Carbohydrate Polymers 92 (2013) 1921–1927

Contents lists available at SciVerse ScienceDirect

Carbohydrate Polymers

journal homepage: www.elsevier.com/locate/carbpol

Mechanical and thermal properties of eco-friendly poly(propylene carbonate)/cellulose acetate butyrate blends

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ARTICLE INFO

Article history:
Received 8 September 2012
Received in revised form
10 November 2012
Accepted 21 November 2012
Available online 28 November 2012

Keywords:
Poly(propylene carbonate) (PPC)
Cellulose acetate butyrate
Polymer blending
Interaction
Physical properties

ABSTRACT

The eco-friendly poly(propylene carbonate) (PPC)/cellulose acetate butyrate (CAB) blends were prepared by melt-blending in a batch mixer for the first time. PPC and CAB were partially miscible because of the drastically shifted glass transition temperatures of both PPC and CAB, which originated from the specific interactions between carbonyl groups and hydroxyl groups. The incorporation of CAB into PPC matrix enhanced not only tensile strength and modulus of PPC dramatically, but also improved heat resistance and thermal stability of PPC significantly. The tensile strength and the modulus of PPC/CAB = 50/50 blend are 27.7 MPa and 1.24 GPa, which are 21 times and 28 times higher than those of the unmodified PPC, respectively. Moreover, the elongation at break of PPC/CAB = 50/50 blend is as high as 117%. In addition, the obtained blends exhibited good transparency, which is very important for the package materials. The results in this work pave new possibility for the massive application of eco-friendly polymer materials.

1. Introduction

Poly(propylene carbonate) (PPC) is a new type of completely biodegradable aliphatic polyester and it is synthesized from propylene oxide and carbon dioxide (CO_2) (Inoue, Koinuma, & Tsuruta, 1969). The wide application of PPC material not only reduces the dependence on petroleum but also lowers the massive emission of CO_2 that has been considered to be the main factor causing the greenhouse effect in the world. However, as an amorphous polymeric material, PPC suffers many drawbacks such as a low glass temperature, poor mechanical properties and a very low thermal resistance. These drawbacks make PPC neither a typical engineering material nor a typical rubber (Luinstra, 2008), thus the unmodified PPC is still far away from practical application. Some efforts have been made to use PPC as a plasticizer/additive for rubber, thermoplastics, or surfactants (Huang et al., 1996; Huang, Wang, Liao, Chen, & Cong, 1997; Pang, Liao, Huang, & Cong, 2002; Wang, Huang, & Cong, 1997). Investigations have also been carried out to modify the physical properties of PPC by melt compounding. Most of the studies focused on PPC blends with other biodegradable polymers to fabricate completely biodegradable materials (Li, Lai, & Liu, 2004; Luinstra & Molnar, 2007; Ma, Yu, & Wang, 2006; Pang, Qiao, Jiao, Wang, Xiao, & Meng, 2008; Wang, Peng, & Dong, 2005; Yang & Hu, 2008; Zhang et al., 2006, 2007), such as poly(L-lactide) (PLLA) (Luinstra & Molnar, 2007; Ma et al., 2006), poly(butylene succinate) (PBSU) (Pang et al., 2008; Zhang et al., 2007), and polyhydroxybutyrate (PHB) (Yang & Hu, 2008; Wang et al., 2005). On the other hand, few works focused on PPC blends with a non-biodegradable polymer (Wang, Du, Jiao, Meng, & Li, 2007; Zhang, Mo, Zhang, Wang, & Zhao, 2003) or low-molecular-weight compound (Yu et al., 2008). One of us (Li & Shimizu, 2009) reported the enhanced mechanical and thermal properties of PPC by incorporation of both poly(methyl methacrylate) (PMMA) and poly(vinyl acetate) (PVAc). The nanocomposites based on PPC and organoclay have also been reported (Du, Qu, Meng, & Zhu, 2006; Shi & Gan, 2007; Zhang, Lee, Lee, Heo, & Pittman, 2008). However, there is very limited improvement for the investigated compounds using PPC as the dominant component. Gao, Chen, Wang, Deng, Zhang, Bai, and Fu, (2011) recently have prepared PPC/graphene oxide nanocomposites and found that the prepared PPC nanocomposites exhibit superior physical properties.

Cellulose is one of the promising natural polymers with renewable and biodegradable characteristics. However, cellulose is insoluble and inmeltable because of three dimensional physical networks formed by the very strong H-bonding interactions as a multi-hydroxyl polymer (Wertz, Mercier, & Bedue, 2010). Esterification of cellulose induces solubility and melts processability. We consider that the derivative of cellulose is a good candidate to compound with PPC because of the natural resourced materials and the excellent physical properties. Zhang et al. (2006) have studied the thermal behaviors of PPC/ethyl cellulose (EC) blends

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0144-8617/$ – see front matter © 2012 Elsevier Ltd. All rights reserved.
http://dx.doi.org/10.1016/j.carbpol.2012.11.058

by casting from dichloromethane solution and they found that the incorporation of ethyl cellulose enhanced the thermal stability of PPC/EC blends drastically. However, EC is unsuitable for common uses as a thermoplastic plastic because of their high melt temperatures, viscosities, and relatively low decomposition temperatures. Therefore, no physical properties of PPC/EC blends have been reported because it is difficult to fabricate massive sample by melt mixing for the physical property evaluation. As an important derivative of cellulose, cellulose acetate butyrate (CAB) is a type of rigid and thermoplastic material, which shows a high glass transition temperature and excellent mechanical properties. It is therefore considered that CAB can be used to improve both the mechanical and thermal properties of PPC. In the present study, cellulose acetate butyrate (CAB) is incorporated into PPC matrix with different weight percentages by the melting blending to fabricate the PPC/CAB blends. It is found that the hydroxyl groups of CAB have specific interactions with the carboxyl groups of PPC, leading to partially miscible PPC/CAB blends. The incorporation of CAB into PPC matrix not only improved the thermal properties but also enhanced the mechanical properties of PPC/CAB blends significantly. Moreover, the PPC/CAB blends could remain good transparency because of the close refractive index, which can be used to produce biodegradable and transparent packing materials.

2. Experimental

2.1. Materials and sample preparation

The PPC with the type of AAA-01-1000 and CAB with the type of 381 were purchased from Empower Co., Ltd. (USA) and Eastman Co., Ltd. (USA), respectively. All the samples were dried in the oven at 80 °C for 24 h before blending. The composites were prepared by direct mixing the PPC with various amounts of CAB in a batch mixer (Haake Polylab QC) with a twin screw at a rotation speed of 50 rpm at 190 °C for 5 min. After melt-mixing, all samples were hot-pressed at 190 °C under a 14 MPa pressure into films 300 μm thick, followed by cool-pressing at room temperature. The obtained sheets were directly used for the following characterization.

2.2. Characterization

Melt rheology was investigated on an Andor Parr equipment in a plane-plane configuration, using 1 mm thick sample discs of diameter 25 mm. Experiments were carried out at 190 °C, i.e. the same temperate as that used for blend processing, in the frequency range 10^{-1} Hz to 10^{2} Hz, applying a shear strain of 1%.

The phase structure of the blends was observed using field-emission scanning electron microscope (FESEM). A H4800 (Hitachi, Japan) scanning electron microscope was used for the measurements at an accelerating voltage of 10.0 kV. All samples were fractured by immersion in liquid nitrogen for about 5 min. The fracture surface was then coated with a thin layer of gold before observed. Transmission electron microscopy (TEM) was performed using a Hitachi HT-7700 instrument operating at an accelerating voltage of 80 kV. The blend samples were first ultramicrotomed to obtain a section with a thickness of about 70–100 nm and then stained with ruthenium tetraoxide (RuO_4) for 3 h.

Differential Scanning Calorimeter (DSC) was carried out using a TA-Q2000 calorimeter. Before sample scanning, the heat flow and temperature of the instrument were calibrated with sapphires and pure indium, respectively. The samples were first heated to 190 °C and held there for 5 min to eliminate previous thermal history. The samples were then cooled down to −50 °C, followed by the heating again to 190 °C. Both the cooling and heating rates were 10 °C/min and the experiments were conducted under a continuous high-pure nitrogen atmosphere.

Dynamic Mechanical Analysis (DMA) was carried out (Q 800, TA) in multi-frequency strain mode. Dynamic loss (tan δ) was determined at a frequency of 5 Hz and a heating rate of 3 °C/min from −50 °C to 150 °C at liquid nitrogen atmosphere.

Thermogravimetric analysis (TGA) was carried out (Q 500, TA) at a heating rate of 20 °C/min from room temperature to 650 °C at continuous high-pure nitrogen atmosphere.

The structures of PPC, CAB, and composites were studied by Fourier transform infrared spectroscopy (FTIR, Bruker Tensor) instrument. FTIR spectra were recorded at the resolution of 1 cm^{-1} and 32 scans from 4000 cm^{-1} to 400 cm^{-1}. The FTIR measurements were carried out by a transmittance mode and the samples for FTIR were prepared directly by the melt press.

Tensile tests were carried out using an Instron universal material testing system (model 5966) at 23 °C with gauge length of 18 mm and crosshead speed of 20 mm/min.

3. Results and discussion

3.1. Rheological behavior

Fig. 1 shows the complex viscosity as a function of frequency for the two neat polymers and the blends at 190 °C. It is found

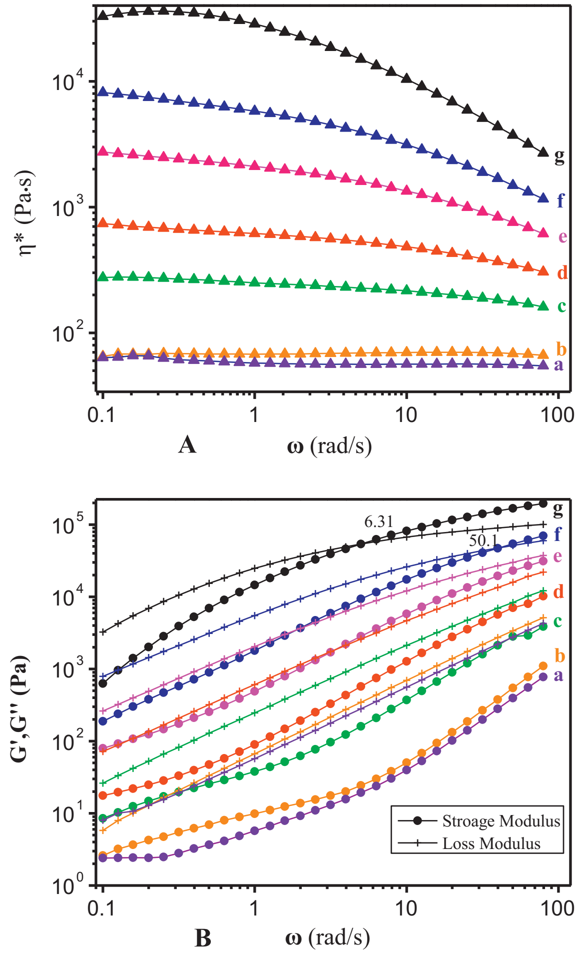

Fig. 1. Frequency dependence of complex viscosity (A) and storage modulus as well as loss modulus (B) at 190 °C for pure PPC, pure CAB, and PPC/CAB blends, (a) PPC/CAB(100/0); (b) PPC/CAB (80/20); (c) PPC/CAB(60/40); (d) PPC/CAB(50/50); (e) PPC/CAB(40/60); (f) PPC/CAB(20/80); (g) PPC/CAB(0/100).

that CAB displays typical newtonian behaviors at low shear rate, followed by the shear thinning at high shear rate, similar to most of thermoplastic polymers. On the other hand, the viscosity of neat PPC is much lower than that of CAB at 190 °C. Morover, almost no shear rate dependence of the viscosity for PPC has been observed. Such PPC rheological behavior may be attributed to the partial thermal-decomposition of PPC at 190 °C because the onset degradation temperature of PPC is only 186 °C at the heat rate of 20 °C/min (as shown in Section 3.5). The viscosity of blends lies in between that of homopolymers in the whole shear rate region investigated. The viscosity of the blends increases with increase in amount of CAB in the blend. The storage modulus and the loss modulus of the blends are also much dependent on the compositions.

3.2. Morphologies

Phase morphologies of the cryo-fractured PPC/CAB blends in the whole compositions are shown in Fig. 2. Clearly, for blends with predominant PPC content, i.e. 80/20 and 70/30 blends, the morphology consists of CAB particles dispersed in the PPC matrix. It is noteworthy that the size of the CAB particles ranges from micrometer to sub-micrometer. It is also worth noting that the interface between PPC and CAB phases are pretty good because all the domains are very well imbedded in the matrix without any gaps and some physical adhesion exists at the PPC–CAB boundary. This is an indication of the rather good compatibility between PPC and CAB due to low interfacial free energy.

For the PPC compositions in the range 40–60%, CAB displays a phase structure with irregular shapes and CAB phase connects in the whole material. This is a typical co-continuous structure. It is also seen that the co-continuous structure is very stable and can exist over a large composition range, which again indicates that the good compatibility between PPC and CAB. Indeed, in the case of high interfacial free energy between components in a blend, the co-continuous phase structure in an irregular structure would involve a highly unstable situation (Sundararaj & Macosko, 1995).

For the blends with CAB compositions more than 70%, PPC appears as a globular dispersion in a CAB matrix. It is again to be noticed that the PPC domains are mainly in the range of sub-micrometers. This is a confirmation of the good compatibility of the unlike species again. The good physical adhension between the two phases can effectively transfer the loadings upon suffering the tensile or impact stress and results in a good mechanical properties of the final materials. TEM measurements have been carried out to investigate the detail morphology of PPC/CAB blends, as shown in Fig. 3. CAB was observed as the dark area and PPC was observed as the white part because CAB is more readily stained by RuO_4 than PPC. For the PPC/CAB = 80/20 blend, the CAB domains are finely dispersed in the PPC matrix with the size of less than 1 μm. The PPC/CAB = 20/80 blend shows the morphology with the PPC domains dispersed in the CAB matrix. The phase size is also in the range of sub-micron meter. The irregular phase structure was observed for the PPC/CAB = 50/50 blend and the dark CAB phase connects in the whole material, indicating a similar co-continuous structure. The TEM results are much consistent with those from SEM.

3.3. Thermal analysis

Fig. 4A shows the DSC thermograms for neat PPC, neat CAB, and PPC/CAB blends with a heating speed of 10 °C/min. Both CAB and PPC are amorphous materials with no crystallization and melting peaks. The glass transition temperatures (T_gs) of neat PPC and neat CAB from DSC curves are 18 and 132 °C, respectively. The T_g corresponding to PPC component in the PPC/CAB blends increases with increasing the CAB content. On the contrary, the T_g corresponding to CAB shifts to the lower temperatures with the addition of CAB. Fig. 4B displays the T_gs of PPC and CAB as a function of blend composition. It is clear that all the blends have two T_gs and the T_gs of PPC and CAB shift to each other with variation of blend composition, which indicates that the PPC and CAB are partially compatible. The partial miscibility between PPC and CAB accounts for the good interface of the material, leading to the improved mechnical and thermal properties.

Fig. 5 shows dynamic viscoelastic curves for neat PPC, neat CAB, and PPC/CAB blends. The storage modulus (E') versus the temperature for the PPC/CAB blends and the neat components is shown in Fig. 5A. Both PPC and CAB are very rigid and show high storage modulus at the temperature below the T_g of PPC. The E' of neat PPC dropped abruptly to a very low value at about 35–40 °C due to the glass transition, which means that the heat distortion temperature of PPC is very low and PPC alone cannot be used above its T_g. In contrast, neat CAB remains very high storage modulus below its T_g at about 140 °C, indicating the good heat resistance of CAB. For the blends with less than 40% CAB, the blends cannot self-support above the T_g of PPC because PPC is the matrix. With increasing the CAB contents to 40–60%, the samples are self-supported in the region between the T_gs of PPC and CAB. Both PPC and CAB are continuous in this composition range and CAB remains the mechanical strength at the temperature above the T_g of PPC. It can also be found that the modulus of PPC/CAB blends gradually increases with increasing content of rigid CAB between the T_gs of PPC and CAB. When the CAB content is more than 60%, the blends exhibit pretty high storage modulus with a small modulus drop at about 40 °C due to the glass transition of PPC. It is obvious from the DMA curves that the incorporation of CAB in PPC enhances the mechanical properties and heat distortion temperature significantly.

Fig. 5B displays the dynamic loss curves as a function of temperature of the PPC/CAB blends. The glass transition temperatures can also be determined by the peak temperature of the dynamic loss ($\tan\delta$) curves. As observed from the $\tan\delta$ curves, all the blends show two glass transitions, indicating that the blends are not thermodynamically miscible. However, the relaxation peaks of both PPC and CAB shift toward each other when the composition ratios are varied, suggesting that PPC and CAB are partially miscible. The significantly shifted glass transition temperatures for PPC and CAB indicate some molecular interactions between the two components, which will be discussed in the following section. The dependence of the glass transition temperatures of PPC and CAB on the composition by DSC and DMA is shown in Fig. 4B. The tendency of T_gs changing with the composition from DMA is consistent with that from DSC measurements.

3.4. FTIR analysis

The each other shifted glass transition temperature indicates the interaction between PPC and CAB. FTIR has been used to detect such specific interaction in detail. Fig. 6 shows the FTIR spectra of PPC, CAB, and their blends in several specific wavenumber regions. From Fig. 6A, neat PPC shows a strong carbonyl stretching absorption at about 1744 cm^{-1}. With the addition of CAB, the —C═O absorption peak shifts to high wave numbers by 2–4 cm^{-1}. For the PPC/CAB 60/40 blends, the —C═O absorption peak locates at 1747 cm^{-1}, which is 3 cm^{-1} higher than that of neat PPC. CAB is a polymer that has been partially esterified but still has large number of hydroxyl groups. The shifted carboxyl group absorption of PPC indicates that there is specific interaction between —C═O of PPC with the the hydroxyl group in CAB. Fig. 6B shows the FTIR spectra at the hydroxyl groups absorption regions. Neat CAB has broad —OH absorption located at 3549 cm^{-1}. The blending with

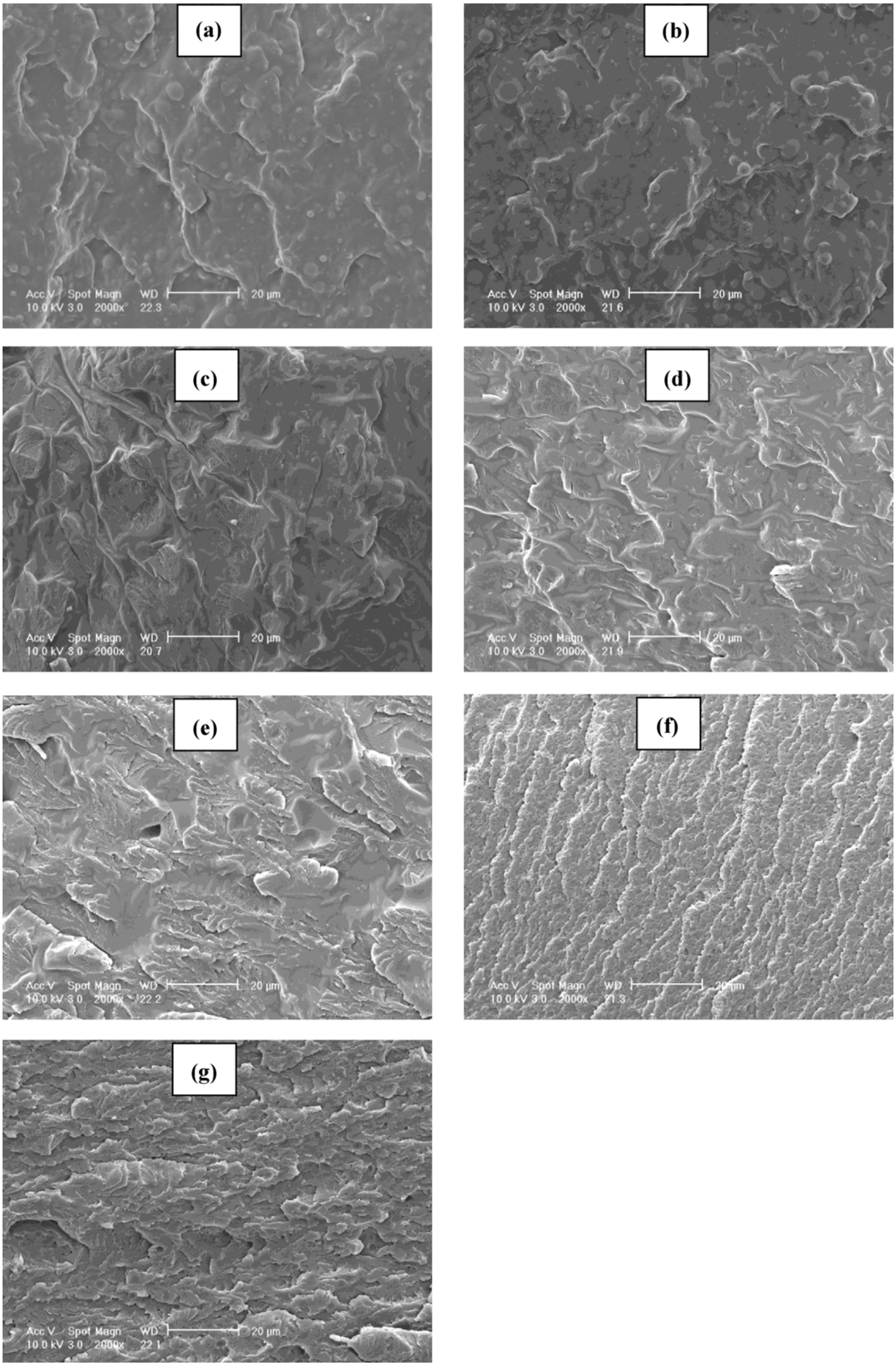

Fig. 2. SEM images of (a) PPC/CAB(80/20), (b) PPC/CAB(70/30), (c) PPC/CAB(60/40), (d) PPC/CAB(50/50), (e) PPC/CAB(40/60), (f) PPC/CAB(30/70), and (g) PPC/CAB(20/80).

PPC induces obvious shifting of the —OH absorption at about 3481 cm^{-1}. The lower wavenubmer shifted absorption indicates the formation of H-bond between PPC and CAB. It is therefore considered that the —OH groups of CAB form hydrogen bonds with the —C=O groups of PPC. Such interaction accounts for the partial miscibility between PPC and CAB. Therefore, the improved mechanical and thermal properties can be achieved for the PPC/CAB blends.

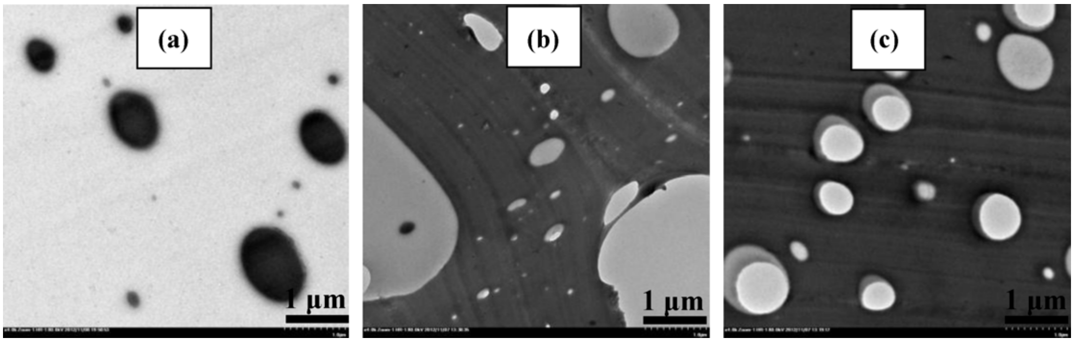

Fig. 3. TEM images of (a) PPC/CAB(80/20), (b)PPC/CAB(50/50), (c) PPC/CAB(20/80).

3.5. Thermal decomposition behavior of PPC/CAB blends

PPC shows very low thermal decomposition temperature, which impedes the melt processing of PPC (Inoue et al., 1969a, 1969b) .The thermal degradation behavior of the neat PPC, neat CAB, and PPC/CAB blends at various compositions was investigated with thermogravimetric analysis (TGA). The corresponding TGA and DTG thermograms are shown in Fig. 7. Both neat PPC and neat CAB show one-step decomposition profile with a single maximum transition temperature. Neat CAB has much higher thermal stability than PPC, because it degrades at 353 °C (Td5%) and maximum decomposition rate occurs at 390 °C, whereas PPC starts degrading at 186 °C (Td5%) and maximum degradation temperature is 251 °C. For the all blends, two steps degradation can be observed, indicating that PPC and CAB degrade separately in the blends. The degradation at the lower temperature corresponds to the thermal decomposition of PPC-rich phase, while the higher temperature one originates from the decomposition of CAB-rich phase. It is further found that the incorporation of CAB into PPC enhances the degradation temperature significantly. The maximum degradation temperature increases from 251 °C for neat PPC to about 297 °C with incorporation of 10 wt.% CAB. The significantly increased thermal stability is critically important for the melt processing of PPC. Obviously, the enhanced thermal stability of PPC in the blend with CAB comes from the specific interaction between CAB and PPC.

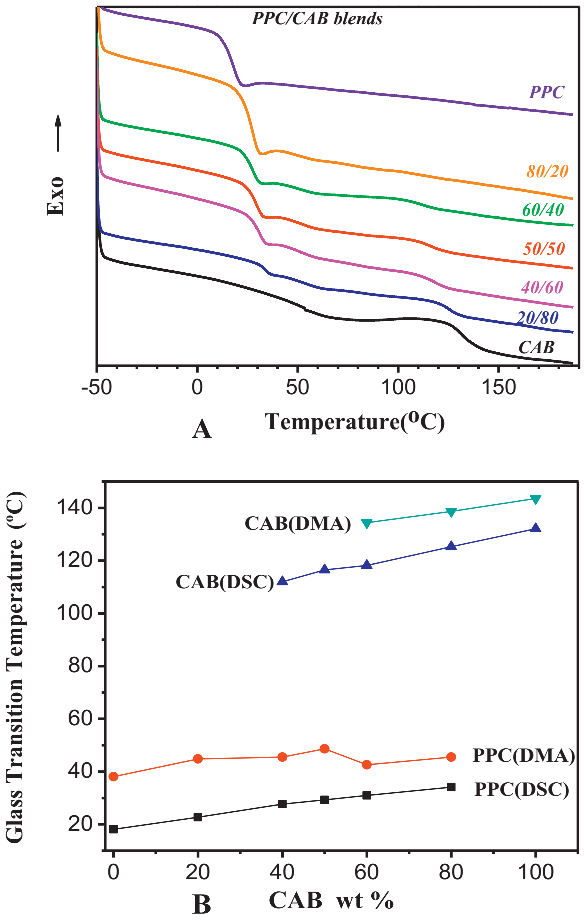

Fig. 4. (A) DSC thermograms of neat PPC, neat CAB, and PPC/CAB blends; (B) the variation of glass transition temperature of PPC and CAB phase in the PPC/CAB blends versus the content of CAB component.

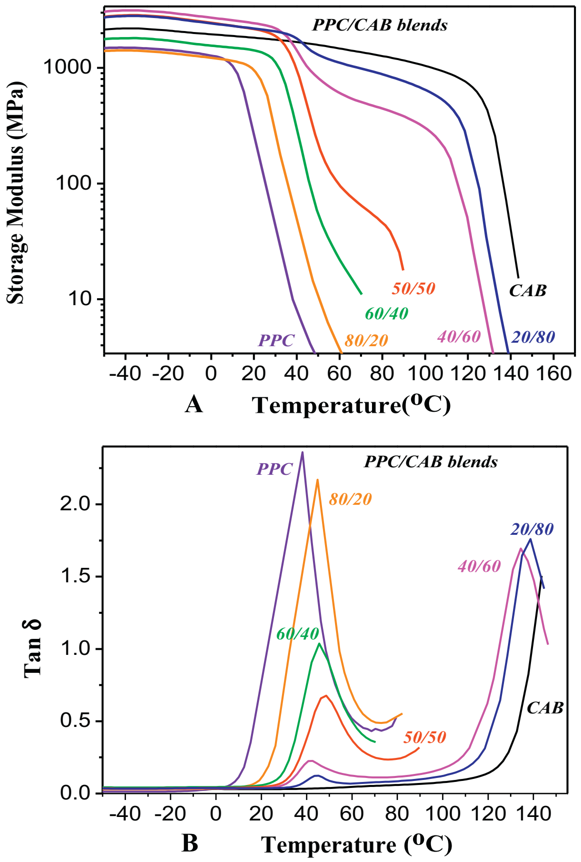

Fig. 5. Storage modulus (A) and tan (δ) (B) for neat PPC, neat CAB, and their blends as a function of temperature by DMA.

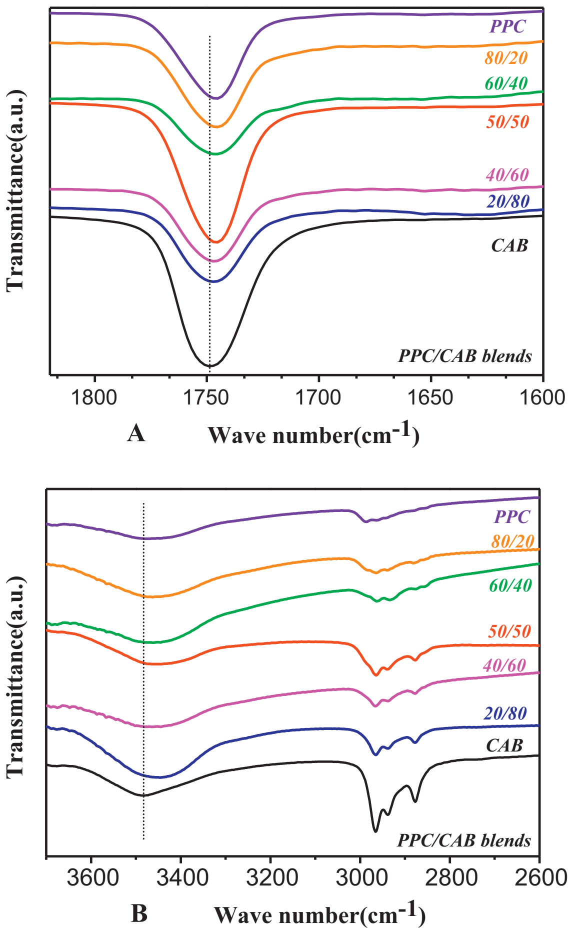

Fig. 6. FTIR spectra of pure PPC, pure CAB, and their blends in carbonyl group (—C=O) region (A) and hydroxyl group (—OH) (B).

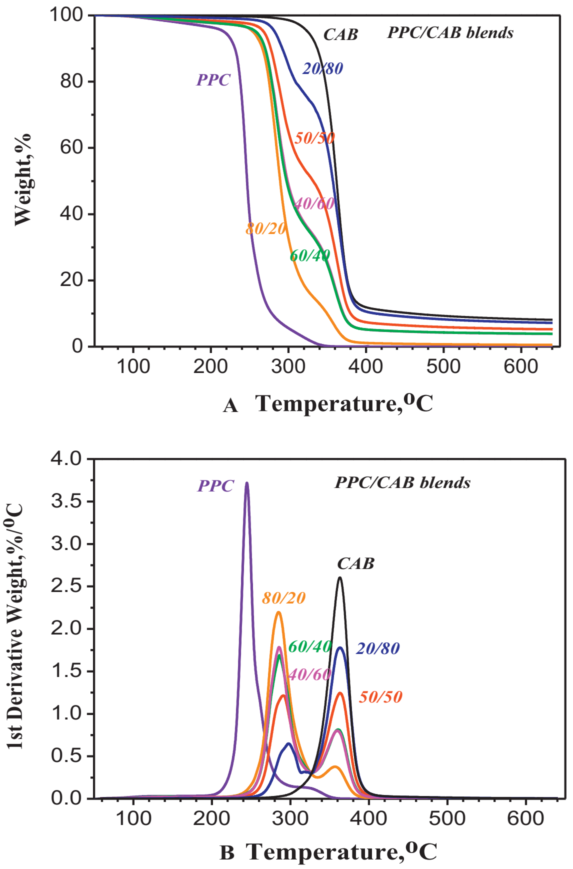

Fig. 7. Thermogravimetric analysis of neat PPC, neat CAB, and PPC/CAB blends (A) TG curves; (B) DTG curves.

3.6. Tensile properties

Pure PPC exhibits rubber-like features including an extremely low elastic modulus and a very low tensile strength but a high elongation at break. The low physical performance characteristics render pure PPC far from practical use without any modification. In contrast, pure CAB has an extremely high both elastic modulus and tensile strength with an elongation at break of about 78%. From the mechanical properties points of view, PPC and CAB show the nice complementarity. It is therefore interesting to measure the mechanical properties of the PPC/CAB blends. Fig. 8 shows the strain–stress curves for pure PPC, pure CAB, PPC/CAB blends measured at room temperature. The main tensile properties such as static modulus, tensile strength, and elongation at break determined from these curves are presented in Table 1. It is obvious that both the elastic modulus and the tensile strength are dramatically increased with the addition of CAB content in the PPC/CAB blends, compared with the pure PPC. This results indicated that the drastically strengthening effects of CAB for PPC material. The tensile strength and the modulus of PPC/CAB = 50/50 blend are 27.7 MPa and 1.24 GPa, which are 21 times and 28 times higher than those of the unmodified PPC, respectively. It should be noted that the elongation at break of the blend is also ranged within the elongation at break of neat PPC and neat CAB, depending on the component ratio. The impoved mechanical performance for the PPC/CAB blends are obviously attributed to the good compatibility between PPC and CAB, as demonstrated in FTIR and DMA measurements.

Table 1
Basic mechanical properties of PPC, CAB, and PPC/CAB blends.

Samples	Elastic modulus (MPa)	Tensile strength at break (MPa)	Elongation at break (%)
PPC/CAB(100/0)	44	1.33	801
PPC/CAB(80/20)	619	6.14	444.5
PPC/CAB(60/40)	845	12.94	149
PPC/CAB(50/50)	1240	27.7	117
PPC/CAB(40/60)	1110	32.69	115
PPC/CAB(20/80)	1327	43.55	96.5
PPC/CAB(0/100)	1460	50.84	78

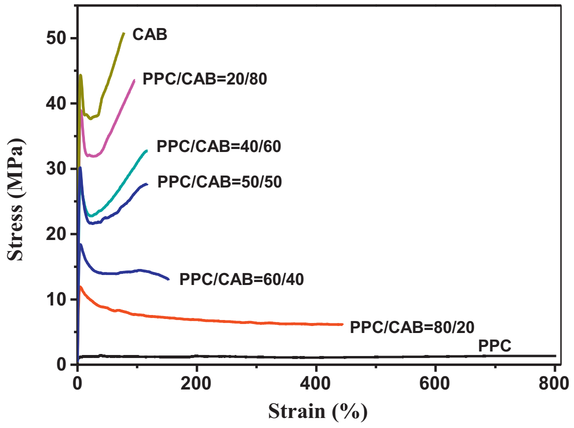

Fig. 8. Typical strain–stress curves for pure PPC, pure CAB, and PPC/CAB blends.

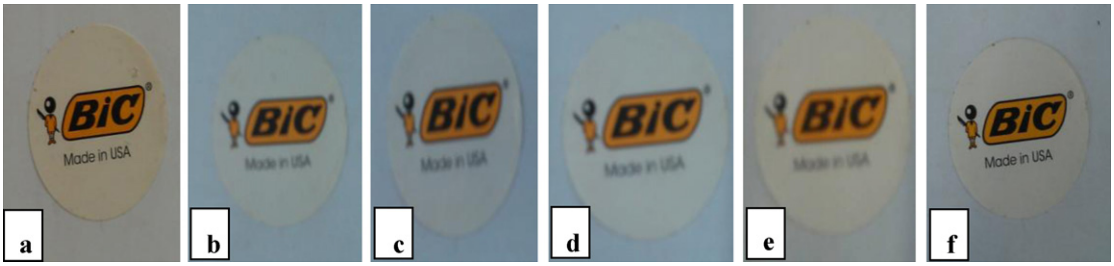

Fig. 9. Photographs of transparency for pure PPC, pure CAB, and PPC/CAB blends, (a): PPC/CAB(100/0); (b) PPC/CAB (80/20); (c) PPC/CAB(60/40); (d) PPC/CAB(40/60); (e) PPC/CAB(20/80); (f) PPC/CAB(0/100) (each film with the thickness about 0.3 mm covers on the whole picture).

3.7. *Optical properties*

Transparency is an attractive properties for the packaging materials. Both PPC and CAB are amorphous polymers and exhibit very nice transparency. The incompatible polymer blends are ususally opaque due to the light scattering and reflection at the interface. However, the prepared PPC/CAB blends in the whole composition range exhibit a very nice clearance, as shown in Fig. 9. The refractive index of PPC is 1.46 and that of CAB is reported to be 1.47. Therefore, the nice transparency of the blends is attributed to the very close refractive index for the neat PPC and the neat CAB. Although the blends are phase separated, the good transparent CAB/PPC blends can be obtained. It is considered that the transparency is very important for the application of PPC/CAB blends as a package material.

4. Conclusions

Novel eco-friendly PPC alloys with drastically improved mechanical and thermal properties were successfully prepared by melt-blending of PPC with CAB. Morphological investigation revealed a pretty good physical adhesion between PPC and CAB phase. Both DSC and DMA analysis demonstrated the each other shifted glass transition temperature, indicating the partially miscible or processing compatibility for PPC and CAB. FT-IR study suggests that the hydroxyl groups of CAB form the H-bonding with carboxyl groups of PPC, so a low interfacial tension between PPC and CAB phase was achieved. The partial miscibility and good interface were mainly responsible for the significant increase in both mechanical and thermal properties. Moreover, we also found that the blends exhibit excellent transparency, which is very important for their application in packaging industry.

Acknowledgements

This work was financially supported by the National Natural Science Foundation of China (51173036, 21074029), Zhejiang Provincial Natural Science Foundation of China (R4110021), and Zhejiang Xinmiao Project of China (2012R421046).

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