Thermal and Photochemical Degradation of PPO/HIPS Blends

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ABSTRACT: In general, polymer blends show a degradation behavior different from a simple combination of the individual components, making any forecast difficult without experiments. Interactions between polymers can sensibilize or stabilize the blend against degradation. In this work, the thermal and photooxidative degradation of blends of poly(2,6-dimethyl-1,4-phenylene oxide) (PPO) and high impact polystyrene (HIPS) have been studied under accelerated conditions. The extent of degradation was accompanied by infrared spectroscopy (FTIR) and Raman spectroscopy (FT-Raman) and impact resistance and strain–stress testing followed its influence on the macroscopic properties of the blends. The results showed that HIPS and the blend containing 60 wt % of PPO are more susceptible to thermal and photochemical degradation,

INTRODUCTION

Polymer blends are mainly manufactured with the purpose of improving properties of components, decreasing expenses, and producing new materials. The physical and chemical behavior of polymer blends is normally different from a simple combination of the properties of the individual components, being difficult to predict without experiments.¹ The degradation of polymer blends is an example of the difference between the behavior of individual components and of the components combined in the blends.² The degradation of one component of the blend can directly influence the degradation process of other polymer. For example, low molar mass compounds resulting from degradation of a component can accelerate or deaccelerate the degradation of the second. Photochemical and photophysical interactions between excited states induced by thermal or light energy can also occur, causing sensibilization of the degradation of one polymer by the other.²

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while the blends containing 40 and 50 wt % of PPO are more stable. Infrared and Raman spectroscopic analyses showed that the degradation of HIPS and its blends is caused not only by degradation of the polybutadiene phase. Effects of interactions, such as exchange of energy in excited state between the PPO and PS components of the polymeric matrix may also be responsible for the degradation and loss of mechanical properties of the PPO/HIPS blends. The chemical degradation directly affects the mechanical properties of the samples with photodegradation being more harmful than the thermal degradation at 75°C. © 2007 Wiley Periodicals, Inc. J Appl Polym Sci 104: 3269–3276, 2007

Key words: blends; photochemical degradation; thermal degradation; mechanical properties

Miscible blends are characterized by the existence of specific and strong chemical interactions between the components, such as dipole–dipole interactions and H-bonding.¹ These interactions may influence the degradation of the polymers in the blends. In immiscible blends, the degradation behavior is generally an addition of the properties of the pure components, but coreactions in the polymer interface, controlled by morphology, can also take place, making the degradation of these systems very complex.²

An important effect in influencing the overall material stability is the difference of the degradation temperature of the components in the blend and of the components themselves. For example, in blends of polystyrene (PS) with poly(vinylidene chloride) (PVDC), polyacrylonitrile (PAN), polybutadiene (PB), and polyphenyleneoxide (PPO), the decomposition of PS occurs at temperature higher than that of PS, due to deactivation of PS macroradicals through intermolecular reactions with structural units of the second component. On the other hand, in blends of PS and poly (ethylene glycol) (PEG), the PS degradation is accelerated by reactions of PS chains with radicals produced from PEG decomposition.³

In this work, the thermal and photochemical degradation of blends of poly(2,6-dimethyl-1,4-phenylene oxide) (PPO) and high impact polystyrene (HIPS) is studied. HIPS is a high impact thermoplastic

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composed of polystyrene and a dispersed elastomer phase of polybutadiene. The polybutadiene phase of HIPS consists of different isomers (1,4-cis; 1,4-trans and 1,2-vinyl), which present different stabilities against the degradation.⁴ Because double bonds represent sites vulnerable to chemical attack,⁴ the component principally responsible for the photo- and thermal degradation of HIPS is usually considered to be the polybutadiene, although some authors have demonstrated the difficulty of proving this hypothesis by using infrared spectroscopy,⁵ nuclear magnetic resonance,⁶ and thermal analysis.⁷

Despite the presence of the polybutadiene disperse phase in the PS matrix of HIPS, the PS phase is miscible with PPO in the PPO/HIPS blends.⁸ This feature is a consequence of specific interactions between PS and PPO components.^{8–11} Fluorescence experiments showed the existence of a mechanism of energy transfer between the excited states of polystyrene and PPO.¹² Therefore, in the degradation studies of HIPS/PPO blends, the behavior of the matrix, a miscible phase of PS/PPO, as well as the behavior of the polybutadiene disperse phase, must be taken into consideration.

The aromatic ring present in the polymer chains of both polymers, PPO and PS, is responsible for light absorption in the ultraviolet region and for their susceptibility to photodegradation at long wavelengths ($\lambda > 300$ nm). PPO presents typical chromophore properties, having maximum light absorption above 325 nm.¹³ The initial step of PPO degradation can occur through two pathways. In the first one, polymer chain scissions take place as a consequence of ultraviolet or thermal energy absorption, forming poly(2,6-dimethylphenoxy) radicals (PDMP). In the second, mainly through thermal energy absorption, elimination of a hydrogen atom belonging to the methyl groups of the aromatic ring occurs, generating a benzylic-type radical.¹³ In the absence of oxygen, a complex set of reactions occurs, resulting in loss of methyl groups, monomers, and opening of the aromatic rings. In the presence of oxygen, the PDMP radicals can directly react with it, leading to the quinone formation. These quinones absorb light at wavelengths longer than 400 nm, being responsible for the yellow coloration of the material. The quinone easily activates groups and accelerates the polymer degradation or decomposes through ringopening reactions.13,14

The thermal and photodegradation of polystyrene in the presence of oxygen is well established. A hydrogen bonded to the carbon of the benzyl group is labile and its loss occurs by the absorption of energy, producing a radical that undergoes subsequent reactions such as depolymerization or oxidation.^{5,15–17}

Birkisnhaw et al.¹⁸ studied the thermal degradation of blends of syndiotatic polystyrene and PPO. The blends were prepared by coprecipitation from solution, followed by compression molding. Because the PS used to prepare these blends was a syndiotatic one, the blends presented two phases: a crystalline PS phase and a miscible and amorphous phase of PS and PPO. Thermogravimetry was used to compare the thermal stability of the polymers and their blends. The authors found that the degradation of PS in the blends was shifted to higher temperature in comparison with the PS, while degradation of PPO in the blends was accelerated relative to pure PPO. The mechanism proposed by the authors to explain the results is that the PPO acts as a radical acceptor, interrupting the intermolecular transfer process in the degrading polystyrene.

In this work, the thermal and photooxidative degradation of blends of poly(2,6-dimethyl-1,4-phenylene oxide) (PPO) and high impact polystyrene (HIPS) have been studied under accelerated conditions. The blends were prepared by mechanical mixing by GE Plastics South America. The extent of degradation was accompanied by infrared spectroscopy (FTIR) and Raman spectroscopy (FT-Raman) and its influence on the macroscopic properties of the blends was followed by impact resistance (IR), strain–stress testing (SST), and thermogravimetry (TGA).

EXPERIMENTAL

HIPS and its blends containing 40, 50, and 60 wt % of PPO, namely PPO 40, PPO 50, and PPO 60, were supplied by GE Plastics South America (Campinas, Brazil). The samples, containing processing additives, were used without purification. Injection molded specimens with dimensions of 125 mm; 13 and 3.2 mm were submitted to thermal and photochemical aging in accordance to the procedures described in ASTM-D5510 and ASTM-G53, respectively. PPO was provided in a powdered form. Specimens for impact resistance testing were prepared by the compression molding process. Specimens for impact resistance tests were mechanically notched using an Emic notching machine (São José dos Pinhais, Brazil) in accordance to ASTM 256 (0.25 \pm 0.12 radius), before thermal and photoaging.

Thermooxidative degradation was carried out using a TECNAL TE 394/1 (Piracicaba, Brazil) ventilated oven at 75° C.

Photodegradation was carried out in a special apparatus¹⁹ composed of PHILIPS model CLEO performance 80 WR mercury Lamps, with emission in the range from 315 to 400 nm. The aging program consisted of cycles of 24 h irradiation at room temperature, followed by condensation at 40° C for 2 h. Only one side of the specimens was irradiated.



Figure 1 Impact resistance retention $IR(t_n)/IR(t_0)$ as a function of the aging time: HIPS (**I**), PPO40 (**•**), PPO50 (\triangle), PPO60 (\bigtriangledown), and PPO (\bigcirc): (a) thermal aging and (b) photochemical aging.

The aged samples were submitted to impact resistance testing using an EMIC AIC 1 apparatus (São José dos Pinhais, Brazil) in accordance to ASTM 256 using Izod notched bars. Specimens were mechanically notched before using thermal and photoaging. At least 10 specimens of each composition were analyzed.

Strain–stress tests were performed on an EMIC LA 2000 universal machine (São José dos Pinhas, Brazil) at a low strain rate (4.5 mm/min). At least 10 specimens of each composition were analyzed.

Fourier transformed infrared spectra (FTIR) were recorded using a Bomen B100 spectrometer (Quebec, Canada) at 4 cm⁻¹ resolution, using 16 scans. The degraded surface of the samples was scratched with a sharp blade, and the resulting powder was dispersed in KBr at 1 wt %. The HIPS spectra were normalized with respect to the intensity of the aromatic band at 696 cm⁻¹, while the spectra of the other samples were normalized using the intensity of the C—O stretching band of PPO at 1186 cm⁻¹. Raman spectra (FT-Raman) were recorded using a Bomen FT DA8 (Quebec, Canada) with laser excitation at 1064 nm and an InGaAs detector. For each spectrum, 64 scans were used.

RESULTS AND DISCUSSIONS

The impact resistance of HIPS, PPO, and their blends as a function of the thermal and photochemical aging time is shown in Figure 1. This property is expressed as the ratio between the impact resistance at the aging time t_n (IR(t_n)) and the initial impact resistance (IR(t_0)). The absolute values of impact resistance as a function of thermal and photochemical aging are listed in Table I.

Thermal aging leads to a continuous decrease in the impact resistance [Fig. 1(a)], while the initial stage of photochemical aging is marked by intense changes in the impact resistance [Fig. 1(b)]. The impact resistance of the blend PPO40 submitted to thermal aging for 6990 h decreases about 6%, while for the PPO50 blend the decrease was 16%. HIPS and PPO60 blend showed a more pronounced decrease in the impact resistance (about 40%) under the same conditions. A similar dependence of impact resistance on blend composition was observed for samples submitted to 2928 h of photochemical aging [Fig. 1(b)]. However, for similar exposition times, the photochemical degradation was more intense than the thermal degradation. Even though the photochemical aging effects are more pronounced, the same order of stability or retention of mechanical proper-

TABLE I Impact Resistance (J/m) as a Function of Blend Composition and Aging Time

	Thermal aging							
Sample	0	48 h	168 h	1397 h	2900 h	6990 h		
HIPS PPO40 PPO50 PPO60 PPO	$125 \pm 469 \pm 481 \pm 350 \pm 542 \pm 4$	$ \begin{array}{r} 119 \pm 6 \\ 66 \pm 4 \\ 82 \pm 3 \\ 56 \pm 6 \\ 44 \pm 3 \\ \end{array} $	115 ± 4 76 ± 1 76 ± 6 43 ± 4 42 ± 3 hotochem	102 ± 3 62 ± 4 76 ± 3 40 ± 4 38 ± 2 ical aging	$ \begin{array}{r} 106 \pm 4 \\ 71 \pm 1 \\ 75 \pm 4 \\ 36 \pm 4 \\ 33 \pm 1 \end{array} $	49 ± 2 65 ± 2 68 ± 3 22 ± 1 23 ± 2		
	0	48 h	168 h	672 h	1440 h	2928 h		
HIPS PPO40 PPO50 PPO60 PPO	$125 \pm 4 \\ 69 \pm 4 \\ 81 \pm 3 \\ 50 \pm 5 \\ 42 \pm 4$	90 ± 6 71 ± 5 77 ± 3 47 ± 6 49 ± 3	$\begin{array}{c} 81 \pm 3 \\ 61 \pm 4 \\ 67 \pm 2 \\ 26 \pm 3 \\ 35 \pm 2 \end{array}$	$71 \pm 4 51 \pm 4 64 \pm 2 13 \pm 1 24 \pm 2$	$74 \pm 3 57 \pm 2 62 \pm 3 26 \pm 4 34 \pm 4$	$\begin{array}{r} 45 \ \pm \ 3\\ 51 \ \pm \ 2\\ 55 \ \pm \ 1\\ 20 \ \pm \ 0\\ 21 \ \pm \ 2 \end{array}$		



Figure 2 Impact resistance retention $IR(t_n)/IR(t_0)$ as a function of composition at similar aging times: (\triangle) thermal aging (2900 h) and (\bigcirc) photochemical aging (2928 h).

ties is observed in both at the end of the experiments: PPO40 > PPO50 > PPO > PPO60 > HIPS. The PPO40 and PPO50 blends present very similar behaviors, while the behaviors of the impact resistance with aging time of the PPO60 blend and HIPS are also similar.

The PPO50 blend presents the highest impact resistance among the studied blends and this feature is maintained after aging (Table I). The better mechanical properties of the PPO50 blend are attributed to the cavitations of the elastomeric particles and shear band formation in the deformed zones ahead of the crack tip of the PPO50 blend. The cavitations and shear band formation dissipate bulk strain energy and their formation is responsible for the highest value of observed mechanical properties.²⁰

The impact resistance of the PPO/HIPS blends is intermediate between the values found for PPO and HIPS. For HIPS, this property presents a pronounced drop with aging, assuming values of impact resistance lower than those of PPO40 and PPO50 blends after 6990 h and 2828 h for thermal and photochemical aging, respectively, (Table I).

Figure 2 shows the dependence of $IR(t_n)/IR(t_0)$ on the blend composition (w_{ppo} %) at similar thermal (2900 h) and photochemical (2928 h) aging times. The impact resistance retention of samples after thermal and photochemical aging presents the same dependence on the blend composition, despite the intensity of the aging effects being dependent on the aging method.

Table II summarizes the results of the stress–strain tests as a function of composition and aging time. Strain–stress tests were not conducted for PPO because it was not possible to mold specimens through the usual methods. Elongation at break was the most affected strain–stress property due to aging. The ratio between the elongation at break at aging time $t_n(E(t_n))$ and the elongation at time t_0 , before the aging process ($E(t_0)$) is plotted as a function of aging time for each sample (Fig. 3). The elongation at break decreases with increasing aging time, as observed for the impact resistance. The decrease of the elongation at break at the initial stage was more accentuated for photoaged samples, and PPO50 presented higher retention of the elongation property at break in comparison with the other blends.

The decreasing order of the retention of the elongation at break after 6990 h of thermal aging is: PPO50 > PPO40 > PPO60 > HIPS, while for samples photochemically aged for 2928 h it is: PPO50 >PPO60 > PPO40 > HIPS.

	0			1	0 0			
	Thermal aging							
Sample	0	48 h	168 h	1397 h	2900 h	6990 h		
HIPS	39 ± 1	27 ± 2	26 ± 1	23 ± 1	19.7 ± 0.8	13.0 ± 0.8		
PPO40	21 ± 1	21 ± 1	20.0 ± 0.6	18 ± 1	15.3 ± 0.6	12.8 ± 0.4		
PPO50	22.9 ± 0.9	21.1 ± 0.4	20.4 ± 0.3	17 ± 1	20.4 ± 0.6	18.2 ± 0.8		
PPO60	15.7 ± 0.7	13 ± 1	11.5 ± 0.8	8.0 ± 0.4	8.4 ± 0.2	6.0 ± 0.2		
	Photochemical aging							
	0	48 h	168 h	672 h	1440 h	2928 h		
HIPS	39 ± 1	9.3 ± 0.9	3.1 ± 0.1	6.9 ± 0.1	5.7 ± 0.1	5.5 ± 0.1		
PPO40	21 ± 1	20.0 ± 0.1	7.7 ± 0.9	9.3 ± 0.2	10.7 ± 0.4	7.5 ± 0.4		
PPO50	22.9 ± 0.9	17 ± 1	14.8 ± 0.8	13.6 ± 0.8	13.6 ± 0.4	10.5 ± 0.4		
PPO60	15.7 ± 0.7	7.7 ± 0.9	4.5 ± 0.3	7.3 ± 0.6	6.6 ± 0.1	6.4 ± 0.1		

 TABLE II

 Elongation at Break (%) as a Function of Composition and Aging Time



Figure 3 Elongation at break retention $(E(t_n)/E(t_0))$ as a function of the aging time for: HIPS (\blacksquare), PPO40 (\bullet), PPO50 (\triangle), PPO60 (\bigtriangledown): (a) thermal aging and (b) photochemical aging.

The mechanical results show that HIPS is more sensitive to thermal and thermooxidative degradation. A pronounced drop of the mechanical performance of HIPS was also described by Singh et al.²¹ who studied the oxidative degradation of HIPS with different polybutadiene contents and found a slight increase in tensile strength during the early stages of exposure; thereafter, a continuous drop was observed. The increase was attributed to the dominance of crosslinking reactions over chain scission during the early stages.

Figure 4 shows the infrared spectra of HIPS and PPO60 at different thermal aging times, focusing on the carbonyl band ($\approx 1700 \text{ cm}^{-1}$), which is usually used to analyze the oxidation of polymers in aging process. The carbonyl band for all samples was not

very expressive, in spite of the long thermal aging times. A low intensity carbonyl band is observed for HIPS thermoaged for 6990 h.

In contrast to the thermal aging, the changes in the infrared spectra of the samples submitted to photochemical aging were intense (Fig. 5).

HIPS is the most sensitive to thermal and photoaging. Figure 5(a) shows the evolution of carbonyl band near 1700 cm⁻¹ as the photoaging time increases. Among the blends, the formation of carbonyl groups is more expressive for the PPO60 blend [Fig. 5(d)]. These results are in agreement with the mechanical testing, which showed that the loss of mechanical properties as a consequence of the extent of degradation is more intense for the PPO60 blend and for HIPS.



Figure 4 Infrared spectra at different thermal aging times: (a) HIPS and (b) PPO60.



Figure 5 Infrared spectra at different photochemical aging times: (a) HIPS, (b) PPO40, (c) PPO50, (d) PPO60, and (e) PPO.

Figure 6 presents the carbonyl index (IOx) as a function of the photochemical aging time. The carbonyl index was calculated as a ratio of the intensity of the carbonyl band at 1700 cm⁻¹ and the intensity of the band at 1186 cm⁻¹ for PPO and its blends. The band at 696 cm⁻¹ was used as an internal reference for HIPS. The carbonyl index (IOx) increases as the photoaging time increases and, for the same experimental time, HIPS presents the highest IOx, while PPO presents the lowest. The blends presented intermediate values of IOx. The order of increasing photooxidative degree (IOx) after 2928 h of photoaging is: PPO < PPO50 < PPO40 < PPO60 < HIPS. The retention of the impact resistance and of the elongation at break is inversely proportional to the IOx.

Raman spectroscopy is a very sensitive technique to analyze C=C chemical groups. This feature allows following the photochemical aging effects on the unsaturated polybutadiene phase in HIPS. Figure 7 shows the Raman spectra of nonaged and photoaged HIPS. The Raman spectrum of HIPS shows a



Figure 6 Carbonyl index of photochemical aging: HIPS (\blacksquare), PPO40 (\bullet) PPO50 (\triangle), PPO60 (\bigtriangledown), and PPO (\bigcirc).



Figure 7 Raman spectra of HIPS after different photochemical aging times showing the absorption band of the polybutadiene phase.

broad band in the region of 1630 to 1690 cm⁻¹ that corresponds to the overlap of absorption of the trans, cis, and 1,2-vinyl isomers of polybutadiene. As the photoaging time increases, the Raman spectra of the HIPS showed that the C=C bonding of the polybutadiene phase presents some changes. The spectra were normalized with respect to the intensity of the band attributed to C=C bonds of the phenyl ring at about 1600 cm⁻¹. Therefore, a direct comparison of the C=C band area is possible. The different isomers of polybutadiene present characteristic C=C absorption bands: 1,2-vinyl C=C at 1640 cm⁻¹; cis-1,4 at 1655 cm⁻¹, and *trans*-1,4 at 1666 cm⁻¹.²² The C=C band becomes narrower with aging and the intensity in the region corresponding to absorption of the 1,2-vinyl and cis-1,4 isomers decreases. Therefore, the degradation affects preferentially these isomers, even though only a small fraction of the C=C bonds of the polybutadiene phase was involved. This result is in agreement with the shift of the glass transition to higher temperature because trans-1,4 polybutadiene presents a higher T_g than the other isomers.²³

The differences between the Raman infrared spectra in the region characteristic of the absorption of the C=C bond for HIPS and photoaged HIPS do not justify the intense absorption at 1720 cm⁻¹ observed in the infrared spectrum shown in Figure 7(a), attributed to C=O. This fact, associated with the drop of mechanical performance of HIPS, is strong evidence that the degradation of the matrix is also important. This should also be true for blends with PPO, as previously verified from dynamic mechanical analyses.²³

CONCLUSIONS

Thermal and photochemical aging studies on PPO, HIPS, and their blends showed a dependence of the degradation extent on the composition and aging method. Photochemical aging was shown to be more aggressive than thermal aging at 75°C for these materials, but the dependence of the aging on the blend composition is similar for both aging methods. PPO50 and PPO40 blends were more stable against degradation, while the degradation behavior of the PPO60 blend and HIPS is very similar, with both PPO60 and HIPS being very susceptive to degradation. The differences between the samples can be caused by synergistic interactions of the PPO and HIPS components. Infrared and Raman spectroscopic analyses showed that the degradation of HIPS and its blends is caused not only by degradation of the polybutadiene phase. Effects of interactions, such as exchange of energy in excited state between the PPO and PS components of the polymeric matrix may also be responsible for some of the degradation and loss of mechanical properties of the PPO/HIPS blends. The chemical degradation directly affects the mechanical properties of the samples. Photo degradation is more harmful to the chemical and mechanical properties than is thermal degradation at 75°C.

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