

# PPO-PC Block-Copolymers Used as Compatibilizers in PS/PC Blends

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**ABSTRACT:** Block-copolymers containing poly(2,6-dimethyl-1,4-phenylene oxide) (PPO) and polycarbonate of bisphenol A (PC) segments were employed as compatibilizers in polystyrene (PS)/PC blends. Block-copolymers were prepared starting from oligomeric diols-terminated PPO and PC. The poly(phenylene ethers) was obtained by oxidative coupling of 2,6-dimethyl-phenol in presence of tetramethyl bisphenol A. The copolymers were obtained with a chain extension reaction between the starting oligomers using bischloroformate of bisphenol A or phosgene as coupling agent. PS/PC blends, cast from chloroform solutions or mixed by melt, were studied by differential scanning

calorimeter (DSC), dynamic-mechanical thermal analysis (DMTA), and optical microscopy (OP). The thermal and morphological analyses showed a clear compatibilization effect between PS and PC, if PPO-PC copolymer is added when blending is performed in the melt; in addition, also mechanical properties are increased when compared with blends without PPO-PC. © 2006 Wiley Periodicals, Inc. *J Appl Polym Sci* 100: 4654–4660, 2006

**Key words:** polycarbonate; poly(2,6-dimethyl-1,4-phenylene oxide); polystyrene; blends

## INTRODUCTION

Polymer blending is a simple method for designing and controlling the performance of polymeric materials, using easy available polymers. This procedure makes it possible to develop new polymeric materials of synergetic performance. In this context, polymeric alloys of polystyrene (PS) and polycarbonate of bisphenol A (PC) are of potential interest to improve the mechanical and thermal properties of PS as well as of high impact polystyrene (HIPS). As in almost all simple polymer blends, PC and PS blends fail to compatibilize with each other and the improvement in interfacial adhesion strength is the key factor for developing new polymeric materials by polymer blending.

In general, the most representative compatibilization method is the addition of a block or graft copolymer, and this has been applied to several immiscible polymer systems.<sup>1–3</sup> In PS/PC compatibilization, polystyrene copolymers having segments or functional groups able to interact with PC<sup>4,5</sup> and statistical copolycarbonates based on bisphenol A and tetramethyl bisphenol A have been already used.<sup>6</sup>

In a previous research, we developed new poly(2,6-dimethyl-1,4-phenylene oxide)-copolycarbonate of bisphenol A (PPO-PC) block-copolymers with high molecular weight ( $\overline{M}_w > 20,000$  g/mol) starting from PPO ( $\overline{M}_w < 10,000$  g/mol) and oligomeric PC ( $\overline{M}_w < 3000$  g/mol).<sup>7</sup> All the synthesized materials showed a single glass transition temperature between 180°C and 190°C. These  $T_g$  values are very close to those of a low molecular weight PPO, but our final product shows an increment in workability range when compared with a PPO of similar molecular weight.

PPO/PS blends are one of the few known examples of miscible blends and are the basis of materials of great technological importance. Considering the complete miscibility existing between PPO and PS,<sup>8,9</sup> the prepared PPO-PC block copolymers having a well controlled molecular structure could be of interest in the field of PS/PC compatibilization. The purpose of this work is, therefore, to evaluate the effect of PPO-PC block-copolymer as compatibilizer for the immiscible blend system PS/PC. Morphological, calorimetric, dynamic-mechanical thermal analyses, and mechanical flexural tests were carried out.

## EXPERIMENTAL

### Instruments

The infrared spectra were recorded by a 5300 Jasco FTIR spectrophotometer on films obtained by casting

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from solvent or by compression molding.  $^1\text{H}$  NMR and  $^{13}\text{C}$  NMR spectra were conducted in deuterated chloroform with a 300 MHz Bruker AC300 spectrometer, using TMS as internal reference. The viscosity measurements were performed in chloroform at  $32^\circ\text{C}$  with a Schott Geräte mod AVS 310 semiautomatic viscometer.

The molecular weight distribution (MWD) was characterized by a size exclusion chromatography (SEC) system equipped with two on-line detectors, a multi-angle laser light scattering (MALS), and a differential refractometer (DRI) used as concentration detector. The SEC system consisted of: Alliance 2690 separation module from Waters (Milford, MA, USA), columns oven, MALS and a Waters 410 DRI. The running SEC conditions were: two PLgel Mixed C columns, chloroform as mobile phase, 0.6 mL/min of flow rate, and  $35^\circ\text{C}$  temperature. The MALS photometer uses a vertically polarized He-Ne laser ( $\lambda = 632.8$  nm) and simultaneously measures the intensity of the scattered light at sixteen angles ranging in chloroform from  $19.2^\circ$  to  $138.3^\circ$ . The calibration constant was calculated using Toluene as standard assuming a Rayleigh Factor of  $1.406 \times 10^{-5} \text{ cm}^{-1}$ . The angular normalization was performed by measuring the scattering intensity of a narrow MWD polystyrene standards ( $M_p = 10.3$  kg/mol,  $M_w/M_n = 1.03$ ) in the mobile phase assumed to act as an isotropic scatterer. It is well known that the on-line MALS detector measures, for each polymeric fraction eluted from the columns, the molecular weight, and when the angular dependence of the scattered light is experimentally measurable also the molecular size is measured, generally known as gyration radius ( $R_g$ ). The SEC-MALS system was described in detail elsewhere.<sup>10,11</sup>

The differential scanning calorimetry (DSC) analyses were carried out using TA instrument Q100 DSC. A sample weight of about 10 mg and a heating rate of  $10^\circ\text{C}/\text{min}$  in the range of  $40$ – $250^\circ\text{C}$  or  $40$ – $220^\circ\text{C}$ , under nitrogen flow (50 mL/min) were used. A discontinuous mixer (Brabender) equipped with a 60 cm<sup>3</sup> chamber was used to prepare melt mixed blends. Dynamic-mechanical thermal analyses (DMTA) were performed with Polymer Laboratories DMTA. The data were obtained at a frequency of 1 Hz and using a heating rate of  $0.8^\circ\text{C}/\text{min}$ .

Flexural tests were performed with a 4302 Instron dynamometer at  $23^\circ\text{C}$  and 0.01 mm/min strain rate. Specimens with dimensions  $2 \times 7 \times 50$  mm<sup>3</sup> in a three-point bending configuration were tested.

Optical microscopy (OM) was performed using a Nikon Labophot-2 (Nikon Corp.) microscopy equipped with a Sony color video camera DXC-950P (Sony Corp.).

Thermogravimetric analyses (TGA) were run on a Mettler TC11 Thermal Analyser equipped with a Mettler TG50 microbalance heating a sample of about 10

mg in an alumina crucible, from room temperature to  $650^\circ\text{C}$  at  $20^\circ\text{C}/\text{min}$  under nitrogen or air flow (20 mL/min).

## Methods

### Blends by casting

Binary and ternary blends were prepared via solution casting from chloroform (bp  $61^\circ\text{C}$ ) at level of 10 wt % of solids. The casting solutions were dried at  $25^\circ\text{C}$  and 760 mmHg for 2 days, at  $60^\circ\text{C}$  and 1 mmHg for 7 days, and at  $240^\circ\text{C}$  and 1 mmHg for 2 min.

### Blends by melt

Binary and ternary blends were also prepared by melt mixing at  $240^\circ\text{C}$  and 60 rpm, with a mixing time of 2 min. Some materials were then compression molded in a hot press at  $240^\circ\text{C}$  for 2.5 min.

## Starting materials

PS having  $\overline{M}_n = 132,000$  g/mol and  $\overline{M}_w = 189,600$  g/mol ( $T_g = 99.6^\circ\text{C}$ ), PC having  $\overline{M}_n = 20,100$  g/mol, and  $\overline{M}_w = 37,400$  g/mol ( $T_g = 150.0^\circ\text{C}$ ), were purchased from Aldrich, and was used as received. Chloroform and *N,N*-diisopropylethylamine (99 wt % purity) were purchased from Aldrich and dried over calcium hydride for 48 h. 4-Dimethylaminopyridine (99 wt % purity), phosgene solution (20 wt % in toluene), 1-methylimidazole, 2,2-bis(4-hydroxyphenyl)propane, 2,2-bis(4-hydroxy-3,5-dimethylphenyl)propane, 2,6-dimethylphenol, and bisphenol A bis(chloroformate) were purchased from Aldrich, and were used as received.

## Synthesis of PPO and PC diol-terminated and of PC-PPO block-copolymer

### Preparation of PC-2OH

Toluene solution (74 mL, 0.140 mol; 20 wt %) of phosgene was placed, under nitrogen atmosphere, in a flask (500 mL) equipped with a magnetic stirrer, a gas inlet that can be lifted at several levels, a gas outlet connected with a sodium hydroxide trap, and a dropping funnel with pressure-equalizing side arm into which a global mixture of 2,2-bis(4-hydroxyphenyl)propane (32.9 g, 0.144 mol), *N,N*-diisopropylethylamine (49 mL, 0.28 mol) and 4-dimethylaminopyridine (8.55 g, 0.07 mol) in THF (88 mL) had been charged. The flask was cooled by means of an external bath at  $5^\circ\text{C}$  and the solution in the funnel was added drop wise to the phosgene solution under stirring. After the addition, the reaction mixture was maintained at  $5^\circ\text{C}$  for 8 h under stirring, and then for further 10 h while allowing it to rise to room temper-

**TABLE I**  
Molecular and Thermal Properties of Starting Oligomers and Copolymer

Sample	$M_n$ (g/mol)	$M_w$ (g/mol)	$\eta$ (dL/g)	$T_g$ (°C)
PC-2OH	1980	2980	0.10	88.4
PPO-2OH	6700	9380	0.16	181.0
PPO-PC	9000	22500	0.26	189.1

ature. After this time, the solvent was eliminated by evaporating under vacuum. The final product was then dissolved in chloroform, precipitated with an excess of isopropanol and immediately washed with ether. The composition and molecular characteristics of the synthesized product were performed by FTIR,  $^1\text{H}$  NMR,  $^{13}\text{C}$  NMR, SEC, viscosimetric, and calorimetric analyses (see Table I).

#### Preparation of PPO-2OH

All the following solutions were prepared under magnetic stirring and at room temperature.

- *Solution A:* To a solution of toluene/acetonitrile (68 mL, 8:2 v/v),  $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$  (968 mg, 4 mmol) and 1-methylimidazole (9.6 mL, 120 mmol) were added;
- *Solution B:* 2,6-dimethylphenol (488.7 mg, 4 mmol) was dissolved in toluene/acetonitrile (8:2 v/v) solution (5 mL) with  $\text{NaOCH}_3$  (800  $\mu\text{L}$ , 4 mmol) and was added to a toluene/acetonitrile (8:2 v/v) solution (15 mL) containing tetramethyl bisphenol A (1.138 g, 4 mmol);
- *Solution C:* 2,6-dimethylphenol (9.77 g, 80 mmol) was dissolved in toluene/acetonitrile (25 mL, 8:2 v/v) solution.

Solution B was added under stirring to solution A, then solution C was added drop wise in 60 min, under oxygen atmosphere and at 20°C. The reactive mixture was maintained at room temperature under stirring and oxygen atmosphere for 60 min. After this time, the product was precipitated in methanol, dissolved in chloroform and reprecipitated in methanol–1% HCl. The product was dried at room temperature and 1 mmHg. The composition and molecular characteristics of the synthesized product were performed by FTIR,  $^1\text{H}$  NMR,  $^{13}\text{C}$  NMR, SEC, viscosimetric, and calorimetric analyses (see Table I).

#### Preparation of PPO–PC block copolymer

Chloroform (20 mL) with bisphenol A bis(chloroformate) (1.77 g, 5 mmol) were placed in a flask under nitrogen atmosphere. At 5°C and under stirring, chloroform solution (20 mL) of PC-2OH (3.25 g, 2.5 mmol)

and *N,N*-diisopropylethylamine (0.88 mL, 5 mmol) were added drop wise in 20 min. First the reactive solution was kept under stirring and at 5°C for 30 min, then a chloroform solution (60 mL) of PPO-2OH (20 g, 2.5 mmol), *N,N*-diisopropylethylamine (0.88 mL, 5 mmol) and 4-dimethylaminopyridine (0.305 g, 2.5 mmol) was added drop wise in 30 min. The reactive solution was kept at 5°C for other 60 min, it was then allowed to reach the room temperature and the product was precipitated in methanol, dissolved in chloroform, and reprecipitated in methanol–1% HCl.

The composition and molecular characteristics of the synthesized product were performed by FTIR,  $^1\text{H}$  NMR,  $^{13}\text{C}$  NMR, MALDI-TOF, SEC, viscosimetric and calorimetric analyses. The spectroscopic and MALDI-TOF characterizations confirm the multi-block structure<sup>12</sup>; viscosimetric, calorimetric, and SEC data are reported in Table I.

## RESULTS AND DISCUSSION

### Synthesis and thermal properties of PPO–PC block-copolymers

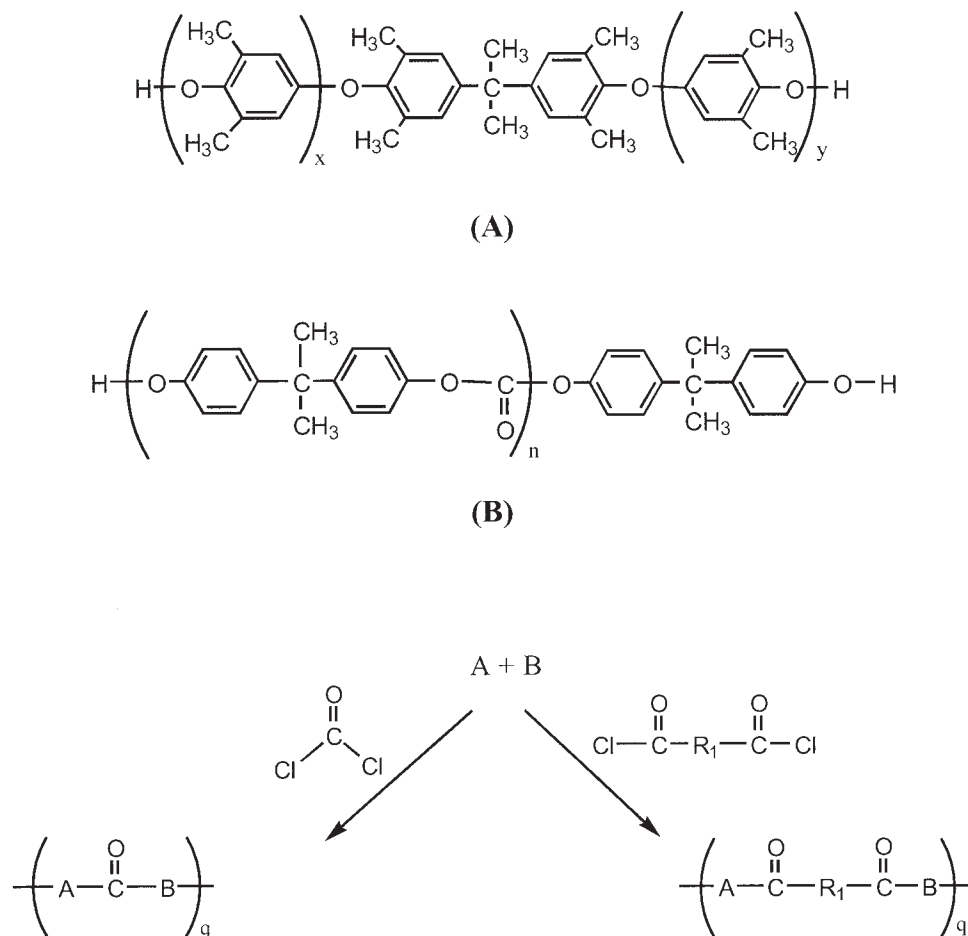
New materials having a multi-block structure were synthesized by a coupling reaction between oligomeric poly(2,6-dimethyl-1,4-phenylene oxide) diol-terminated (PPO-2OH) (named A in the Scheme 1) and oligomeric polycarbonate of 2,2-bis(4-hydroxyphenyl)propane (bisphenol A) diol-terminated (PC-2OH) (named B in the Scheme 1).

Either bisphenol A bis(chloroformate) or phosgene was employed as coupling agent in presence of tertiary amines. Three steps are involved in the general synthetic Scheme 1. Namely, first oligomeric PPO-2OH is prepared by oxidative coupling of a mixture of 2,6-dimethylphenol and 2,2-bis(3,5-dimethyl-4-hydroxyphenyl)propane using a copper catalyst<sup>7</sup>; second, oligomeric PC-2OH is synthesized by a polycondensation reaction between bisphenol A and phosgene (molar ratio diol/phosgene > 1). Finally, a copolymer having a well controlled alternated multi-block structure is obtained by means of a coupling reaction using a bischloroformate or phosgene.

The composition and molecular characteristics of all synthesized material were performed by FTIR,  $^1\text{H}$  NMR,  $^{13}\text{C}$  NMR, MALDI-TOF, SEC, viscosimetric and calorimetric analyses. The spectroscopic and MALDI-TOF characterizations confirm the multi-block structure<sup>12</sup>; viscosimetric, calorimetric and SEC data are reported in Table I.

The DSC analysis of copolymer shows a single  $T_g$  close to oligomeric PPO (see Table I), this suggests that a homogeneous material was obtained.

Since blending by melt requires thermal stability of the used components at the process conditions, thermo gravimetric analyses (TGA), respectively, in



Scheme 1 Synthetic pathway of PPO-PC multi-block copolymers.

nitrogen and air, were carried out on copolymers, starting oligomers and, for comparison, on typical commercial PPO. In Table II, the inflection temperature ( $T_i$ ) evaluated from the TGA traces and the related weight losses at the end of the step ( $\Delta W\%$ ) are reported. It should be noted that the copolymer shows a thermal stability in nitrogen when compared with commercial PPO. On the other hand in air, commercial PPO shows a small step at  $T_i = 281^\circ\text{C}$ , with an increas-

ing in weight of 2.6 wt %. This could be due to the oxidation process of aromatic methyl groups.<sup>13</sup> Analogous effect was not highlighted from the copolymer TGA trace.

### PS/PC blends

Polystyrene/polycarbonate blends, with and without compatibilizer, were prepared by casting and by melt mixing according to the following procedures.

The casting method foresees two evaporation steps from a 10 wt % of total mass chloroform solution: the first one at room temperature for 48 h, the second under vacuum for 7 days at  $60^\circ\text{C}$  and later at  $240^\circ\text{C}$  for 2 min.

According to this procedure, a series of blends containing a multi-block-copolymer, exactly 10% by weight of total polymer, was prepared. For comparison purpose, an analogous series without compatibilizer was prepared too. Composition and calorimetric data of all blends are summarized in Table III.

First of all, it should be noted that all blends always show two glass transition temperatures. In the series

TABLE II  
Thermogravimetric Analyses of Starting Oligomers and Copolymer

Sample	Nitrogen		Air	
	$T_i$ ( $^\circ\text{C}$ ) <sup>a</sup>	$\Delta W$ (%) <sup>b</sup>	$T_i$ ( $^\circ\text{C}$ ) <sup>a</sup>	$\Delta W$ (%) <sup>b</sup>
PPO-2OH	456	63.7	422	19.1
PC-2OH	495	83.5	480	70.0
PPO-PC	465	55.0	449	46.7
PPO <sup>c</sup>	456	59.5	281	-2.6
			454	32.2

<sup>a</sup> Inflection point.

<sup>b</sup> Total weight loss at the end of the step.

<sup>c</sup> Commercial product.

**TABLE III**  
**Composition and Calorimetric Data of PS/PC Blends**  
**Prepared by Casting (A), Melt Mixing (B), and Moulded**  
**(M), with and without PPO-PC Block Copolymer (C)**

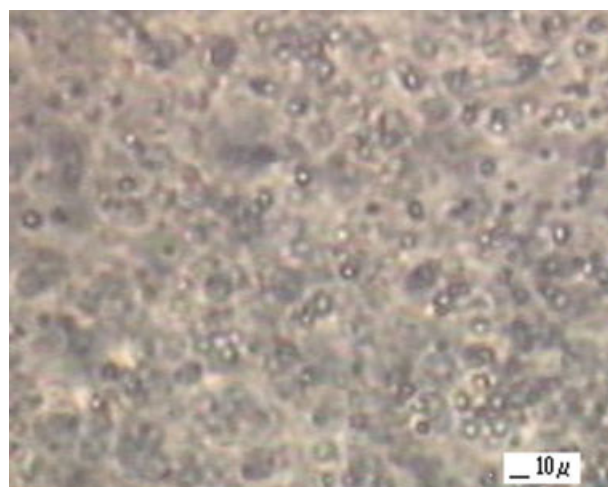
Blend	PS <sup>a</sup> (wt %)	PPO-PC <sup>b</sup> (wt %)	Low $T_g$ (°C)	High $T_g$ (°C)	$\eta$ (dL/g)
A3	30	—	101.7	144.3	—
A5	50	—	100.0	143.7	—
A7	70	—	100.0	140.0	—
AC-3	30	10	106.7	144.6	—
AC-5	50	10	104.6	142.3	—
AC-7	70	10	104.5	140.0	—
B5	50	—	101.4	142.1	—
B5-C	50	1	102.8	141.1	—
B5-CII	50	2	102.8	140.7	—
M5	50	—	101.4	143.5	0.59
M5-C	50	1	101.8	140.0	0.56
M5-CII	50	2	102.5	138.2	0.51

<sup>a</sup> PS:  $M_n = 132,300$  g/mol,  $M_w = 189,800$  g/mol,  $T_g = 99.6^\circ\text{C}$ ; PC:  $M_n = 20,100$  g/mol,  $M_w = 37,400$  g/mol,  $T_g = 150^\circ\text{C}$ .

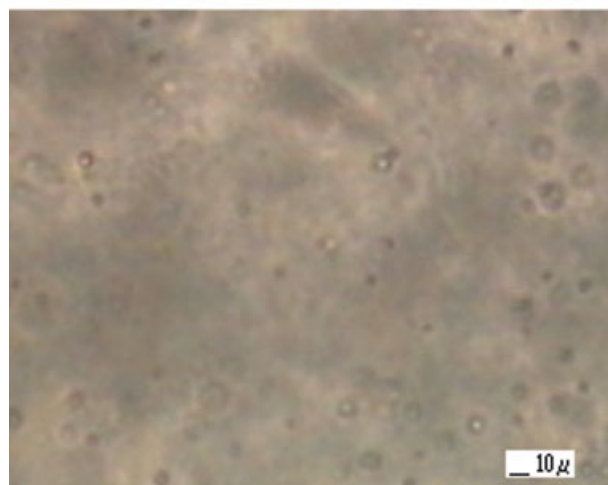
<sup>b</sup> PPO-PC:  $M_n = 10,600$  g/mol,  $M_w = 30,500$  g/mol,  $T_g = 189.1^\circ\text{C}$ .

obtained via casting without compatibilizer, the PC phase  $T_g$  of blends having 30, 50, and 70 wt % of PS are 144.3, 143.7, and 140.0°C respectively, while those of the PS phase are 101.7, 100.0, 100.0°C. The shifts of low and high  $T_g$  respect to the pure components (PS  $T_g = 99.6^\circ\text{C}$ ; PC  $T_g = 150.0^\circ\text{C}$ ) are more significant when PS and PC are present at lower concentration (30 wt %). This is consistent with the effect of blend composition on miscibility when the chemical structure and the molecular mass of components are fixed.<sup>14</sup> The addition of PPO-PC block-copolymer (10 wt % on total mass) to the same PS/PC blends seems to act only on PS phase causing a significant shift of  $T_g$  (106.7, 104.6, and 104.5°C in the order for 30, 50, and 70 wt % PS blends), while PC phase  $T_g$ s are slightly or are not affected compared to the values of the corresponding uncompatibilized blends. This could be explained considering the length of PPO and PC blocks in PPO-PC copolymer; in fact the PC segments are probably too short to improve the overall copolymer interaction with PC phase. This is in agreement by DSC analyses carried out on PS/PPO-PC and PC/PPO-PC (50/50 wt %) binary blends, cast from chloroform. In fact, PS/PPO-PC blend shows only one glass transition temperature (133.4°C), while PC/PPO-PC shows two  $T_g$  respectively, at 141.1 and 187.8°C. This suggests that while the PPO units of the copolymer effectively interact with PS chains, the PC units seem unable to lead to similar interactions. On the other hand the presence of the copolymer markedly affects blends morphology. The morphological analysis performed by optical microscopy on blends prepared by casting, highlights always a heterogeneous systems, but finer dispersed domains are observed if compatibilizer is added (Fig. 1).

Blends prepared by melt were obtained using a discontinuous mixer at 240°C with a mixing rate of 60 rpm and for a residence time of 2 min. In particular, PS/PC blends (50/50 wt %), having respectively, 0, 1, and 2% by weight of PPO-PC as compatibilizer, were prepared and subsequently press molded to obtain specimens for DMTA and flexural mechanical tests. DSC analyses were conducted at different stages of the material processing. From DSC data, summarized in Table III, it should be noted that both for the melt mixed and the press molded blends the low and the high  $T_g$  approach each other if the block copolymer is added. This is also observed by DMTA analysis (Fig. 2), which confirms an evident increase of the low  $T_g$  and a less evident decrease of the high  $T_g$  when the compatibilizer is added. Moreover, the PC phase  $T_g$ s

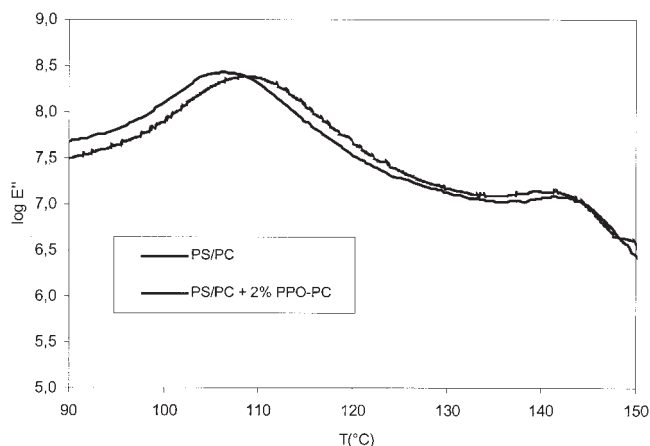


(a)



(b)

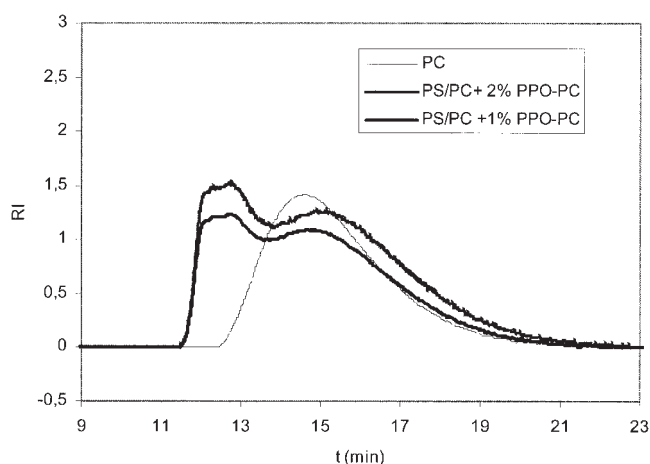
**Figure 1** Optical microscopy of 50/50 PS/PC blends without compatibilizer (a) and with 2% by weight of PPO-PC (b). [Color figure can be viewed in the online issue, which is available at [www.interscience.wiley.com](http://www.interscience.wiley.com).]



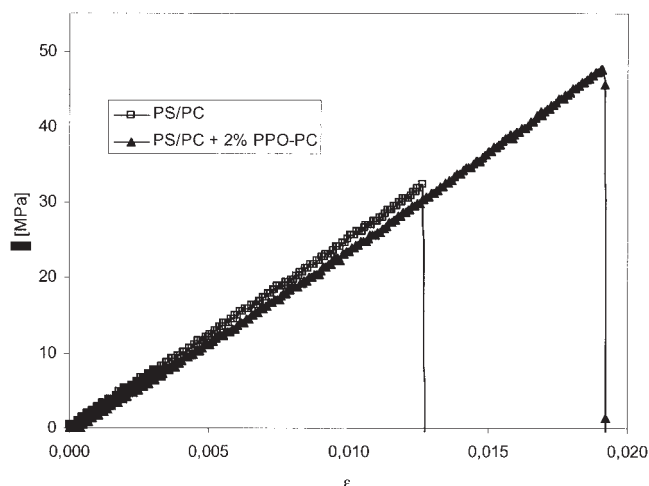
**Figure 2** Log  $E''$  versus  $T$  ( $^{\circ}\text{C}$ ) of 50/50 PS/PC blends without compatibilizer and with 2% by weight of PPO-PC.

of melt mixed blends are lower in comparison with those obtained from casting, and the value decreases further if the material is press molded.

All data suggest that a higher compatibilization effect is obtained if blends are subjected to thermal treatment. This is probably due to the occurrence of a transesterification reaction between the starting PC and polycarbonate segments of the compatibilizer.<sup>6</sup> The viscosities of press molded blends are reported in Table III (performed at  $32^{\circ}\text{C}$  in chloroform solution) and show a decrease of molecular weight increasing PPO-PC content. The hypothesis of a transesterification reaction that involves the PC phase is confirmed by size exclusion chromatography (SEC). As shown in Figure 3, increasing the PPO-PC content the maximum of PC phase peak is shifted to higher retention time. A further confirmation comes from DSC data of a binary blend PC/PPO-PC (50/50 wt %), cast from chloroform solution, subjected to three thermal cycles:



**Figure 3** SEC traces of commercial PC and 50/50 PS/PC blends with 1 and 2 wt % of PPO-PC.



**Figure 4** Flexural tests of 50/50 PS/PC blends without compatibilizer and with 2% by weight of PPO-PC.

one between 40 and  $250^{\circ}\text{C}$  to eliminate PC crystallinity and two from 40 to  $220^{\circ}\text{C}$ , with a scanning rate of  $10^{\circ}\text{C}/\text{min}$ . After the second scan, 141.1 and  $187.8^{\circ}\text{C}$  turn out as low  $T_g$  and high  $T_g$ , while after the third scan 143.7 and  $184.2^{\circ}\text{C}$  are respectively, obtained.

Mechanical properties were evaluated by flexural tests to highlight the effect of PPO-PC block copolymer as compatibilizer on PS/PC blends. Typical flexural strength-strain curves for 50/50 PS/PC blend without compatibilizer and with 2% by weight of PPO-PC are shown in Figure 4. A brittle behavior was observed for all materials, although higher strain at break is obtained in compatibilized blends. The compatibilizer does not affect much of the flexural modulus value, which goes from  $2800 \pm 240$  MPa to  $2700 \pm 210$  MPa if PPO-PC is added, but significantly affects the break load increasing the stress from  $35 \pm 4$  MPa to  $51 \pm 3$  MPa, respectively. The use of PPO-PC block copolymer increases the flexural strength of about 50% compared with the uncompatibilized blend and this is probably related to the higher phases adhesion and dispersion. These results further confirm that our new PPO-PC gives effective compatibilization increasing mechanical properties of high incompatible PS/PC blends.

## CONCLUSIONS

Multi-block-copolymers containing PPO and PC segments are useful systems to increase the compatibility in PS/PC blends. The synthetic scheme used to prepare the copolymers makes possible to tune the molecular structure for optimizing the compatibilization effect.

In this study, the compatibilization effect of a PPO-PC block-copolymer having short PC segments has been studied. The prepared material showed good thermal stability, both in nitrogen and air, and only

one glass transition temperature close to the  $T_g$  of the oligomeric PPO employed.

A significant compatibilization effect was noted in blends prepared by melt mixing, while a lower effect was observed for cast specimens. In the first case, viscosimetric, calorimetric and SEC data gives evidence that PC segments of the copolymer and high molecular weight PC could be involved in a transesterification reaction. Low PPO-PC amount (1 and 2 wt %) added to PS/PC blends was able to increase the mechanical strength of material in a significant way in comparison with uncompatibilized blends. A modification of the copolymer structure, in particular of the PC unit length, may lead to further increase of the compatibilization effects; and this aspect will be further investigated.

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