The Effect of UV-Irradiation and Molten Medium on the Mechanical and Thermal Properties of Polystyrene–Polycarbonate Blends

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ABSTRACT: Polymer materials with improved properties can be obtained through polymer blends. As a polymer mixture is generally immiscible and incompatible, it is necessary to develop new methods to improve the interfacial adhesion. The aim of this work is to find formulations and associated processes to upgrade engineering polystyrene (PS) and polycarbonate (PC) polymer blends with the objective of using the best "process-formulation" couple. In this study, blends of PS/PC were prepared in molten medium using reactive extrusion after UV-irradiation. The effects of UV-irradiation on some properties of blends under molten medium were investigated by differential scanning calorimetry (DSC), fourier transform infrared (FTIR), and thermogravimetric analysis (TGA). The data showed that the presence of polycarbonate in the blend increased the tensile strength and elongation at break with respect to pure PS. The mechanical properties of the blends were improved after irradiation. All irradiated blends are thermally more stable than those nonirradiated. Chemical changes can be clearly seen in FTIR spectra through two bands assigned to C=O and OH groups. The mutual influence between the PS/PC polymer blends compositions during UV-irradiation was studied. PS and PC have different photo-mechanisms due to the larger UV absorption of polystyrene and formation of more stable tertiary carbon radicals. © 2011 Wiley Periodicals, Inc. J Appl Polym Sci 124: 1096–1105, 2012

Key words: polycarbonate; polystyrene; polymer blends; recycling; extrusion

INTRODUCTION

During the last five decades, study of polymer blends has become a special field of research on polymer materials with the aim of recycling with new properties.[1] Blending of immiscible polymers offers attractive opportunities for developing new materials with useful combinations of properties.[2] A good balance between properties and processability which allows reuse and upgrading is absolutely necessary in a recycled material. In addition to the academic interest in this system, there exists an industrial interest for automotive wastes.[3] Moreover, there has been significant interest in the recycling of plastic materials because of environmental and economical concerns in recent years. Some separation processes based on physical and chemical properties of materials have been proposed but they are difficult and expensive to apply at industrial level in most cases.[4,5] Although increasing the number of polymer components does lead to complications, there are several important reasons for studying and developing new high performance materials arising from synergistic interactions. However, simple blends promote the desired segregation of phases; they are also responsible for high interfacial tension, poor adhesion between the phases with poor mechanical properties and unstable morphologies.[6] Compatibilization of such blends is necessary. The thermodynamic miscibility of most polymer blends is quite limited because of the large size of the components and their weak mutual interaction and, on the other hand, the presence of two different glass transition temperatures (T_g) shows a typical sign of an immiscible or partially miscible system.[7,8] Some partially miscible systems that show large T_g changes, which are indicative of high interactions and as a consequence high solubility, show mechanical properties below the additivity rule (PVC/Polystyrene system).[9]

It is well known that styrene polymers (PS) and engineering plastics as polycarbonate (PC) occupy a large part of the plastic market and are some of the most recycled polymer materials.[10,11] PC has a distinguished role in this context, because it is extensively used in engineering applications, which is due to its relatively high toughness under certain conditions and excellent chemical resistance.[12] PS has been used all over the world due to its excellent

physical properties and low production costs but earlier studies have shown that polystyrene degrades under exposure to sunlight in air through two distinct processes: photolysis and oxidation.[13] Usually, these plastics are used or recycled in blends. It is hard to separate these plastics, respectively, from recycled materials. For this reason, it is important to find experimental conditions for mixing different plastics without losing mechanical properties. A proactive alternative is to generate some copolymers *in situ* during the blend preparation through polymer-polymer grafting reactions using functionalized polymers.[16] However, it is often difficult to produce suitable graft or block copolymers for important commercial applications. Irradiation of polymer materials generates some effects depending on the kind of polymer, as well as on the parameters of irradiation and the state of the material under processing.[14–17] The two main reactions which determine the final properties of the polymer include (a) scission of main chain and (b) crosslinking. The yield of scission and crosslinking or more precisely the mutual ratio of these two parameters, are key parameters for the outcome. To achieve the desired objective, irradiation of polymer material can be conducted under special conditions. It is well known that introduction of chromophore such as carbonyl groups to the conventional polymer chain through copolymerization may facilitate polymer degradation.[18] Absorbed light of wavelength up to about 380 nm causes breaking of chemical bonds and free radicals formed this way initiate the polymer decomposition or other chemical and physical transformations.[19] The primary reaction in photodegraded polymer is mainly abstraction of hydrogen atom from a macromolecule and formation of alkyl radicals. The bulk properties of polystyrene and polycarbonate make them ideal for a wide range of structural and functional applications. However, the low surface energy in many cases limits their use. Thus, the pretreatement or modification using UV-irradiation is frequently used for improvement of polymer properties such as adhesion. In this article, we have studied the influence of the processing on the efficiency of UV-irradiation to compatibilize different blends under molten state. Irradiation was applied on the pellets before injection molding. A variety of techniques have been used to identify the behavior of blends versus the time irradiation to verify the changes that take place in the polymer structure, e.g., thermal analysis technique and mechanical properties. The reactions were carried out in a bench top twin-screw micro-compounder. This technique was chosen for its simplicity. This compound was transformed in the reactor under controlled thermomechanical conditions. The results presented in this paper deal with irradiated modified polymers prepared according to this process.

TABLE I
Formulations of the Polycarbonate and Polystyrene Prepared

Formulation	Polymer	Composition (%)	UV-Irradiation (min)
F1	PC	100	0
F2	PC	100	15
F3	PC	100	30
F4	PC	100	60
F5	PC	100	120
F6	PS	100	0
F7	PS	100	15
F8	PS	100	30
F9	PS	100	60
F10	PS	100	120

EXPERIMENTAL

Materials

Polycarbonate (PC) and Polystyrene (PS) were provided by DSM Engineering Plastics Europe (The Netherlands) coming from electrical and electronic equipment. Both materials were characterized and the obtained results for MFI, density and VST were as follows: For polycarbonate: MFI (230°C/5 kg) = 6.06; density = 1.2 g/cm^3; VST = 139.2°C. For polystyrene: MFI (230°C/5 kg) = 20.45; density = 1.05 g/cm^3; VST = 95.3°C.

Blends and sample preparation

Polycarbonate and polystyrene and their blends were prepared using a 15 g-capacity DSM micro-extruder (Midi 2000 Heerlen, The Netherlands) with corotating screws (Tables I and II). The operating gradient temperature of the extruder was 220°C and the rotational speed of the screws was kept constant at 150 rpm during all the extrusion cycle and injected in a 10 cm^3 mold at 85°C to obtain dumb-bell-shaped specimens. This process was repeated 10 times under the same operating conditions. Samples were taken in every extrusion cycle to evaluate the mechanical properties, ageing test, flow index tests and differential scanning calorimetry (DSC) and thermogravimetry (TGA) tests. For molded pieces prepared from irradiated pellets, the time between irradiation and injection-molding was 3 min. Afterwards the changes in morphology were visualized using scanning electron microscopy (SEM). A Cambridge Stereoscan S 200 was used.

UV-irradiation

The UV-irradiation was carried out using a 125 W low pressure mercury lamp without any optical filter. The UV lamp was positioned at a distance of 15 cm from the sample. An electric fan was used to

TABLE II
Formulations of the Blends Prepared

Formulation	Polymer	Composition (%)	Time irradiation (min) (PC)	Time irradiation (min) (PS)
F11	PC/PS	25/75	0	0
F12	PC/PS	25/75	60	30
F13	PC/PS	75/25	0	0
F14	PC/PS	75/25	60	30

maintain the sample at room temperature during irradiation. The intensity of illumination with this set up was 5555 $Jm^{-2} s^{-1}$. The samples were irradiated for different intervals of time. The measurement of free volume was performed on these irradiated samples using the PAL spectrometer. The PAL spectrometer consists of a fast coincidence system using KL236 plastic scintillator coupled to a RCA-8575 photomultiplier assembly as detector and has a time resolution of 340 ps.

Infrared spectroscopic analysis

FTIR spectra were measured with Nicolet iS10 spectrometer in the absorption mode from Thermo Scientific. Each sample was scanned 32 times from 1900 to 1300 cm^{-1}, with a resolution of 4 cm^{-1}. The experiments were done in triplicate and were collected using the KBr pellet method.

Molecular weight and differential scanning calorimetry measurements

The first characterization technique consisted in the determination of the glass transition temperature (T_g) for each set of irradiation conditions. This was accomplished using differential scanning calorimetry (DSC). The instrument used to conduct these evaluations was a TA Q100 DSC. Samples were prepared by slicing sections of unstressed tensile samples used in the tensile tests. The samples measured $\sim 3 \text{ mm} \times 3 \text{ mm} \times 1 \text{ mm}$ thick. The mass of each sample was between 6 and 10 mg. Samples were heated at a rate of 151°C/min under nitrogen. The T_g for each sample was determined using the output data and TA's built in software.

The second method employed to characterize each irradiation condition was to measure the apparent viscosity of the samples at elevated temperature. The instrument used was a Monsanto MPT Processability Tester. Ten grams of pellets were added to the heated cylinder of the MPT. Samples were held in the cylinder for 10 min to allow them to reach the cylinder temperature. The instrument was then activated to start the test. The instrument was programmed to push a heated piston through the cylinder at a rate of 2.54 cm/min. The material was

extruded. The force on the piston was recorded by the instrument which in turn calculated the apparent viscosity. Each conditions set was evaluated at the same rate temperature of 275°C. Three tests were run for each condition and the results were averaged.

Thermogravimetric analysis measurements

Thermogravimentric analysis (TGA) was carried out using a TGA Q500 instrument. The heating was carried out at temperature range from room temperature to 400°C at heating rate of 10°C/min under nitrogen gas atmosphere.

Mechanical testing

Mechanical properties were determined by means of a universal tensile test machine from Instron. Five individual dumbbell-shaped specimens were cut out from the sheets using a steel die of standard width (6.2 mm). The values were determined at a crosshead speed of 10 mm/min on a rubber tensile testing machine.

RESULTS AND DISCUSSION

Mechanisms

Energy absorbed in the UV region produces changes in the electronic energy of the polymer molecule resulting from the transitions of valence electrons and various physical and chemical processes take place in the polymer. Radiation of wavelength shorter than 350 nm in the UV region of the electromagnetic spectrum has sufficient energy to cleave the bonds between the groups in many polymers,

TABLE III
Variation of Glass Transition Temperatures by DSC for Different Blends Compostions For Various Irradiation Time

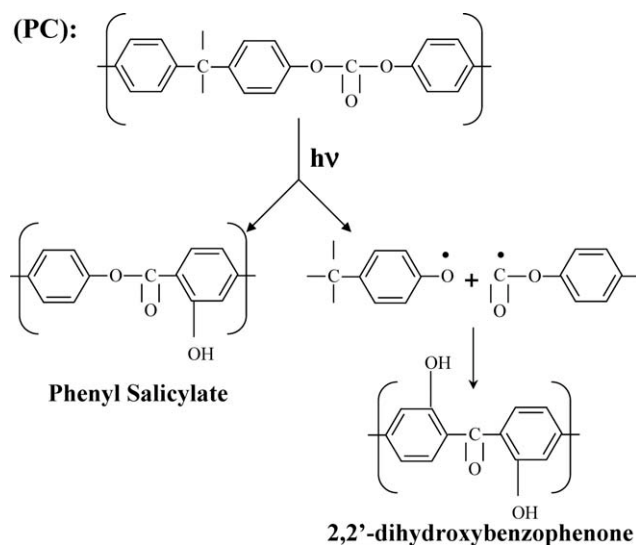| Formulation	T_g (°C) (PC)	T_g (°C) (PS)
F11	134.72	102.19
F12	130.75	96.07
F13	134.53	99.86
F14	128.08	104.18

Figure 1 The mechanism of photo-fries rearrangement in polycarbonate (PC) in UV-irradiated.

thereby producing free radicals.[20] The radicals so produced can initiate further reactions such as crosslinking. The most prominent species are hydrogen, molecular scission products from the end groups, and pendant groups of the polymer. It has been well established that mechanical, physical, and chemical properties changes in polymers are determined by the magnitude of crosslinking and scission and that crosslinking enhances mechanical stability while scission degrades mechanical strength.[21] This provides much of the basis for understanding the materials response to irradiation. The exact effect produced in a polymer depends on the structure of the polymer and on the nature of radiation. In this investigation, we have used Positron Annihilation Lifetime (PAL), a well-established microanalytical tool to characterize the free volume holes in polymers to understand the UV irradiation induced effects on polystyrene and polycarbonate. The samples were irradiated for different intervals of time.

The chemistry of degradation processes in polycarbonates has been studied extensively over the past few decades; however, what is happening under exposures is still under debate, since most of these studies were done under different exposure conditions. In (PC), the chemistry underlying the photodegradation has been described to two different mechanisms: photo-fries rearrangement and photo-oxidation (side chain and ring oxidations). It is suggested that during the initiation of the photodegradation process, the photo-fries rearrangement reaction is the source for free radicals. In the presence of oxygen, formed radicals lead to photo-labile oxidation products such as hydroperoxides and aromatic ketones.[22,23] The hydroperoxides can initiate new oxidation cycles causing autocatalytic photo-

oxidation. The relative importance of these mechanisms depends on the used irradiation wavelengths. Lemaire et al.[24] illustrated that the photo-fries rearrangement reaction is more likely to occur when light with wavelenghts shorter than 300 nm is used, whereas photo-oxidation reactions are more important when light of longer wavelengths (>340 nm) is used. Irradiation of the polymer (PC) with short wavelenghts causes the aromatic carbonate unit to rearrange itself into phenylsalicylate and dihydroxybenzophenone derivatives (Fig. 1). This reaction can either be a concerted or a radical process. In this mechanism, an initiating radical is required to start this autocatalytic oxidation process. Up till now, it is not clear where this radical originates from. The irradiation of polycarbonate were analyzed with FTIR. With this technique, we have examined changes in chemistry during the degradation. In Figure 2, the IR spectra of samples with different irradiation times are displayed. As the irradiation time increases, absorption bands are formed at different wavelengths. The absorption band at 1713 cm^{-1} is assigned to aliphatic chain an acid which is a typical oxidation band. In literature,[25] the phenylsalicylate and dihydroxybenzophenone bands are assigned to the vibration band at 1689 cm^{-1} and 1629 cm^{-1}, respectively. From the increase of the absorption at 1629 cm^{-1} and 1689 cm^{-1} it was suggested that photo-fries products might be present in the irradiated formulations. However, it is also likely that the increase is due to band broadening of the oxidation band at 1713 cm^{-1}.

The PS was exposed to UV light and, as expected, all of them presented alteration in the FTIR spectra as a function of time (Fig. 3). The discussion of the results will focus on the absorption evolution in the
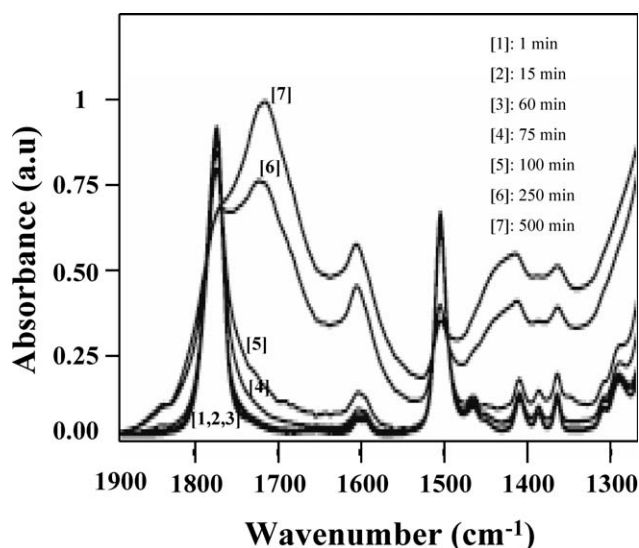

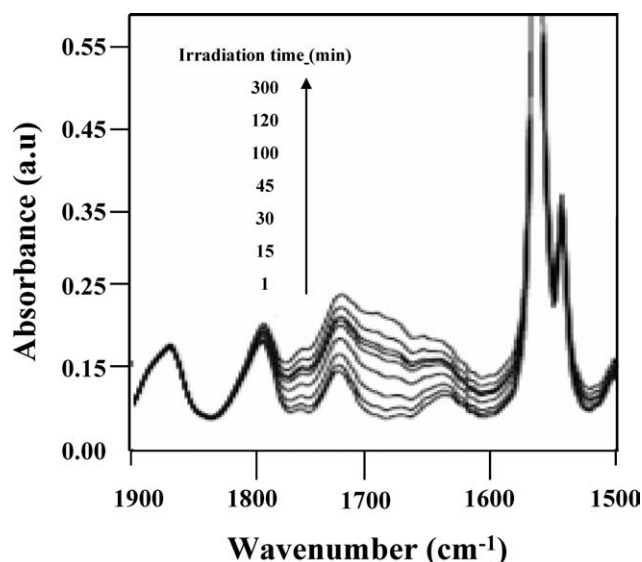**Figure 2** FTIR spectra of irradiated PC in the carbonyl region for various irradiation times.

Figure 3 FT-IR spectra of irradiated (PS) for various irradiation times.

C=O stretching range (assigned to carbonyls), close to 1700 cm^{-1}. The evolution of carbonyl formation for the irradiated PS is represented in Figure 3 as a function of time. This happens due to the larger UV radiation absorption for PS which possesses an aromatic ring in each monomer unit and acetophenone as degradation product, both are chromophore groups. The main initial reactions in UV-irradiated polystyrene were described in Figure 4.

However, the free volume increases in the early stages of UV exposure and thereafter shows a slight decrease for both polymers PC and PS (Figs. 5 and 6). These interesting changes can be explained in the following way. Polycarbonate contains a carbonyl chromophore which has a weak absorption band resulting from the forbidden transition in the 270–
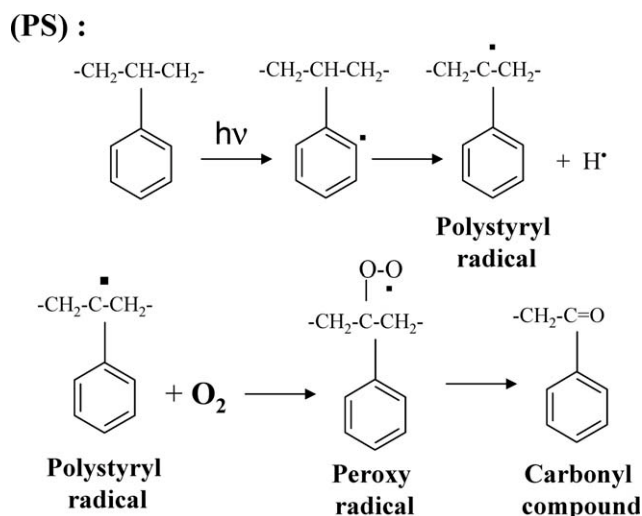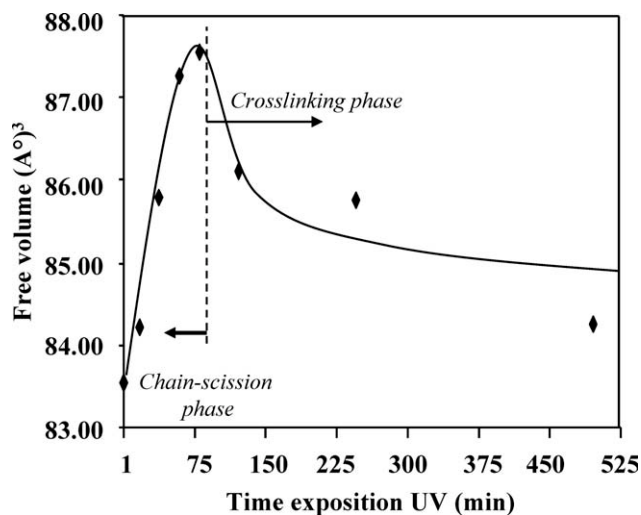

Figure 5 Variation of free volume as a function of UV exposure time for polycarbonate (PC).

300 nm range. Further, the two benzene chromophores have a weak absorption each at 256 nm. The average free volume size in (PC) varies from 83.5 to 87.5 A° under UV irradiation. The initial increase in free volume is due to chain-scissions mainly at the C—O bonds adjacent to the carbonyl group. An earlier investigation by Zimmermann et al.[26] reports that the C—O bonds adjacent to the carbonyl group in the PC chain are the vulnerable bonds, laking the resonance stabilization of the phenyl group. Initially when chain-scission occurs, the available free volume increases because of the rearrangement of the molecules due to chain-scission. Thus, the increase in free volume in the initial stages is essentially due to the increase in the size of the free volume present in the disordered regions. This also indicates that the local electron density near the polymer sites gets
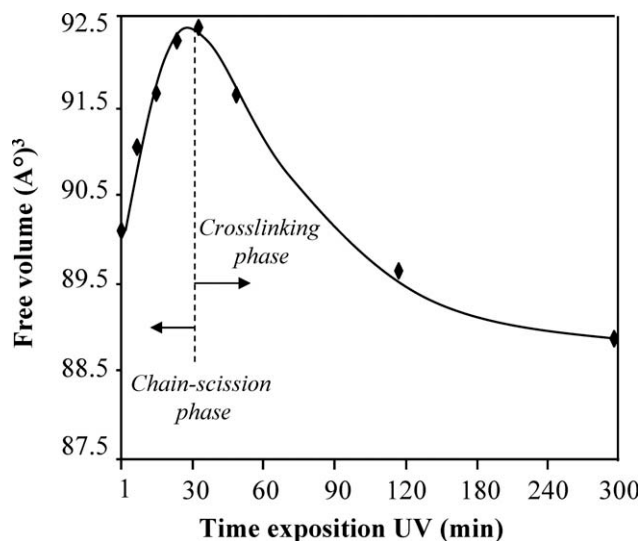

Figure 4 The main initial reactions in UV-irradiated polystyrene.

Figure 6 Variation of free volume as a function of UV exposure time for polystyrene (PS).
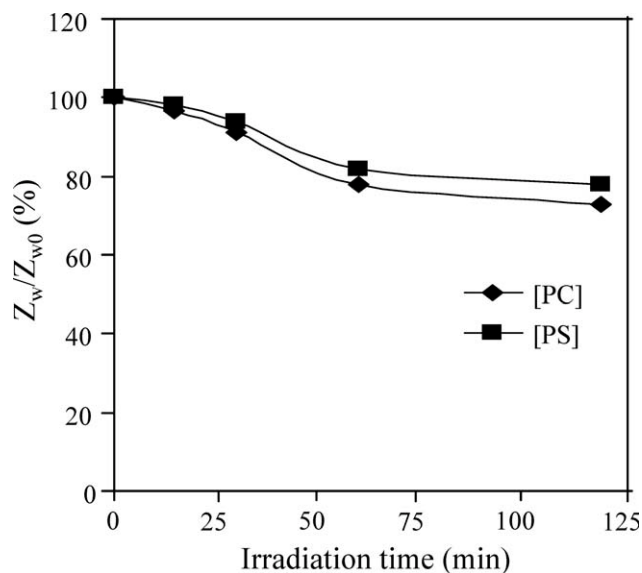
Figure 7 Percentage of molecular weight with respect to a control sample as a function of irradiation time for PC and PS samples.

reduced. After long time of exposures, there is a slight decrease in the size of the free volume perhaps due to mild crosslinking. In the crosslinked state, the polymer chains are expected to have a closer packing hence a reduction in free volume is observed at this stage as seen in Figures 5 and 6.

We can conclude that the results seem to indicate that chain-scission leading to free radical formation is the dominant process in the early stages of UV exposition. The crosslinking reaction occurs after chain-scission in both polymers.

Molecular weight and thermal properties

Melt viscosity analysis was used to characterize the change in molecular weight. The most important structural variable determining the flow properties of polymers is the molecular weight Z. It was well established (Fox et al., 1956) for essentially all polymers studied that for values of Z above a critical value Z_c (which is about 600 for many polymers):

$$\log \eta = 3.4 \, \log Z_w + k$$

Where Z is the melt viscosity, and Z_w is the average molecular weight. Figure 7 displays the relationship between the ratio of average molecular weight with respect to control and time irradiation. It is easily noticed that decrease in molecular weight happens after 60 min indicating that chain scission is a mechanism at higher time irradiation. DSC was also used to characterize the possible evolution of thermal properties (Fig. 8). This study has focused on the analysis of PC and PS blends to estimate the interac-

tion between binary pairs, where composition resins and UV irradiation are decisive on the miscibility study. Both polymers were subjected to the same processing cycle and they were exposed to UV irradiation conditions. In this work, thermal behavior of blends was analyzed to relate macroscopic properties with microscopic behavior. As the work is carried out with amorphous materials, information about interaction phenomena between components is restricted to the study of changes on T_g since the system does not display melting peaks which allow to observe annealing effects typical of PC and PS that can influence mechanical properties. Regarding changes on T_g values, we can observe that PS/PC system displays two glass transitions for all the compositions range (Fig. 9). The dependence with composition indicates certain interaction between components. If the system is immiscible, T_g values of individual polymers would not change; otherwise if ?A3B2 tlsb?> the system is totally miscible, it would display only one T_g. In our case, PC/PS system shows an intermediate behavior, since we can observe two changing T_g values in the whole composition range (Table II). When compared with similar blends before irradiation with amorphous materials, changes in temperature T_g was near to 5°C. This partial miscibility does not imply a phase differentiation sense but we obtained a material with the coexistence of PC and PS. In all PC and PS samples, both processed by extrusion and injection, the DSC results show a unique glass transition temperature at 135.07°C and 103.07°C, respectively. The temperature T_g values decreased slightly as the time of UV exposition increased, due to the already observed decrease in the molecular weight for all the samples during the process. Moreover, these values are in agreement with the behavior observed for the polystyrene
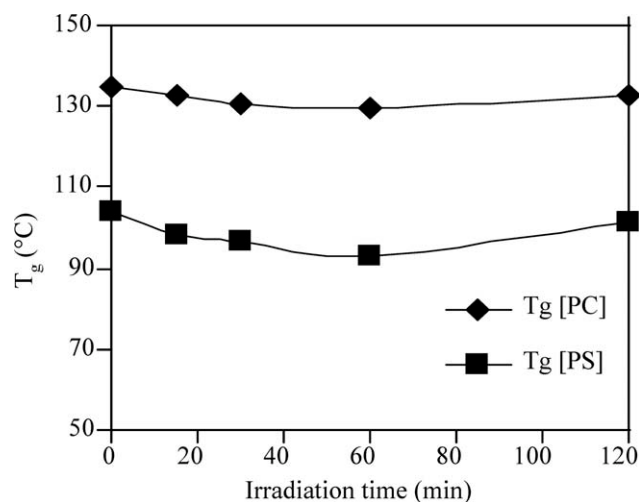

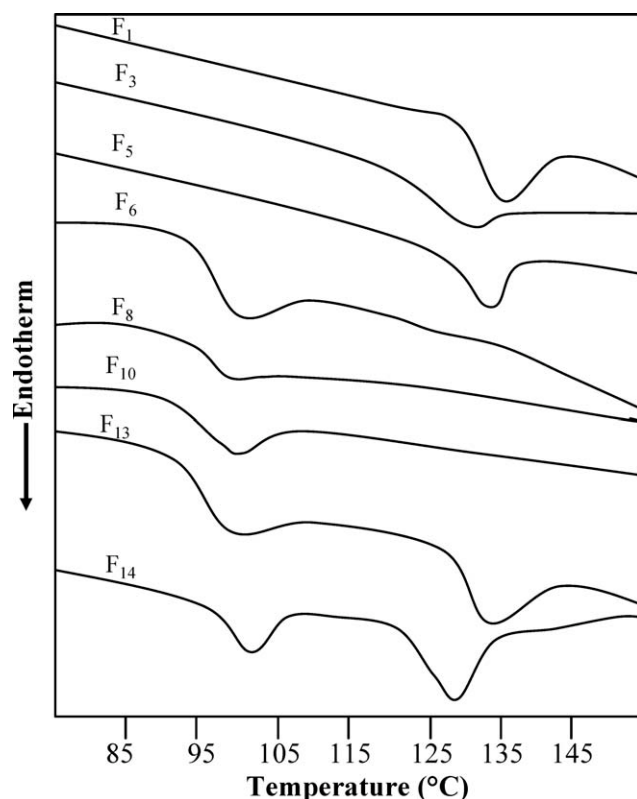**Figure 8** Glass transition temperature as a function of irradiation time for PC and PS samples.

Figure 9 Comparison of DSC curves of different blends compositions for various irradiation time.

fractions i.e., in both cases and an increase of the glass transition temperature took place as the molecular weight increased.

Claude et al.[27] studied the impact of photochemical modifications happened under UV irradiation upon the physical properties of an aromatic polycarbonate, observing that the glass transition temperature, defined at the surface of the irradiated samples, decreased continuously. They attributed this phenomenon to a decrease in the chain mobility. The

formation of crosslinking would happen and depend on the wavelengths of irradiation and on the atmosphere. At short wavelengths and under vacuum, the formation of a highly crosslinked structure, almost depleted of carbonate functionalities, was noticed. Webb and Czanderna[28] postulated that this crosslinked structure could be the result of photophysical interactions between carbonate and phenol groups. These interactions involved an energy transfer from the phenolic group to the adjacent hydrogen-bonded carbonyl and consequently an abstraction of the phenolic proton by the excited carbonyl. Moreover, Ramani and Ranganathaiah[29] in a viscosity measurement on UV-irradiated PC report the change in molecular weight due to chain scission and partial crosslinking. However, from our data we can conclude that the glass transition temperature of the samples seems to depend on the exposition time under UV-irradiation.

Thermal stability by TGA

Thermal stability analysis of polycarbonate, polystyrene, and their blends exposed to UV-irradiation was determined using thermogravimetric analysis in nitrogen (TGA). The thermal decomposition occurs in two stages: the first (minor) at about 80–160°C and the second (dominant) at 320–430°C. The first stage of thermal decomposition below 160°C is connected with a small drop of weight that is usually caused by elimination of low-volatility impurities. The main important stage of thermal decimposition in PS starts at 326°C and corresponds to the breaking of chemical bonds and evolution of radical or molecular products. From the TGA data of no irradiated and irradiated formulation, presented in Figure 10, weight losses are reported. The data gives the percent weight loss of the specimen remained.
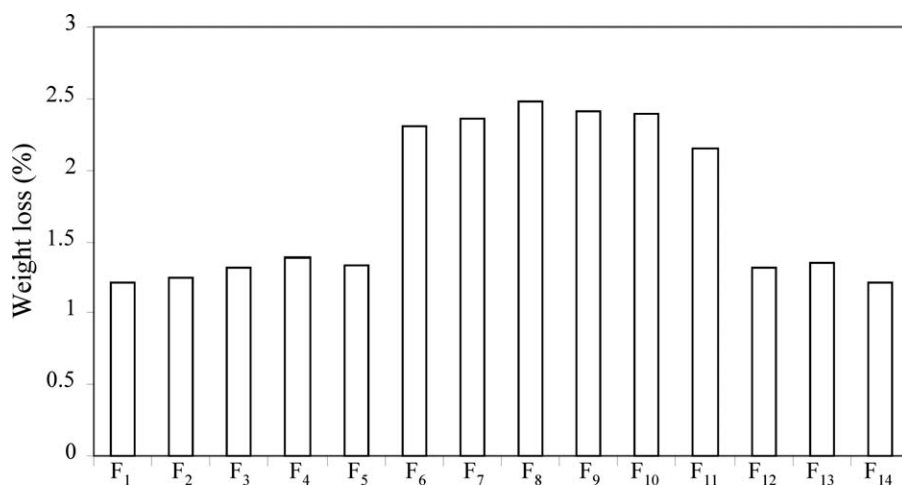

Figure 10 Weight loss measured by TGA for the PS and PC with different blends compositions for various irradiation time.
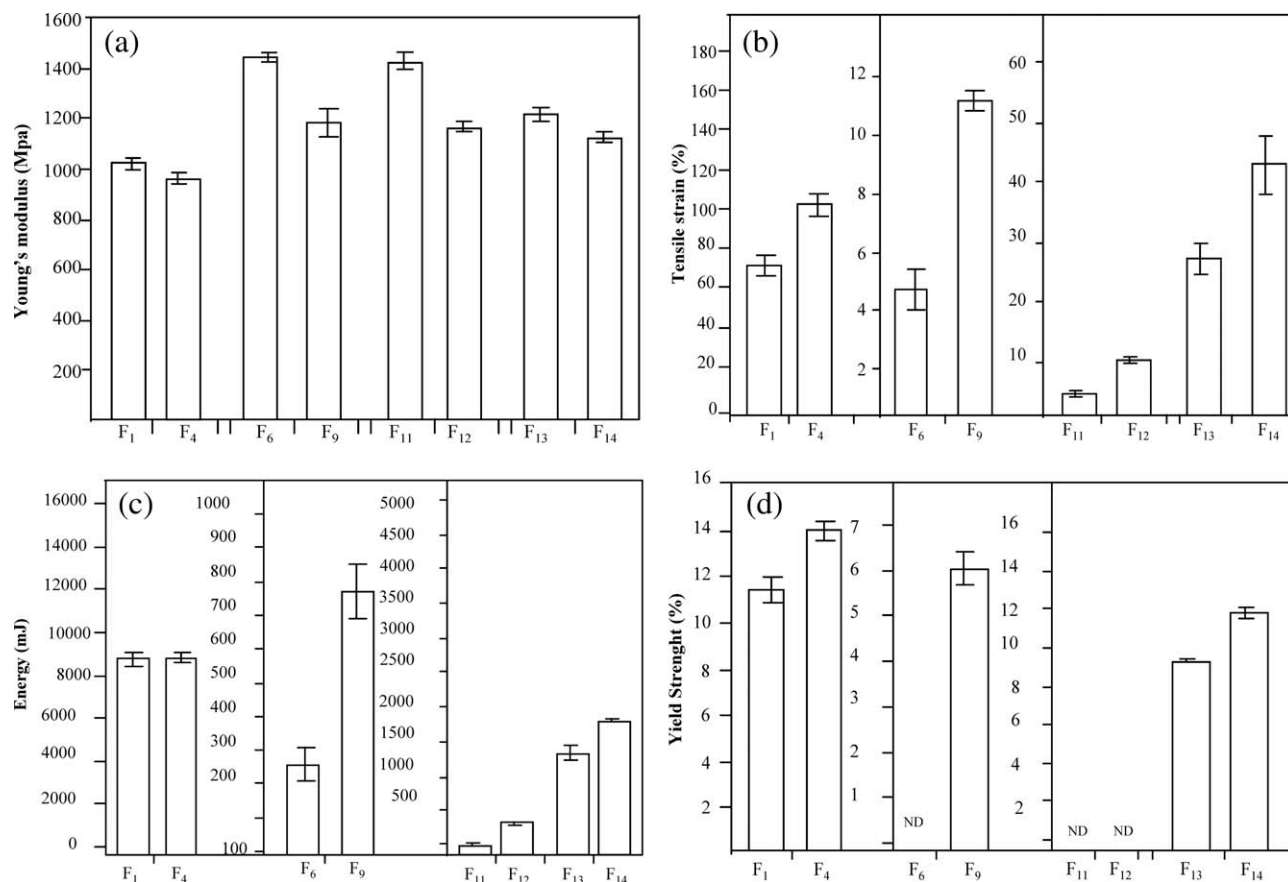
Figure 11 (a) Effect of UV-irradiation and molten medium on the tensile strain of PS, PC, and PS/PC blends. (b) Effect of UV-irradiation and molten medium on the energy (mJ) of PS, PC, and PS/PC blends.

Weight loss of a polymer due to thermal degradation is largely related to oxidation whereby the molecular bonds of a polymer are attacked by oxygen molecules. There is a gradual increase in weight loss as irradiation time increased up to 60 min. A gradual and regular increase in weight loss can be noticed indicating a decrease in thermal stability. This decrease was less important after 60 min maybe due to the crosslinking process. Therefore, from the point of view of its thermal stability, the material could be recycled more or less times depending on the final requirements. This result was expected since an increase in free volume is indicative of low molecular weights and, it was already observed, in both types of polymers. A similar manner was observed for irradiated blends, but interestingly, found to possess better stability compared to all other blends no irradiated. Generally it is accepted that the increase of thermal stability can be achieved by crosslinking which take place in both polymers.

Mechanical properties

This study has been carried out only with the polycarbonate (F_1, F_4), polystyrene (F_6, F_9) and their blends (F_{11}, F_{12}, $F_{13,}$ and F_{14}) processed by injection.

Figure 11(a–d) show the modulus of elasticity, tensile, yield strength and energy values. The mechanical properties parameters were practically affected by UV-irradiation.

Polycarbonate is a tough material but shows brittle behavior in thick-walled applications. It is also notch sensitive, as it fractures brittle when a notch is present. The polystyrene itself does not have very good toughness. The presence of PS in the blend (PC, PS) decreased the tensile strength and elongation at break as with respect to pure PC. This reduction is yet more accentuated with the introduction of 75% of polystyrene. This behavior was partially expected since the tensile strength values of PS are inferior to those of PC. However, the significant reduction of the elongation at break of the blend reveals the incompatibility of this system. The toughness reaches an optimum only when the PC content is around 75% in the blend.

UV-irradiation can improve the toughness of the blends (PC, PS) and the young's modulus decreases slightly. The addition of polystyrene to polycarbonate does not depress the mechanical properties due to the fact that each polymer was exposed to UV irradiation before being melted in the reactive extrusion. The tensile strength at break and the energy *J* increase to
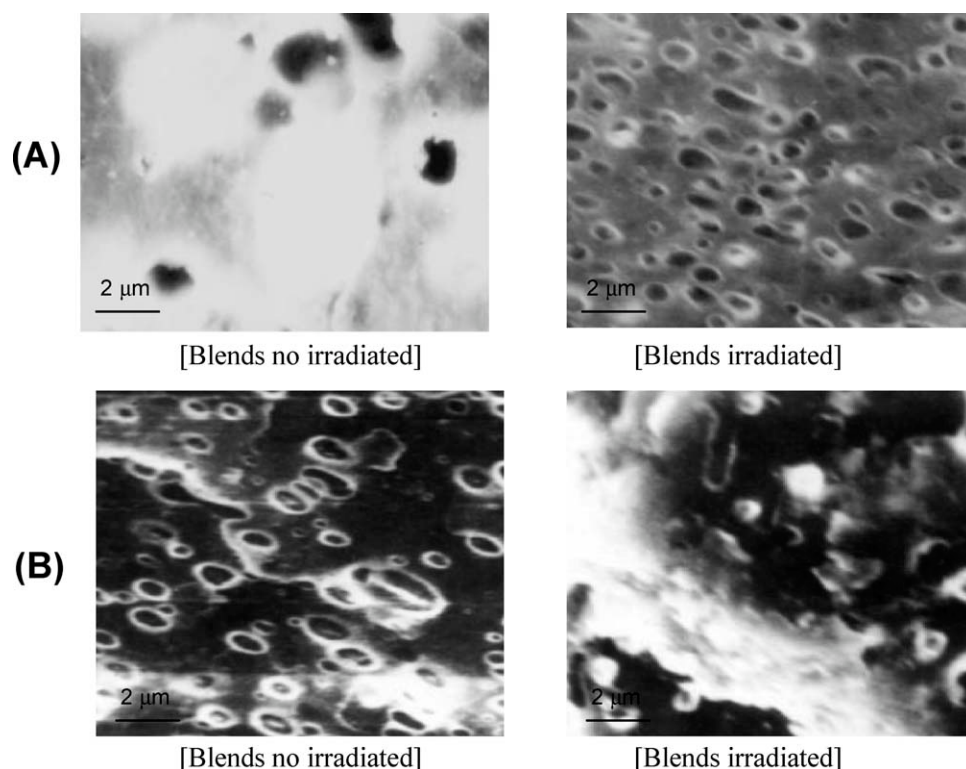
Figure 12 (a) Effect of UV-irradiation and molten medium on the Young's modulus of PS, PC, and PS/PC blends. (d) Effect of UV-irradiationand molten medium on the yield strength of PS, PC and PS/PC blends. ND, not detectable. A: SEM of 75PS/25PC blends, room temperature fracture surface, B: SEM of 25PS/75PC blends, cyclohexane extraction of PS phases.

some extent, so there might be a certain enhancement of interfacial adhesion between PS and PC.

This suggests that mechanical properties are clearly affected by the presence of PC level in the blend and the degradation occurred during irradiation. The increase in mechanical properties observed can be attributed to reaction of radicals between polystyrene and polycarbonate in molten medium which could increase the adhesion between PS and PC.

Scanning electron microscopy

A deep insight into the interfacial situation is provided by SEM of fracture surfaces prepared at room temperature. The lack of adhesion between PC and PS is obvious from the examination of Figure 12, wherein dispersed particles do not adhere to the matrix and leave cavities with a smooth (clear) surface. This phase morphology is strikingly modified after irradiation. The minor component (PS) no longer forms dispersed particles, but a continuous and very thin network firmly anchored into the PC matrix is observed.

CONCLUSIONS

The aricle describes the mechanical and thermal behavior of an immiscible binary blend of the poly-carbonate (PC) and polystyrene (PS) through the UV-irradiation and reactive extrusion.

On the basis of the obtained results the following conclusions were drawn:

(a) When irradiation is performed and is followed by extrusion in molten medium, the free radicals could react: the effect of irradiation is enhanced and lower irradiation times are sufficient. The process is more cost-efficient.
(b) Numerous radicals created by irradiation could react and enhance the modification of structure if a new processing step is performed. The crosslinking by combination of two macroradicals is an intermolecular reaction which depends on radical mobility. Irradiation could lead to a new material in the point of view of the structure and mechanical behavior.
(c) The lack of miscibility will be observed both by the presence of two different T_g and lower mechanical properties. The importance of T_g variation is not indicative of a good compatibility since this variation can be explained by simple physical phenomena. All irradiated formulation is thermally more stable compared with those of nonirradiated polymers.

(d) The presence of PS in the blend decreased the tensile strength and elongation at break as compared with pure PC. The increase in mechanical properties was observed after UV irradiation. The composition 25PS-75PC is most interesting to obtain an industrial material with balanced properties, for different reasons: first, mechanical ductile properties do not increase and second, processing conditions are similar to other styrenic derivatives.

(e) It is possible to conclude the extrusion; injection molding processes and γ-irradiation are effective enough to obtain homogenous blends since heating processes ensure good mixing conditions and final macroscopic homogeneity. Chemical changes can be seeing clearly in FTIR.

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