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Polyethylene–polystyrene grafting reaction: effects of polyethylene molecular weight

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Abstract

Blending of different thermoplastic polymers usually results in segregated and low value materials for almost any mixing condition. Nevertheless, a synergetic combination of properties can be obtained by an adequate compatibilization via reactive blending. In this work a Friedel–Crafts alkylation reaction is used to graft polyethylene chains onto polystyrene. The relation between the initial PE molecular weight (MW) and the structure of the compatibilizer copolymer is studied by a combination of size exclusion chromatography and Fourier transform infrared spectroscopy. The amount of copolymer formed is estimated from the amount of polystyrene reacted. The relative lengths of the grafted polyethylene chains are assessed. It is found that lower MW PE produces, upon reaction, a greater amount of short chain length grafted PE onto PS than higher MW PE. The results are in agreement with theories relating the component MW to the reaction localization at the interface.

The low cost Friedel–Crafts alkylation results in a convenient reaction for the in situ compatibilization of PE/PS blends. It produces enough graft copolymer to compatibilize the phases without causing PS crosslinking and PE chain scission. © 2002 Elsevier Science Ltd. All rights reserved.

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1. Introduction

Polymeric materials based on commodity thermoplastics are attractive, not only for their low cost, but also for their technical performance, versatility of processing-transformation, and simplicity of recycling. In the past, the efforts were aimed at the development of new resins with better properties. However, in recent years, increasing attention has been directed towards polymer–polymer blending and/or polymer-reinforcing filler composites to develop desired final properties. Blends and composites take advantage of synergetic combinations of the difference in properties of resins and fillers. As an example of this trend, it is enough to mention the advance of polymer blends in replacing engineering resins in the automotive industry [1–3]. Thermoplastic polymer consumption has also been favored because of its ability to be recycled either to the original polymer and then conveniently re-used, or transformed into liquid chemicals and fuels [4].

Most frequently used commodity thermoplastics (PE, PP, PVC and PS) exhibit very low entropy of mixing. So, phase segregation will result upon blending them in significant proportions. Formation and coalescence of sizable dispersed phase domains, low adhesion between phases, and poor final properties can be expected from direct blending of thermoplastic commodities [2–6]. Then, some kind of polymer compatibilization is needed to enhance the final properties of commodity thermoplastic blends. These compatibilization processes are based on the improvement of the adhesion between phases, the reduction of interfacial tension and the phase stabilization by the inhibition of collision of sized droplets [6–8]. Co-crystallization and co-crosslinking often result in more stable morphologies, which resist the coalescence [9]. Strong interactions such as acid–base or ion–dipole, hydrogen bonding and transition metal complexation have also been shown to enhance thermodynamic miscibility of suitably functionalized components, resulting in improved compatibility [9, 10]. The addition of copolymers (e.g. block, graft), bearing segments that are compatible to each component, was extensively used to promote blend compatibility. These copolymers may be added separately, or formed in situ by

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blending functionalized polymers (reactive compatibilization). The resulting graft or block copolymers lie at the interface and contribute to reduce the interfacial tension and to fix the morphology [11]. It can be stated that the main purpose of compatibilization processes is to bring the dispersed phase into intimate contact with the matrix, by maximizing the interfacial area. Reactive compatibilizers are often preferred because the products appear to be more stable than those obtained by addition of a previously synthesized copolymer [10,12].

Several important thermoplastic applications and processes require economical reactive compatibilization of resins. Few illustrative examples are: polyolefin–elastomer blending have been extensively used to improve the impact strength of automotive parts and other mechanical applications. Polyamides are rubber toughened to obtain ‘super-tough’ nylon products. Reactive processed polyamide/polypropylene blends offer better dimensional stability and lower permeability than pure polyamide with some economical benefit [12]. Particularly, the economy of municipal plastic waste (MPW) recycling may rely on low cost compatibilization [4]. These plastic wastes contain a large proportion of highly inert polyolefins (PE, PP and PS constitute more than 80% of the total MPW). Then, the compatibilization of two or more of these resins will be potentially useful for the development of a competitive commingled plastic waste recycling method [13,14]. This work deals particularly with the application of Friedel–Crafts reactions to the compatibilization of PE/PS resins.

Neither PE nor PS has functional groups in their structures, which may directly combine to form a copolymer. However, different reactions were reported to generate an in situ compatibilizer copolymer for this pair. Simmons [15] produced a functionalized polyethylene via batch grafting of dimethylamine ethyl methacrylate onto molten linear low-density polyethylene, using a peroxide initiator. A thermally stable, easy to process product, containing about 3% of grafted DMAEMA, was obtained. Although, the compatibilizing effect was not analyzed further. Baker [16] studied the melt blending of reactive polystyrene and reactive polyethylene with oxazoline and carboxylic acid functions, respectively. They reported a good PE/PS adhesion (observed by SEM), but poor elongation to break and impact strength. The grafting of maleic anhydride onto low-density polyethylene, in solution, was also studied [17]. A successful grafting reaction was reported, but PE crosslinking was present in all of the cases presented. The authors postulated that this reaction is fast and improves the compatibilization between PE/PS. Maleic anhydride was also grafted to PE in molten state, using the vector fluid technique. In this case the PE crosslinking was less significant [18,19].

Friedel–Crafts alkylation reactions were previously proposed to compatibilize PE/PS. Early studies were carried out by Carrick in 1970 [20], he worked with LDPE and PS in a boiling cyclohexane solution with aluminum chloride and

observed the formation of copolymer before degradation began. About 20% of the PS added appeared in the graft copolymer. Baker et al. [21,22] also studied Friedel–Crafts compatibilization reactions of PS–PE, carried out both in a batch mixer and in a single screw extruder, using aluminum chloride as catalyst and styrene as co-catalyst. High conversion rates to copolymer were reported, although the possibility of PE chain scission was also suggested.

Theoretical studies were developed, for immiscible polymer blends, aimed at understanding the compatibilization process performed by in situ reactions. The thermodynamic theory proposed by Helfand and Tagami [23], for purely physical blends, predicts that the chain ends of both polymers, as well as the low MW fractions, tend to concentrate at the interface. These results suggest that any reaction between the blend components most likely will occur at the interface, involving mainly short polymer molecules. Also, the reaction site may be located at or near the molecular chain ends.

Kramer [24] modeled the grafting reaction kinetics of a molten polymer at a reactive interface. The author considered two alternatives for grafting control: free chain diffusion through the ‘brush’ of previously grafted chains; and control by the reaction kinetics at the interface. For both alternatives the model predicts exponential decays in grafting density with the degree of polymerization. A grafting density equilibrium is reached after a long reaction time, and this grafting density value decreases with the degree of polymerization. Then, larger grafting density values can be expected from short molecules. Similar results were obtained by O’Shaughnessy [25] for interface reactions between end-functionalized chains of two polymers to produce a diblock copolymer.

In this work a low cost and suitable Friedel–Crafts alkylation reaction is applied to graft PE chains onto PS. Subsequently, the effects of PE MW on the amount and structure of the copolymers formed are studied by a combination of size exclusion chromatography (SEC) and Fourier transform infrared spectroscopy (FTIR). The results are discussed in terms of theories, for blend compatibilization, describing the reaction site and the chain length concentration at the interface.

2. Experimental

2.1. Materials

Polystyrene homopolymer Lustrex HH-103 (M_w : 271 000, M_n : 136 000) was kindly supplied by UNISTAR S.A., further on called PS. Two grades of linear low-density polyethylene came from Dow-Polisur, LLDPE 6200 (M_w : 52 000, M_n : 17 000) and 6500 (M_w : 40 000, M_n : 11 000), further on called PE62 and PE65, respectively.

The alkylation reaction was catalyzed by a system containing anhydrous Aluminum Chloride ($AlCl_3$), (>98%

Table 1
Blends designation and composition (wt%)

Sample	LLDPE	PE/PS	AlCl ₃	Styrene
PB62	6200	80/20	0.0	0.0
RB62	6200	80/20	0.3	0.3
PB65	6500	80/20	0.0	0.0
RB65	6500	80/20	0.3	0.3

purity) from Merk, and styrene (>99% purity). The catalyst was immersed in *n*-hexane to protect it from air moisture.

2.2. Blending

Physical blends. Two sets of PE (80%)/PS (20%) physical blends were prepared. They were called PB62 and PB65, for PE-62 and PE-65, respectively. The blends were performed, under nitrogen atmosphere, in a batch mixer (Brabender Plastograph W50) at 190 °C. Table 1 summarizes the characteristics of the blends prepared. The mixing procedure includes the initial melting of PS (powder form), and subsequent incorporation of PE (pellet form) under nitrogen. Mixing of PB62 blends was carried out at 30 rpm during 12 min, while the PB65 blends were processed at 50 rpm and 20 min. The mixer conditions were adjusted to reach the same PE/PS viscosity ratio and dispersion degree in each blend.

Reactive raw materials and reactive blends. The reaction was carried out in the same batch mixer and at the same temperature, speed and time as used for the physical blends. In all cases the Friedel–Crafts reaction was performed after a complete melting and mixing was reached. A 0.3 wt% styrene was used, followed by addition of 0.3 wt% AlCl₃. The resulting reactive raw materials and reactive blends, prepared with low and high MW PE, were named RPE65, RPE62, RPS, RB65 and RB62, respectively.

2.3. Solvent extraction

A careful phase separation was carried out by selective Soxhlet solvent extraction of PS and PE. PS was first extracted using tetrahydrofuran (THF) for 48 h. Then PE extraction was performed by two steps with *n*-heptane (48 and 96 h, respectively). Also, pure PE samples (PE65 and PE62) and the reactive raw materials (RPE62, RPE65 and RPS) were extracted with THF (48 h) in order to check the solvent selectivity and to verify the occurrence either of crosslinking reaction or chain scission. The respective THF soluble fractions were indicated by placing SF before the corresponding sample name.

2.4. Characterization

Size exclusion chromatography. SEC chromatograms of

Table 2
Average MWs of reactive and pure resins

Sample	M _n ^a	M _w ^a
PE65	11 000	40 000
PE62	17 000	52 000
PS	136 000	270 000
RPE65	11 000	41 000
RPE62	15 000	52 000
RPS	134 000	268 000

^a Determined by SEC.

raw materials, physical and reactive blends, and all the extracted residues were obtained in a Waters Scientific Chromatograph model 150-CV. The samples were dissolved in 1,2,4 trichlorobenzene (0.0125% BHT) at the same initial concentration, and then injected at 135 °C. MW distributions were determined for the starting materials as well as for RPE62, RPE65, RPS, SFPE65 and SFPE62. The average values are shown in Table 2.

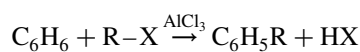
Fourier transform infrared spectroscopy. The PS concentration was estimated by comparing the ratio of infrared absorption peaks on film samples. For samples containing 0–50 wt% PS peaks at 719 cm⁻¹ (PE) and 700 cm⁻¹ (PS) were used, while for 50–100 wt% PS peaks at 720 cm⁻¹ (PE) and 1600 cm⁻¹ (PS) were selected.

Calibration curves were constructed with PS and PE physical blends of known proportions. The data summarized in Table 3 shows average values of 10 film measurements.

3. Results and discussion

3.1. Reaction mechanism

The general scheme of Friedel–Crafts alkylation reactions is [26]:



The benzene ring is susceptible to electrophilic attack primarily because of its exposed π electrons. The reaction occurs in three steps: (1) a carbocation is formed by reaction of an halogenated alkane with aluminum chloride, (2) the carbocation (acting as an electrophile) hits the benzene ring to form an arenium ion, and (3) the arenium ion loses a proton to produce the alkylated benzene.

Table 3
Total WPE and PS content after THF (48 h) extraction

Sample	WPE	PS insoluble fraction ^a	PS soluble fraction ^a
PB65	31.9	3.4	60
RB65	30.4	4.2	50
PB62	22.9	1.9	81
RB62	19.2	7.7	73

^a Determined by FTIR.

Carrick [20] and Baker et al. [21,22] studied applications of Friedel–Crafts reactions to PE–PS blends, suggesting a mechanism where a low MW carbocation is formed first from an AlCl_3 ionic complex. Then, the carbocation hits the PE molecule to yield a macrocarbocation. Finally this one produces, by electrophilic attack on the polystyrene benzene ring, a graft copolymer.

According to these ideas, Friedel–Crafts reactions were performed in this work to form compatibilizing graft copolymers in PE–PS blends. Also, the corresponding raw materials were evaluated to check the occurrence of crosslinking in competence with grafting reactions.

3.2. Qualitative analysis of initial polymers and their blends

Fig. 1 shows the SEC chromatograms for low and high MW PE and PS, as well as the cumulative MW distributions for both PE. It is noted in Fig. 1(a) that the PE and PS peaks are inverted, this comes out from the opposite signs of their TCB solution refractive indexes. So, for any blend the PE contribution will increment the peak height while the PS one will reduce it. Based on this characteristic, a qualitative analysis of PE and PS contributions can be performed by considering the relative positive and negative peak height variations. MW changes are expected in reactive blends due

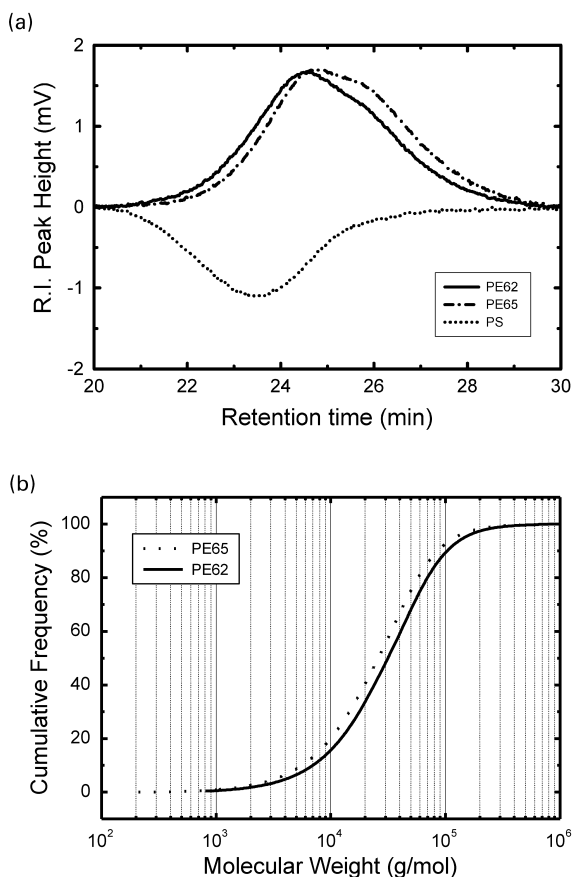


Fig. 1. (a) SEC chromatograms for PS, PE62 and PE65, (b) cumulative frequency of MW for PE62 and PE65.

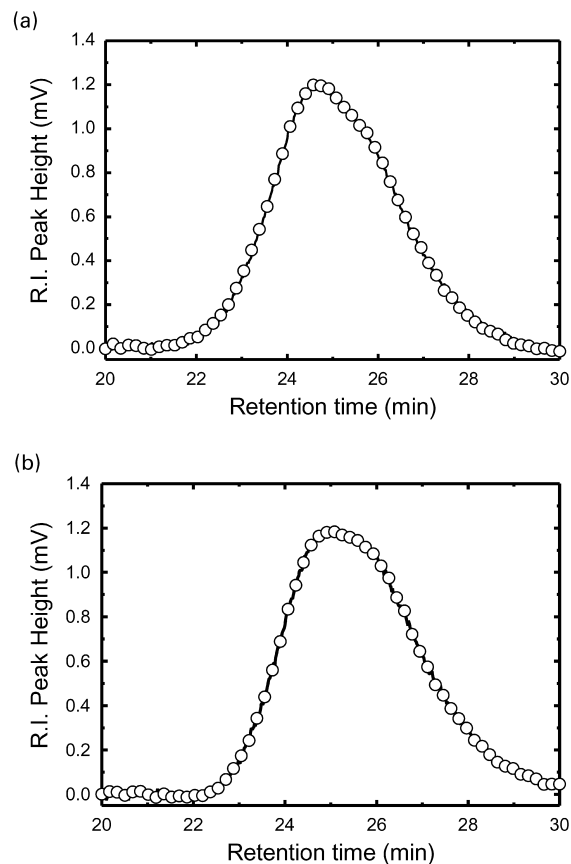


Fig. 2. SEC chromatograms for 80/20 physical blends. Weighed addition of the original components (—) and experimental data (○), (a) PE62/PS and (b) PE65/PS.

to the copolymer formation and possible crosslinking reaction. These changes can be assessed by comparison of reactive to physical blend SEC chromatograms.

It is assumed that there is no chemical interaction between components in physical blends. This condition is verified in Fig. 2. Here, the PB65 and PB62 SEC chromatograms are superposed to corresponding calculated traces, obtained from pure PE and PS chromatograms, weighed in the same proportion. The close matching observed is an indication of pure physical (non-interacting) blending, and also additive character of the blend refractive indexes.

Fig. 1(b) shows the cumulative MW distributions for the two PE studied. PE65 exhibits a considerable low MW tail, starting at 200 g/mol, as compared to PE62 that starts at 800 g/mol. The chain length difference is also observed along the entire distribution. For example, the fractions of MW less than 10 000 g/mol for PE65 and PE62 are 20 and 14%, respectively.

Fig. 3(a) shows SEC chromatograms for both the reactive (RB65, RB62) and physical blends (PB65, PB62). The high MW region (<24 min retention time) of RB65 shows a height increase with respect to the PB65. Such an increase indicates that a reaction occurs, but an increase in MW

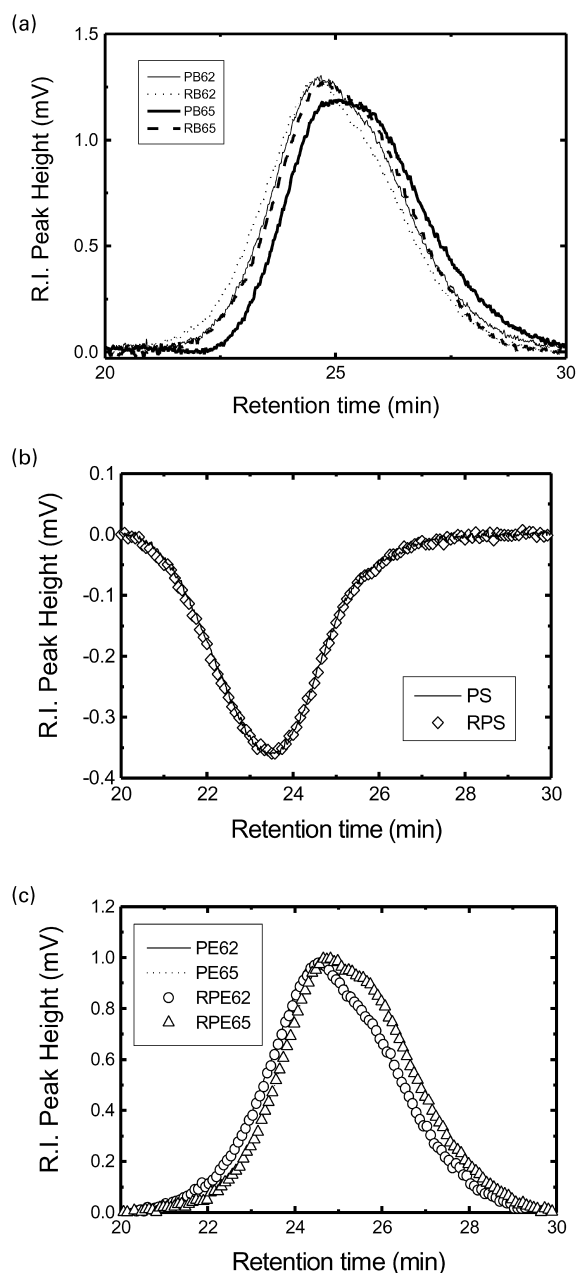


Fig. 3. (a) Physical and reactive blend SEC chromatograms, (b) reactive and pure polystyrene SEC chromatograms (area normalized), (c) reactive and pure polyethylenes SEC chromatograms (area normalized).

could be due to PE grafting onto PS and/or PS crosslinking reaction. To determine the possible appearance of crosslinking, a Friedel–Crafts alkylation was carried out onto pure PS under the same conditions as in the reactive blends. The normalized SEC chromatogram of this reaction product (RPS) is compared, in Fig. 3(b), to the normalized SEC chromatogram of pure PS. The close matching between the SEC distributions of RPS and pure PS clearly shows that crosslinking is not present. Then, it follows that the increment in the high MW region, observed for the reactive blends, is consistent with the presence of copolymers of higher MW than their original homo-

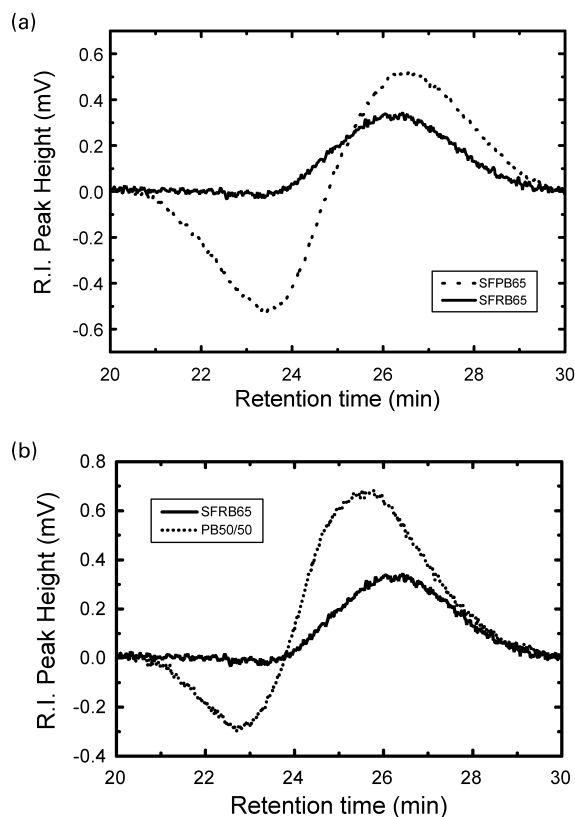


Fig. 4. SEC chromatograms for: (a) SFPB and SFRB from PE65, (b) soluble fraction of RB65 compared with a 50/50 physical blend PE65/PS.

polymers. The RB62 presents a similar behavior but with a smaller increase.

On the other hand, in the low MW region (<26 min retention time), RB65 shows a height reduction with respect to PB65 (Fig. 3(a)). Such reduction may be assigned to short chain consumption by the grafting reaction. The grafted chains are those that appear in the high MW region, incorporated into the larger size copolymer molecules. It has to be noted that a height reduction in this region is consistent with the absence of PE chain scission. This was also verified through RPE62 and RPE65 samples showing the same chain length distribution as that corresponding to pure PE (Fig. 3(c)). Also the average MW M_n and M_w are very similar (Table 2). Once again, the RB62 exhibits a similar behavior but in a less significant proportion.

3.3. Structural analysis

A combination of characterization techniques, performed on the soluble fractions of starting polymers and blends, extracted by THF, was used to characterize the copolymer structure. The polymer species (PS, PE) and their relative amounts were determined by FTIR analysis, while the relative chain lengths of grafted PE were obtained by SEC chromatography.

The soluble fractions (THF 48 h) of physical (SFPB) and reactive (SFRB) blends, as well as polyethylene

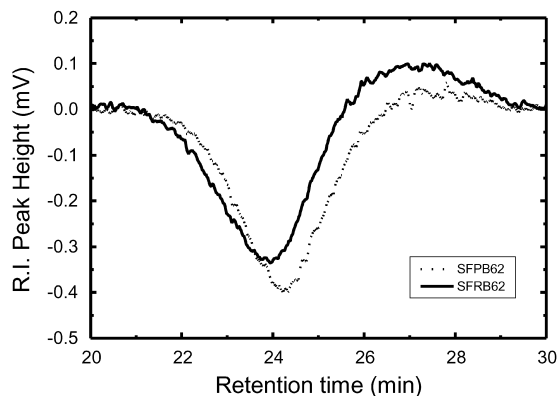


Fig. 5. SEC chromatograms for SFPB and SFRB from PE62/PS.

homopolymers (SFPE) were considered. Low and high MW PE blends exhibit considerable differences in the molecular structure of the copolymers formed, as revealed by SEC chromatography. These differences are explained in terms of the effect of chain length (of the grafted PE) on the copolymer solubility.

The SFPB65 and SFRB65 compositions are close to each other (60 and 50% PS, respectively) as shown in Table 3. Then, similar SEC chromatograms, exhibiting negative (PS) and positive (PE) peaks can be expected from both of them. However, this behavior is observed only for SFPB65 (Fig. 4(a)). The SFRB65 does not present a negative peak in apparent disagreement with the expected trace. Also, the positive peak is lower than that corresponding to SFPB65, even when the PE concentration in SFRB65 is higher (50%) than in SFPB65 (40%).

A physical blend (50% PE/50% PS), which is free of copolymer, was prepared to compare the SEC trace to SFRB65 one. It is noted that a significant negative peak is observed for this comparative blend (Fig. 4(b)). The absence of a negative peak for SFRB65 accounts for the presence of a copolymer of similar size to PS molecules, but having different detector response (different polarizability). Here, the negative contribution from PS is compensated by a positive contribution from the grafted PE molecules. Since these copolymer molecules were dissolved by THF during extraction, then the grafted PE segments should be short (only short PE molecules are dissolved by THF). On the other side, the PE peak reduction observed in Fig. 4(a) can be explained by transference of PE molecules from the PE phase to the copolymer. These results are consistent with theoretical predictions. Helfand and Tagami [23] postulated that the molecular chain ends tend to concentrate at the interface, so the low MW fractions should be forced to the interface. Kramer [24] predicts a greater graft density for shorter chains.

A similar analysis for the high MW PE blends, SFPB62 (81% PS) and SFRB62 (73% PS), reveals that negative and positive peaks appear for both fractions. Also, the peak heights are proportional to PS and PE contents, respectively (Fig. 5).

Table 4

Soluble fraction MW and total WPE of PE65 and PE62 after 48 h in THF

Sample	WPE	M_n^a	M_w^a
PE65	10.9	2500	9000
PE62	6.7	3000	11 000

^a Determined by SEC.

The different behavior shown by RB65 and RB62 is interpreted in terms of copolymer solubility. In view of the PE MW differences, the copolymer formed in RB62 should include longer (and less soluble) PE chain molecules than in RB65. Now, if it can be assumed that SFRB62 does not contain copolymer, then negative and positive peaks should be present in both SFPB62 and SFRB62, in similar proportions to the homopolymer compositions. Experimental evidence, as shown in Fig. 5, supports this assumption. This result is consistent with the MW values for SFPE62 and SFPE65 as shown (Table 4).

The different copolymer structures have different solubility in THF. In consequence some PE65-g-PS appears in SFRB65, while PE62-g-PS does not in SFRB62. This behavior is explained in terms of different PE chain lengths grafted on PS. Then, it is stated that a PE65 copolymer should have a shorter PE chain length than a PE62 copolymer.

3.4. Quantity of copolymer formed

The FTIR spectra of reactive and physical blends were carefully compared to search for band shifts that could indicate the presence of reaction products. A band shift in the aromatic ring substitution zone ($665\text{--}910\text{ cm}^{-1}$) was expected. This region is most important in determining the group positions by indicating the number of adjacent hydrogen atoms. Two adjacent hydrogen atoms, as in 1,4-di-substituted benzenes, absorb between $855\text{ and }800\text{ cm}^{-1}$. Our PE-g-PS has a 1,4-di-substitution and no measurable peaks were found in this region. The absence of such peaks is probably due to the low copolymer concentration and the relatively low sensitivity of the method. In consequence, a more lengthy technique by solvent extraction of the homopolymers had to be used to isolate the graft copolymer.

The quantity of copolymer formed might be estimated by the difference between the amounts of PS extracted (THF) from the corresponding reactive and physical blends. This implies a perfect selectivity of THF for PS. However, as it is observed in Table 3, that requirement is only partially fulfilled. On the one hand, the total weight percent extracted (WPE) from physical blends exceeds the original composition (20% PS), suggesting that part of the PE is dissolved. Moreover, low MW blends (PB65 and RB65) show a larger WPE than the corresponding PB62 and RB62, indicating that more short PE chains are dissolved. On the other hand, the insoluble fraction analysis demonstrates that the wt% PS of reactive blends is larger than that of the physical blends.

This can be explained by the presence of insoluble PS forming part of the copolymer molecules. In consequence, careful consideration of these limitations is needed for the estimation of the amount of copolymer formed.

In the first approximation, mass balances and differences in wt% PS from the insoluble fractions were used to estimate the amount of copolymer formed. For the starting blends the mass of soluble and insoluble fractions is calculated from the WPE (Table 3). Then, the residual PS in reactive and physical blends comes from the wt% PS of the insoluble fraction. Finally, the PS included in the copolymer is estimated by the difference between residual PS in reactive blend and the initial PS mass. This calculation indicates that for RB62 24% of the PS added is in the copolymer phase, while for RB65 it gives only 3% PS, far lower than expected. However, it has to be considered that, in this case, part of the copolymer was dissolved by THF, decreasing the residual PS in the insoluble fraction. In consequence, the correct estimation has to take into account that the PS in the SFRB65 (50%) is distributed between the copolymer and homopolymer phases. This distribution can be estimated by considering SEC chromatograms for physical blends. As shown in Fig. 3 the characteristic negative peak of PS does not appear up to 20% PS content. Therefore, it can be inferred, from Fig. 4(a), that 20% of the SFRB65 is PS homopolymer and 30% PS is in the copolymer. Bringing this value to a mass base and adding it to the residual PS in the insoluble phase, it follows that 48% of the initial PS should be included in the copolymer. This result is in agreement with the theories predicting that short length molecules are preferred for reaction at the interface. As was stated above, PE65 exhibits a greater fraction of low MW than PE62 (Fig. 1(b)). Hence, a larger amount of reaction product can be expected from PE65.

Because of the poor solvent selectivity to separate PS and PE from the copolymer, particularly for PE65, these results should be considered as first approximations for the amount of copolymer formed. Nevertheless, they are qualitatively consistent with theoretical predictions. Alternative extraction techniques are being worked out in order to improve the solvent selectivity.

4. Conclusions

In the reactive compatibilization of polyolefin blends the MW plays an important role. In this work, a low cost and suitable Friedel–Crafts alkylation reaction was performed to compatibilize PE/PS blends. Subsequently, a combination of SEC and FTIR techniques was used to assess the PE molecular chain lengths attached to PS and the amount of copolymer formed. The extent of reaction, as well as the PE chain lengths grafted onto PS, has shown to be dependent on the PE MW. The following conclusions are drawn:

1. Comparative SEC of reactive and physical blends is effective to estimate qualitatively the reaction (grafting and/or crosslinking) occurrence and the size of the PE molecules grafted to PS.
2. PS crosslinking does not occur, as is shown from the close matching between the SEC distributions of RPS and pure PS. Then, Friedel–Crafts reaction on PE/PS blends produces only graft-copolymer.
3. The correspondence between the height reduction in the low MW region, and the height increase in the high MW zone of the SEC chromatograms indicates the prevalence of short PE chain grafting.
4. PE chain scission is not produced as probed by comparing the PE and RPE SEC chromatograms. This conclusion is in agreement with the height decrease (at the lower MW zone) in the SEC chromatograms of reactive blends respect to physical ones.
5. From the analysis of soluble fractions of the blends, extracted by THF, it is determined that the copolymer structures include a greater amount of short PE chains for RB65 than for RB62. This was verified by the different solubility behavior of the respective copolymers. The copolymer coming from PE65 is dissolved by THF, while the one from PE62 is insoluble.
6. The extent of reaction, as estimated in terms of the amount of PS incorporated into the copolymer, is inversely related to the PE MW. In the studied case, the amount of PS reacted for PE65 blend system is double that for the PE62 system.
7. These results are in general agreement with the theory, proposed by Helfand and Tagami, postulating that short chain length molecules concentrate at the blend interface. They are also consistent with the Kramer's predictions that short chains are the most reactive.

The Friedel–Crafts alkylation appears to be a convenient reaction for the in situ compatibilization of PE/PS blends. It produces enough graft copolymer to compatibilize the phases without causing PS crosslinking and PE chain scission.

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