

Inhibition effect of polyvinyl butyral on crystallization of polypropylene

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ABSTRACT

In this work, a series of polypropylene/polyvinyl butyral (PP/PVB) blends were prepared by melt-blending process, at PVB loadings 3 wt%, 10 wt%, and 30 wt%. The effects of PVB on crystallization behavior of PP were investigated by differential scanning calorimetry (DSC), and polarized optical microscopy (POM). The isothermal crystallization kinetics were analyzed by Avrami equations. It was found that the addition of PVB strikingly reduced the overall crystallization rate of PP. The POM results further indicated that the crystallization rate of PP/PVB was significantly reduced by reducing the nucleation density of PP with the addition of PVB. The fractured surface morphology of PP/PVB blends was characterized by scanning electron microscopy (SEM), and the results showed that the PVB was uniformly dispersed in the PP matrix as small spherical particles, with a good dispersion and dimensional stability. **Polyolefins J (2018) 5: 141-151**

Keywords: PP/PVB blend; isothermal crystallization; nucleation density; morphology.

INTRODUCTION

Isotactic polypropylene (iPP) is one of the most important thermoplastic polymers. Due to its outstanding merits including regular structure and high crystallinity, low density, heat resistance, corrosion resistance, and low manufacturing costs [1-3], PP is widely used in automotive, aerospace, packaging, pharmaceuticals, consumer products (toys, electrical appliances), elec-

tronics, cables and wire coatings, thin film materials, thermal and acoustic insulation and other building materials [4-6]. Since there is no polar group in the PP backbone, it displays poor compatibility with other materials during blending, leading to a significant phase separation and limitation in PP applications [7-9]. The perpetual interest is driven by an impetus to improve the poor interactive properties of PP for the application on much high value products, especially polymer

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blends and composites, those in which adhesion and compatibility with other materials are paramount. The investigation on the crystallization behavior and mechanical properties of PP and its blends/composites has been attracting researchers' attention for several decades. Thus, the promising feature has become the focal point of academic and industrial research.

PP has been commonly modified by the incorporation of rubbers [10], thermoplastic polymers [7, 11], block copolymers [8, 12], thermosetting resins [13], nanoparticles [6, 13], organic and inorganic fillers [14], fibers [15] and polymerization of in-reactor iPP catalyzed [15], etc. The modifications can notably improve the performance of PP, such as ductility and impact resistance. Polyvinyl butyral (PVB) is an environment-friendly multi-functional polymer, which is synthesized by the reaction of n-butyl aldehyde with poly(vinyl alcohol) in the presence of acid catalyst [16] and is considered as an excellent component for blends. PVB contains three functional groups: butyral, alcoholic hydroxyl and acetate groups, which can provide strong adhesion. Since PVB has a long side chain, it possesses a good molecule flexibility, and low glass transition temperature. The molecular structure of PVB determines its overall performance with a high tensile strength, excellent impact resistance, great adhesive property, resilience and transparency, as well as good film-forming properties [17,18]. Since PVB contains both hydrophilic and hydrophobic parts in its structure, it can be compatible/miscible with both hydrophilic and hydrophobic polymers. By virtue of its outstanding merits as described above, it is widely used in laminated glass, surface coatings and primers, adhesives and binders, as well as printing inks and toners [19,20].

It has been investigated that PVB displays good compatibility with many materials, such as specific polyamides, thermoplastic polyurethane (TPU) [21], poly(vinyl chloride) (PVC) [22], etc. Moreover, PVB greatly enhances the polymer performance, such as toughness, impact resistance, hydrophilic properties, and so on.

Lee et al.[9] investigated that the addition of recycled PVB film increased the elongation-at-break and reduced the size of disperse phase of PP/PA6 blends. Jarvela et al.[23] studied the dynamic mechanical and

mechanical properties of PP/PVB/mica composites, and demonstrated that PVB had poor compatibility with PP/mica component, and did not display reinforcing effect in the PP/mica binary composites. Costa et al.[24] studied the influence of recycled PVB in PP/wood flour composites, and their results showed that PVB acted as one possible toughening agent for PP, and the energy absorbed by the PP/PVB/wood flour composites showed high-impact performance properties for these composite products. Cascone et al. [25] studied the blends of maleic anhydride-grafted polypropylene (PP-MA) and PVB containing different contents of butyral. The results indicated good compatibility between PVB and PP-MA and showed that the mechanical performance of PP-MA was improved. However, few research studies have been reported on PVB/polyolefin binary blends, particularly PP/PVB blends.

In the present work, a series of PP/PVB blends were prepared with melt blending, and the effect of PVB on the crystallization behavior and surface morphology of PP was studied systematically by differential scanning calorimetry (DSC), polarized optical microscopy (POM) and scanning electron microscopy (SEM) techniques. It is amazing that a small amount of PVB can remarkably reduce the crystallization rate of PP. Indeed, it is proved that the mechanism of the inhibition of crystallization is due to reduction in the nucleation density of PP. The crystallization morphology and fractured surface morphology of PP and PP/PVB blends were observed by POM and SEM, respectively.

EXPERIMENTAL

Materials and blend Preparation

A commercial grade isotactic PP with a melt flow rate (MFR) of 3.4 g/10min was provided by Lanzhou Petrochemical Company. Polyvinyl butyral (PVB) (MW = 40,000-70,000), a terpolymer composed of vinyl butyral (80 wt%), vinyl alcohol (VA, 18 wt%), and vinyl acetate(VAc, 2 wt%), was purchased from Aladdin Industrial Corporation. Ethanol (AR) was purchased from Sinopharm Chemical Reagent Co., Ltd. Beijing. A series of PP/PVB blends were prepared by an internal mixer (Polylab OS Haake) at a processing

temperature of 210°C for 8 min, and at a rotor speed of 50 rpm. The ratio of PVB to PP was 3%, 10%, and 30% by weight fraction. The pellets of PP/PVB blends were molded into rectangular bars (83×12.6×1.65) using a micro injection molding apparatus (MinJet system MiniJet II Haake) at an injection pressure of 180 bar. The molding temperature was set at 40°C and the barrel zone temperature ranged from 190 to 200°C. The rectangular bars were used for SEM observations.

Characterization

DSC measurement

Isothermal crystallization process of PP/PVB was performed using a Perkin Elmer Pyris Diamond DSC under nitrogen atmosphere. The flow rate of nitrogen was 50 ml/min. The samples (3–5 mg) were first heated to 200°C at a heating rate of 50°C/min and then kept at that temperature for 5 min to erase the previous thermal history. The samples were then rapidly dropped from 200°C to the isothermal crystallization temperature (T_c) at a cooling rate of 300°C/min, and keeping for a period of time until the samples were crystallized completely, and finally heating to 200°C at a rate of 10°C/min.

The half crystallization time ($t_{1/2}$) of the samples can be obtained from DSC curves and more information like the rate of crystallization, the dimension of spherulites, and growth and nucleation kinetics can be determined by Avrami equation [26]:

$$1 - X_t = \exp(-kt^n) \quad (2.1)$$

where k is the crystallization rate constant depending on nucleation and growth rate, and n is the Avrami exponent subjected to the combined effects of the nature of nucleation, nucleation density and growth geometry of the crystals.

Optical microscopic observations

The crystalline morphology of PP and the PP/PVB blends was observed using an Olympus-BX51 polarizing optical microscope (POM) equipped with a temperature controller (Linkam THMS600) under nitrogen atmosphere. The cooling rate was controlled with the aid of liquid nitrogen. The samples used for POM

observation were films with the thickness of ca. 30 μm , which were prepared by a hot stage. The samples were heated to 200°C at a rate of 50°C/min, and then maintained at that temperature for 5 min to eliminate the thermal history. The study of the isothermal crystallization process was made at 130°C after erasing the thermal history of the samples with a cooling rate of 100°C/min. The polarized optical micrographs were taken when the samples reached to 130°C, with an interval of 5 sec to record the spherulite growth rate of the samples. The software Image-pro Plus 6.0 was used to measure the spherulite radial growth rate of the samples.

Scanning electron microscope observation

A Japan JSM-6700F scanning electron microscope (SEM) with an accelerating voltage of 5.0 kV was used to observe blends morphology. Specimens were prepared by brittle fracturing under liquid nitrogen; then the PVB was etched with anhydrous ethanol for about 48 h. Fracture surfaces were plated with gold using a sputter coater (Bal-Tech, SCD 500) before examination.

RESULTS AND DISCUSSION

Isothermal crystallization kinetics analysis

The isothermal crystallization processes of PP and PP/PVB blends were performed by DSC. Figure 1 shows the development of relative crystallinity with crystallization time for PP and PP/PVB blends at different crystallization temperatures. As shown, all of the curves are S-shaped. Meanwhile, the complete crystallization time of all samples increased with raising crystallization temperature, indicating the crystallization rate of PP and PP/PVB blends was reduced by increase in the crystallization temperature. The thermal motion of molecules becomes more active with the increase of the temperature, which result from the form of nuclei turned more difficult or more unstable.

As for formula 2.1, taking the logarithm on both sides twice, it can be deformed as following:

$$\ln[-\ln(1 - X_t)] = \ln k + n \ln t \quad (3.1)$$

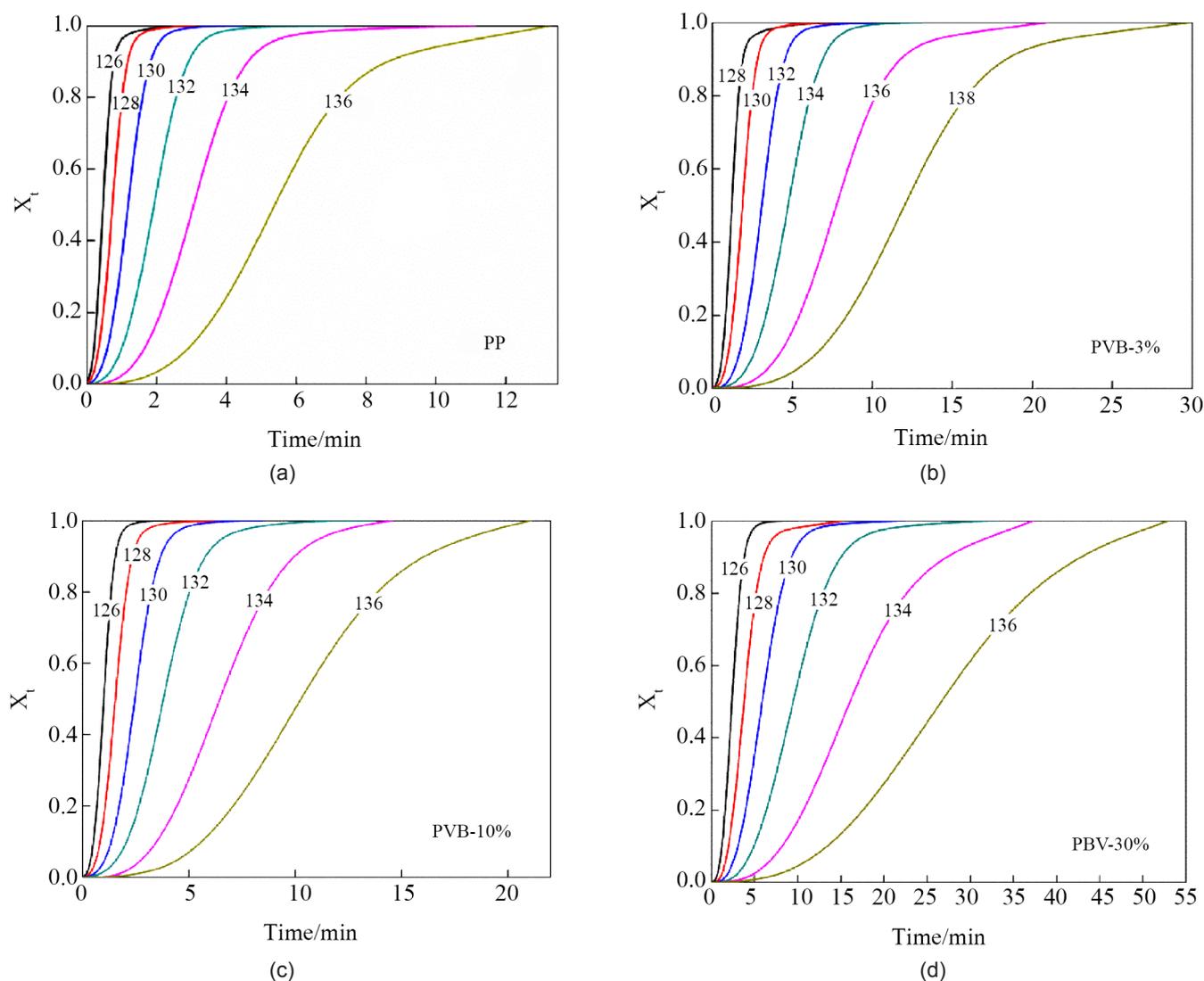


Figure 1. Development of relative crystallinity with crystallization time for (a) PP, (b) PVB-3%, (c) PVB-10% and (d) PVB-30%.

The Avrami plots of PP and PP/PVB blends (plots of $\ln[-\ln(1-X_t)]$ vs. $\ln t$) are shown in Figure 2. The Avrami parameters n and k were obtained from the slopes and intercepts, respectively. It can be seen that a series of straight lines are obtained, which means that the Avrami method can describe the plots of relative degree of crystallinity against crystallization time very well. The values of Avrami parameters for PP and PP/PVB blends are summarized in Table 1. As it is shown, the crystallization half-time ($t_{1/2}$) of the PP/PVB blends increases with increasing PVB content, indicating that the addition of PVB reduced the crystallization rate of PP. What's more, the crystallization rate of PP decreased with increasing contents of PVB. When the content of PVB was more than 50%, we could hardly

see the crystallization peak from the DSC curves of isothermal crystallization within the same temperature (graphs are not shown). Pang et al. [6] studied the ductile-brittle transition (DBT) controlled by isothermal crystallization of PP and its blend with poly(ethylene-co-octene)(PEOc), it was demonstrated that the addition of PEOc decreased the crystallization rate of PP and delayed the DBT occurrence for the PP/PEOc blend. The addition of amorphous components to a crystalline polymer can significantly reduce the rate of crystallization of the crystalline polymer, which has been reported before [28-30]. There are three reasons: firstly, the amorphous regions of PVB can limit the shift of PP molecular chains, and the crystallization of PP may also be restricted by the increasing content of PVB; secondly, the viscosity of the PP/PVB increases

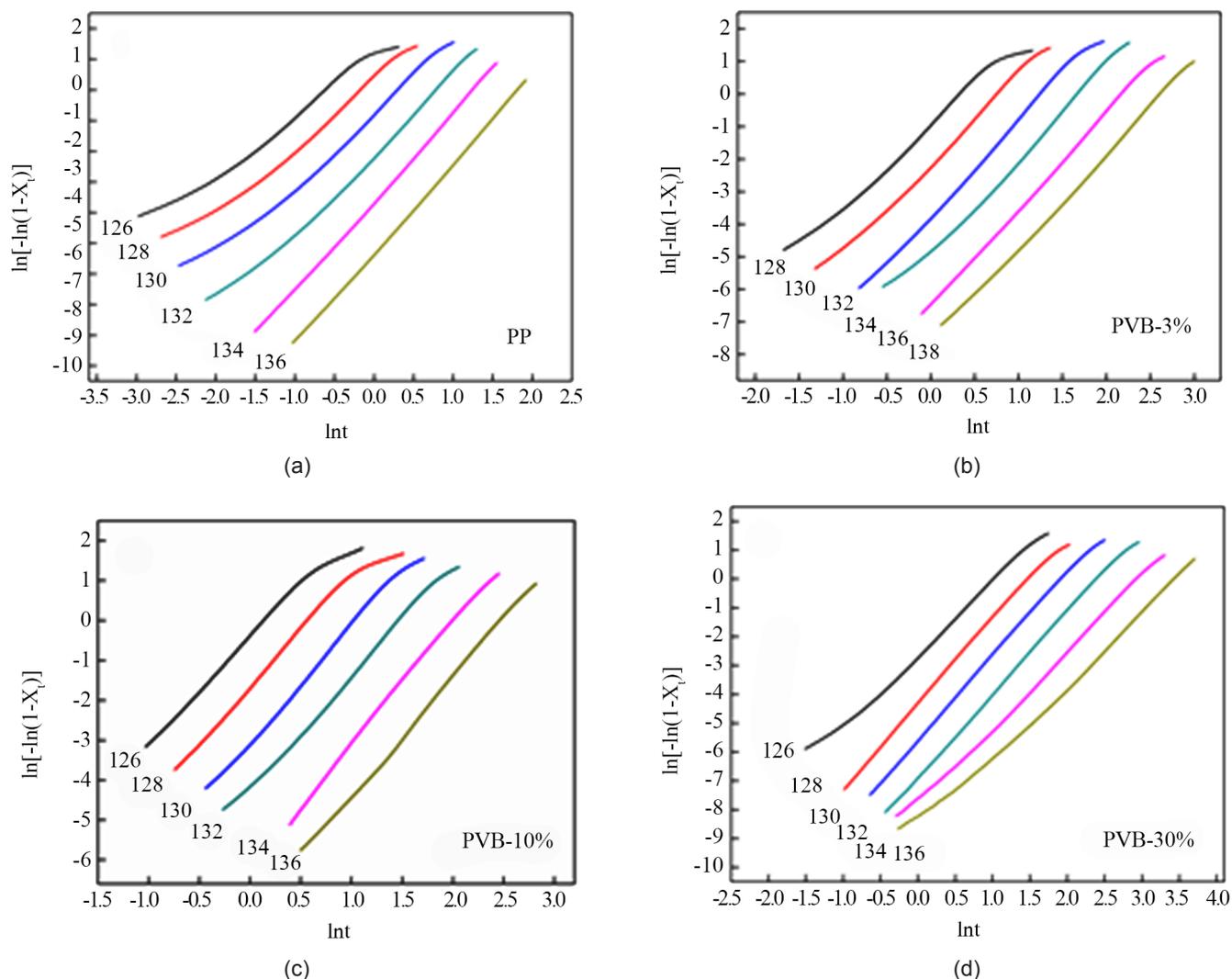


Figure 2. Avrami plots of (a) PP, (b) PVB-3%, (c) PVB-10% and (d) PVB-30%.

with the increasing of PVB content, and the rate of diffusion of the molecules to the growing crystals is obstructed, causing the decrease of the overall crystallization rate; thirdly, the PVB acts as a dilution of the PP at the growth front.

From Table 1, the values of n are not integers for all the samples, which can be ascribed to the secondary crystallization processes or the mixed growth or surface nucleation modes. The values of n are between 2 and 3 for PP, indicating a relative heterogeneous nucleation and spherical growth during the observed temperatures. As to the PP/PVB blends, n was around 3, which was similar as PP. It was illustrated that the PVB could not change the nucleation form and the growth dimension of PP in the PP/PVB blends. According to the value of n , we can speculate that the

mode of polypropylene crystallization is heterogeneous nucleation in the blends.

Crystallization Morphology of PP and PP/PVB blends

Figure 3 shows the POM micrographs of PP and PP/PVB blends for different time at the isothermal crystallization temperature of 130°C. The figure reveals the development of birefringent disc-like formations growing from a central nucleus uniformly, in all spatial directions, as well as the spherulites with a classical Maltese cross pattern can be observed in PP and PP/PVB blends. Then, the spherulites become larger and come into contact with each other, and finally the growth fronts of the spherulites result in irregular polyhedron confined by straight or curved lines. In the blends, the amorphous component of PVB was

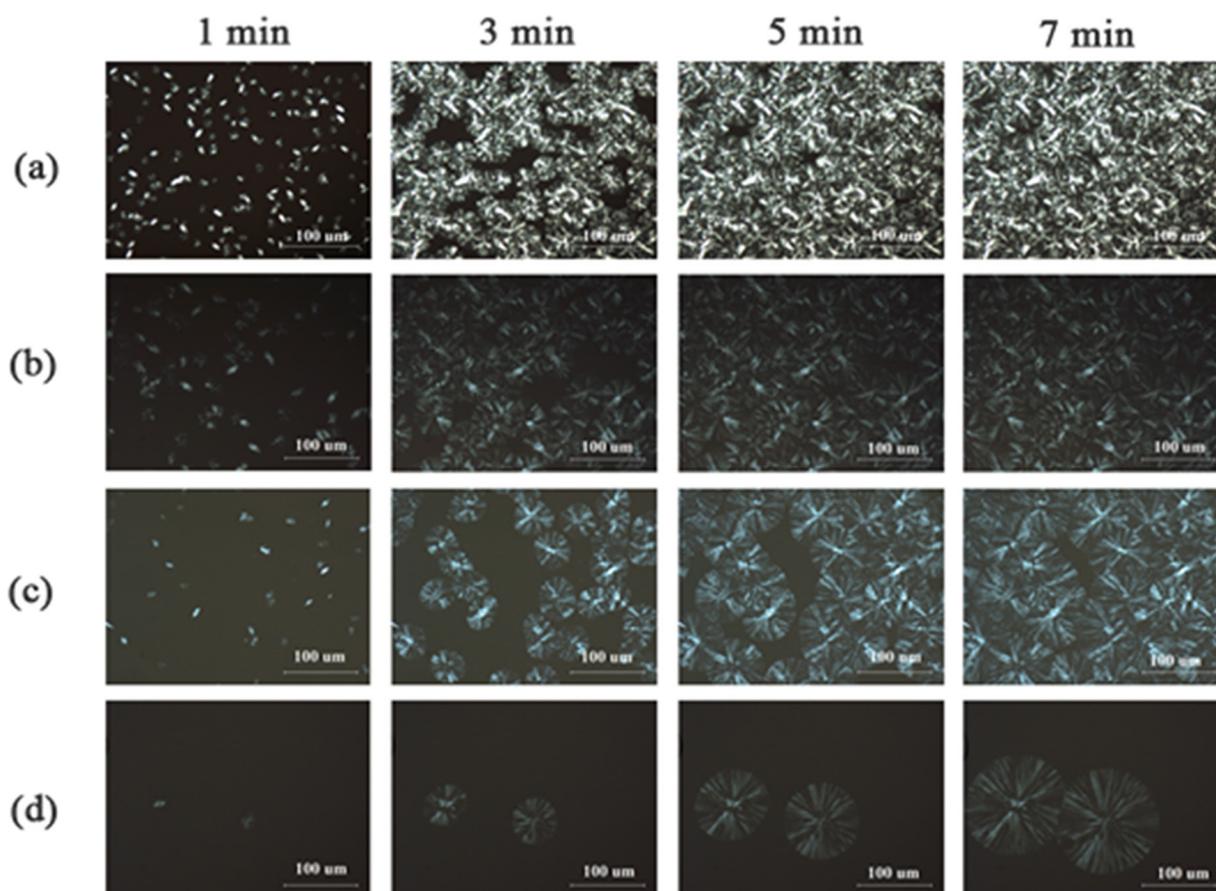
Table 1. Crystallization kinetics parameters of neat and PP/PVB blends crystallized at different temperatures.

Sample	PP			3%			10%			30%		
	$t_{1/2}/\text{min}$	n	$\ln k/\text{min}^{-n}$									
126	0.47	2.07	1.23	-	-	-	1.00	2.48	-0.44	2.39	2.68	-2.71
128	0.74	2.16	0.36	1.22	2.51	-0.94	1.55	2.58	-1.61	3.78	2.90	-4.27
130	1.18	2.36	-0.72	1.88	2.76	-2.13	2.46	2.98	-3.08	5.89	2.86	-5.51
132	1.93	2.63	-2.07	3.09	2.90	-3.73	3.82	2.88	-4.25	9.46	2.87	-6.86
134	3.07	2.93	-3.66	4.74	2.93	-4.93	6.47	3.03	-6.09	16.06	2.77	-8.06
135	4.02	2.86	-4.38	6.15	2.87	-5.69	8.07	3.03	-6.73	21.96	2.81	-9.06
136	5.38	2.94	-5.36	7.75	2.98	-6.53	10.28	3.00	-7.41	26.63	2.69	-9.21
138	-	-	-	12.01	2.95	-7.74	-	-	-	-	-	-

dispersed well in PP spherulites, and the PVB was dispersed between the regions of the spherulites, and finally the spherulites almost filled the entire region. As can be seen from the images, the size of PP spherulites becomes larger with the increase of PVB in the blends, and the nucleation density is decreased apparently from PP/PVB (30 wt%) to PP. When the content of PVB was 30%, there were only two spherulites in the region (Figure 3(d)). At the same time, the time

to complete crystallization is also prolonged with the increase of PVB. It is clear that the nuclear density decreases and the spherulite radials increase with the increasing of the content of PVB. There have been many reports with regard to the phenomenon of the reduction of nucleation density because of the addition of second component in the blends [28-30].

The crystallization rate of PP was affected by the spherulite growth rate and the nucleation rate. The

**Figure 3.** Polarized optical micrographs of PP and PP/PVB blends crystallized at 130°C for different time (a) PP; (b) PP/PVB-3%; (c) PP/PVB-10%; and (d) PP/PVB-30%.

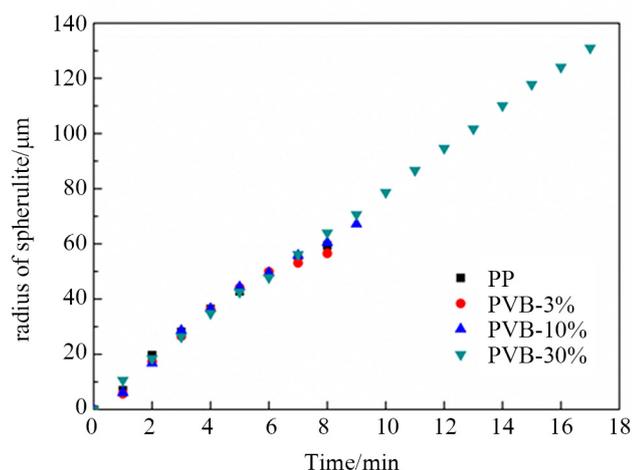


Figure 4. Spherulitic radial growth rate of PP and PP/PVB blends versus time at 130°C.

software of Image-Pro Plus 6.0 was used to measure the spherulite radial growth rate of samples (the radius was taking the average of same spherulite for 5 times). Figure 4 shows the function of the spherulite radial growth rate of PP and PP/PVB blends versus

time at the crystallization temperature of 130°C. It can be seen that the spherulite radial growth rate of the samples grows linearly over time, and the radial growth rates ($G = dr/dt$) of all samples are equal to a constant value of 7.55 $\mu\text{m}/\text{min}$. Moreover, the radial growth rate of spherulite does not change with the increase of PVB. The results consistent with the rule of theoretical predictions reported in the literature, which studied the growth rate of spherulites with impurities [31,32]. The rate of spherulite growth may be limited by the addition of PVB, but the effect of this limitation was very small and could be ignored within the experimental error.

Kuo et al.[33] studied the crystallization kinetics and morphology of binary phenolic/poly(ϵ -caprolactone) (PCL) blends, and showed that the phenolic was miscible with PCL, as well as a depression of the overall crystallization rate and the spherulite growth rate of PCL as crystallizable component was found with the addition of phenolic as amorphous component. What's

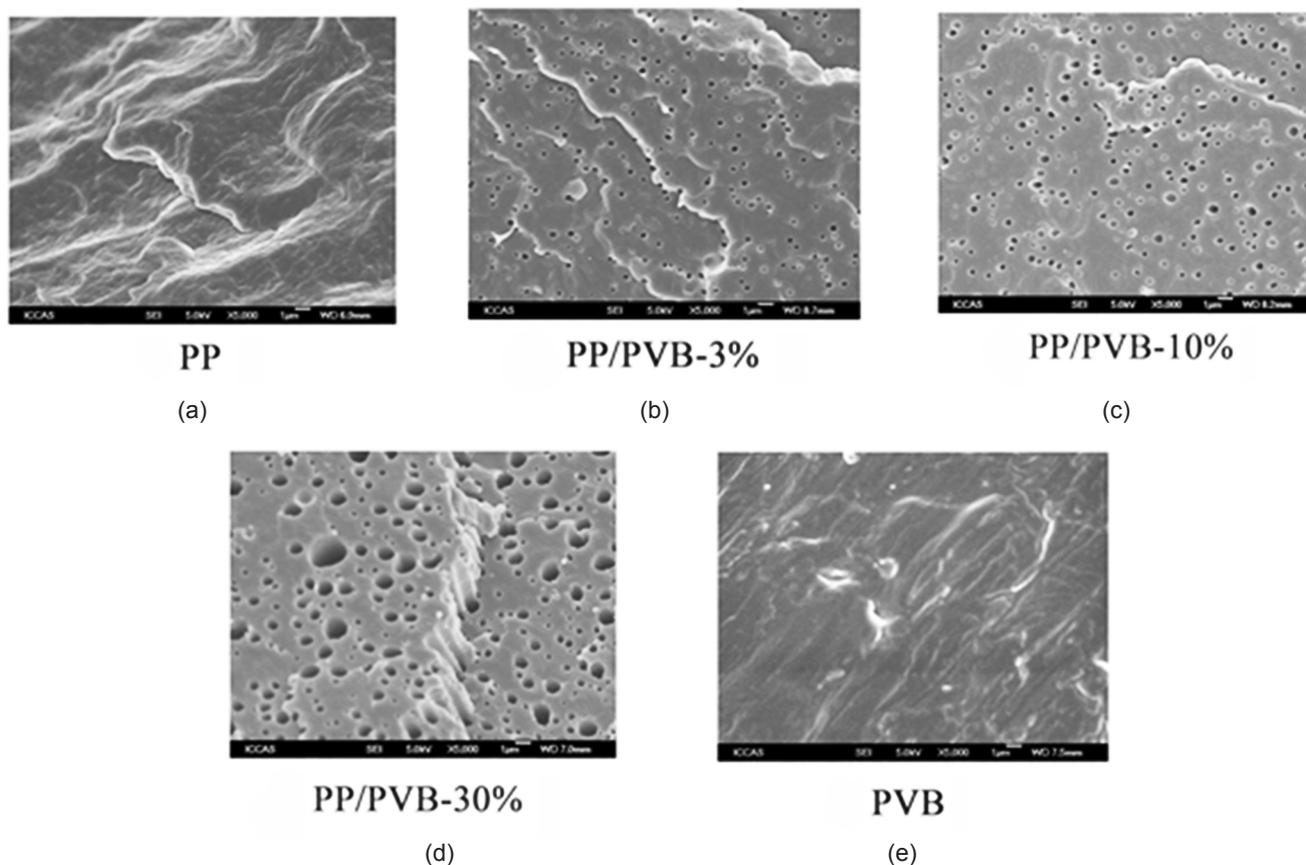


Figure 5. SEM images of fractured surfaces of PP, PVB and PP/PVB blends: (a) PP, (b) PP/PVB-3%, (c) PP/PVB-10%, (d) PP/PVB-30%, and (e) PVB. (PP/PVB blends were etched with ethanol).

more, the crystallization ability of PCL decreases with increasing phenolic content in the blends. The addition of the phenolic component to PCL causes a depression in both the overall crystallization rate and the spherulite growth rate of PCL.

In this work, the DSC and POM results showed that, the addition of PVB had an inhibited effect on the crystallization rate of PP and that it was because of the introduction of PVB which could remarkably reduce the nuclear density of PP. It was worth mentioning that the spherulite growth rate of PP/PVB blends did not change with increasing PVB content. The reason for that can be that part of the impurities causing crystallization of PP which was encased in PVB, so the crystallization point of PP decreased, which caused the

nucleation density of PP to reduce. While the spherulite growth rate of PP/PVB blends was almost unaffected. Since there are large numbers of $-OH$ groups on the PVB structure, the impurities and PVB interact with each other and some impurities may be encased in PVB, making a strong force between the impurity and the PVB. To confirm this hypothesis, the fractured surface morphology of PP and PP/PVB was observed.

3.3 Fractured surface morphology observation

The phase morphologies of PP, PVB and PP/PVB blends are displayed in Figure 5 at $\times 5000$ magnification. The fracture surfaces of samples were pressed and etched with ethanol for 48 h after fracturing in liquid nitrogen.

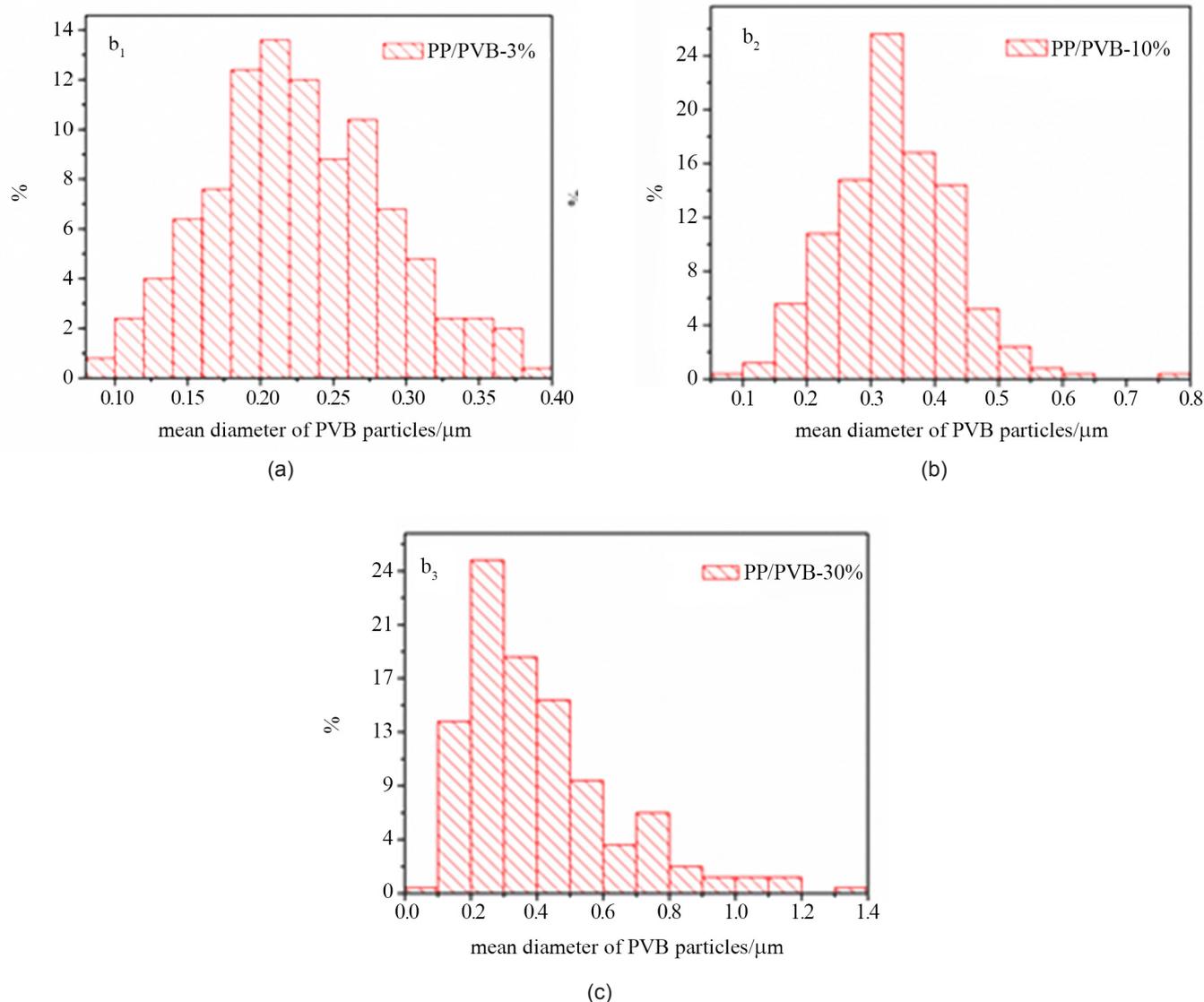


Figure 6. Histograms of particles size distribution of (a) PP/PVB-3%, (b) PP/PVB-10%, and (c) PP/PVB-30%.

Table 2. Average particle size, maximum particle size, and minimum particle size of PVB.

Particle size Sample	Mean size/ μm	Max. size/ μm	Min. size/ μm
PP/PVB-3%	0.22	0.39	0.09
PP/PVB-10%	0.33	0.78	0.09
PP/PVB-30%	0.40	1.38	0.08

As seen from these images in Figure 5, the PVB particles are uniformly dispersed in the PP matrix as pillared pore structure. And from Figure 5(d), a cylinder- or string-like structure can be clearly seen in the blend. Few authors, to our knowledge, have studied these structures. Zou et al. [34] reported that the formation of the cylinder or string phase structures will result in a phase-separated polymer blend under steady shear flow where the applied shear rate is not too large. And the further increase of shear rate leads to a blurred cylinder- or streak-like phase. In our system, there were circular edges around PVB particles, also the interface between PP and PVB was vague and a phenomenon of stress whitening was emerged between PVB phase and the matrix phase of PP, indicating good compatibility between the two phases. Moreover, it was also suggested that when the blend was subjected to an impact force, the strength could be transmitted from the matrix phase to the PVB phase, confirming that there was a strong force between the interface of PP and PVB. The distribution of particles size of the dispersion phase is shown in Figure 6. It was indicated that the particle size uniformity and the mean particle size get slightly larger as the PVB content increased. The average particle diameter, maximum particle size, and minimum particle size of PVB are shown in Table 2. It revealed that the particle size distribution was widened as the PVB content increased, and that the PVB maximum particle size was increased from 0.39 to 1.38 μm . At the same time, there was a gradually increase in the average particle size from 0.22 to 0.4 μm . From the point of phase size, PVB had a good compatibility with PP. These results indicated that the dispersion and dimensional stability of PVB in PP matrix was fine.

CONCLUSION

The crystallization kinetics and fractured surface mor-

phology in PP and PP/PVB blends were investigated. The crystallization behavior of PP was strongly influenced by PVB. The addition of the PVB could dramatically reduce the overall crystallization rate of PP, but the crystallization mechanism and crystal structure of PP did not change with the addition of PVB. The spherulitic growth rates of PP and PP/PVB blends nearly were the same, while the size of spherulite became larger and the nucleation density became significantly smaller with the incorporation of PVB. The interface between PP and PVB was vague and there was a strong force between the interface of PP and PVB, and fine dispersed phase sizes and string-like structure could be found in PP/PVB blends.

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