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ORIGINAL PAPER

Effect of acrylonitrile–butadiene–styrene copolymer (ABS) on *β*-nucleation in *β*-nucleated polypropylene/ ABS blends

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Abstract *β*-PP/acrylonitrile–butadiene–styrene (ABS) blends were prepared with PP, ABS and a novel supported *β*-nucleating agent or *β*-PP and ABS. The effect of ABS on the *β*-nucleation of PP and crystallization and melting behavior of *β*-PP/ ABS blends were investigated by differential scanning calorimeter, wide angle X-ray diffraction, and polarized light microscopy. Results suggested that addition of low content of ABS has no effect on the *β*-nucleation of PP and crystallization behavior, and melting characteristic of *β*-PP/ABS blends. However, the increasing content of ABS decreases the *β*-nucleation, crystallization temperatures, and spherulite size of PP in the blends. However, the blends with the *β*-PP content above 80 % were obtained at the content of ABS below 40 %.

Keywords Polypropylene · Acrylonitrile–butadiene–styrene (ABS) · *β*-Nucleating agent · *β*-Nucleation

Introduction

In recent years, polymer blending has been considered a convenient route for the development of newer polymeric materials with a wide range of properties. PP has poor impact strength but high elongation and also good chemical resistance, whereas acrylonitrile–butadiene–styrene (ABS) has poor elongation but high impact strength. Hence, incorporation of ABS in a PP matrix [1–9] would be desirable to achieve higher impact strength without losing important properties of PP.

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However, α-PP is generally formed in the PP blends. It has been widely reported that β-PP exhibits a superior performance characteristic, including improved elongation at break, impact strength, and higher heat distortion temperature [10–24]. Yet the yield strength and elastic modulus of β-PP are lower than those of α-PP. In order to improve the yield strength and elastic modulus of β-PP, β-PP blending with other polymers, which has high yield strength and elastic modulus, shall become a highly effective method, such as LDPE [25], PVDF [26], PA-6 [26–31], and PET [32]. However, incorporation of the second component suppresses the formation of β-PP, e.g., in the β-nucleated PP/PVDF and PP/PA-6 blends, the β-PP cannot form even in the presence of a highly effective β-nucleated agent due to the strong α-nucleating ability and the selective or preferable encapsulation of β-nucleating agent (β-NA) by the polar second components.

Although β-PP blends with other crystalline polymers have been investigated, β-nucleated PP/ABS blend has not been reported. The polar part acrylonitrile of ABS may affect the efficiency of β-NA and the formation of β-PP in the blends, so β-nucleated PP/ABS blends were prepared with addition of a highly efficient nano-$CaCO_3$-supported β-NA in our lab [10–12]. In this article, effects of the ABS content and preparation method on the non-isothermal crystallization behavior, melting characteristics, and the β-crystal content and the morphology of the β-nucleated PP/ABS blends were investigated by differential scanning calorimeter (DSC), wide angle X-ray diffraction (WAXD), and polarized light microscopy (POM).

Experimental

Materials

A commercial grade of isotactic polypropylene (PP, N-T30S) used in the study was supplied by Sinopec Group, Maoming petroleum Chemical Industry Limited Company, China, and the MFI of PP was $3.2\ g \times 10\ min^{-1}$. ABS (PA-747) was purchased from Chi Mei Industrial Factory, Taiwan, and the MFI of ABS was $12\ g \times 10\ min^{-1}$. A nano-$CaCO_3$-supported β-NA was prepared by mass ratio of nano-$CaCO_3$/pimelic acid (100/1) in our lab [10–12].

Specimen preparation

Before blending, all the materials were adequately dried in a vacuum oven at 80 °C for 12 h. β-PP/ABS blends were prepared by two different methods. In method one, the components PP, β-NA, and ABS were simultaneously mixed and β-PP/ABS blends were prepared using a HL-200 internal mixer (Jinlin University Science and Education Instrument Factory, China) at temperature of 240 °C, and 50 rpm for 5 min. The composition and the mark of β-PP/ABS blends are listed in Table 1. In method two, β-PP was first prepared by adding 5 wt% β-NA into PP matrix on a twin-screw extruder at temperature of 190 °C. Extrudates were cooled in a water bath and cut into pellets by a pelletizer, and then mixed with ABS pellets on the HL-200 internal mixer. The obtained β-PP/ABS blends were marked as β-PP-X, where X noted the ABS content in β-PP/ABS blends.

Table 1 The composition of β-PP/ABS blends prepared by different methods

Sample		PP (wt%)	β-NA (wt%)	ABS (wt%)
Method one	Method two			
PP	PP	100.00	0	0
β-PP	β-PP	95.00	5.00	0
PP-5	β-PP-5	90.25	4.75	5.00
PP-10	β-PP-10	85.50	4.50	10.00
PP-20	β-PP-20	76.00	4.00	20.00
PP-30	β-PP-30	66.50	3.50	30.00
PP-40	β-PP-40	57.00	3.00	40.00

Apparatus and characterization procedures

DSC measurements were made on a Perkin-Elmer DSC-7 DSC, the temperature calibrated with indium in nitrogen atmosphere. About 5 mg sample was weighted. It was heated to 240 °C at heating rate of 100 °C min^{-1}, held there for 5 min, and then cooled to 50 °C at cooling rate of 10 °C min^{-1}. The sample was reheated to 200 °C at heating rate of 10 °C min^{-1} for melting characteristics study.

WAXD experiment was conducted with a Rigaku Geigerflex Model D/Max-IIIA rotating anode X-ray diffractometer. Graphite monochromatic Cu Kα radiation was employed as a radiation source. The scanning range was 5°–30° with the rate of 4° min^{-1} and a step length of 0.02. In order to remain the thermal history as same as the DSC measures, the samples used in WAXD measures were prepared in DSC by heating up to 240 °C and held there for 5 min, then cooled to 50 °C at scanning rate 10 °C min^{-1}. The k_β value representing the content of β-PP in such blends was calculated from X-ray diffractograms according to Turner-Jones et al. [33]

$$k_\beta = \frac{H_{\beta(300)}}{H_{\alpha(110)} + H_{\alpha(040)} + H_{\alpha(130)} + H_{\beta(300)}}, \qquad (1)$$

where $H_{\alpha(110)}$, $H_{\alpha(040)}$, and $H_{\alpha(130)}$ are the intensities of α-diffraction peaks corresponding to angles 2θ equals 14.2°, 17.0°, and 18.8°, respectively, and H_β is the intensity of β-diffraction peak at 2θ equaling 16.2°.

The observation of crystallization morphology for samples was performed using a Leitz Orthoplan Pol microscope equipped with a Linkam THMS-600 hot stage. The thermal history of samples for crystallization morphology investigation was the same as that of WAXD samples.

Results and discussion

β-Nucleation of β-PP/ABS blends prepared by method one

Figure 1 shows DSC crystallization (a) and melting (b) thermograms of β-PP/ABS blends prepared by method one, the relative data are listed in Table 2. It can be seen that the crystallization peak temperature (T_{cp}) of PP increases from 119.6 to

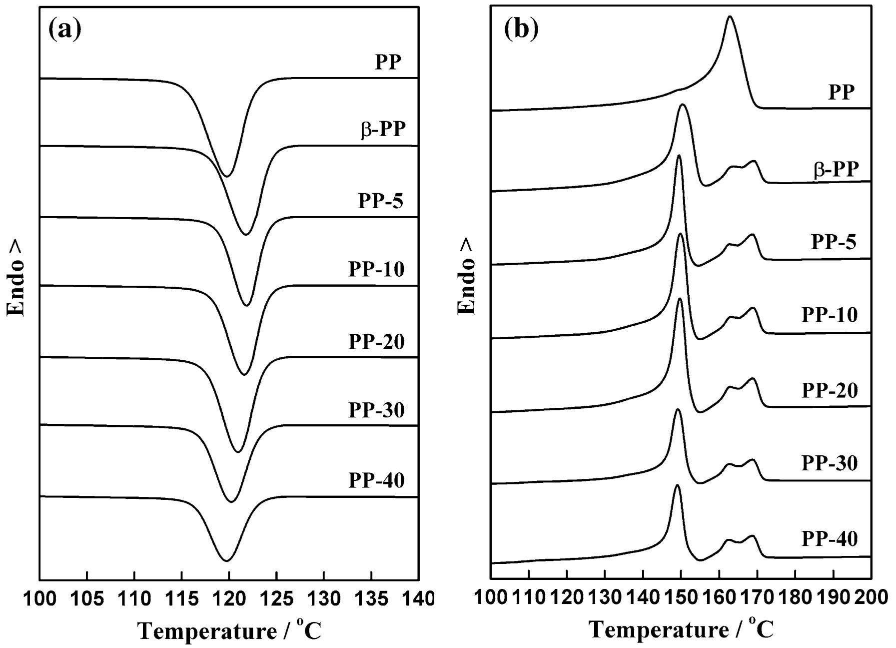


Fig. 1 DSC cooling (**a**) and heating (**b**) thermograms of neat PP, *β*-PP, and *β*-PP/ABS blends prepared by method one

Table 2 DSC data of neat PP, *β*-PP and *β*-PP/ABS blends prepared by method one

Sample	Crystallization			Melting					k_β
	T_c^{on} (°C)	T_{cp} (°C)	ΔH_c (J g^{-1})	T_{mp} (°C)			$\Delta H_{m(\beta)}$ (J g^{-1})	$\Delta H_{m(\alpha)}$ (J g^{-1})	
				β	α_1	α_2			
PP	122.4	119.6	107.5	–	162.8	–	–	102.7	0
β-PP	124.5	121.8	87.9	150.6	163.8	169.6	74.6	24.6	0.99
PP-5	124.1	121.9	85.8	149.5	162.5	168.7	93.2	27.7	0.96
PP-10	124.1	121.6	83.4	149.9	162.8	168.8	83.6	24.6	0.98
PP-20	123.6	121.0	76.0	149.7	162.6	168.7	68.9	23.3	0.95
PP-30	123.3	120.3	66.9	149.2	162.3	168.7	58.8	22.0	0.97
PP-40	122.9	119.7	56.7	149.1	162.1	168.7	46.5	18.9	0.98

121.8 °C by incorporation of *β*-NA. Moreover, addition of ABS has little influence on the T_{cp} of PP in *β*-PP/ABS blends, which is almost the same with *β*-PP as the ABS content goes below 10 wt%. However, it decreases down to 119.7 °C as the ABS content comes up to 40 wt%. It suggests that amorphous ASB has no heterogeneous nucleation for PP crystallization, but the crystallization is baffled by excessively high content of ABS.

From DSC melting thermograms, it can be observed that pure PP presents only one melting peak at temperature of 162.8 °C, attributed to the fusion of *α*-crystal.

β-PP and β-PP/ABS blends show three melting peaks: One strong melting peak at low temperature of 150 °C is due to the fusion of β-crystal, and the others at high temperature of 162 and 168 °C are corresponded to the fusion of α_1- and α_2-crystal, respectively [34]. The melting peak of β-crystal is stronger than that of α-crystal in all β-PP and β-PP/ABS blends. Compared to β-PP, addition of ABS has little effect on the melting peak temperatures (T_{mp}) and the relative intensity of β-crystal, α_1-crystal, and α_2-crystal of PP. It indicates that the presence of ABS phase and content of ABS have little influence on the β-nucleation and melting behavior of PP in the β-PP/ABS blends. The β-PP/ABS blends with strong β-nucleation and high β-crystal content can be easily obtained by method one.

Figure 2 presents the X-ray diffraction diagrams of β-PP/ABS blends. It shows that β-PP and β-PP/ABS blends mainly form β-crystal, while PP only forms α-crystal. The β-crystal content (k_β) calculated from the Eq. (1) based on Fig. 2 is listed in Table 2. The results show that the β-PP containing β-crystal content of 0.99 was obtained for PP filled by 5 wt% β-NA. Although the β-crystal content in β-PP/ABS blends prepared by method one slightly decreases, the k_β values are still higher than 0.95. It is considered that the ABS content has little influence on the β-crystal content in β-PP/ABS blends. All the above results indicate that β-NA prepared in our lab possesses high efficient β-nucleation for PP crystallization, induced PP to form almost pure β-PP. Addition of ABS has little influence on the β-nucleation of β-PP, and β-PP/ABS blends with the β-crystal content above 0.95 could be easily prepared by simultaneously mixing PP, β-NA, and ABS.

β-Nucleation of β-PP/ABS blends prepared by method two

Figure 3 presents DSC crystallization (a) and melting (b) thermograms of β-PP/ABS blends prepared by method two, the corresponding data are listed Table 3. It

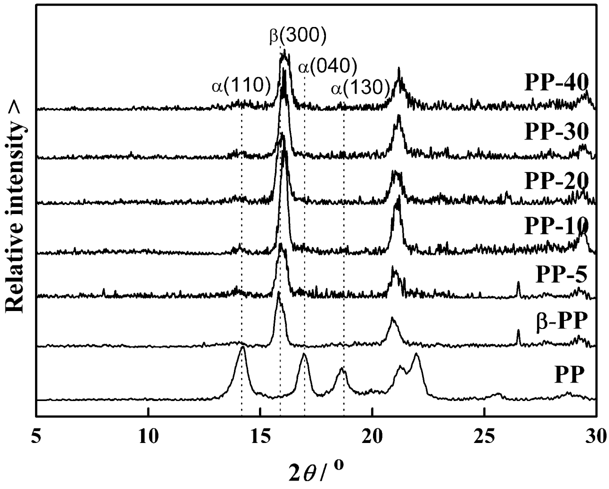


Fig. 2 WAXD spectra of neat PP, β-PP, and β-PP/ABS blends prepared by method one

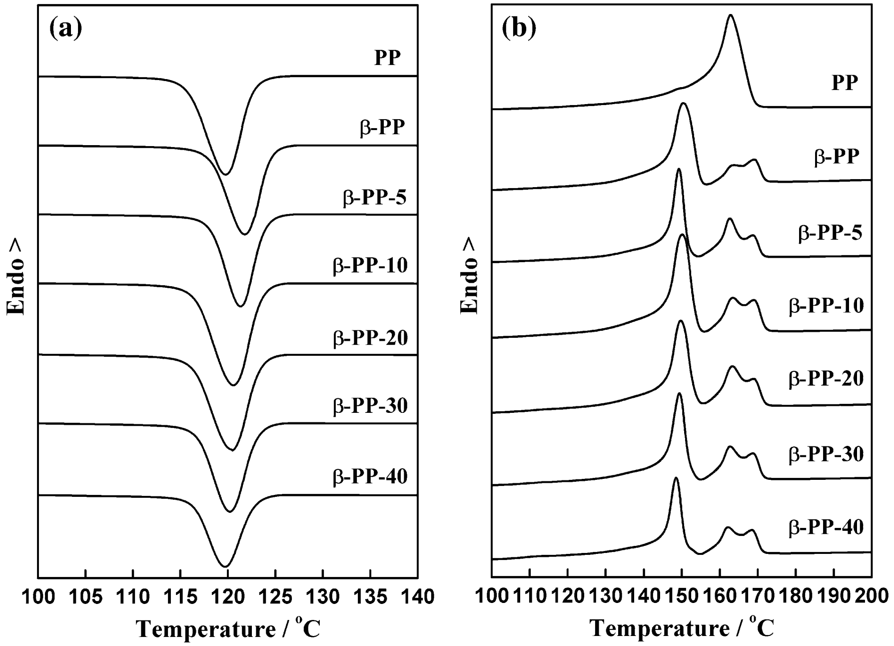


Fig. 3 DSC cooling (**a**) and heating (**b**) thermograms of neat PP and β-PP/ABS blends prepared by method two

Table 3 DSC data of neat PP and PP in β-PP/ABS blends prepared by method two

Sample	Crystallization			Melting					k_β
	T_c^{on} (°C)	T_{cp} (°C)	ΔH_c (J g^{-1})	T_{mp} (°C)			$\Delta H_{m(\beta)}$ (J g^{-1})	$\Delta H_{m(\alpha)}$ (J g^{-1})	
				β	α_1	α_2			
PP	122.4	119.6	107.5	–	162.8	–	–	102.7	0
β-PP	124.5	121.8	87.9	150.6	163.8	169.6	74.6	24.6	0.99
β-PP-5	124.0	121.3	86.9	149.2	162.6	169.1	58.4	23.9	0.84
β-PP-10	123.6	120.6	80.7	150.1	163.4	169.4	57.9	25.2	0.83
β-PP-20	123.4	120.5	75.7	149.7	163.4	169.3	45.2	25.2	0.80
β-PP-30	123.2	120.2	67.6	149.4	162.7	169.1	41.5	23.6	0.85
β-PP-40	122.8	119.7	56.8	148.5	162.2	168.8	35.9	20.6	0.90

can be seen that the crystallization and melting behavior of β-PP/ABS blends prepared by method two is similar to those of β-PP/ABS blends prepared by method one. Addition of ABS slightly decreases the T_{cp} of PP, all the melting thermograms of β-PP and β-PP/ABS blends show three melting peaks, corresponding to the melting of β-, α_1-, and α_2-crystal, respectively, whose T_{mp}'s slightly decrease with increasing ABS content. However, compared with β-PP/ABS blends prepared by method one, the melting peak of α_1-crystal is stronger than that of α_2-crystal for β-

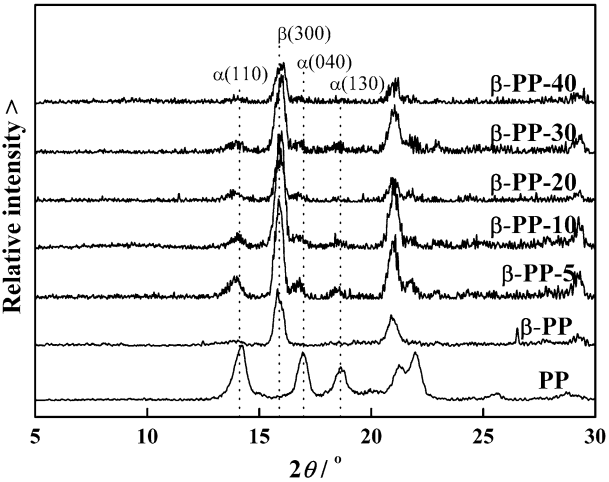
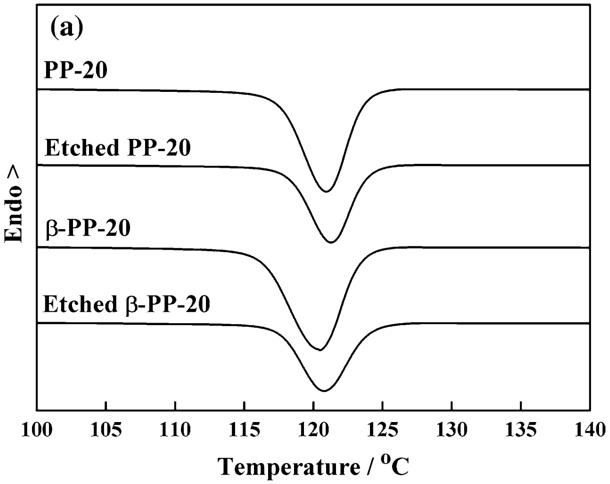


Fig. 4 WAXD spectra of neat PP and β-nucleated PP/ABS blends prepared by method two

PP/ABS blends prepared by method two. Varga [35] suggested that the low temperature α_1-peak corresponds to the melting of the α-crystal formed during the primary crystallization, but the high temperature peak reflects to melting of the α-crystals formed as a result of β to α recrystallization during the partial melting of the β-crystal. It suggests that the β-PP/ABS blends prepared by method two crystallizes in more α-crytal during crystallizaiton process, which was proved by WAXD measurement. Based on Eq. 1 and Fig. 4, the β-crystal content is listed in Table 3. The β-crystal content in β-PP/ABS blends prepared by method two is lower than that by method one. Nevertheless, the k_β values are higher than 0.80. The above results indicate that addition of ABS has a little influence on the β-nucleation of PP in such blends prepared by method two and PP in such blends mainly forms β-crystal, too. β-PP/ABS blends with the β-crystal content above 0.80 can be obtained by mixing β-PP with ABS.

β-Nucleation of etched β-PP/ABS blends

In order to confirm the dispersion of β-NA, the blends were etched with sulfuric acid to remove ABS phase. Figure 5 shows DSC crystallization (a) and melting (b) thermograms of β-PP/ABS blends etched with sulfuric acid, the relative data are listed in Table 4. The PP-20 and β-PP-20 (β-PP/ABS blends with 20 wt% ABS) prepared by methods one and two, respectively, were selected. It can be seen that the T_{cp} of β-PP/ABS blends etched by sulfuric acid, in spite of preparative method, is similar to that of β-PP/ABS blends without treatment. Although the etched blends also present three melting peaks, compared with the blends without treatment, the melting-peak intensity of β- and α_2-crystal decreases while that of α_1-crystal increases, resulted in the decreased β-crystal content. The β-crystal content, calculated in Fig. 6 and listed in Table 4, also indicates that sulfuric acid etching decreases the β-nucleation of β-PP/ABS blends, especially in the blend prepared by



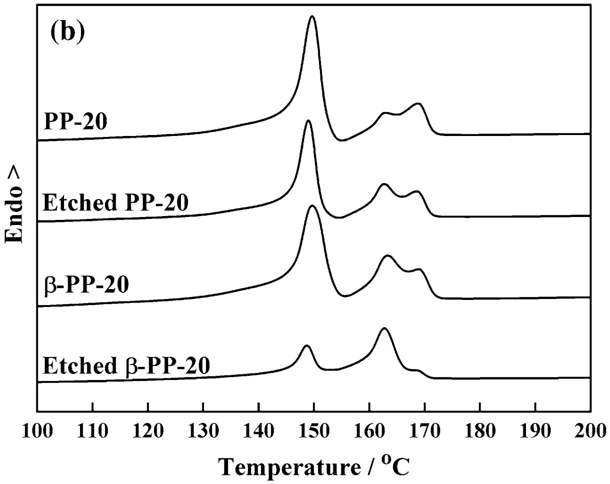


Fig. 5 DSC cooling (**a**) and heating (**b**) thermograms of β-PP/ABS etched by sulfuric acid for 12 h

Table 4 DSC data of β-PP/ABS blends etched by sulfuric acid for 12 h

Sample	Crystallization			Melting					k_β
	T_c^{on} (°C)	T_{cp} (°C)	ΔH_c (J g^{-1})	T_{mp} (°C)			$\Delta H_{m(\beta)}$ (J g^{-1})	$\Delta H_{m(\alpha)}$ (J g^{-1})	
				β	α_1	α_2			
PP-20	123.6	121.0	76.0	149.7	162.6	168.7	68.9	23.3	0.95
Etched PP-20	124.3	121.2	74.7	149.0	162.7	168.9	51.4	27.2	0.90
β-PP-20	123.4	120.5	75.7	149.7	163.4	169.3	45.2	25.2	0.80
Etched β-PP-20	125.9	120.8	71.4	148.7	162.8	–	25.8	31.1	0.49

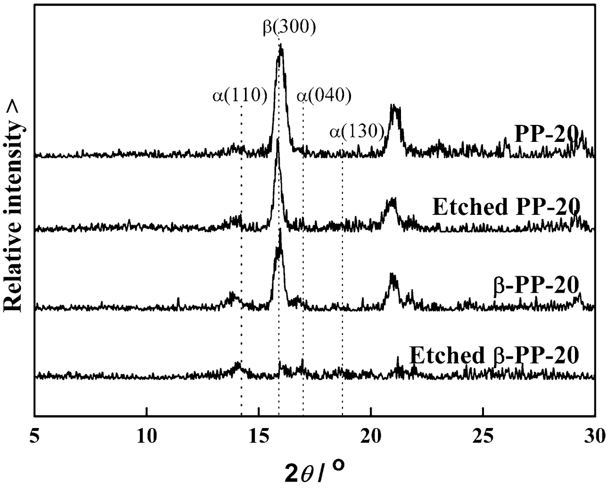


Fig. 6 WAXD spectra of *β*-PP/ABS blends etched by sulfuric acid for 12 h

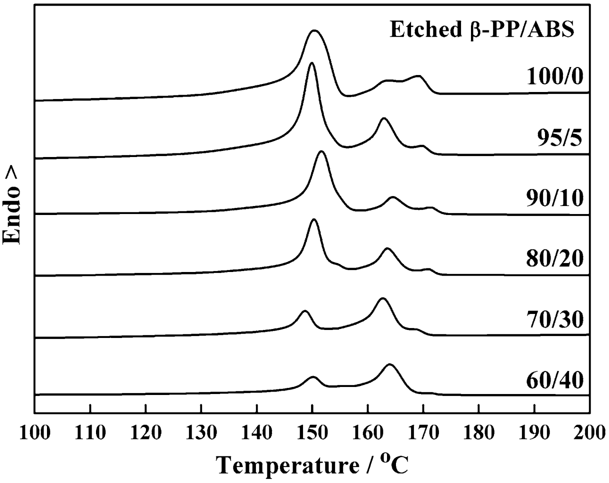


Fig. 7 DSC heating thermograms of *β*-PP/ABS blends (prepared by method two) etched by sulfuric acid for 12 h

method two. Therefore, *β*-PP/ABS blends with different ABS contents prepared by method two (noted as *β*-PP-X) were selected to etch by sulfuric acid. Figure 7 shows their DSC heating thermograms, and the relative data are listed in Table 5. It can be seen that in the *β*-PP-X etched by sulfuric acid, the melting-peak intensities of *β*- and α_2-crystal significantly decrease while that of α_1-crystal (formed during the primary crystallization) obviously increases with increasing ABS content, indicated that the *β*-crystal content decreases with the increasing ASB content.

Table 5 DSC heating data of β-PP/ABS blends (prepared by method two) etched by sulfuric acid for 12 h

β-PP/ABS	$T_{m(\beta)}$ (°C)	$T_{m(\alpha1)}$ (°C)	$T_{m(\alpha2)}$ (°C)	$\Delta H_{m(\beta)}$ (J g^{-1})	$\Delta H_{m(\alpha)}$ (J g^{-1})
100/0	150.6	163.8	169.6	74.6	24.6
90/5	149.9	163.0	170.0	47.3	16.4
90/10	148.7	162.1	168.8	50.3	31.3
80/20	148.7	162.8	–	25.8	31.1
70/30	148.5	162.3	–	15.7	29.5
60/40	150.1	164.1	–	9.1	25.5

However, that in the β-PP/ABS without treatment is little affected by ABS content as previously mentioned. According to the above analysis, it is considered that due to the polar interaction the β-NA mainly dispersed in the dispersed phase of ABS or the interface between PP and ABS. Therefore, the β-NA was etched along with ABS, resulted in lower β-nucleation for PP crystallization and less formation of β-PP.

Crystal morphology of β-PP/ABS blends

Figure 8 shows the POM of β-PP/ABS blends prepared by method two. Spherulites of weak and positive birefringence are of the α-crystal, which is a consequence of a characteristic lamellar branching, while bright, negatively birefringent spherulites are of the β-crystal [36]. It can be observed that perfect β-spherulites are obtained for PP filled by β-NA, indicating the sample nearly crystallizes completely in β-crystal. The addition of ABS has little influence on the β-spherulite morphology of PP, as the ABS content goes below 10 %. However, it can be seen that the ABS phase disperses in β-spherulite of PP as small spherular particles. As the ABS content further increased, the dispersed ABS phase forms a larger irregular morphology dispersed in the PP matrix, and retarded the growth of β-spherulite, resulting in forming irregular β-spherulites of PP in such blends and reducing integrity with increasing the content of ABS.

Conclusion

1. β-Polypropylene/ABS blends with high content of β-crystal were prepared by mixing PP, ABS, and $CaCO_3$ supported β-NA or β-PP and ABS, and the former method is more effective.
2. Addition of ABS and the increasing content of ABS have little influence on the β-nucleation and crystallization behavior and melting characteristics of β-nucleated PP, and the β-crystal content in all blends is up to above 0.80.

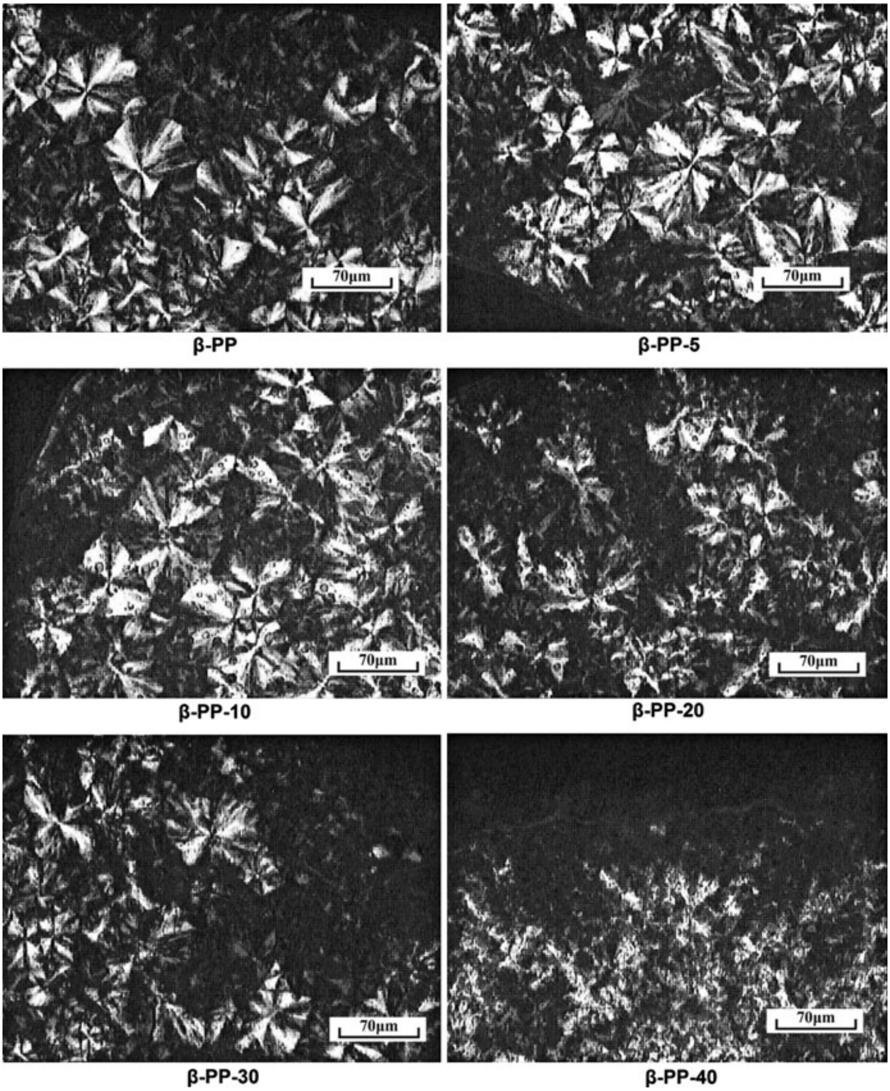


Fig. 8 POM micrographs of β-PP/ABS blends prepared by method two

3. Due to the polar interaction the β-NA mainly disperses in the dispersed phase of ABS or the interface between PP and ABS, so the β-NA was etched along with ABS, resulted in lower β-nucleation for PP crystallization.

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