# Crystallization Behavior of Polypropylene in Polypropylene/Nylon 6 Blend

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**ABSTRACT:** This article describes the crystallization behavior of polypropylene (PP) in the presence of a crystallizable polymer, namely, nylon 6, in the binary blend of PP/nylon 6 in the composition range from 0 to 30 wt % of nylon 6 content in the blend. The crystallization behavior was studied through variation of the crystallinity with the blend composition and changes in the crystallization exotherms were recorded by differential scanning calorimetry (DSC) and the spherulite morphology was observed via polarized light microscopy (PLM). Comparison of the crystallization exotherms and melting endotherms revealed some differences which are attributed to the role of a sufficiently high thermal energy of the nylon 6 crystals on the melting of PP. The crystallinity of PP decreased in the presence of nylon 6, whereas the crystallinity of nylon 6 increased considerably in the presence of PP. The rate of nucleation of PP on addition of nylon 6 decreased rapidly in the region 0-10 wt % nylon 6 content, and, thereafter, at a higher nylon 6 content, decrease of the nucleation rate was relatively slow. PLM observation revealed the presence of composite spherulites with PP spherulites grown on the surface of the already-formed nylon 6 spherulites. © 1999 John Wiley & Sons, Inc. J Appl Polym Sci 71: 1153-1161, 1999

Key words: crystallization; polypropylene; nylon 6; polypropylene/nylon 6 blend

## INTRODUCTION

Polypropylene (PP), due to its high technical and economic significance, has generated enormous scientific interest. The impact toughness and other mechanical properties of its blend with other polymers has been found to be influenced by its crystallization and morphology in addition to the physical and mechanical properties of each component, state of dispersion, interfacial adhesion, etc. The effect of blending on the crystallization of PP has been extensively studied.<sup>1–10</sup> The crystallization behavior of PP in its blends with various polymers, such as polybutadiene (PBu)<sup>11</sup> and the styrene–ethylene–butylene–styrene copolymer (SEBS)<sup>12</sup> studied by one of the authors showed distinct effects of the presence of dispersed domains of the other polymer on the nucleation and growth of crystallization, thereby influencing the crystallinity, crystal size, and crystal-size distribution. Recently, blends of PP with polyamides were investigated by several authors.<sup>9,10,13–20</sup> These works were mainly confined to the study of the mechanical properties, ability to cocrystallize, degree of crystallinity, compatibility in the amorphous phase, and some new phenomena like "fractionated" and "concurrent" crystallization.

In this article, we report a study of the crystallization behavior of PP in a PP/nylon 6 blend. The results revealed variation of the nucleation rate with the blending ratio depending on the range of the blend composition. These effects were different from the effects of other amorphous polymers on the crystallization of PP.<sup>11,12</sup> Unlike the cases reported in refs. 11 and 12, the blend in the

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present case involves a crystallizable polymer (i.e., nylon 6) as the second component. The work reported here is based on the analysis of differential scanning calorimetric (DSC) crystallization exotherms as used earlier.<sup>11,12,21,22</sup> Additional studies based on polarized light microscopy (PLM) are also presented to show the role of nylon 6 crystallites in the crystalline morphology of PP.

## **EXPERIMENTAL**

#### **Materials**

The PP used in this work was Koylene S3030 of Indian Petrochemicals Corp. (Baroda, India) and nylon 6 (Gujlon M28RC) was a molding-grade product of the Gujarat State Fertilizer Corp. (Baroda). The MFIs obtained under similar testing conditions, that is, at 230°C and 2.16 kg load, were 18.2 and 25.0 g/10 min for PP and nylon 6, respectively.

#### **Blend Preparation**

PP/nylon 6 blends containing 0, 10, 20, and 30 wt % of nylon 6 were prepared using a twin-screw extruder (Brabender Plasticorder PLE 651). PP and nylon 6 were dried in a vacuum oven at 70°C for over 24 h. The granules were dry-mixed in appropriate ratios and extruded at 230°C and at a low screw speed of 10 rpm to ensure a longer residence time, thus providing good mixing. The extruded strands were cooled by quenching in a water bath at 30°C and then were granulated and injection-molded into the test specimens.

## Measurements

Crystallization exotherms and melting endotherms were recorded on a DSC (DuPont 9900 thermal analyzer having a 910 DSC module) at a cooling and heating rate of  $10^{\circ}$ C/min. Samples obtained by cutting small chips from injectionmolded specimens of about 5 mg were used. All the samples were first run through a heating cycle from ambient to 230°C and then through a cooling cycle after holding for 2 min at the highest temperature, that is, 230°C, to destroy any previous thermal history and crystallization. The degree of crystallinity (X) was calculated from the DSC data using the following relationship:

$$X = (\Delta H_c / \Delta H_c^{\circ}) \times 100$$
 (1)

where  $\Delta H_c$  is experimental heat of fusion and  $\Delta H_c^{\circ}$ is the heat of fusion for a 100% crystalline polymer. Values of  $\Delta H_c^{\circ}$ , which are taken from the literature, are 207 J/g for  $PP^{23}$  and 204.8 J/g for nylon 6.24 Hot-stage PLM measurements were made on a Mettler FP82 using a Leitz polarizing microscope. Samples as flat chips of about a 5-mm<sup>2</sup> area cut from the injection-molded specimens were crystallized on the microscope hot stage and the formation of spherulites and their growth were observed and photographed at identical crystallization times under polarized light. The crystallization samples were melted and annealed for 2 min at 230°C in order to destroy all self-seeding nuclei and then rapidly cooled to the desired isothermal crystallization temperature, that is, 195 and 125°C, corresponding to the crystallization temperature of nylon 6 and PP, respectively.

### **RESULTS AND DISCUSSION**

#### **Differential Scanning Calorimetry**

#### General Observations on Crystallization Behavior

The crystallization exotherms of the PP/nylon 6 blends and their pure components are shown in Figure 1. The pure PP and nylon 6 show single peaks around 112 and 188°C, respectively, whereas the exotherms of the blends show two separate peaks, each corresponding to the individual components. The area of the nylon 6 peak increases and that of the PP peak decreases with increasing nylon 6 content of the blend. These peak areas, representing the enthalpy of the concerned exothermic process, are proportional to the crystallinity of the concerned polymer according to eq. (1), which is shown as (X) in Table I. It is seen that the crystallinity of the concerned polymers in the blend does not vary in the same proportion as does the weight fraction of the corresponding polymer in the blend, implying a considerable effect of blending on the crystallization of each individual component of the blend or, in other words, it suggests that the crystallization of any one component is influenced by the presence of the second component.

The degree of crystallinity of PP in the blend is smaller than its values calculated on the basis of its weight fraction in all the blend compositions studied. This suggests that the presence of the nylon 6 component (already in a solidified state at the crystallization temperature of PP) has reduced the degree of crystallinity of PP in the blend, whereas the presence of PP, which is in a



**Figure 1** DSC crystallization exotherms, recorded during cooling cycle at cooling rate of 10°C/min, for PP/nylon 6 blend at various compositions (wt % nylon 6 content): (a) 0%; (b) 10%; (c) 20%; (d) 30%; (e) 100%.

molten state during the crystallization of nylon 6, has increased the overall degree of crystallinity of the nylon 6 in the blend. This effect is much more pronounced at a higher nylon 6 content of the blend. At 30 wt % nylon 6 content, the degree of crystallinity achieved for nylon 6 is about two times higher than its value calculated on the basis of its weight fraction. Variations of the degree of crystallinity of the individual components of the blend, as a function of the weight fraction of the respective components in the blend, were plotted [Fig. 2(a,b)] and their best linear fits were determined by a regression analysis which yield the following relationships for the two components of the blend:

$$(X)_{\rm PP} = 49.4 \ \phi_{\rm PP} - 11.9 \tag{2}$$

$$(X)_{\text{nvlon 6}} = 62.6 \phi_{\text{nvlon 6}} - 0.59 \qquad (3)$$

where X represents the degree of crystallinity, and  $\phi$ , the weight fraction of the respective components denoted by the subscripts. This observed difference in the slopes, that is, the rate of increase of the crystallinity with the weight fraction of the respective polymer, may be attributed to the effect of one component on the crystallization behavior of the other. The increase of the crystallinity of PP with the weight fraction of PP in the blend is slower than is the increase of the crystallinity of nylon 6 with the weight fraction of nylon 6 in the blend. This indicates that the crystallization of nylon 6 encounters less restriction due to the presence of PP in its molten state than does the crystallization of PP which occurs in the presence of the already-solidified nylon 6 component.

#### Analysis of Crystallization Exotherms

A comparison of the crystallization exotherms recorded under identical experimental conditions

PP	Crystallization Exoth	erm	Nylon 6 Crystallization Exotherm			
$\phi_{ m (PP)}$ (Wt Fraction)	$X_{(\mathrm{PP})} \ (\mathrm{Experimental}) \ (\%)$	$X_{ m (PP)} \  m (Calculated)^a \  m (\%)$	$\phi_{(nylon \ 6)}$ (Wt Fraction)	$X_{(nylon \ 6)} \ (Experimental) \ (\%)$	$X_{(\mathrm{nylon}\;6)}$ $(\mathrm{Calculated})^{\mathrm{a}}$ $(\%)$	
1.0	39.1	39.1	0.0	0.00	0.00	
0.9	31.1	35.2	0.1	6.4	3.3	
0.8	25.8	31.3	0.2	8.7	6.5	
0.7	24.4	27.4	0.3	20.1	9.8	
0.0	0.0	0.0	1.0	32.5	35.5	

 Table I
 Comparison of Exotherm Peak Areas of PP/Nylon 6 Blends as a Function of Weight Fraction of the Respective Polymers

<sup>a</sup> Calculated on the basis of "in proportion to the weight fraction of the respective polymer."



**Figure 2** Variation of crystallinity  $X_{\rm PP}$  and  $X_{\rm nylon6}$  with weight fraction  $\phi_{\rm PP}$  and  $\phi_{\rm nylon6}$  of the corresponding components of the PP/nylon 6 blend: (a) for PP; (b) for nylon 6.

and normalized for identical sample weights leads to additional information about the behavior of the individual components of the blend. An analysis of the PP exotherms is presented below to study the crystallization behavior of PP at various compositions of the blend. This analysis is based on the changes in the exotherm parameters defined in Figure 3 on a schematic exotherm. These parameters and their relationships with the crystallization process and morphology are stated below:

- 1.  $S_i$ , the initial slope of the exotherm. This is influenced by the initial process of crystallization, namely, nucleation. The faster the nucleation, the greater will be the  $S_i$ .
- 2.  $\Delta W$ , the width of the exotherm at its halfheight. This is dependent on the crystallite-size distribution such that the narrower the crystallite-size distribution the smaller will be the  $\Delta W$ . The observed similarity in the trends of the variation of the widths of the melting endotherm and the crystallization exotherm in various PPbased blends studied earlier<sup>11,12,21,22</sup> and the well-accepted origin of the variation of the width of the melting endotherm suggest that the width of the crystallization exotherm may be attributed to the crystallite-size distribution.
- 3. A/m, the area (A) of the exotherm divided by the mass (m) of the concerned polymer



**Figure 3** Schematic diagram of a crystallization exotherm recorded during the cooling cycle and the various parameters characterizing it.



**Figure 4** DSC crystallization exotherms of PP at various blend compositions (wt % nylon 6 content): (—) 0%; (-··) 10%; (-··) 20%; (-··-) 30%.

in the blend. This is proportional to the degree of crystallinity.

- 4.  $T_{\text{onset}}$ , the temperature at which the thermogram departs from the baseline at the beginning of the exotherm. Its higher value implies the occurrence of the process at a higher temperature, which is an indication of the faster rate of the process.
- 5.  $T_p$ , the exotherm peak temperature. This is dependent on the overall rate of crystallization, as it is a measure of supercooling. Supercooling,  $T_m - T_p$ , where  $T_m$  is the melting temperature, is known to govern the rate of crystallization and the morphology.

This five-parameter model of analysis would provide information on the crystallization behavior and the resulting morphology of the crystalline phase. However, its essential requirement is that the exotherms be recorded under identical experimental conditions. Self-consistency of this model of analysis is inherent in the mutually opposite variation of  $S_i$  and  $\Delta W$ , which implies that an increase of  $S_i$ , that is, an increase of the rate of nucleation, should result in a decrease of  $\Delta W$ , that is, a narrower distribution of the crystallite size. The reason for this is that faster nucleation involves an almost simultaneous creation of most crystallites which on subsequent growth produce a narrow crystallite-size distribution, whereas slow nucleation involves the creation of nuclei at different instants of time which subsequently grow to widely varying sizes and thus produce a wide distribution of crystallite size. This self-consistency is obeyed by all the previously studied systems.<sup>11,12,21,22</sup>

The data of Figure 1 were analyzed for these five parameters by replotting the exotherms on a larger scale (identical for all samples) as shown in Figure 4, for greater accuracy of the parameters calculated. Variations of the parameters,  $S_i$ ,  $\Delta W$ , A/m,  $T_{\text{onset}}$ , and  $T_p$ , for the PP exotherm in the PP/nylon 6 blend as a function of the blend composition are shown in Figure 5. On the initial addition of nylon 6 (i.e., on going from 0 to 10% nylon 6 content of the blend), both  $T_{\text{onset}}$  and  $T_p$ show a sharp decrease from their values for unblended PP, and, thereafter, both  $T_{\text{onset}}$  and  $T_p$ increase slightly with increasing nylon 6 content. This is due to the fact that only a small quantity of nylon 6 is enough for nucleation of the crystallizing PP component of the blend. Variation of  $T_p$ is much smaller than that of  $T_{\text{onset}}$ , implying that the blending with nylon 6 has a greater effect on the initial process (i.e., nucleation) than on the overall crystalline growth.

The initial slope,  $S_i$ , decreases in the blend composition range from 0 to 10% nylon 6 content and, thereafter, it increases between 10 and 30% nylon 6 content. This implies that the presence of



**Figure 5** Variations of crystallization exotherm parameters,  $S_i$ ,  $\Delta W$ , A/m,  $T_p$ , and  $T_{\text{onset}}$  with blend composition (wt % nylon 6 content).

nylon 6 in a small quantity (i.e., less than 10%) slows down the nucleation rate. This slow rate of nucleation is accompanied by the decrease of  $T_{\rm onset}$ , confirming thereby a kinetically slower process. The  $\Delta W$  increases in this range of blend composition, which fulfills the above-mentioned self-consistency condition and indicates an increase in the crystallite-size distribution as compared to that in unblended PP.

The situation for a nylon 6 content greater than 10% is quite different. The trends of variation of these parameters are reversed such that  $S_{\nu}$   $T_{p}$ , and  $T_{onset}$  increase while  $\Delta W$  decreases with increasing nylon 6 content, implying an increase in the rate of nucleation with its accompanying effect of widening the crystallite-size distribution. The magnitude of the variation of  $T_p$  is smaller than that of  $T_{\text{onset}}$ , which implies that the effect of blending is less prominent in the overall crystallization than on the nucleation. It seems, at a nylon 6 content greater than 10%, the nylon 6 component enhances the nucleation rate while crystallinity shows little variation or a slight decrease with increasing nylon 6 content of the blend, as seen from the variation of A/m in Figure 5.

The degree of crystallinity of PP, that is, the related parameter A/m, decreases rapidly on in-

creasing nylon 6 content of the blend from 0 to 10% and, thereafter, it stabilizes or slightly decreases with increasing nylon 6 content of the blend (Fig. 5). The rate of nucleation is considerably affected by the initial addition of nylon 6 (i.e., 10%) and, thereafter, the effect becomes insignificant, because for nucleation a large nylon 6 content is not required. Thus, the above analysis of variations in the DSC thermograms reveals a clear distinction of the crystallization behavior and morphology in the two regions of the blend composition, namely, at low and high nylon 6 contents:

- (i) At low nylon 6 content (i.e., 0-10%), the rate of nucleation decreases on addition of nylon 6 accompanied by a broader crystallite-size distribution. This occurs due to sporadic creation of nuclei, which, in turn, results in the coexistence of small and large crystallites with a wide size distribution. Furthermore, the overall crystallization rate of PP shows a quite small decrease and the crystallinity of PP decreases in this region of blend composition.
- (ii) In the region of high nylon 6 content (i.e., above 10 wt % nylon 6 content), the variations in  $S_i$ ,  $T_p$ ,  $T_{onset}$ , A/m, and  $\Delta W$  are relatively smaller than in region (i), indicating that the crystallization of PP varies very little with the blending ratio.

# Analysis of Melting Endotherms

Some interesting effects of the blend composition dependence of melting behavior are observed in these blends. For this study, the melting endotherms of these samples were recorded after going through identical crystallization cycles, which was achieved by freshly crystallizing the sample from the melt during a cooling run in the DSC experiment using an identical cooling rate for all the samples. The peak area ratio (area of nylon 6 peak/area of the PP peak) at any given blend composition is identical irrespective of whether it is for the crystallization exotherm peak or the melting endotherm peak, as shown in Table II. This indicates that the degree of crystallinity achieved during the cooling cycle remains unchanged before commencement of the melting experiment or, in other words, no additional crystallization takes place during this time gap. Similar behavior is observed on comparison of the ratio of the peak widths of the crystallization exotherms ( $\Delta W$  of nylon 6/ $\Delta W$  of PP) and that of

	Crystallization Exotherm			Melting Endotherm		
Nylon 6 Content (wt %)	$A_1/A_2^a$	$\Delta W_1/\Delta W_2{}^{\rm b}$	$\begin{array}{c} \text{Decrease}^{\text{c}} \text{ of } T_c \\ \text{of PP} \ (^{\circ}\text{C}) \end{array}$	$A_1 / A_2^{a}$	$\Delta W_1/\Delta W_2{}^{\rm b}$	$\begin{array}{c} \text{Decrease}^{\text{c}} \text{ of } T_m \\ \text{ of PP (°C)} \end{array}$
10	0.023	1.56	0.9	0.024	1.60	8.4
20 30	$\begin{array}{c} 0.084\\ 0.310\end{array}$	$0.78 \\ 0.75$	$\begin{array}{c} 0.4 \\ 0.1 \end{array}$	$\begin{array}{c} 0.086\\ 0.322\end{array}$	$0.89 \\ 0.94$	$\begin{array}{c} 8.5\\ 8.1\end{array}$

Table II DSC Exotherms and Endotherms Data of PP/Nylon 6 Blend

<sup>a</sup> $A_1/A_2$ : peak area of nylon 6/peak area of PP.

<sup>b</sup>  $\Delta W_1 / \Delta W_2$ : width at half-height of nylon 6/width at half-height of PP.

<sup>c</sup> Decrease with respect to the value for PP.

the melting endotherms ( $\Delta W$  of nylon  $6/\Delta W$  of PP). However, the variation of the melting peak temperature is much larger than is the variation of the crystallization exotherm peak temperature.

The melting peak temperature of PP decreases by about 8°C in the studied blend composition range, which is greater than the similar shift of the crystallization exotherm peak temperature of PP which changes by 1 °C around its value of 112°C. This may be attributed to the fact that the crystallization of nylon 6 occurs at a higher temperature than that of PP, and, thus, during the crystallization of PP in the cooling cycle, crystallites of nylon 6 are already present in the melt which give rise to the formation of composite crystals (with PP spherulites grown over the surface of nylon 6 spherulites, as will be discussed subsequently), whereas in the case of melting, since the crystallites are composite, the melting of one component may affect the melting of the crystallites of the second component.

It seems that nylon 6 crystals or the amorphous phase of nylon 6 owing to its sufficiently high thermal energy in the vicinity of the temperature of the melting endotherm of PP destabilizes the crystalline structure of PP and eases its melting. During the cooling cycle, the crystallization of the nylon 6 component precedes the crystallization of PP. Hence, the presence of nylon 6 crystallites may influence the crystallization of the PP melt. Furthermore, PP has been shown to form transcrystallites growing around the foreign particles such as glass fiber.<sup>25</sup> A similar phenomenon may be expected to occur in this case also where nylon 6 crystallites may act as the sites for the growth of PP spherulites around them in a manner similar to transcrystallization.

## **Polarizing Light Microscopy**

The formation of spherulites for PP and nylon 6 and their growth in the PP/nylon 6 blend at various blend compositions were studied by PLM. A birefringent spherulite structure truncated by impingement was observed when a sample of pure PP was crystallized from the melt isothermally. The birefringent patterns displayed a Maltese cross whose arms are in the direction of the planes of the analyzer and of the polarizer [Fig. 6(a)], whereas a pure nylon 6 sample upon cooling from a molten state did not show such well-developed spherulites like PP. In fact, the crystallization of nylon 6 at 195°C was so fast that



**Figure 6** Polarized light micrographs of (a) unblended PP crystallized at 125°C and (b) unblended nylon 6 crystallized at 195°C.



**Figure 7** Polarized light micrographs of PP/nylon 6 blend crystallized at 125°C at various blend compositions (wt % nylon 6 content): (a) 10%; (b) 20%; (c) 30%.

it led to instantaneous formation of a very large number of small spherulites which covered the entire area of the sample [Fig. 6(b)].

Figure 7(a–c) shows PLM micrographs of the PP/nylon 6 blend at various blend compositions, which clearly illustrates the formation and growth of PP spherulites in the presence of nylon 6 inclusion. Two types of spherulites are seen from these PLM micrographs, which are formed at two different temperatures, corresponding to the crystallization of the two components of the blend. At 195°C, the molten nylon 6 became crys-

tallized and formed a very large number of small spherulites which are well dispersed in the amorphous PP matrix. By cooling the sample and allowing its isothermal crystallization at 125°C, within a few minutes, the PP spherulites were formed and grew linearly with time. At a lower nylon 6 content of the blend [Fig. 7(a)], it is seen that nylon 6 crystallites are more uniform and well dispersed in the PP matrix. However, at higher nylon 6 contents [Fig. 7(b,c)], the nylon 6 crystallites vary in size and exist as larger droplets dispersed in the PP matrix. At a nylon 6 content of 10%, not much interaction exists between the PP spherulites and nylon 6; hence, it seems that the formation of PP spherulites are independent of the nylon 6 crystallites. However, at a higher nylon 6 content of the blend, much more interaction can be seen between the nylon 6 particles and the PP spherulites which are mostly nucleated and grown around the already-crystallized domains of nylon 6. The number of PP spherulites is very low for the blend sample containing 10% nylon 6, but it increases at a higher nylon 6 content. These could be the reasons for the increase in the nucleation rate of the PP spherulites in the region of a higher nylon 6 content of the blend.

# CONCLUSIONS

The crystallinity of both components of the blend do not vary in the same proportion as does the weight fraction of the corresponding polymer in the blend, implying a considerable effect of the presence of the second component of the blend on the crystallization of each of the individual components of the blend. The crystallization of PP decreases in the presence of nylon 6, whereas the crystallinity of nylon 6 increases considerably in the presence of PP. The effect of nylon 6 on the crystallization behavior of PP is such that it decreases the nucleation rate of PP and, consequently, broadens the crystallite-size distribution and decreases the crystallization rate and the crystallinity of PP. These effects are more pronounced on the initial addition of 10 wt % nylon 6. At a higher nylon 6 content, the nucleation rate of PP does not show much variation with the blending ratio. Since the crystallization of PP occurs only at a temperature when nylon 6 has already crystallized, the observed variation of the crystallization behavior of PP is attributed to the role of nylon 6 crystallites on the crystallization behavior of PP. The spherulite growth detected by PLM shows the formation of PP spherulites surrounding the nylon 6 spherulites, which confirms the role of the nylon 6 crystallites. At a high nylon 6 content, the spherulites of nylon 6 provide a greater surface area for the formation of PP crystals; hence, the decrease of the nucleation rate becomes slower in the region of high nylon 6 content of the blend.

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