Optimization of PE/Binder/PA Extrusion Blow-Molded Films. II. Adhesion Properties Improvement Using Binder/EVA Blends

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ABSTRACT: The second part of this series shows that the incorporation of EVA in the tie layer of extrusion blow-molded three-layer (PE/binder/PA) improves the adhesion properties, while reducing the film cost by 6% and preserving the performances previously (Part I) obtained by addition of EVA in the PE layer only, such as optimal improved seal ability, optical properties, and satisfactory mechanical properties. The improvement of adhesion between the binder and the PA layers could be related to a modification of the type and the density of the intermolecular interactions between binder, PA, and EVA. © 2006 Wiley Periodicals, Inc. J Appl Polym Sci 101: 118–127, 2006

Key words: adhesion; blends; blown film; coextrusion; heat sealing; mechanical properties; optical properties; peeling; tearing

INTRODUCTION

Developing multilayer PE/PA films is industrially interesting but also challenging as it consists in combining the specific properties of each polymer. The major concern is actually the combination of use properties, such as food preservation, recyclability, or impermeability to water steam and oxygen, with high mechanical, optical, adhesion, and sealing properties, at the best performance/cost ratio.

Except for some particular cases, of often limited interest, the association of polymers with very different properties generally requires the use of adhesive materials. Their function is to assure the binding between naturally incompatible polymers. Depending on the case, different adhesion mechanisms[1–9] may be involved.

Most of the time, more than one mechanism contributes to adhesion (mechanical anchoring, electrical, wetting, diffusion or interdiffusion, chemical bonds, low cohesion layer models).[8,9] Good adhesion is reached when the compatible polymer molecules diffuse through the interface to create entanglements.[10] However, a mutual interdiffusion of incompatible polymers, such as polyethylene and polyamide, is limited and the binding forces are generally weak.

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Therefore, manufacturing of PE/PA film structures requires the use of a reactive compatibilizer, or functionalized polymer. Either a functionalized PE or a functionalized PA can be chosen. In the first case, there would be interactions with PA; in the other, interactions with PE. Considering PE and PA chemical structures, functionalized PE will be preferred. It is indeed relatively difficult to operate a chemical reaction on a PE without bringing other reactive elements. On the other hand, amine functions remain in the PA and react easily with acids or anhydrides. This property is exploited to choose a grafted copolymer acting as a reactive compatibilizer agent.[2–4,11,12] The copolymer is a polyolefin chain grafted with functional groups (butyl acrylate or maleic anhydride), which reacts with the PA amine groups.[13,14] This justifies the choice of a grafted maleic anhydride PE (PEgMAH) as binder for the coextrusion blow molding of a PE/binder/PA multilayer film. In this case, the PE part of the copolymer is supposed to diffuse in the PE phase, whereas the maleic anhydride functions react with the NH_2 end groups of the PA chains (Fig. 1).

Part I of this series[15,16] showed that the addition of EVA in the PE layer resulted in the improvement of the seal ability and the optical properties of PE/PEgMAH/PA films while preserving satisfactory mechanical performances. In addition, the price of EVA is lower than that of PEgMAH and presents a compatibilizing character for the pair PE/PA.[15–20] Consequently, the incorporation of EVA both in PE and tie layers should theoretically improve the adhesion
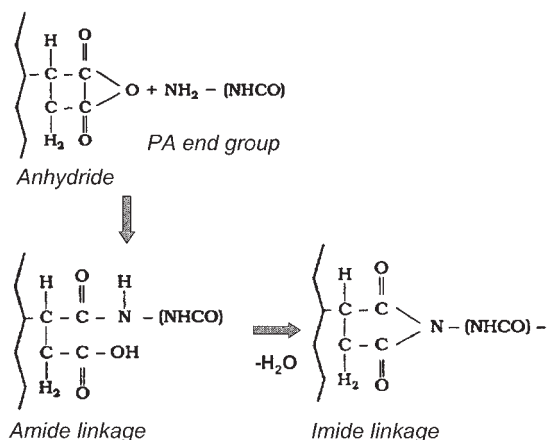
Figure 1 Interaction between PEgMAH and polyamide.[11]

properties of PE/PA films and reduce the total cost of such sealed and transparent packages.

However, so far no research program has been conducted on the theme of joint modification of the PE and PEgMAH layers of three-layer PE/PEgMAH/PA films through EVA addition in these two layers. Thus, the objective of the present study is to quantify the incidence of variable EVA contents in both the PE and the binder layers on the peel strength of a PE/PEgMAH/PA structure. We will focus in particular on the identification of the phenomena and the mechanisms involved in these modifications.

EXPERIMENTAL

Materials and manufactured structures

Table I shows the main characteristics of polymers used in this study. PE/binder/PA (internal/central/external layer) film structures with EVA contents varying from 0 to 50% in weight in both the PE and binder layers have been manufactured (Table II). The PE and PA layer thicknesses are set to 50 and 30 μm, respectively. The optimal tie layer thickness has been determined by varying this thickness (pure PEgMAH) from 5 to 20 μm.

Manufacturing equipment and conditions

The 470 mm wide films (blow up ratio 1.5) are manufactured on a three-layer coextrusion line made of three extruders (see main characteristics in Table III), a take up equipment (speed 11.5 m/min), and a radiant channels three-layer die (diameter 200 mm, gap 0.8 mm), set at 235°C. The extrusion temperatures of the various layers (internal, central, external) are maintained constant for all experiments (Table I).

Equipment and processing conditions for heat sealing

The sealing between two PE internal layers is carried out perpendicular to the extrusion direction by heating jaws using a THIMONIER thermal sealing machine (type SI5–63 CA). The experimental seal strength characterization procedures as well as the failure modes (separation, peeling and tearing) have been previously described in detail.[15]

Characterization equipment and conditions

The experimental details of the procedures for the measurement of the optical and mechanical properties, and the seal and slip ability have been described in the first part of this article series.[15]

Mechanical behavior in tension

The mechanical tests have been carried out following the ISO 527.3 standard using a ZWICK tensile machine (dimensions of the samples 150 mm × 15 mm). The same equipment is also used for the peel and tear performance evaluation as well as for the slip and seal ability tests.

Peel strength

The characterization of the adhesion between the various layers is carried out by peel tests at 180° and 100 mm/min. The peeling of samples (150 mm × 15 mm)

TABLE I
Materials Characteristics

Material	Supplier	Reference	Melt flow index (g/10 min)	Density (g/cm^3)	Melting temp. (°C)
LDPE	ENICHEM	R FF20	0.65	0.92	112
PA 6/6,6	BASF	U C35	8.01[a]	1.13	195
PEgMAH	DP	B 4288	4.7	0.923	104
EVA 9% vinyl acetate	ENICHEM	GFF35	0.8	0.931	102

[a] Measured at 215°C, 2.16kg.

TABLE II
Composition of the Studied PE/PEgMAH/PA Films

	wt% EVA content	Internal layer		Central layer		External layer
		wt % LDPE in the support	wt % EVA in the support	wt % PEgMAH in the tie layer	wt % EVA in the tie layer	wt % PA in the external layer
Reference	0	100	0	100	0	100
EVA in PE and binder layers	10	90	10	90	10	100
	20	80	20	80	20	100
	30	70	30	70	30	100
	40	60	40	60	40	100
	50	50	50	50	50	100

is carried out following the procedure previously described[15] (Fig. 2).

Tear strength

The tear strength is measured following the NF T 54–108 standard, in the extrusion and transverse directions (dimensions of the samples 30 mm × 50 mm, crosshead speed 100 mm/min).

Slip ability

The slip ability is estimated from the static (Ks) and dynamic (Kd) friction coefficients (internal face/internal face, i.e., PE/PE) measured following the NF T 54–112 standard.

Optical properties

The gloss of the films is measured by means of a Gardner Micro Gloss 45 reflectometer (ASTM D 2457), haze and clearness by means of a Haze Gard Plus equipment (ASTM D 1003).

IR analysis

The analysis is carried out by Fourier transform infrared spectroscopy in reflexion ATR mode, using a Perkin–Elmer 1720X equipment, and by infrared microscopy in specular transmission mode, using a Spectratech Avantage equipment coupled to a Nicolet 460 ESP Protégé FTIR spectrometer.

To measure the maleic anhydride and vinyl acetate content on the surface and in the thickness, transmittances at 1792 cm^{-1}, 1735 cm^{-1}, and 1465 cm^{-1} were, respectively, taken as references for C$=$O groups of the PEgMAH, C$=$0 groups of the EVA, and CH$_2$ groups of the PE chains.

Influence of the tie layer thickness

There is a relationship between the tie layer thickness and the adhesion performance.[4,7,8] Therefore, the first goal is the reduction in the cost and the thickness of the film while maintaining satisfactory adhesion properties. Taking into account the manufacturing constraints, the tie layer thickness has to be higher than or equal to 5 μm.

Influence on film cost

Dividing by 4 the tie layer thickness obviously decreases the material cost (by 75% for that layer and 25% for the whole film) (Fig. 3). However, it is necessary to choose the lowest possible thickness preserving the other properties, mainly the adhesion performance.

Influence on adhesion properties

In the range of the tested structures, the adhesion performance strongly increases with the tie layer thickness (Fig. 4) for the low thicknesses. Then it stabilizes beyond a thickness of 8 μm.

TABLE III
Main Characteristics of the Coextrusion Line and Manufacturing Conditions

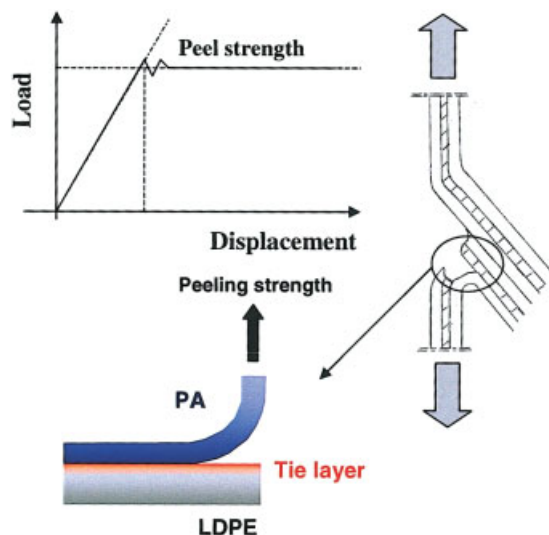| Layer	Extruder Reference	Screw		Extrusion conditions		
		Diameter D (mm)	Length	Thickness (μm)	Flow rate (kg/h)	Extrusion temperature (°C)
Internal	DOLCI[45]	45	22 D	50	30	210
Central	REIFENHAUSER[35]	35	31.4 D	5 to 20	6	235
External	DOLCI[40]	40	32 D	30	22	240

Figure 2 Measurement of the adhesion strength.[15,16] [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

Various causes can explain the results observed for the lowest thicknesses (<8μm).[4,7] First, the tie layer stiffness is not sufficient to absorb the strain difference of the two adjacent layers. Moreover, the tie layer thickness becomes too small to fill the surface microdefects existing at the interface. Finally, the total quantity of polar groups in the binder, likely to be directed towards the interface, is too low because of the limited amount of end groups.

The plateau observed for thickness higher than 8 μm corresponds to a saturation of the substrate (PA) polar groups by their corresponding groups coming from the binder (maleic anhydride).

In conclusion, there is a minimal tie layer thickness that guarantees a maximum peel strength. The further investigations have thus been carried out with a cen-
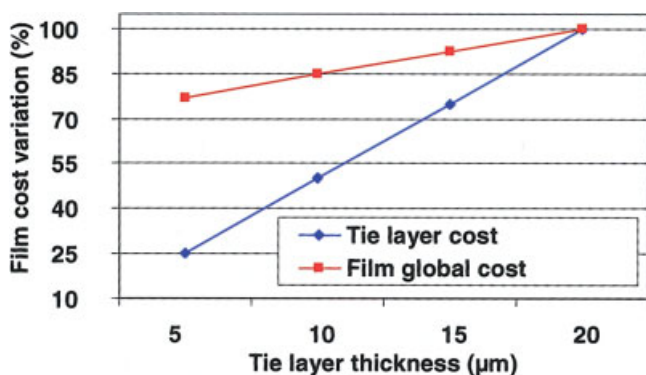

Figure 3 Effect of the tie layer thickness on the complex film (PE/PEgMAH/PA) cost. [Color figure can be viewed in the online issue, which is available at www.interscience. wiley.com.]
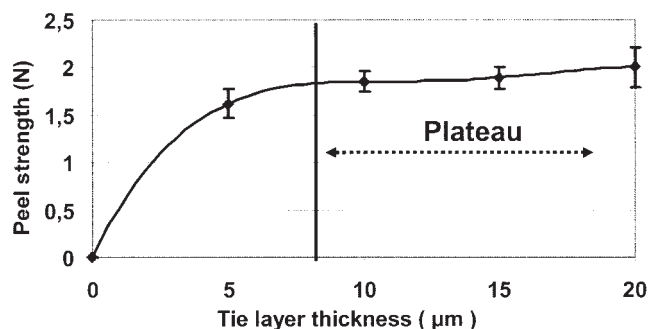

Figure 4 Influence of the tie layer thickness on the peel strength of the complex film (PE/PEgMAH/PA).

tral layer thickness set at 10 μm (corresponding to a 15% film cost reduction).

EVA Incorporation in the tie layer

A previous study[15,16] showed that EVA incorporation in the PE layer appreciably improves the film seal ability, but leads to an increase in the film cost that has to be taken into account in the final assessment. This expense can be attenuated or suppressed by addition of EVA to the PEgMAH layer. However, such modification should not penalize the properties (especially adhesion) previously obtained by addition of EVA in the PE layer only.

Effect on film cost

Considering the price and the required amount of the different components, the addition of EVA in the tie layer appreciably reduces the film cost. The cheapest solution (Fig. 5) corresponds to the maximum EVA content in the PE and tie layers (with about 6% reduction in the film cost compared to the reference structure) in spite of an internal layer cost increase of almost 1%.
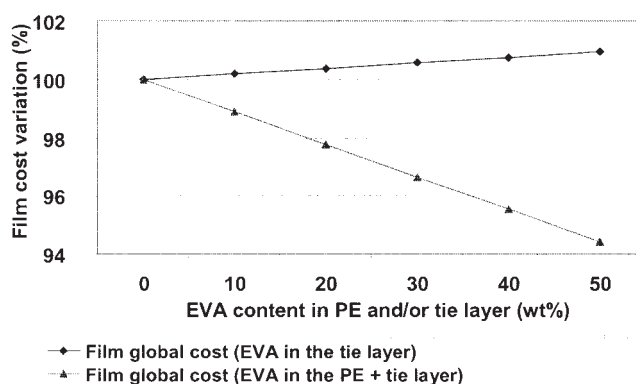

Figure 5 Effect of the EVA incorporation in the PE and tie layers on the whole complex film cost (PE/PEgMAH/PA).
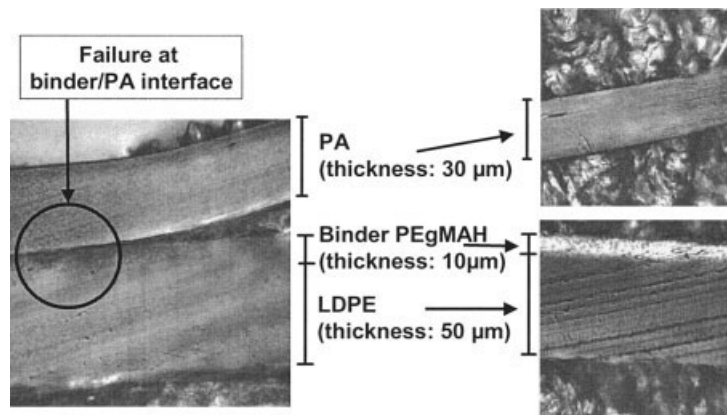
Figure 6 Failure mode of PE/PEgMAH/PA films after incorporation of EVA in the PE and tie layers.

Effect on adhesion properties

Considering the failure mode obtained on the initial structure (failure at the binder/PA interface), a modification of the tie layer composition may potentially enhance the adhesion properties of the multilayer structure.

In practice, whatever the EVA content is, EVA incorporation in the tie layer does not affect the failure mode, which always occurs at the binder/PA interface (Fig. 6). Nevertheless, the effect of EVA incorporation in the tie layer on the peel force is highly significant. This means that the peel strength can be multiplied by 4.5 by changing the EVA contents (Fig. 7 and Table IV).

Effect on optical properties

The haze, clearness, and gloss of films containing 0 to 50 wt % of EVA in the PE and tie layers are listed in Table IV and reported in Figure 8 according to the EVA content, in base 100 (value 100 corresponding to the internal and tie layers made of pure PE and PEg-MAH).
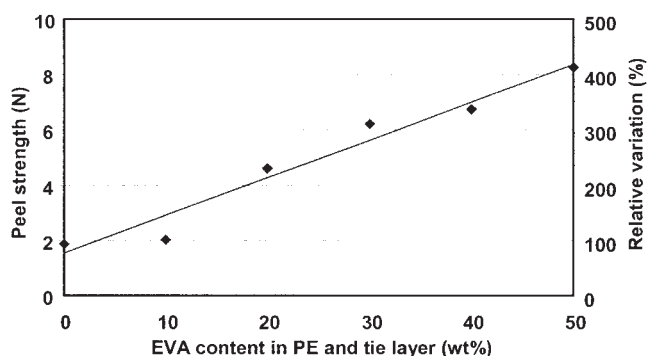
A significant improvement of these optical properties is noticed in proportions similar to those obtained when the PE layer only is modified by blending with EVA.[15] Thus, the modification of the central tie layer alone has no or very little influence on these properties. This is not surprising as the tie layer is very thin.

Effect on mechanical properties in tension and on tear strength

Table IV presents the mechanical characteristics and the tear strength of films according to the EVA content in the PE and tie layers. Considering data scattering, the differences between the various materials are not significant. Thus, the mechanical properties in tension (modulus, stress, and elongation at break) and the tear strength of the three-layer structure are not affected by these composition modifications of the coextruded films.

Knowing that the EVA incorporated in the PE layer only has no effect on the mechanical properties of the films,[15] we can deduce that, added solely in the tie layer, it does not affect these performances either.

Effect on seal ability

Films containing 0 to 50 wt % of EVA in the PE and tie layers were sealed, under the conditions described in a previous article.[15] Figure 9 presents, in base 100, the effect of the sealing temperature (from 130 to 190°C) on the seal strength for the various tested compositions (value 100 corresponds, for each film, to the maximum seal strength obtained over the considered sealing temperature range).

Whatever the amount of EVA incorporated in the PE and tie layers is, three sealing temperature zones leading to different failure modes can be distinguished: separation for sealing carried out at temperatures lower than 140°C, peeling between 140 and 160°C, and tearing above 160°C. This result is not

Figure 7 Influence of the EVA incorporation in the PE and tie layers on the peel strength of the complex film (PE/PEgMAH/PA).

TABLE IV
Influence of the EVA Incorporation in the PE and Tie Layers on the Tensile, Mechanical, and Optical Properties, on the Slip Ability, and on the Peel and Tear Strengths for Complex PE/PEgMAH/PA Structures

EVA content in PE and tie layers (wt %)	Friction coefficients		Optical properties			Tear strength (N)	
	Static	Dynamic	Haze	Clearness	Gloss	Long. dir.	Trans. dir.
0	0.92 ± 7.69%	0.72 ± 0.56%	14.2 ± 5.1%	84.6 ± 0.9%	62.1 ± 2.6%	19.79 ± 5.9%	25.57 ± 5.9%
10	0.82 ± 11.7%	0.82 ± 3.25%	11.6 ± 4.3%	89.3 ± 1%	66.0 ± 6.3%	21.12 ± 5.2%	25.81 ± 2.2%
20	1.32 ± 25%	0.98 ± 2.31%	10.4 ± 4.4%	91.1 ± 1.1%	71.2 ± 1%		
30	1.17 ± 1.7%	1.02 ± 0.68%	10.5 ± 4.5%	92.1 ± 3.2%	71.3 ± 1.1%	21.60 ± 5.5%	26.03 ± 3.6%
40	1.30 ± 32%	1.09 ± 9.48%	9.5 ± 4.5%	93.2 ± 1.7%	72.2 ± 1.9%		
50	2.13 ± 8.5%	1.29 ± 2.75%	9.6 ± 3.7%	91.9 ± 0.7%	75.5 ± 2.1%	20.08 ± 2.4%	24.85 ± 3.6%

EVA content in PE and tie layers (wt %)	Peeling force (N)	Young's modulus (MPa)		Tensile strength (MPa)		Tensile strain at break (%)	
		Long. dir.	Trans. dir.	Long. dir.	Trans. dir.	Long. dir.	Trans. dir.
0	1.87 ± 4.51%	239 ± 6.1%	270 ± 3.7%	35 ± 6.9%	25 ± 6.4%	330 ± 6.1%	320 ± 3.4%
10	2.02 ± 6.1%	243 ± 10.6%	271 ± 6.3%	35 ± 7.2%	35 ± 4.0%	349 ± 5.5%	384 ± 6.0%
20	4.6 ± 3.7%	262 ± 4.0%	275 ± 1.5%	30 ± 18.6%	23 ± 17%	306 ± 11.1%	302 ± 7.3%
30	6.23 ± 0.6%	259 ± 2.7%	229 ± 5.9%	27 ± 10.5%	33 ± 4.6%	291 ± 5.7%	370 ± 1.6%
40	6.73 ± 9.3%	234 ± 1.6%	241 ± 5.6%	36 ± 7.6%	32 ± 3.4%	347 ± 7.2%	368 ± 2.7%
50	8.27 ± 13%	230 ± 2.7%	248 ± 3.5%	35 ± 7.6%	30 ± 12%	349 ± 11.2%	358 ± 5.3%

surprising as the composition of the internal sealing layers is identical to that of the films studied previously.[15]

The seal strength passes by a maximum for sealing temperatures higher than 160°C, but tends to stabilize at its maximum value. In addition, as soon as the sealing temperature is higher than 160°C, the seal resistance becomes higher than 94% of the maximum accessible strength.

Consequently, whatever the composition of the structure is, the sealing temperature window is as broad as that observed when EVA is incorporated only in the PE layer[15] (about 30°C). Thus, the additional EVA incorporation in the tie layer does not degrade the better film seal ability obtained by its blending solely in the PE layer.

As a consequence, blending EVA in both the PE and binder layers still allows significantly reducing the

sealing time (40%), at equivalent seal quality and optimized pressure (3 bar), and appreciably increasing the seal strength (5%), at constant sealing time.

In these conditions, the optimal sealing temperature has been estimated at 170°C. This value is higher than the lowest temperature guaranteeing a maximal seal strength, but does not require a too constraining thermal regulation of the process.

Effect on slip ability

The static (K_s) and dynamic (K_d) friction coefficients of structures containing 0 to 50 wt % EVA in the PE and tie layers are presented in Table IV in absolute values and in Figure 10 in relative values.
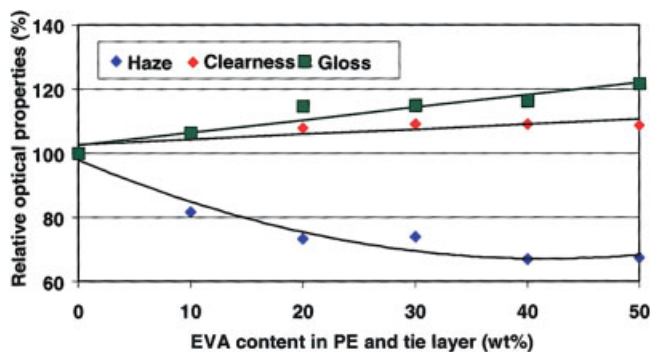

Figure 8 Effect of the EVA incorporation in the PE and tie layers on the optical properties of the complex film (PE/PEgMAH/PA). [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]
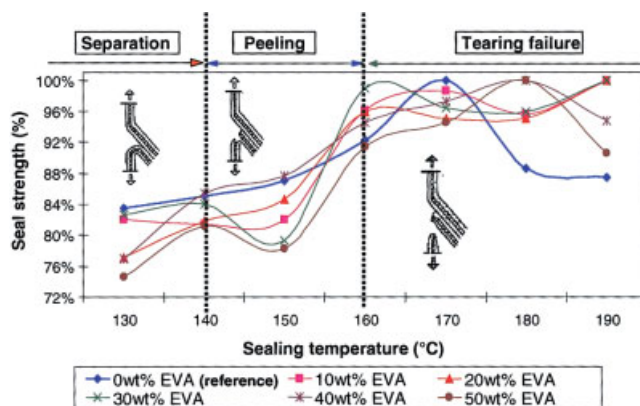

Figure 9 Influence of the sealing temperature and the EVA content in the PE and tie layers on the seal strength. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]
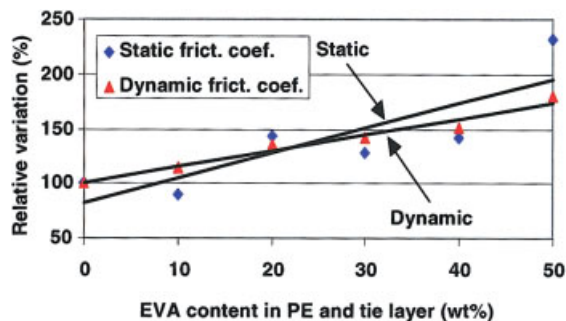
Figure 10 Influence of the EVA incorporation in the PE and tie layers on the static and dynamic friction coefficients (internal face/internal face). [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

As previously noted in the case of EVA/PE blending,[15] Ks and Kd linearly increase with the EVA content with the same orders of magnitude.[15] Considering the high data scattering, it is assumed that blending EVA in the tie layer does not have any additional effect on the film slip ability. Thus, the aforementioned penalizing effect of EVA (blocking during the blown film opening operation) is still present, and it requires further investigations to find a suitable solution (use of slipping agents, for example).

EVA incorporation in the PEgMAH tie layer significantly affects (in a positive way) the binder/PA interface strength (in a ratio of approximately 4.5) and the film cost only.

Considering its thickness, the tie layer (modified or not) takes very little part in the multilayer structure global properties. The EVA/PEgMAH blend used in the tie layer has no complementary effect on the optical and mechanical properties nor on the slip ability. These performances remain at the level imposed by the addition of EVA in the PE layer. In particular, the seal ability increases, in similar proportions.[15]

DISCUSSION

Adhesion essentially results from interatomic and intermolecular interactions occurring at the layer interfaces. Chemical bonds between PA- NH_2 end groups and PEgMAH maleic anhydride groups are created at the binder/PA interface (Figs. 11 and 12). This explains the high MAH reactive groups concentration observed by FTIR at the binder/PA interface (Fig. 13-1). Thus, at the PE/binder interface, the PE chains diffuse and cocrystallize (Figs. 12 and 13–2).

Insofar as blending EVA in the tie layer reduces the reactive end groups amount, it should also reduce the covalent bonds (highly energetical) number between PA NH_2 end groups and PEgMAH maleic anhydride groups (Figs. 11 and 12). As a result, the binder/PA adhesion performances should be reduced as well.

However, experience shows that the addition of EVA in the tie layer appreciably increases (by a factor of 4.5) the peel force. This unexpected trend can be related, on the one hand, to the migration of maleic anhydride groups to the binder/PA interface, and, on the other hand, to the creation of hydrogen bonds between EVA and PA (Fig. 14).

Complementary tests were carried out to evaluate the joint variations of MAH content on the tie layer surface and the total MAH contained in the tie layer after dilution by PE over a range from 0 to 100%. The choice of the dilution of the binder by PE is due to the fact that the PEgMAH and EVA characteristic peaks are superimposed and do not allow a direct analysis (Fig. 15).

The dilution of PEgMAH by LDPE up to approximately 50 wt % does not affect the potential chemical interactions between PA and the binder, as the MAH grafting ratio on the tie layer surface remains identical (Fig. 15). Considering the very close nature of PE and EVA, we can suppose that MAH migration takes place and compensates for the potential reduction in the number of covalent bonds between the PA and MAH groups.

However, this mechanism cannot explain by itself the evolutions of adhesion properties. The peel strength variations can then be related to the hydrogen bonds creation between the NH of PA and the vinyl acetate groups of EVA (Fig. 14). These bonds are not very energetical but they are much more numerous than the PA chain ends.

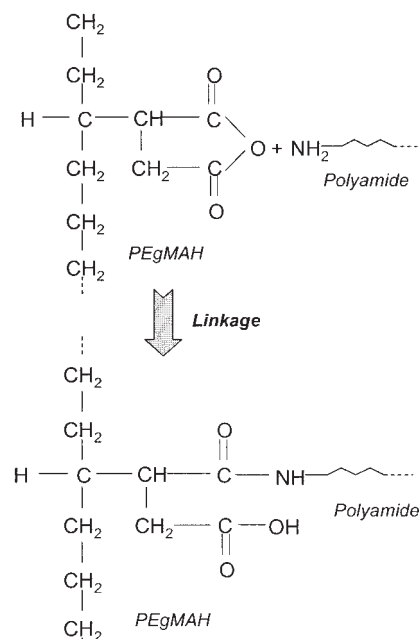Figure 16 presents the (PEgMAH+EVA/PA) peel force, the vinyl acetate content at the (PEgMAH+EVA/

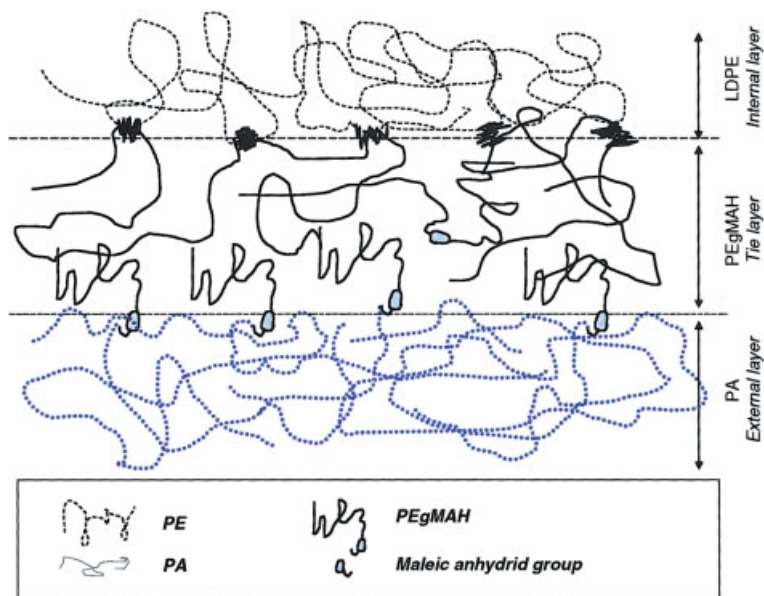**Figure 11** PEgMAH/PA interactions.

Figure 12 PEgMAH, PA, and PE layers interactions. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

PA) interface and the MAH content at this interface after the dilution of the tie layer with PE (Fig. 15) as a function of the EVA content in the tie layer.

For EVA incorporation ratio ranging from 0 to 50 wt %, it is possible to estimate that the second mechanism (hydrogen bonds) is dominating, ex-
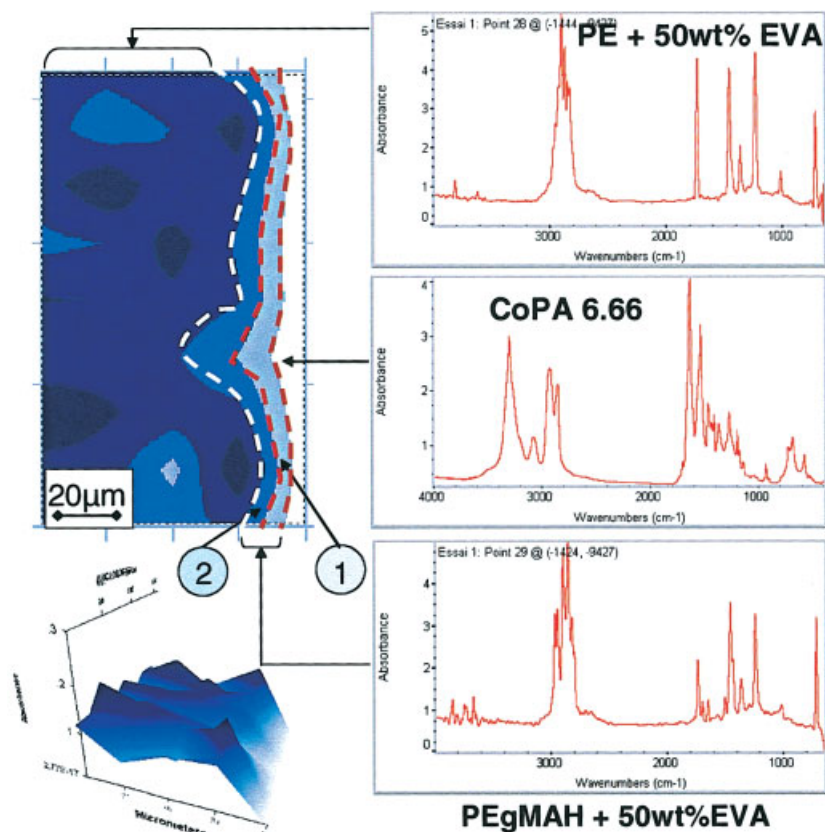

Figure 13 Through-the-thickness FT-IR image of the (PE+EVA)/(PEgMAH+EVA)/PA complex film. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]
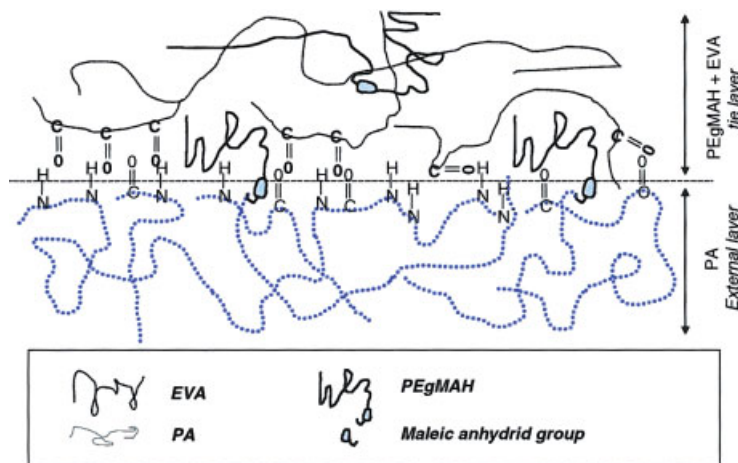
Figure 14 Bonds between (PEgMAH/EVA) and PA layers. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

plaining the increase in adhesion with increasing EVA content.

However, for higher EVA contents, the secondary bonds (between EVA and PA) cannot counteract the molecular slip between the two layers any more, because of a lack of stabilizing chemical bonds. If interfacial adhesion mainly involves dispersion forces between the EVA and the PA, a molecular slip at the interface can occur during the failure process, resulting in a low peel resistance. The dispersion forces between polymers are too weak (1–5 kcal/mol), compared to the chemical bonds of higher energies (50–250 kcal/mol), to counteract the molecular slip at the interface.

Thus, the formation of chemical bonds between the (PEgMAH) tie layer and the PA layer contributes more to the stability of the assembly than to their intrinsic energy. The addition of EVA in PEgMAH, up to 50 wt %, increases this energy due to

addition of less energetic but much more numerous hydrogen bonds. Above this value, the lack of chemical bonds involves a significant reduction in adhesion.

The other properties variations (optical and mechanical properties, and seal and slip ability) have already been explained by modifications of the melting temperature, the morphology, and the surface topology.[15,16]

CONCLUSIONS

The results presented here show that the incorporation of EVA in both the PE layer and tie layer of PE/PEgMAH/PA films improved the adhesion and the optical properties, without adversely affecting the mechanical properties or the seal ability, compared to incorporation in only the PE layer.

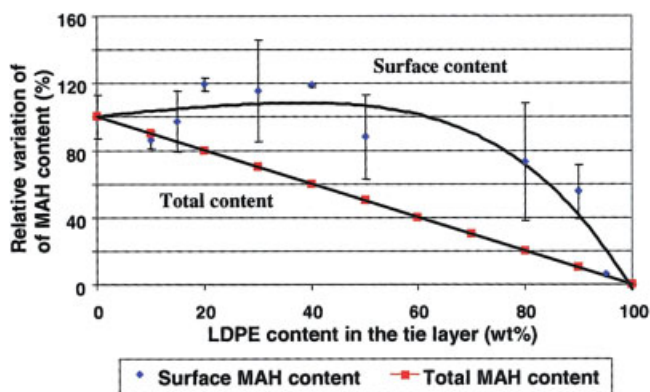The adhesion between binder and PA reached a maximum for an EVA content around 50 wt %. At

Figure 15 Influence of the LDPE content added in the tie layer on the total and surface maleic anhydride content. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]
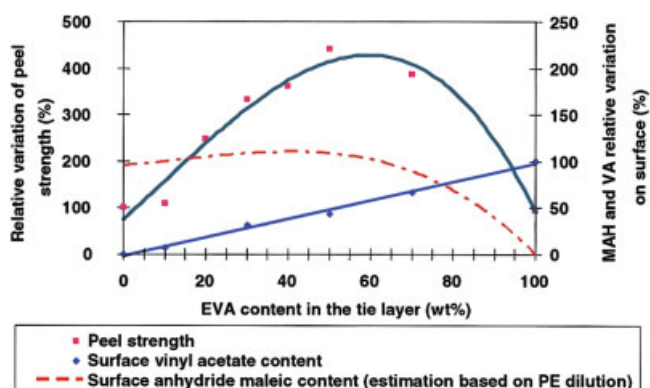

Figure 16 Influence of the EVA content in the tie layer on the anhydride maleic content, vinyl acetate content, and peel force. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

low EVA contents, the decrease in the density of chemical bonds between the NH_2 end groups of PA and the maleic anhydride groups of the binder can be compensated by creation of hydrogen bonds between the vinyl acetate and the PA. The high number of these bonds makes up for their low energy, leading to an improvement of the adhesion. Beyond this critical dilution level, the total energy of these secondary bonds becomes too weak to compensate for the energy of the stabilizing primary bonds, resulting in a decrease in the adhesion properties.

The improvement of the optical properties can be connected to a decrease in the crystallinity and in the size of the crystallites in the central layer.

Thus, with similar or even higher performance, the addition of EVA in both the PE and PEgMAH layers brings a 6% reduction in the film cost (most favorable case and optimized binder thickness), while notably increasing the adhesion between the PA and the PEgMAH binder (+450%). The sealing time can also be sensibly reduced (−40%). This decreases the production costs related to this step, while maintaining a wide flexibility in the settings.

Parts I and II of this study clearly point out the industrial interest of using EVA based blends in PE/binder/PA films to optimize the production costs (linked to both raw materials costs and cycle time), the adhesion properties, and the seal ability, while preserving satisfactory mechanical and optical performances. Only the slip ability remains slightly impaired compared to a standard PE/binder/PA multilayer film.

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