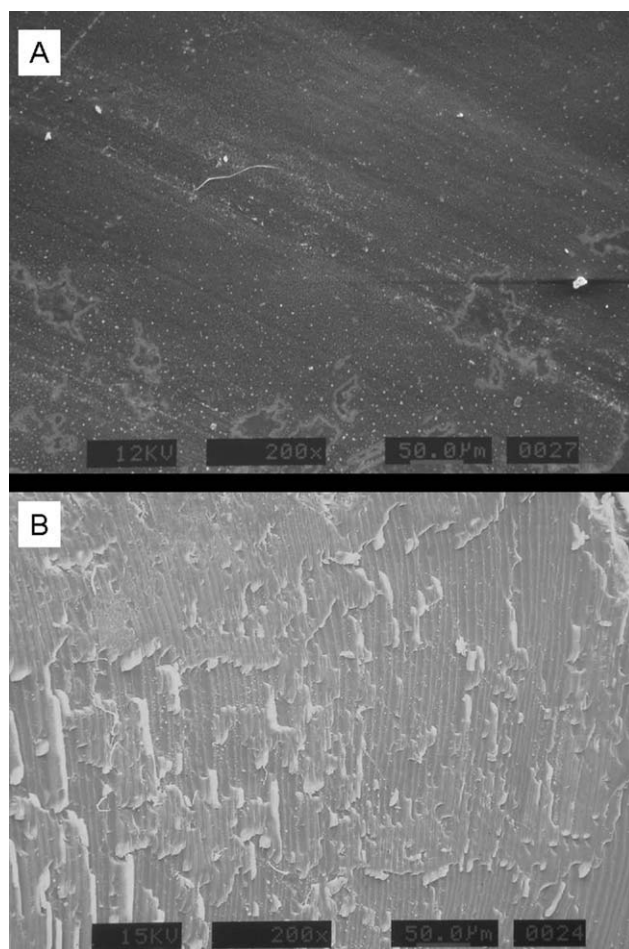
The peel strength of samples bonded for 60 s (Fig. 5) exhibit a slight adhesion improvement compared to samples bonded for 30 s. Apparently, PP-g-NH<sub>2</sub>-12 film has a better adhesion to PC at 60 s of bonding time, however there was a remarkable decreases in adhesion when was used PP-g-NH<sub>2</sub>-12p. If the unreacted diamine migrate to the surface during the lamination process and react with reactive sites available on the PC, would significantly reduce the chemical reaction between free-amine groups grafted into PP-g-NH<sub>2</sub> and PC. This behavior was more evident in samples laminated at 260°C, in which adhesion values did not improve as expected. As reported previously,<sup>21</sup> the control laminated film of PP-g-MA/PC did not show adhesion between layers, which confirms that amine groups grafted to PP are good compatibilizers to PC. It has been reported that in PC blends with polymers containing primary amine groups, an aminolysis is the main reaction mechanism between carbonate and primary amine groups.<sup>31,32</sup> Similar kind of reaction could be expected in the experiments conducted in this study, however it was difficult to confirm due to low levels of grafting.



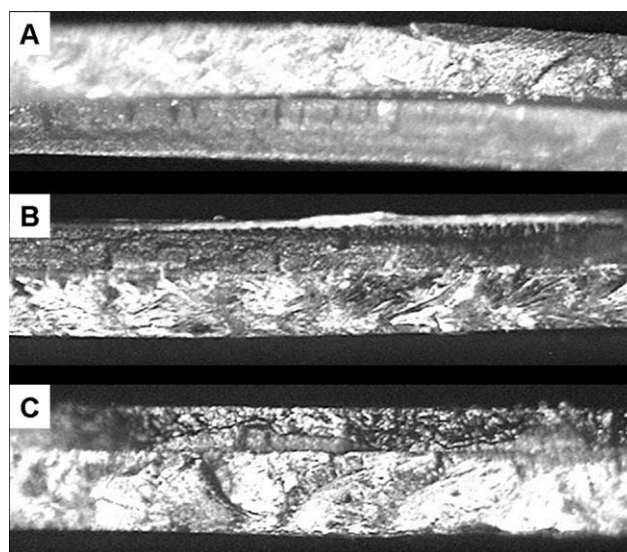
**Figure 6** FTIR-ATR spectra of polypropylene side from delaminated surfaces of (a) PP-g-NH<sub>2</sub>-4/PC bonded at 220°C during 30 s; (b) PP-g-NH<sub>2</sub>-12/PC bonded at 260°C during 60 s; (c) PP-g-MA/PC bonded at 260°C during 60 s and (d) PC film surface.

### Characterization of delaminated film surface

Delaminated film surface of all PC/PP-g-NH<sub>2</sub> were evaluated by FTIR-ATR. Figure 6 shows ATR spectra of the polypropylene surface after delamination: films with the lowest adhesion (PP-g-NH<sub>2</sub>-4/PC bonded at 220°C during 30 s), films with the highest value of adhesion (PP-g-NH<sub>2</sub>-12/PC bonded at 260°C during 60 s) and control sample (PP-g-MA/PC bonded at 260°C during 60 s). The characteristic FTIR-ATR peaks in each spectrum are<sup>12</sup>: 1770 cm<sup>-1</sup> for C=O carbonyl stretching vibration of PC, 1250 cm<sup>-1</sup> for O—C—O carbonate stretching vibration, 1460 cm<sup>-1</sup> and 1380 cm<sup>-1</sup> for deformation of methyl (—CH<sub>3</sub>) and methylene (—CH<sub>2</sub>—) of PP. The ATR spectrum for PP-g-NH<sub>2</sub>-4/PC (Fig. 6 curve a), shows peaks relative to —CH<sub>3</sub> and —CH<sub>2</sub>— for the delaminated PP-g-NH<sub>2</sub>-4 surface which confirm our results about poor adhesion to PC film. In contrast, the ATR spectrum of the delaminated surface from PP-g-NH<sub>2</sub>-12/PC (Fig. 6, curve b) shows O—C—O carbonate stretching vibration and became more intense than —CH<sub>3</sub> and —CH<sub>2</sub>— of PP-g-NH<sub>2</sub>-12. This result suggests that PC was deposited on the PP surface.



**Figure 7** SEM images for the peeled surface morphology of: (A) PP-g-MA and (B) PP-g-NH<sub>2</sub>-12.



**Figure 8** Cross-sectional image of fractured laminated samples bonded at 260°C during 60 s. (A) PC/PP-g-NH<sub>2</sub>-4; (B) PC/PP-g-NH<sub>2</sub>-6; (C) PC/PP-g-NH<sub>2</sub>-12.

The spectrum of delaminated control film PP-g-MA/PC (Fig. 6, curve c) only shows —CH<sub>3</sub> and —CH<sub>2</sub>— of PP, confirming that there is not carbonate groups on the delaminated PP-g-MA surface, otherwise peaks O—C—O would have been showing in ATR spectrum.

The delaminated SEM surface images of PP-g-MA and PP-g-NH<sub>2</sub>-12 side (bonded to PC at 260°C during 60 s) are shown in Figure 7. The PP-g-MA surface, [Fig. 7(A)] shows a uniform texture with visible features, which correspond to surface texture of mold during the compression molding operation. As nonadhesion between PC and PP-g-MA was possible under our lamination processing conditions, no residue of PC was evident in the surface image of PP-g-MA. In contrast, the delaminated surface of PP-g-NH<sub>2</sub>-12 shows a rough texture which is presumably PC deposited on surface during lamination process.

Cross-sectional morphologies for each laminated film of PC with PP-g-NH<sub>2</sub>-4, PP-g-NH<sub>2</sub>-6 and PP-g-NH<sub>2</sub>-12, are compared in Figure 8. A poor adhesion of PP-g-NH<sub>2</sub>-4 /PC can be seen in Figure 8(A), this is due to a separation of specific areas after fracturing at liquid N<sub>2</sub> temperature. In contrast, images corresponding to PP-g-NH<sub>2</sub>-6/PC and PP-g-NH<sub>2</sub>-12/PC, showed a uniform interface, indicating a much better adhesion between layers.

### CONCLUSIONS

Surface properties of PP-g-NH<sub>2</sub> films confirmed that unreacted diamine could migrate to the surface, decreasing polarity of the film. Adhesion between PP-g-NH<sub>2</sub> and PC films was improved when bonding temperature was increased. The decreasing trend



of peel strength was evident as unreacted diamine saturated the interface between films reducing reactive sites from each one.

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