Online Monitoring of the Influence of the Chemical Structure of Hindered Amines on the Hydrolysis of Polycarbonate in a Polycarbonate/Poly(acrylonitrile– butadiene–styrene) Blend by Ultraviolet–Visible Spectroscopy

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ABSTRACT: Without stabilization, polycarbonate (PC)/ poly(acrylonitrile-butadiene-styrene) (ABS) blends are susceptible to a loss of mechanical properties after a few days of exposure to weathering conditions. ABS can be stabilized against terrestrial light by the use of hindered amines in combination with a UV absorber; such hindered amines cannot be used when PC is present in the polymer blend. The hydrolysis of PC is accelerated when a small amount of a hindered amine light stabilizer (HALS) is incorporated into the resin and is exposed to elevated temperatures. In this study, three different HALSs (Tinuvin 123, Tinuvin 770, and Tinuvin 765, Ciba, Basel, Switzerland) were used as UV stabilizers for PC/ABS blends, and their effects on the PC phase were observed with online ultraviolet-visible spectroscopy on extruded flat films. These stabilizers were compounded with the blends in a corotating twin-screw extruder at 240°C. The molecular weight of

the compounded samples was determined by gel permeation chromatography. The extent of degradation induced by the HALSs on the PC phase was found to be a function of its chemical structure. Tinuvin 123 with an amino ether functional group enhanced degradation in comparison with Tinuvin 770 and Tinuvin 765. Tinuvin 770, a secondary amine, was apt to be more reactive than Tinuvin 765, a tertiary amine, because less steric hindrance was experienced by the former. Accelerated aging of the compounded samples was performed. Decreased degradation was observed for the samples containing hindered amines; however, the HALSs alone were not effective in protecting the PC/ABS blends against harmful UV light. © 2010 Wiley Periodicals, Inc. J Appl Polym Sci 119: 491–499, 2011

Key words: blends; compounding; degradation; HALS; UV–vis spectroscopy

INTRODUCTION

Polymer blends provide a valuable balance of mechanical properties, melt-processing behavior, and cost. As a result, blends have gained wide industrial acceptance. One such system is polycarbonate (PC) blended with rubber-modified styrenic poly(acrylonitrile-butadiene-styrene) (ABS). PC provides high mechanical and thermal properties,¹ whereas ABS allows for a lower processing temperature and imparts enhanced low-temperature impact strength.² PC blends are widely used in demanding applications such as automotive parts, power tools, and office equipment. Automotive specifications, in particular, are stringent with respect to color retention

for PC/ABS blends. However, adequate stabilization of PC/ABS blends against terrestrial light remains problematic.³⁻¹¹ The loss in the properties of these blends is mainly due to UV-induced photooxidation of the polybutadiene portion of ABS. A UV absorber at a high concentration is often used, but it results in high costs for PC blends.¹² Although a UV absorber is beneficial for shielding against UV light, it does not scavenge radical species arising from UVinduced or thermally induced degradation of the PC or ABS components in the blend. Hindered amines are well known to be radical scavengers for many classes of polymeric materials.¹³ They are extensively used to protect PC/ABS blends from photooxidation and thermooxidative degradation.^{14,15} The mechanism of protection, although not yet completely clarified, involves oxidation of the amines to nitroxide radicals, which by a reaction with carbon-centered radicals from the polymer give rise to hydroxylamines and alkylated hydroxylamines. These, in turn, can regenerate the nitroxide and restart the

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Figure 1 Stabilization mechanism of the HALSs in the polymers (X is an alkyl group).

protective cycle many times before the additive, in its various forms, is completely depleted. The intermediate nitroxide radicals are very long-living species, especially in a solid matrix like that of the host polymer. A simplified mechanism of action that is often used is depicted in Figure 1.¹⁶

However, hindered amines cannot be used in polymeric substrates that are sensitive to chemical reactions with the basic nitrogen atom in the light stabilizer.¹⁵ Such polymeric substrates include PC. Indeed, a severe reduction in mechanical properties is reported when hindered amines are incorporated into PC/ABS blends during the compounding stage.¹⁷

The objective of this study was to examine the effects of three different hindered amine light stabilizers (HALSs) on the PC phase during the compounding of PC/ABS blends in a twin-screw extruder at 240°C and thereby screen the stabilizers as harmful or not harmful to the PC phase. Tinuvin 123 (T123), Tinuvin 770 (T770), and Tinuvin 765 (T765; Ciba, Basel, Switzerland) were used as the stabilizers for this study. These stabilizers were compounded with PC/ABS blends and extruded into films with a thickness of 0.35 mm. The effects of HALSs on the polymer phase were monitored by online ultraviolet-visible (UV-vis) spectroscopy during the compounding stage. The molecular weights of the compounded samples were determined with gel permeation chromatography (GPC). The effectiveness of the HALSs at protecting the samples against UV radiation was tested by accelerated weathering in an Atlas Suntest apparatus. Because the main objective of this work was to monitor the effects of different hindered amines on the backbone of the PC phase, the effect of water molecules on the whole process was limited to measure the total moisture content present in the PC polymer.

EXPERIMENTAL

Material

The polymers used in the investigation were PC (Calibre 300-10, Dow Benelux, Terneazen, The Netherlands) and ABS (Magnum F641, Dow Benelux). A schematic overview of the stabilizers is shown in Table I. The additives exhibited no significant absorption within the wavelength range used for UV–vis spectroscopy (350–900 nm). The lower wavelength for the UV–vis measurements was restricted by the large scattering from the ABS phase.

PC and ABS were first preblended with a weight ratio of 65/35. In the second step, this material was compounded with the additives. Because the amount of additives was quite small, a master batch was prepared in ABS powder to ensure that the

 TABLE I

 Schematic Overview of the Stabilizers Used with Their Structures and Suppliers



required amount of the additive was accurately fed. A small increase in the ABS content during compounding was therefore explained when the additives were fed into the preblend. The stabilizer concentration varied from 0 to 1 wt %.

Compounding experiments

A production-size corotating twin-screw extruder (ZSK-32 Mc MEGA compounder, Coperion Werner and Pfleiderer GmbH, Stuttgart, Germany) with 32-mm screws was equipped with gravimetric feeding units (Brabender, Duisburg, Germany). These units were capable of feeding the minor component with throughputs from 0.2 to 2.5 kg/h. The barrel temperature was set to 240°C. A screw speed of 600 rpm and a throughput of 35 kg/h were chosen. A standard screw configuration was used for the compounding.

The film extrusion line (type 136-350S chill roll, Dr. Collin GmbH, Ebersberg, Germany) had a roll face width of 350 mm preceding a slit die with a width of 250 mm and a lip that was adjustable between 0.3 and 2 mm. The roller conveyor following the chill roll had a length of 3300 mm and could be adjusted to throughput velocities between 0.5 and 5 m/min. The film extrusion line offered enough space for a number of high-throughput analysis methods. The PC/ABS film extrusion was conducted at a velocity of 5 m/min, and the thickness of the films was 0.35 mm.

Testing procedure

The molar mass of unaged PC in PC/ABS blends was determined by size exclusion chromatography (GPC) in tetrahydrofuran with toluene as the internal flow marker. The GPC setup consisted of a Waters 515 (Milford, Massachusetts) isocratic high performance liquid chromatography (HPLC) pump, a TSP AS 100 autosampler, a Waters UV 486 detector operated at 254 nm, and a Waters 410 differential refractometer. Three poly(styrene)/divinylbenzene (SDV) columns (Polymer Standard Service GmbH, Mainz, Germany) with nominal pore sizes of 10^6 , 10^5 , and 10^4 Å (30×0.8 cm²) were used. Calibration was performed with narrow-distribution polystyrene standards. Data acquisition and processing were performed with WINGPC software version 6.2.

The online UV–vis spectroscopy apparatus consisted of a MultiSpec 2-channel process spectrometer system (tec5 AG, Oberursel, Germany) equipped with a 1024-pixel diode array detector. The UV–vis transmission probes measuring the film were connected to the spectrometer by optical fibers.

The films were aged in an Atlas Suntest XLS+ apparatus containing xenon lamps with daylight filters with a wavelength range starting at 298 nm. The black standard temperature was set at 65°C. The irradiance level was 765 W/m^2 at a band width of 300–800 nm. The samples were aged for 450 h under dry conditions.

The moisture content of PC was measured with an Aboni FMX Hydrotracer (HESS Werkstoff–Prüfgeräte, Richard HESS MBV GmbH, Sonsbeck, Germany). Each sample was weighed and filled into the sample tray. A heater warmed the sample to the chosen temperature (175°C). The evaporating water reacted with the powdery reagent, calcium hydride, which transformed the water into hydrogen. The concentration of hydrogen within the reactor atmosphere was a measure of the water content within the reactor. A gas sensor detected the hydrogen concentration.

RESULTS

Compounding of PC/ABS with the UV stabilizers

In this section, the compounding of PC/ABS with the UV stabilizers T770, T123, and T765 and their effects on the PC phase are discussed.

The effects of two different screw speeds, 600 and 1200 rpm, on the UV-vis spectra of the films without any additives are shown in Figure 2. Throughout the article, curves are plotted as difference spectra, which are obtained by the subtraction of the reference spectrum (the spectrum of the blend extruded at 600 rpm without additives) from the spectrum of the blend extruded with or without additives under different conditions. The films compounded at 1200 rpm exhibited spectra of different shapes. The films had absorbance values below the reference curve for wavelengths roughly between 500 and 700 nm. This was attributed to the better dispersion of the ABS phase within the blend and therefore less scattering at higher screw speeds. In the near-infrared region (>700 nm) and at wavelengths shorter than 500 nm,



Figure 2 Variation of the UV–vis spectra as a function of the screw speed.

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Figure 3 Variation of the UV–vis spectra as a function of the concentration of T770.

the absorbance increased remarkably. The increasing absorbance in the short-wavelength region corresponded to a yellow coloring of the films, which could be seen visually. The shear forces at 1200 rpm caused a high melting temperature, which favored thermal degradation of the polymer. The degradation products absorbed mainly at short wavelengths and in the nearinfrared region. Because the compounding at 1200 rpm caused severe degradation of the films, further compounding with additives was done only at 600 rpm.

The effect of increasing the concentration of T770 on the UV–vis spectra of the films is shown in Figure 3 with compounding at a screw speed of 600 rpm. An overall increase in the absorbance, independent of the wavelength, could be seen. This was the additional scattering due to the increased ABS content from the feeding of the additives with ABS powder. In addition, there was a small tendency for increasing absorbance with increasing T770 content for wavelengths below 400 nm. This corresponded to slight yellowing. This yellowing was due to the degradation products formed by the reaction of T770 with the polymer.

The influence of increasing the amount of T123 on the UV–vis spectra is shown in Figure 4. The samples were compounded at a screw speed of 600 rpm. Even with 0.05% T123, the absorbance increased in the short-wavelength region and led to the yellowing of the film. With higher T123 contents, the shapes of the spectra became similar to those of the PC/ABS samples compounded at 1200 rpm (Fig. 2). Visually, the samples were yellow and brittle. This showed severe degradation of PC/ABS caused by T123 during compounding.

The online UV-vis spectra for different concentrations of T765 compounded at 600 rpm are shown in Figure 5. An overall increase in the absorbance,

Figure 4 Variation of the UV–vis spectra as a function of the concentration of T123.

attributed to the feeding of ABS together with the additive, can be seen. No increased absorbance with increasing T765 content below 400 nm was observed. This showed that no yellowing due to the degradation of the polymer occurred. Visually, the samples were white and tough, and this correlated with the observations of the UV–vis spectra.

To compare and evaluate the effects of the three different HALSs on the deviation of the difference spectra from the reference line and thus on the damage, independent of the scattering background originating from the fed ABS, a compressed operational parameter was defined. To get rid of the scattering background, each spectrum was mean-centered, and a squared deviation integral (χ^2 = mean-centered square deviation integral; s^2 = square deviation integral) was calculated:



Figure 5 Variation of the UV–vis spectra as a function of the concentration of T765.







Figure 6 Squared deviation integral (χ^2) as a function of the Tinuvin content.

$$\chi^{2} = \int_{\lambda_{\min}}^{\lambda_{\max}} \left(\Delta A(\lambda) - \langle \Delta A(\lambda) \rangle \right)^{2} d\lambda$$
 (1)

 λ is the wavelength where as λ_{max} and λ_{min} are wavelength at 900 and 350 nm

$$\langle \Delta A(\lambda) \rangle = \int_{\lambda_{\min}}^{\lambda_{\max}} \Delta A(\lambda) d\lambda / (\lambda_{\max} - \lambda_{\min})$$
 (2)

where ΔA is the absorbance difference and λ_{\min} and λ_{\max} are 350 and 900 nm, respectively.

The χ^2 values calculated for the curves shown in Figures 3–5 are plotted as a function of the Tinuvin content in Figure 6. For T770 and T765 at 600 rpm, the χ^2 values were mainly below 0.15. The samples without Tinuvin had a χ^2 value of 0 by definition. With concentrations of T123 greater than 0.07%, χ^2 values higher than 10 were observed.

Comparing Figures 3–6, we can conclude that the degradation caused by T770 or T765 was similar but low in extent with processing at 600 rpm. Severe degradation occurred for the polymer when the χ^2 values were greater than 1.

Molecular weight determination

The weight-average molecular weight (M_w) values of PC/ABS samples compounded with T770, T123, and T765 were determined for different concentrations. The results are depicted in Figure 7. Similar to PC, one of the components of ABS, styrene acrylonitrile absorbed at 254 nm because of the presence of an aromatic ring during the measurement of the molecular weight. ABS is known to not undergo hydrolysis during compounding with UV stabilizers, but it can show a reduction in the molecular weight during

processing due to thermomechanical degradation. However, in this case, the degradation of PC and ABS due to thermomechanical effects in the extruder was not taken into account because all the experiments were performed under the same processing conditions except for the usage of different stabilizers; therefore, a comparative molecular weight was obtained that reflected mainly the hydrolytic effect of UV stabilizers on the PC phase. The molecular weight decrease was prominent for T123, whereas for the other two stabilizers, it was marginal. There was a sharp decrease in the molecular weight for T123 until a concentration level of 0.3%; afterward, it leveled off. The reduction of M_w finally corresponded to a value of 27%. T770 caused a slight decrease corresponding to a reduction of only 3%, whereas with T765, there was practically no decrease; a reduction in M_w less than 1% was found.

With the trends for the molecular weight, the conclusions drawn from the online spectroscopy were very well supported.

Accelerated aging of the PC/ABS blends by UV radiation

Samples of the PC/ABS blend, for which degradation during compounding was minimal, were irradiated in the Atlas Suntest XLS+ apparatus. All the T770 and T765 samples as well as the blends with no more than 0.07% T123 were tested. The UV–vis spectra are shown in Figures 8–11. To focus only on the changes caused by the irradiation, the plotted curves are difference spectra obtained by subtraction of the spectrum of the nonirradiated sample from the spectrum of the irradiated sample.

In Figure 8, the curves for the T770 samples are shown. The T770-containing samples as well as the sample without Tinuvin showed a pronounced



Figure 7 Molecular weight as a function of the concentration of the hindered amines.

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600 rpm

0 %

0.1 % 03%

0.5%

Figure 8 Variation of the UV–vis spectra of the irradiated PC/ABS samples as a function of the T770 content.

increase in absorbance for wavelengths below 400 nm. This visually corresponded to yellowing. For wavelengths higher than 400 nm, there was a bleaching effect; that is, the absorbance decreased. Both effects, the yellowing and the bleaching, were more pronounced for the sample without T770. The additive stabilized the blend against UV light, but the T770 content seemed to be of minor importance because no significant correlation with the Tinuvin content could be seen. This was due to the high efficiency and long lifetime of the HALS, which was due to the cyclic process mechanism by which the HALS was regenerated rather than consumed during the stabilization process. Therefore, significant levels of stabilization were achieved at relatively low concentrations, and no specific layer thickness or concentration limit needed to be reached to guarantee good results.

600 rpm

---- 0 % 0.07 %



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Irradiated samples

Tinuvin 123

0.15

0.10

0.05

-0.10

-0.15

-0.20

4 0.05
4 0.00
4 0.05
6 0.05



600

Wavelength (nm)

700

800

900

500

Irradiated sample

Tinuvin 765

0.15

0.10

0.05

0.00

-0.05

-0.10

400

A_{UV}-A_{non}

The curves of the samples with 0 or 0.07% T123 are shown in Figure 9. The spectra of the samples with T123 basically looked similar to the curve of the sample with no additive. It can be concluded that no stabilization against UV light was achieved with low concentrations of T123, whereas higher concentrations of T123 caused severe degradation during compounding.

The UV-vis spectra of the irradiated PC/ABS polymer blend compounded with T765 at different concentrations are depicted in Figure 10. The samples showed a yellowing effect at wavelengths below 400 nm and bleaching at higher wavelengths. With increasing T765 content, the yellowing and bleaching effects decreased.

To get a compressed operational measure of the extent of the spectral changes and thus of the damage caused by the irradiation, a squared deviation integral (s^2) was calculated:

$$s^{2} = \int_{\lambda_{\min}}^{\lambda_{\max}} \left[(A_{\rm UV} - A_{\rm non})(\lambda) \right]^{2} d\lambda \tag{3}$$

 A_{non} = Absorbance of non-irradiated samples; A_{uv} = Absorbance of uv-irradiated samples.

This is analogous to eqs. (1) and (2) but without subtraction of the mean spectral value because any deviation from the zero horizontal was assumed to be caused by the irradiation.

The parameter s^2 is plotted in Figure 11 for the curves shown in Figures 8-10. Above a certain amount of T770 or T765, the parameter s^2 sharply decreased from a value of approximately 7 and leveled off at values of approximately 3 and 1.5, respectively. These samples, therefore, showed some resistance to photodegradation. The blend with 0.07% T123 had an s^2 value of approximately 9, and this confirmed that no protection against UV light was





Figure 11 Squared deviation integral (s^2) as a function of the Tinuvin content. The lines serve as guides to the eyes.

achieved with low concentrations of T123. Because s^2 for the T765 samples leveled off at 1.5, the protection against UV was better compared to that of the T770 samples, for which s^2 leveled off at values around 3.

Although an increase in the stabilization of the PC/ABS blends was seen with T765 and T770, weak protection was found for the samples against harmful UV radiation, as shown by the yellowing and bleaching effects, which were still apparent even with higher stabilizer contents.

DISCUSSION

In this study, the degradative effect of the three UV stabilizers (T770, T123, and T765) on the PC/ABS blends was measured by means of online UV–vis spectroscopy and molecular weight determination.

It is known that hindered amines cannot be used in polymeric substrates that are sensitive to chemical reactions with the basic nitrogen atom in the hindered amines. A severe reduction of the mechanical properties has been reported when hindered amines have been incorporated into PC/ABS blends during compounding. This is explained¹⁵ by the acceleration of the hydrolysis reaction in PC due to the presence of weak bases such as HALSs at elevated temperatures. Water remains in PC even after drying before compounding, and a concentration of 0.17% was found for all the compounding experiments performed in this research. The mechanism by which the hydrolysis of PC with a weak base forms acid carbonate and its further decarboxylation are described in Figure 12.17-19

This process can, therefore, be described as random chain scission with an accumulation of phenolic chain ends. Subsequent oxidation of the phenolic end groups led to yellow products that were observed in the samples with T123 (Fig. 4) and to a lesser extent in those with T770 or T765 (Figs. 3 and 5). The degree of base-catalyzed hydrolysis was markedly affected by the structure of the light stabilizer.

On the basis of the UV–vis spectra of the UV stabilizers (Figs. 3–5), the χ^2 plot (Fig. 6), and the molecular weight determination (Fig. 7), we found that the extent of degradation caused by the UV stabilizer in the PC phase showed the following trend:

T123 >> T770 > T765

This reactivity series can be explained by the structure and the different groups attached to the basic nitrogen atom of each stabilizer and therefore by the reactivity of each stabilizer.

The UV-vis spectra and molecular weight determination studies showed that of the two conventional light stabilizers used, T765 (a tertiary amine)



Figure 12 Mechanism of the hydrolysis of PC with a weak base.

and T770 (a secondary amine), the PC phase was less hydrolyzed by the former. The lower reactivity of T765 versus T770 can be explained as follows.¹⁵ The basic strength of amines in aqueous media depends on steric and hydration effects. The steric effect refers to the crowding of alkyl groups around the nitrogen atom, which hinders the attack of protons on the amine molecule; this decreases its basic strength. Because the crowding of alkyl groups around the nitrogen atom increases from primary amines to tertiary amines, the basic strength of amines decreases in the following order: primary > secondary > tertiary. The hydration effect refers to the stabilization of the protonated amine by water molecules. The water molecules form hydrogen bonds with the protonated amine and release energy called hydration energy. The greater the extent of hydrogen bonding is in the protonated amine, the greater its stabilization will be, and consequently, the greater the basic strength of the corresponding amine will be. Hydration due to hydrogen bonding is maximum in monoalkyl ammonium ions, protonated cations of a primary amine. It is less in dialkyl ammonia ions and still less in trialkyl ammonium ions. Thus, the basic strength decreases as follows: primary > secondary > tertiary. These explanations show that the basic strength and hence hydrolysis reaction of the secondary amine are greater than those of the corresponding tertiary amine.

T123 is a free-radical scavenger based on an amino ether (NOR) functional group. Because of the NOR functionality, T123 has lower basicity than conventional HALSs, and this makes it noninteracting with acidic species. The hydrolysis effect of T123 on the PC phase should, therefore, be minimal because of the aforementioned property. However, we found that the degradation effect of T123 on the PC phase was far more pronounced than the effect of the conventional HALSs. A sharp decrease in the molecular weight of PC and an exponential increase in the squared deviation integral (χ^2) were observed with an increase in the concentration of T123. These results showed that the hydrolysis mechanism alone could not account for the degradation effect of T123.

It is known that the thermolysis of NOR hindered amines follows two seemingly similar but distinctly different reaction pathways. The two cleavage processes can be described as follows:²⁰



The thermally induced breakdown of NOR hindered amines occurs at high temperatures, whereas compounding leads to the formation of either alkyl and nitroxy radicals [Eq. (4)] or alkoxy and aminyl radicals [Eq. (5)]. Aminyl and alkoxy radicals are very reactive and are involved in the free-radical chemical reactions. The radicals generated by NOR hindered amines can cause degradation to the PC polymer by a number of degradation mechanisms.²¹ These reactions explain the greater degradation caused by NOR in the PC phase during compounding.

The accelerated aging studies performed on PC/ ABS samples showed that none of the three stabilizers was truly effective in protecting them from photodegradation (Figs. 8–11). This was because the HALSs absorbed no light in the wavelengths between 300 and 400 nm and acted only by catalyzing the termination step of the oxidation cycle.²² For polyolefins, this process is very effective; however, it generally is less effective for aromatic polymers for which the rate of initiation is high and the number of propagation steps in the propagation cycle of autoxidation is small.

CONCLUSIONS

The effect of three different HALSs (T123, T770, and T765) on PC/ABS blends during compounding in a twin-screw extruder was analyzed with online UVvis spectroscopy. This method turned out to be a powerful and nondestructive tool for obtaining immediate information on degradation during compounding experiments. UV-vis spectroscopy was especially sensitive to minute color changes. From the changes in the spectra, we observed that T123 and, to a much lesser extent, T770 reacted with the blends. This was confirmed by GPC, which showed a large decrease in the molar mass of PC in the samples with T123 versus those with T770. T765 seemed to react with the PC phase to a very small extent. This was also shown by the UV-vis spectra and by GPC: no reduction of the molecular weight of the compounded samples could be found. It could be concluded that the chemical structure of the hindered amine played an important role in the hydrolysis of the PC phase. T123 with a NOR functional group enhanced degradation versus T770 and T765. T770, a secondary amine, was susceptible to being more reactive than T765, a tertiary amine, because less steric hindrance was experienced by the former and this made the basic nitrogen more reactive. It could also concluded that the HALSs alone were not effective in protecting the PC/ABS blends from harmful UV light even though decreased degradation was observed during irradiation experiments.

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