Fire Retardancy, Thermomechanical and Thermal Properties of PP/PC Blends

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This work investigates the fire retardancy, the thermal and thermomechanical properties of PP/PC blends using commercially available grafted polyolefins with functional groups as compatibilizing agent. In the first part, the effect of compatibilizer on the morphology has been correlated with the thermomechanical properties. The blends, containing 75% of PP and 25% of PC or 22% PC plus 3% compatibilizer have been prepared during the melt processing in a mixer. The compatibilizers were grafted maleic anhydride polypropylene (PP-g-MA) and a terpolymer ethylene/acrylic ester/maleic anhydride (EBuAMA). The thermal stability was improved up to 400°C when using compatibilizer in comparison with PP/PC blend. The thermomechanical properties were improved for PP/PC/PP-g-MA blend. It was found that the addition of compatibilizer slightly decrease the particle size of the minor PC phase. The crystallization temperature of PP in PP/PC/PP-g-MA increases which may explained the improvement of the properties of the blend in comparison with PP/PC/EBuAMA. In a second part, fire retardant formulation are developed. The fire retardant properties of the blend are slowly affected by the presence of a compatibilizer.

Keywords polypropylene/polycarbonate blend, thermomechanical properties, thermal stability, fire retardancy, intumescence

Introduction

To upgrade some properties of polyolefins, a well-known method consists in blending polyolefins with engineering plastics such as polyamide or polycarbonate. However, because of their non-polar structure, polyolefins are incompatible with polar polymers which strongly limit their use in blends. In order to achieve suitable morphology and to improve interfacial adhesion between the blend components, it is necessary to compa-

tibilize them (1–7). The compatibilizers across the interface control the physical and chemical interactions between the immiscible polymers of the blend reducing the interfacial tension and increasing phase adhesion.

Blends of polypropylene (PP) and polyamide 6 (PA 6) have been the subject of a large number of studies (8–10). In these systems, polypropylene functionalized with maleic anhydride (MA) is often used as a compatibilizing agent. The structural stability and morphology of the PP/PA 6 blends are greatly improved by PP–PA6 grafted copolymers that are formed by the *in situ* reaction of anhydride groups with the amino end groups of PA6.

More recently, PP and polycarbonate (PC) blends have been investigated (11–17). Favis and co-workers have demonstrated that the size and shape of PC domains in the blend are closely linked with the processing conditions, as well as on the contribution of particular components. The use of PP grafted with glycidyl methacrylate (GMA) as a compatibilizer during the melt processing has been investigated by Zhihui and co-workers. It has been reported that using PP-g-GMA as a compatibilizer makes it possible to significantly decrease the size of the PC domains and leads to a change in the crystal form of PP (from α and β into unique α crystal form). Moreover, it has been shown that PP-g-GMA acts as a nucleating agent in PP enhancing the crystallization temperature of PP. Tang and co-workers have studied the use of functionalized polyolefin elastomer (FPOE) as a compatibilizer in PP/PC blends. The authors show that with the addition of FPOE, the size of the PC domains is reduced and interaction between different phases are greatly improved.

It is generally accepted that olefins based co- and graft polymers with functional groups may act as compatibilizers between polyolefins and high performance engineering polymers. In particular, grafted maleic anhydride or grafted acrylic acid polyolefins are widely used and commercially available.

On the other hand, the use of polymer blends to develop intumescent fire retardant systems is particularly interesting. Previous work done in our laboratory (18–21) demonstrates the effectiveness of the concept. Intumescent systems are based on the development when heated with a charred layer acting as a physical barrier on the surface of the material, which slows down heat and mass transfer between the gas and condensed phase (22). To develop such systems, three ingredients are needed: a carbonization agent, an acid source, and a blowing agent. Polyols are widely used as a carbonization agent, but present a number of limitations (exudation, low thermal stability,...), that is a break to the development of intumescent polymers. The use of a polymer as carbonization appear as an interesting way to solve this problem. Polyamide (18) and polyurethanes (21) have previously demonstrated high efficiency in PP-based polymer, whereas polycarbonates have not yet been investigated.

The aim of the present work is first to investigate the thermal and thermomechanical properties of PP/PC blends compatibilized with commercially available grafted polypropylene and, second to develop intumescent fire retardant PP-based materials. The blends, containing 75% of PP and 25% of PC or 22% PC plus 3% compatibilizer have been prepared during the melt processing in a mixer. The compatibilizers were grafted maleic anhydride polypropylene (PP-g-MA) and a terpolymer ethylene/acrylic ester/maleic anhydride (EBuAMA). The thermal and thermomechanical properties are first studied using thermogravimetric analyses (TGA) and dynamic mechanical analyses (DMA), respectively. Then, the morphological properties are investigated using differential scanning calorimetry (DSC), scanning electron microscopy (SEM), and wide angle X-ray diffraction analyses. In a second part, the fire retardant performance of intumescent PP-based materials are investigated.

Experimental

Materials and Blends Preparation

The PP used was a commercial product (PPH7060–homopolymer) from Atofina with MFI = 12 g/10 min. The PC used was a commercial product (Lexan 151 R) from GEP. Two compatibilizing agents have been used: a terpolymer ethylene/acrylic ester/maleic anhydride EBuAMA (Lotader 3410) supplied by Atofina with MFI = 5 g/10 min; a PP functionalized with maleic anhydride PP-g-MA (Polybond 3150) supplied by Uniroyal Chemical with MFI = 50 g/10 min.

The blends of PP and PC were melt mixed at 230°C in a Brabender internal mixer with a rotor speed of 50 rpm for 10 min. The compositions of the blends were PP/PC 75/25, PP/PC/PP-g-MA 75/22/3, and PP/PC/EBuAMA 75/22/3.

Intumescent Fire Retardant PP-based Materials

The ammonium polyphosphate (APP) used in this study was a commercial product (Exolit AP422) from Clariant presenting a crystalline form II and a soluble fraction in water <1%.

The materials were melt mixed at 230°C in a Brabender internal mixer with a rotor speed of 50 rpm for 10 min. The compositions of the materials were PP/APP 70/30, PC/APP 70/30, PP/PC/APP 53/17/30, PP/PC/PP-g-MA/APP 53/15/2/30, and PP/PC/EBuAMA/APP 53/15/2/30.

Thermogravimetric Analysis

Thermogravimetric analysis (TGA) were carried out in a Setaram thermobalance (Model TGA 92) under a flow of synthetic air (Air Liquide grade) at a heating rate of $10°C \cdot min^{-1}$. Samples (about 10 mg) were positioned in open vitreous silica pans.

Diffrential Scanning Calorimetry

Differential scanning calorimetry (DSC) measurements were performed using a Setaram DSC 92 under nitrogen atmosphere. The samples (about 10 mg) were heated from 30 to 250°C at a heating rate of $3°C \cdot min^{-1}$. The temperature was calibrated using standards (indium, bismuth, zinc, and lead) supplied by Setaram.

Dynamic Mechanical Analysis

The dynamic mechanical properties were studied on a compression-molded bar $(40 \times 4 \times 1 \, mm^3)$ with a Metravib dynamic mechanical analyzer in the tensile mode. The samples were tested from −40 to 140°C at a heating rate of $3°C \cdot min^{-1}$ and a frequency of 1 Hz.

Blend Morphology

The morphology was studied using a scanning electron microscope (Jeol JSM 5300 apparatus). Samples were fractured under liquid nitrogen and the fractured surfaces were coated with gold to avoid charge by electron beam. The accelerating voltage was 20 kV.

Crystalline Structure

Structure investigation were carried out by means of wide-angle X-ray scattering (WAXS) using CuKα radiation ($\lambda = 15418$ Å).

Fire Testing

Limiting Oxygen Index (LOI) was measured using a Stanton Redcroft instrument on sheets ($100 \times 10 \times 3$ mm^3) according to ASTM 2863 (23).

UL-94 tests were carried out on $127 \times 12.7 \times 3$ mm^3 sheets according to the UL-94 (24).

Heat Release Rates (HRR) are measured using oxygen consumption calorimetry. Samples are exposed to a Stanton Redcroft Cone Calorimeter according to ASTM 1356-90 and ISO 5660 under a heat flux of 50 kW/m^2, which corresponds to the heat evolved during a fire (25). Conventional data (Heat Release Rate (HRR), Total Heat Release (THR), CO and CO_2 production, Total Smoke Rate (TSR), and residual weight (RW)) are obtained using a software developed in our laboratory. When measured under 50 kW/m^2, all data are reproducible to within $\pm 10\%$. The results presented in the following are averages from two experiments.

Results and Discussion

Thermal Degradation

The TG curves and the first derivative curves of PP, PC and PP/PC blends are shown in Figure 1. Characteristic degradation temperatures obtained from these thermograms are summarized in Table 1.

Base Polymers

The thermo-oxidative decomposition of PP occurs in one step with an initial degradation temperature T_i of 240°C. The thermal decomposition of PP has been extensively studied (26–28) and in the presence of oxygen, the well-known initiation (28), propagation and termination sequences occur as presented in Figure 2. Once an alkyl radical is formed, it will react with oxygen, even at very low partial pressures, to give the alkylperoxy radical. This is converted by radical exchange to a hydroperoxide, which in turn will cleave to an alkoxy radical. In PP, fragmentation of the tertiary alkoxide radical to a ketone occurs. This regenerates a primary alkyl radica, which, after rearrangement, can once again take part in the original scheme. Polycarbonate exhibits a three-step thermal decomposition process. The major step of decomposition occurs between 400°C and 550°C and two overlapped steps are observed successively between 550°C and 600°C and between 600°C and 680°C. It has been previously reported (28) that the degradation of polycarbonate occurs in three stages; oxidative degradation above 310°C, depolymerization between 340°C and 380°C with a characteristic endothermic peak, and thermal degradation above 500°C, which yields aromatic hydrocarbons, phenolic compounds, and tar. The oxidative degradation (29) involves at least oxidation and decomposition of the isopropenyl groups and cross-linking reactions through the oxidative coupling of bisphenol A units to form biphenyl groups.

Blends PP/PC

When PP and PC are blended together without any compatibilizer, smaller values of T_i and T_{max} are achieved. The blends compatibilized with PP-g-MA and EBuAMA also
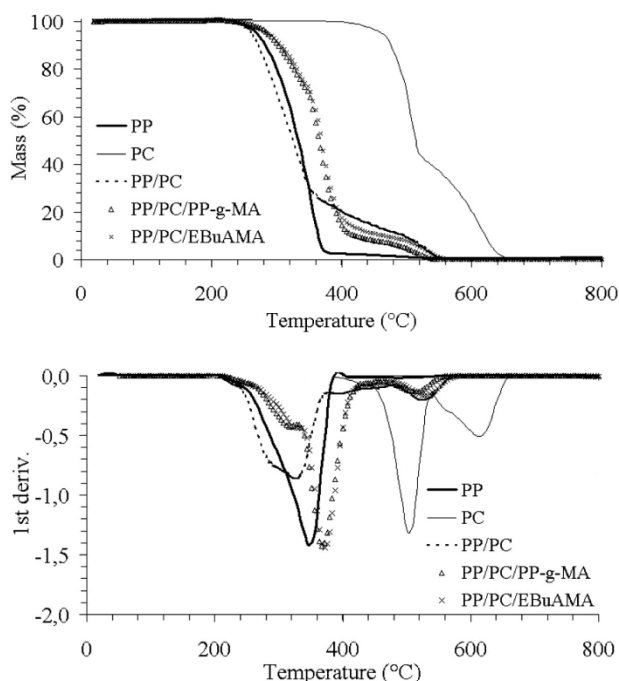
Figure 1. TG and first derivative curves of PP, PC and blends PP/PC, PP/PC/PP-g-MA, PP/PC/EBuAMA.

exhibit smaller values of T_i whereas the T_{max} increase marginally (respectively, 370 and 375°C). The first step of decomposition may be attributed to the degradation of PP since the temperature T_{1max} for pure PP is 350°C and 320°C for PP/PC blend without any compatibilizer and 370°C for PP/PC with compatibilizer. The thermal decomposition of PP and PC does not overlap. The final stage of blend degradation occurs between 400 and 560°C and may be assigned to PC component degradation in blend. In the blends, PC component degrades in a single step with a maximal decomposition temperature T_{2max} between the two decomposition temperatures of pure PC.

Table 1
Parameters evaluated from the TGA and DTG curves of PP, PC and blends PP/PC, PP/PC/PP-g-MA and PP/PC/EBuAMA

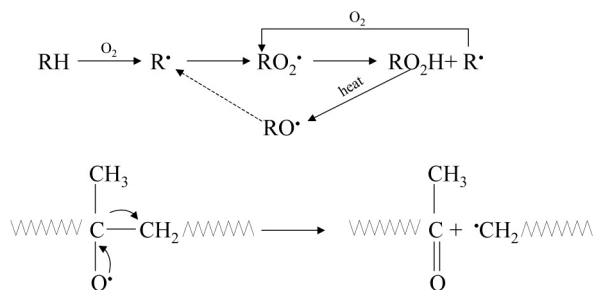| System	Initial decomposition temperature T_i (°C)	Maximal decomposition temperature (°C)		Final decomposition temperature T_f (°C)
		T_{1max}	T_{2max}	
PP	240	350	—	380
PC	400	500	610	650
PP/PC	220	320	530	550
PP/PC/PP-g-MA	230	370	510	540
PP/PC/EBuAMA	230	375	530	560

Figure 2. Mechanism of degradation of PP.

Figure 3 shows comparison between the experimental curves and the theoretical curves. Those curves have been constructed by adding the weighted contributions of each pure polymer curve. In this way, the theoretical curve represents one, which should be expected from the system when no interactions are present between the blend components. The PP/PC blend shows a lower stability than expected. This has already been observed in the literature and attributed to the incompatibility of the system leading to a very poor interaction between the phases (30, 31). For the PP/PC blends with compatibilizer, the thermal stability is higher up to 400°C. Such an increase in the thermal stability may be attributed to an increase in the adhesion between the components of the blend as previously reported for other systems (30). It may be due to the formation of a new material formed by chemical and/or physical interactions between the blend constituents. As an example, the polyolefins grafted with highly electrophilic MA moiety can react readily with the nucleophilic amine end groups of polyamides to form amic acid, followed by ring closure to form a thermally stable imide at the elevated temperature under normal melt processes. In order to check if chemical reactions occur between the components of the PP/PC blends, FTIR analysis is carried out and reported in Figure 4. It clearly appears that there is no additional peak on the spectra of the compatibilized blend in comparison with the PP/PC blend. As a consequence, the increase in thermal stability in the PP/PC/PP-g-MA and PP/PC/EBuAMA materials is somewhat attributed to a difference in morphology of the blends rather than in strong chemical interactions. This will be discussed in a latter part of this paper.

Thermomechanical Stability for PP/PC Blends

Dynamic mechanical analysis is often used to study the relaxation of the polymer. Visco-elastic spectra of PP, PC and blends in terms of storage modulus E' and tan δ are given in Figure 5. At lower temperatures, E' has a greater value for pure PP and ternary blend PP/PC/PP-g-MA when compared with PC and other blends (Table 2). Pure PC presents the steadiest value of E' between −40°C and 140°C. From ambient temperature to 140°C. At T = 100°C, the storage modulus (E') had a smaller value for the PP and PP/PC/EBuAMA when compared with PP/PC/PP-g-MA, and PC. The highest E' value is for the PC. Apparently, the two compatibilized blends do not behave identically.

For pure PP, the main relaxation of PP attributed to β-transition, appears around 3°C (T$_g$ PP). PP also presents an α-transition around 120°C (32). For PP/PC and PP/PC/EBuAMA blends, the T$_g$ (PP) shifts to smaller temperatures (Figure 5). On the contrary, T$_g$ for the blend PP/PC/PP-g-MA displaces to higher temperature (Table 2). The blend with PP-g-MA also presents the smaller decrease in storage modulus after PP relaxation.
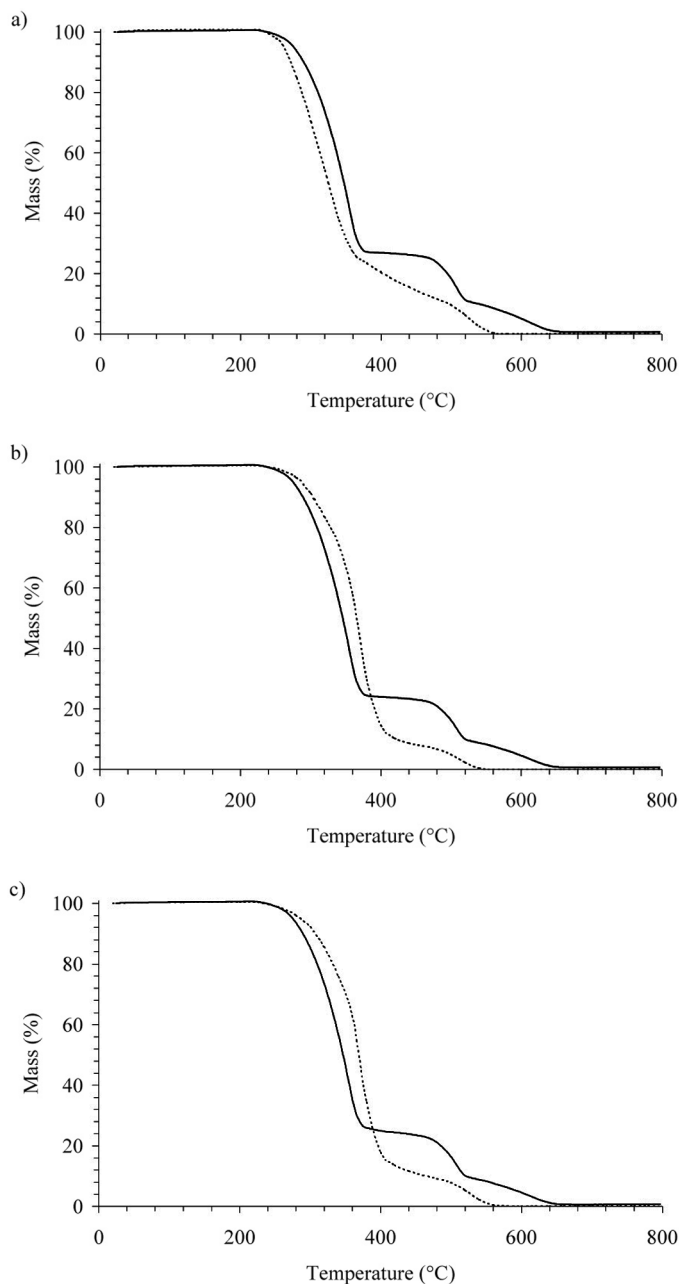
Figure 3. Experimental (– – –) and theoretical (——) TGA curves of PP/PC (a), PP/PC/PP-g-MA (b), and PP/PC/EBuAMA (c).

Morphology and Structure

The effect of the type of compatibilizer can be seen comparing the morphologies of the blends. Figure 6 shows blends with and without the presence of the two compatibilizing agents used. Figure 6(a) displays the micrograph of the noncompatibilized blend
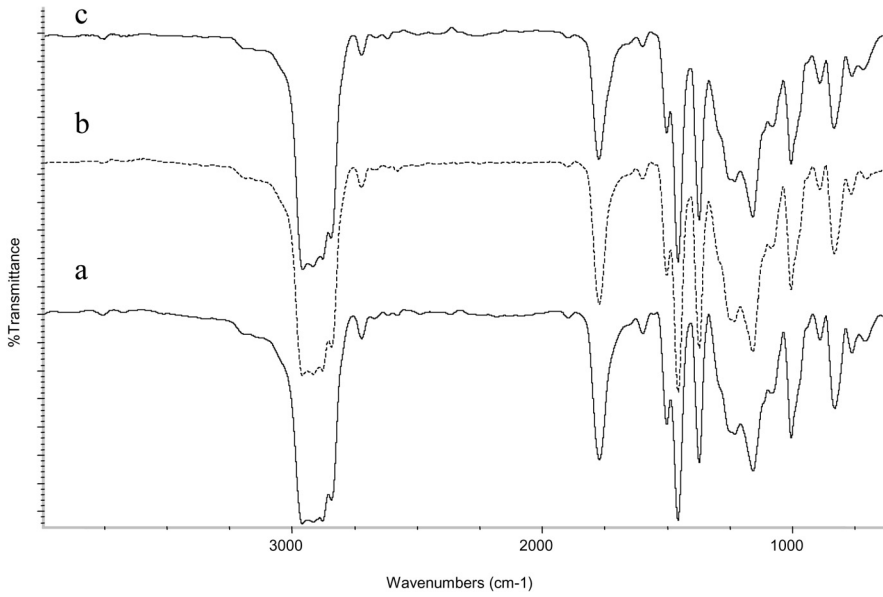
Figure 4. FTIR spectra of PP/PC (a), PP/PC/PP-g-MA (b), and PP/PC/EBuAMA (c).
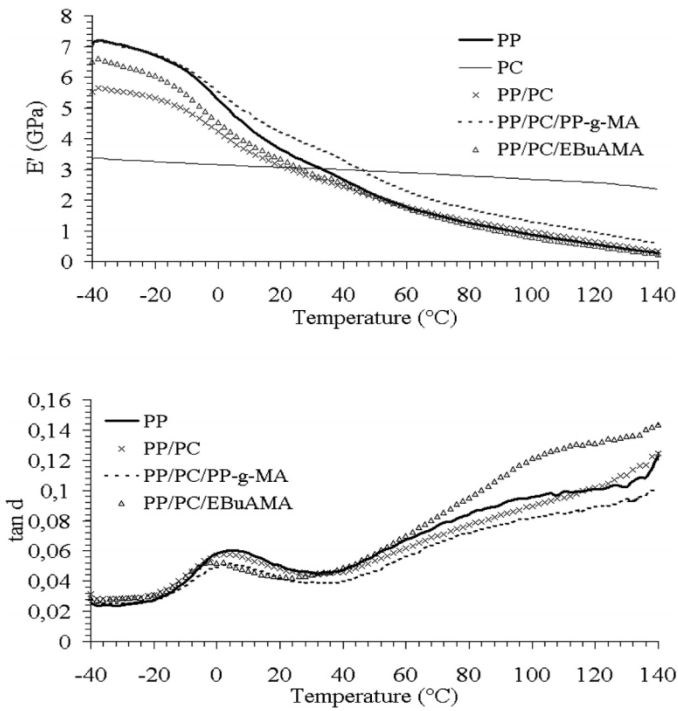

Figure 5. Temperature dependence of storage modulus E′ and tan d of PP, PP/PC, PP/PC/PP-g-MA, and PP/PC/EBuAMA.

Table 2
DMA data for PP, PC, blends PP/PC, PP/PC/PP-g-MA, and PP/PC/EBuAMA

System	T_g (°C)	E′ (GPa)		
		T = −40°C	T = 20°C	T = 100°C
PC	—	3.36	3.07	2.68
PP	3.20	7.22	3.73	0.88
PP/PC	2.40	6.91	4.00	1.21
PP/PC/PP-g-MA	6.40	7.21	4.22	1.28
PP/PC/EBuAMA	1.60	6.70	3.33	0.78

PP/PC. The immiscibility of the two components resulted in phase segregation, where the minor phase was dispersed in large spherical domains with diameter ranging from 5 to 10 μm. This may explain why the degradation of the blends is dominated by the matrix. The presence of holes proves the retreat of particles, indicating that there was no adhesion between PP and PC.

The addition of PP-g-MA or of EBuAMA to the blend PP/PC leads to a decrease in the particule size as can be observed in Figures 6(b) and (c). However, there is still poor adhesion between the immiscible phase, since holes are still present in a large quantity. This behavior is not in agreement with the results obtained for thermomechanical properties that show different behavior for PP-g-MA and EBuAMA. The improvement in thermomechanical stability obtained in the presence of PP-g-MA can not only be
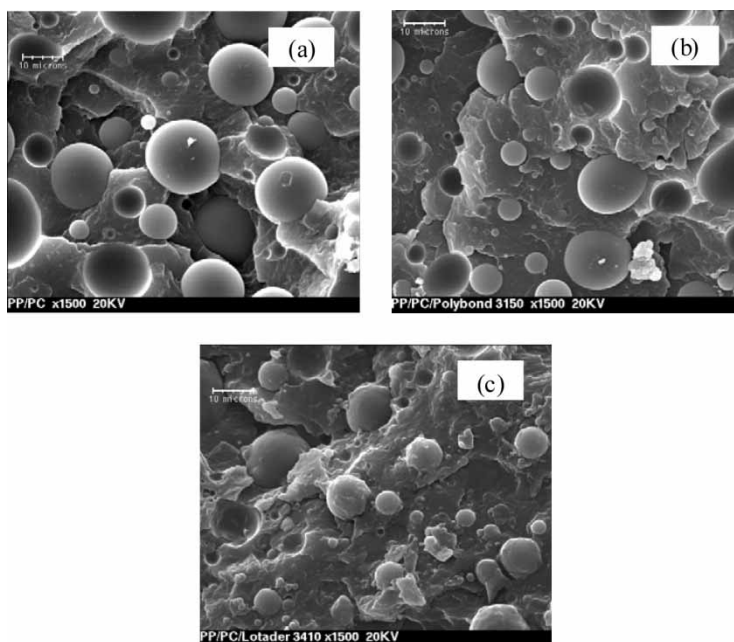

Figure 6. SEM micrographs of (a) PP/PC, (b) PP/PC/PP-g-MA, and (c) PP/PC/EBuAMA.

assigned to the weak improvement of the homogeneity of the PP/PC/PP-g-MA system (i.e., decrease in the particle size).

The WAXS patterns of a selected sample are presented in Figure 7. Plain PP exhibits peaks at $2\theta = 14.2$; 17.0; 18.6; 21.1 and 22.0 degrees, which correspond to the α crystal form (monoclinic phase). The addition of PC and compatibilizing agent does not affect the crystal structure of PP in the blends.

Thermal Properties

The crystallization of PP in PP/PC blends was studied by differential scanning calorimetry (DSC).

The DSC cooling thermograms of pure PP and PP/PC blends are shown in Figure 8. Characteristic thermal properties are summarized in Table 3.

Crystallinities were calculated according to Equation (1).

$$\Phi_i = \frac{\Delta H_m}{\Delta H_m^0} \times 100 \tag{1}$$

where ΔH_m^0 and ΔH_m are respectively an extrapolated value of enthalpy corresponding to the melting of 100% crystalline PP and the value of enthalpy corresponding to the melting of PP ($\Delta H_m^0 = 209\,J/g$).

Effects of amorphous PC on the crystallinity of PP in blend are shown in Table 3. For all the blends, the degree of crystallinity of PP component decreases. The crystallization temperature of PP (T_c PP) is lower than T_g of PC (about 145°C) (17). That means when PP begins to crystallize, the PC component has already become rigid and therefore may affect
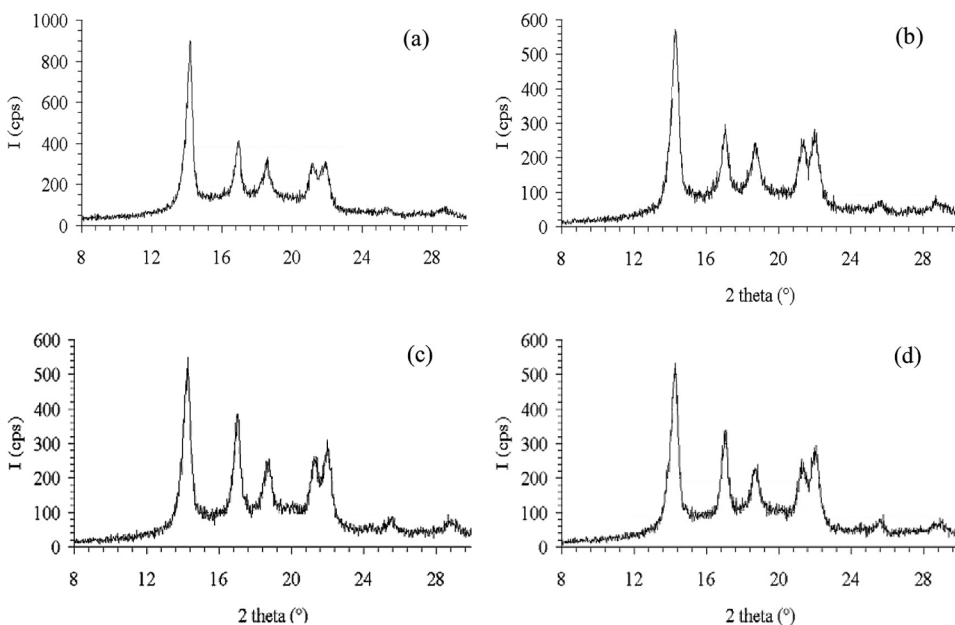

Figure 7. Wide-angle X-ray scattering patterns of (a) PP, (b) PP/PC, (c) PP/PC/PP-g-MA, and (d) PP/PC/EBuAMA.
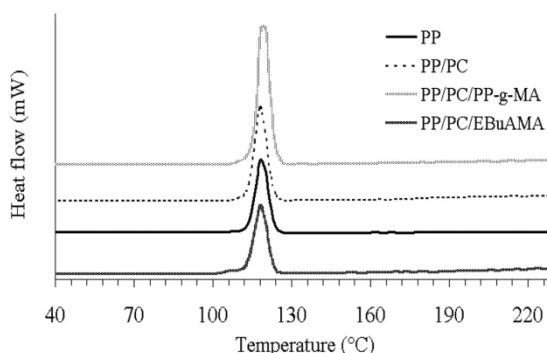
Figure 8. DSC crystallization behaviour of pure PP and PP/PC blends.

the crystallization process of PP in blend. The blend PP/PC/PP-g-MA presents the smaller decrease of crystallinity. For this blend, the crystallization peak temperature is higher than the crystallization peak of pure PP (118.4°C). A similar shift of T_c to a higher temperature has been reported by Karger et al. (8) and Ghijsels et al. (4) for PP/EPDM blends, and they suggested the dispersed phase of EPDM acted as a weak nucleating agent. For the blend PP/PC/PP-g-MA, the increase of crystallization peak temperature of PP and the limited decrease of PP crystallinity show that PP-g-MA seems to act as a nucleating agent. This behavior has already been reported for PP-g-GMA (17).

The melting temperature of PP and PP/PC blends are similar whatever the material (Table 3).

Fire Retardant Properties

The heat release rate (HRR) curves of PP, PC, and PP/PC blends are shown in Figure 9 and the residue collected after the experiments are presented in Figure 10. Characteristic fire retardant properties are summarized in Table 3.

Base Polymers

The addition of ammonium polyphosphate in the base polymer matrices leads to an improvement of the flammability of the materials in particular in the case of PC. The

Table 3
Thermal parameters of pure PP and PP in blends PP/PC, PP/PC/EBuAMA and PP/PC/PP-g-MA

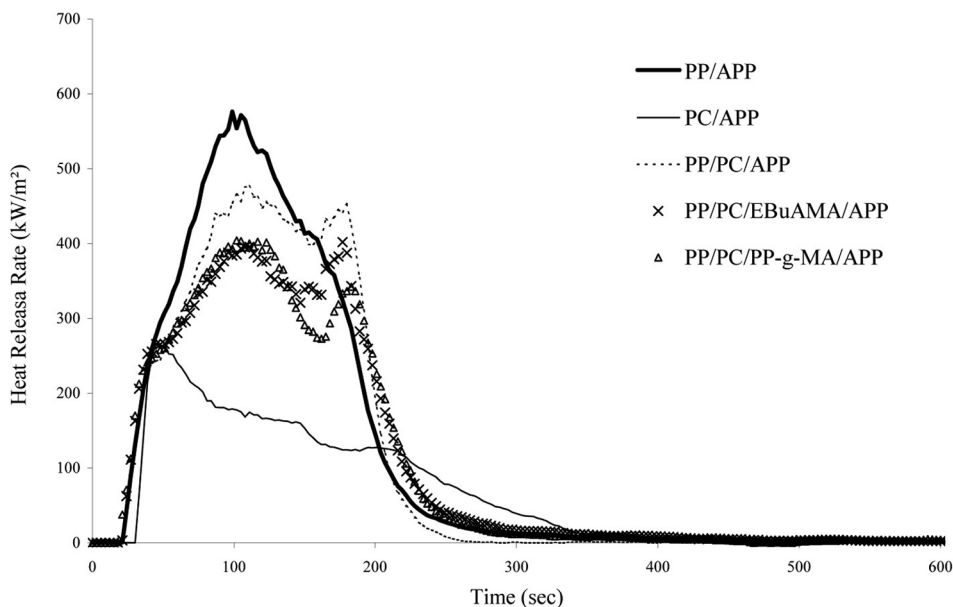| System	T_m PP (°C)	T_c PP (°C)	$T_o - T_c$ (°C)	ΔH_c PP (J/g)	ΔH_m PP (J/g)	ϕ_i (%)
PP	160.9	118.4	5.0	-91.5	88.1	42
PP/PC	161.2	118.2	4.9	-81.0	79.1	38
PP/PC/EBuAMA	160.1	118.0	4.9	-76.1	69.7	33
PP/PC/PP-g-MA	162.2	119.0	4.6	-86.2	83.3	40

Figure 9. HRR curves vs. time of intumescent materials (heat flux $= 50\,kW/m^2$).

improvement is particularly important in the case of the PC matrix. The addition of ammonium polyphosphate into the PP increase the LOI of around 3 vol % and the PkHRR decreases from around 50% (virgin PP–LOI = 17 vol. %, PkHRR = 1300 kW/m^2 (33)). A black carbonaceous residue (17 g/g-%) is obtained at the end of the cone calorimeter experiment (Figure 10), whereas the virgin PP burns totally. However, the material does not successfully pass the UL-94 tests due to a high combustion time (40 sec) and to dripping. In the case of PC/APP, a UL-94 V0 rating is achieved (compared to V-2 for virgin PC (34)). PC appears as an interesting carbonization agent as demonstrated by the residue obtained at the end of the cone calorimeter tests (Figure 10, an expanded intumescent structure, is obtained). Moreover, the addition of APP into PC leads to an increase of the LOI of 8 vol. % (pure PC presents a LOI of 30 vol. % (34)) and the heat release rate remains very low during all the cone calorimeter experiments ($<270\,kW/m^2$). The high values of TSR and PkCOP for PC/APP material demonstrates that, in that case, incomplete combustion are favored leading to an increase of the opacity and toxicity of the smoke release.
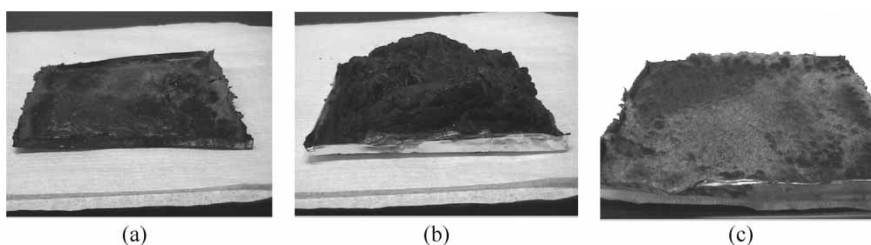

Figure 10. Residue ((a) PP/APP; (b) PC/APP; (c) PP/PC/PP-g-MA/APP) from cone calorimeter experiments.

PP/PC Blends

The addition of APP into PP/PC blends improve the fire retardant properties of the material. Either, if the LOI of the PP/PC blends compares to PP-based material does not sharply increase, the V-2 classification and the improvement of the cone calorimeter parameters are noted. When compared with PP/APP, it is observed that the PkHRR decreases from 20% when no compatibilizer is used to 30% when PP-g-MA compatibilizes the blend. The addition of the interfacial agent has an effect on the fire retardant performance of the blend as previously observed in PP/PA-6 blends (33). The best performances are obtained when PP-g-MA is used. It may be assumed that in the case of the compatibilized blend, the better dispersion of PC into the PP matrix enables us to improve the interactions between the APP particles and the PC domains. Further study is needed to confirm this hypothesis. The HRR curves vs. time present two peaks, which is characteristic of intumescent materials. The first peak is attributed to the formation of the intumescent shield and the second to its destruction (either mechanical or thermal degradation). The compatibilized systems present a lower first PkHRR confirming the previously described hypothesis. Moreover, the compatibilized blends present an increase in the residual weight and a decrease in PkCOP and $PkCO_2P$ compared with PP/PC/APP. A carbonaceous residue is obtained whatever the PP/PC blends (Figure 10).

Conclusion

In the first part, the effects of two commercially available compatibilizer on the thermal and thermomechanical properties of PP/PC blends have been investigated. It has been shown that the compatibilizing effect of PP-g-MA for PP/PC blends are more efficient than the terpolymer EBuAMA leading to higher thermomechanical properties. The particle size of the minor phase in the PP/PC blends decrease in a similar way whatever the compatibilizer. The crystalline form of the PP is not affected by the presence either of PC or of the compatibilizer. However, the crystallinity increase when PP-g-MA is added in comparison with EBuAMA which may be bright together with the improvement of the thermomechanical properties. In a second part, intumescent fire retardant PP/PC blends have been developed. A UL-94 V2 material has been obtained. It is observed that the compatibilizing agent affects the fire retardant properties of the material and the best performance is achieved when PP-g-MA is used. Further study will focus on the optimization of this formulation.

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