

Rheological Properties, Crystallization, and Morphology of Compatibilized Blends of Isotactic Polypropylene and Polyamide

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ABSTRACT: Blends of isotactic polypropylene (iPP) with the polyamide nylon-6 (N6), prepared by extrusion, were studied with a composition of up to 30% by weight polyamide. In the case of a 70/30 iPP/N6 blend, the influence of a compatibilizing agent based on polypropylene functionalized with maleic anhydride (PP-*g*-MA), with compositions of 1, 3, 5, and 10% by weight in polypropylene, was followed. The influence of the concentration of N6 and the compatibilizing agent on the rheological and thermal properties, and the morphology of the blends, was analyzed by monitoring the melt viscosity at different shear rates, differential scanning calorimetry, and polarized light microscopy. Vibrational spectroscopy was used to characterize the blends and to study the effect of the compatibilizing agent. The viscosity–composition curves for the iPP/N6 blends, in the composition and shear rate ranges analyzed, show a negative deviation from the additive rule, while the opposite trend is observed for the blends compatibilized with PP-*g*-MA. Important variations in the spectroscopic behavior was observed between compatibilized and noncompatibilized blends, which varied as a function of the compatibilizing agent concentration. The crystallization rates of iPP in the iPP/N6 blends, under both dynamic and isothermal conditions, are much greater than are those observed for pure iPP and are directly related to the nucleating activity of the polyamide. This effect is much smaller in the presence of the compatibilizing agent. The isothermal crystallization of the polyamide N6 in compatibilized blends is affected by the presence of iPP, reducing the crystallization rate due to the diluent effect of the polypropylene. © 1997 John Wiley & Sons, Inc. *J Appl Polym Sci* **65**: 2665–2677, 1997

Key words: polymer blends; nylon-6/polypropylene; compatibilizing agent; crystallization; crystalline morphology; differential scanning calorimetry; vibrational spectroscopy

INTRODUCTION

Polymer blending is a well-recognized method for the design of macromolecular materials with

properties defined for specific applications, and in the majority of cases, it is a more versatile and economic alternative to the synthesis of new polymeric materials. For this reason, there is a great deal of interest in the industrial development of blended polymer systems.

It is accepted that due to the low entropy of blending the product resulting from a blend of two polymers is an immiscible system composed of two phases, where the component with higher concentration forms the continuous matrix and the other

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component is heterogeneously dispersed in small domains. These systems present high surface tension and poor adhesion, resulting in poor mechanical properties.¹⁻³ To improve the properties of this type of blend, a series of compounds denominated compatibilizing agents were added to the blends. These compounds generate interfacial interactions between the components, resulting in a reduction in the energy between the distinct phases and an increase in adhesion and improved mixing during the blending process.⁴

Blends of isotactic polypropylene and polyamide have received much attention over recent years,⁵⁻⁷ since they combine the environmental moisture-stability and ease of processing of the former with the thermomechanical characteristics of the latter. Unfortunately, both polymers are incompatible because of their different polarities and crystalline morphologies. In an attempt to increase the compatibility between these polymers, different compatibilizing agents have been employed as a third component in the blend. These compounds are generally based on polypropylene functionalized with acrylic acid,⁸⁻¹⁰ maleic anhydride (MA),¹⁰⁻²¹ or other modified polar thermoplastics such as ionomers²²⁻²⁵ or functionalized styrene block copolymers.¹⁸⁻²⁰ Despite the importance of understanding the correlation between the structure and properties of this type of blend, especially in terms of the effect of compatibilization on the crystalline structure of the materials, little information is available.^{9-11,15-19,26}

In a previous study,²⁷ we investigated the effect of compatibilization on the crystallization process and on the mechanisms of nucleation and growth in iPP/N6 blends compatibilized with maleic anhydride-modified polypropylene (PP-*g*-MA), prepared in a Brabender torque rheometer. In the present study, this work was extended to iPP/N6 blends prepared on an industrial scale by extrusion, both in the presence and absence of PP-*g*-MA. The effect of the blending method on the flow properties, thermal properties, dynamic and isothermal crystallization processes of both materials, and the crystalline morphology are studied.

EXPERIMENTAL

Preparation of the Blends

Polypropylene homopolymer was supplied by Repsol, S.A., and presented an isotacticity of 92% (determined by NMR), a melt flow index (MFI)

of 7.0 g/10 min (ASTM D-1238, condition-L), and a density of 0.9058 g/cm³. Polyamide (nylon-6), with a density of 1.13 g/cm³, was supplied by Akzo Chemicals. The compatibilizing agent used was PP-*g*-MA with 0.4% MA and an MFI of 22 g/10 min (ASTM D-1238, condition-M), supplied by Exxon Chemicals. Blends were prepared in a Werner & Pfleiderer Model ZSK-30 extruder with a length/diameter ratio of 26, at a temperature of 230°C and a screw speed of 100 rpm. The effect of the compatibilizer concentration was studied in the 70/30 blends of iPP/N6 with 1, 3, 5, and 10% by weight PP-*g*-MA. The weights of PP and PP-*g*-MA were added up to 70% of the total blend weight.

Rheological Properties

The apparent melt viscosities were determined in a CEAST Rheoscope 100 capillary rheometer with a length/diameter ratio of 20 at 230°C and shear rates between 6 and 3800 s⁻¹.

Thermal Analysis

The thermal properties were analyzed under both dynamic and isothermal conditions. In the first case, a Mettler TA4000 differential scanning calorimeter with a DSC-30 oven with an automatic temperature control and thermal flow calibration was used, with a heating/cooling rate of 10°C/min under a nitrogen atmosphere. The sample weight in all cases was around 10 mg, and the peak maxima were considered as the transition temperatures. The enthalpic calculations were performed on a PC using Mettler TA72 software. The isothermal studies were performed on a Perkin-Elmer DSC7/UNIX system, previously calibrated with indium ($T_m = 156.5^\circ\text{C}$, $\Delta H_m = 28.45 \text{ J/g}$) and zinc ($T_m = 419.5^\circ\text{C}$, $\Delta H_m = 108.4 \text{ J/g}$) standards, in a nitrogen atmosphere, using a sample weight of 10 mg. The general principles described in the literature were employed in the kinetic studies.²⁸ The samples were heated to 10°C above their corresponding melting points for 10 min and then cooled at a rate of 64°C/min to the predetermined crystallization temperature. The corresponding crystallization exotherms were recorded as a function of time to baseline variations of less than $5 \times 10^{-3} \text{ mW}$. The partial areas, corresponding to a particular degree of crystalline transformation, were determined using the Perkin-Elmer-DSC7 analysis software. At the end of the isothermal

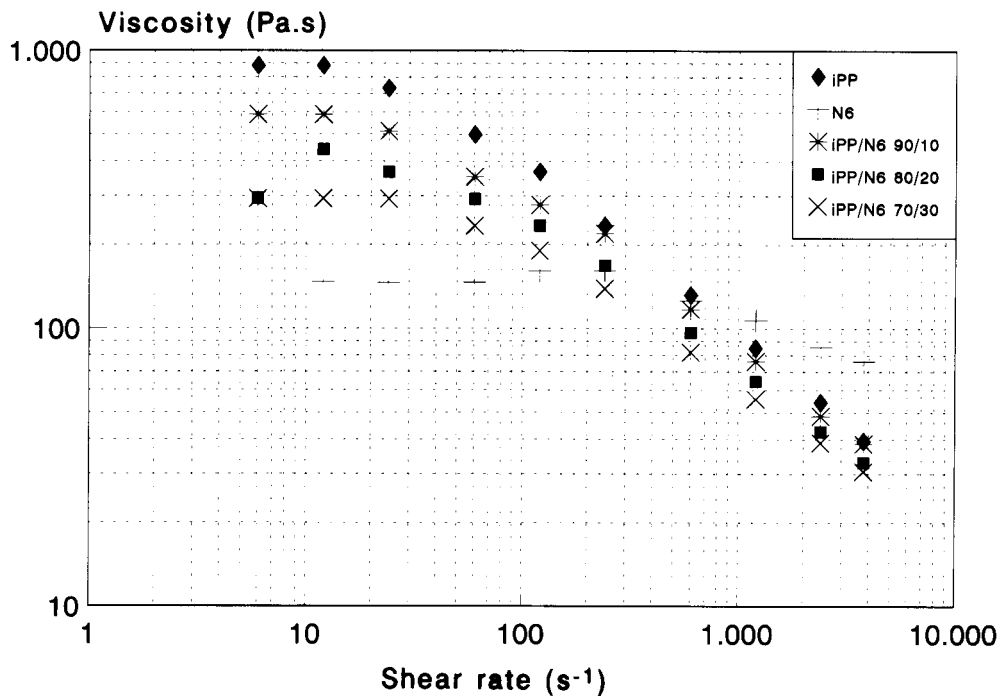


Figure 1 Variation in the melt viscosity versus the shear rate for noncompatibilized iPP/N6 blends.

crystallization process, the samples were heated at a rate of 10°C/min to determine the corresponding melting points. Crystallinities were calculated

using values of $\Delta H_m = 137.9$ J/g (ref. 29) and 190 J/g (ref. 30) for 100% crystalline iPP and N6, respectively.

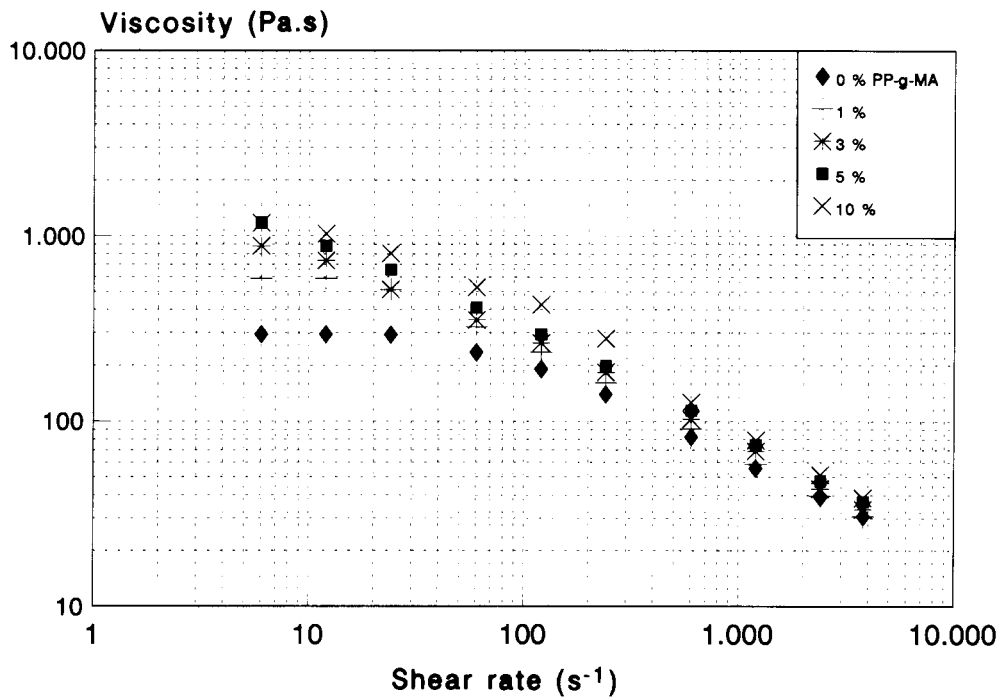


Figure 2 Variation in the melt viscosity versus shear rate for the 70/30 iPP/N6 blend, at different concentrations of PP-*g*-MA.

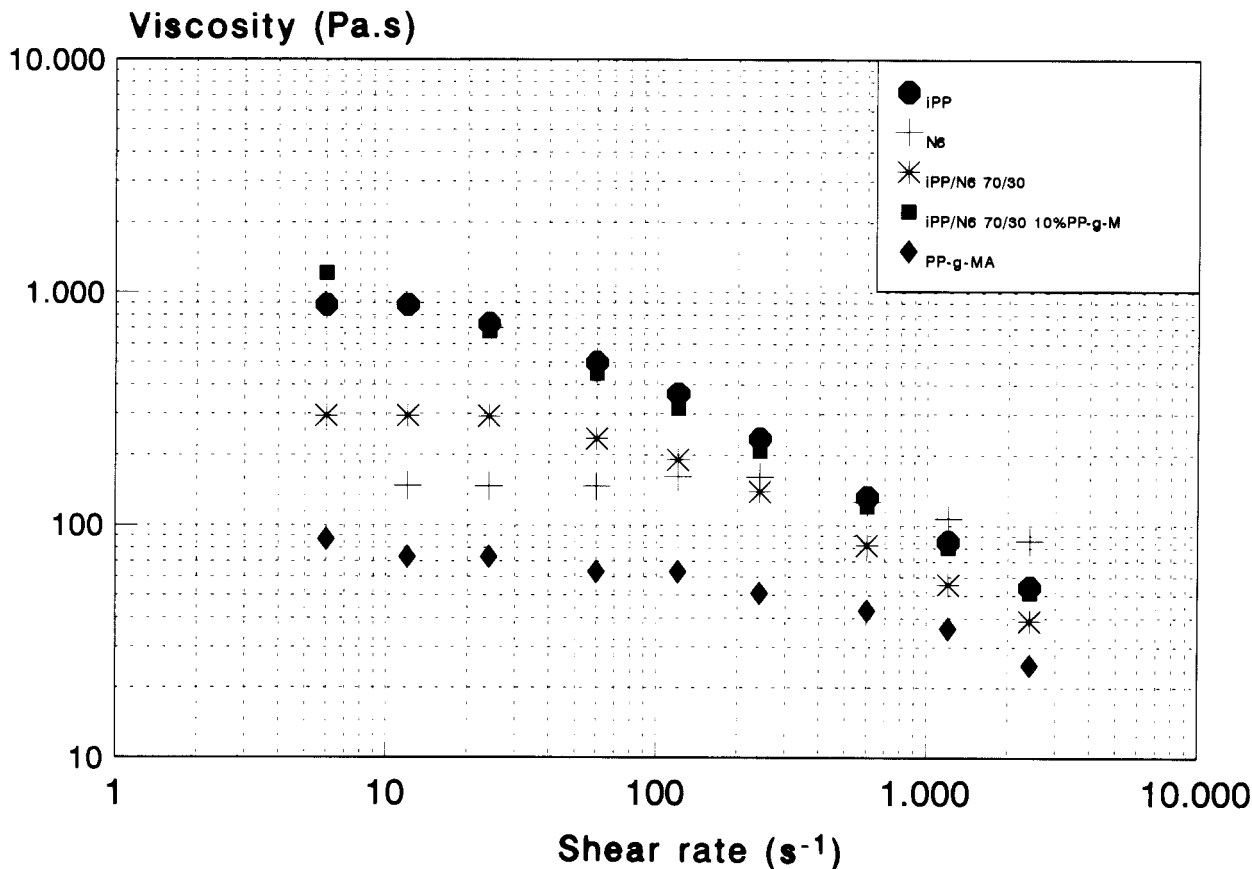


Figure 3 Influence of the compatibilizing agent on the melt viscosity.

Wide-Angle X-ray Diffraction

The diffractograms were obtained from films prepared at 240°C and a pressure of 100 bar for 2 min, using a Rigaku Geigerflex-D/max diffractometer with a Rigaku RU-200 rotating anode generator and Ni-filtered $\text{CuK}\alpha$ radiation, registered between $2\theta = 5^\circ\text{--}35^\circ$.

Polarized Light Microscopy

Microscopy and thermo-optical analysis were performed using a Reichert Zetopan Pol light microscope, with a Mettler FP82HT temperature cell. The images obtained, both under dynamic and isothermal conditions, were recorded in real-time using a Sanyo VC2512 CCD camera and a Sony SVT-5000P time-lapse video recorder, analyzing the data with a Leica Quantimet-500 image analysis system.

Vibrational Spectroscopy

Infrared spectra were recorded from thin films (thickness between 20–60 μm) prepared in a hot

press at 240°C and a pressure of 100 bar for 4 min on a Perkin-Elmer System 2000 FTIR module equipped with a DTGS detector, using between 20 and 50 scans at a resolution of 2 cm^{-1} . Raman spectra were recorded from samples both in the form of beads, and as the previously described films, on the Perkin-Elmer System 2000 FT-Raman module, using a $\text{Nd}^{3+}:\text{YAG}$ ($\nu_0 = 9394 \text{ cm}^{-1}$) laser source with a power of 200 mW at the sample and an InGaAs detector operating at room temperature. Spectra were recorded at a resolution of 2 cm^{-1} and 200 scans were collected.

RESULTS AND DISCUSSION

Figure 1 shows the variation of the melt viscosity as a function of shear rate for iPP, N6, and their blends in the absence of the compatibilizing agent. The behavior of noncompatibilized iPP/N6 blends is intermediate to that of each of the pure components, presenting a negative deviation to the additive rule over the composition range analyzed. This is indicative of the absence of adhesion be-

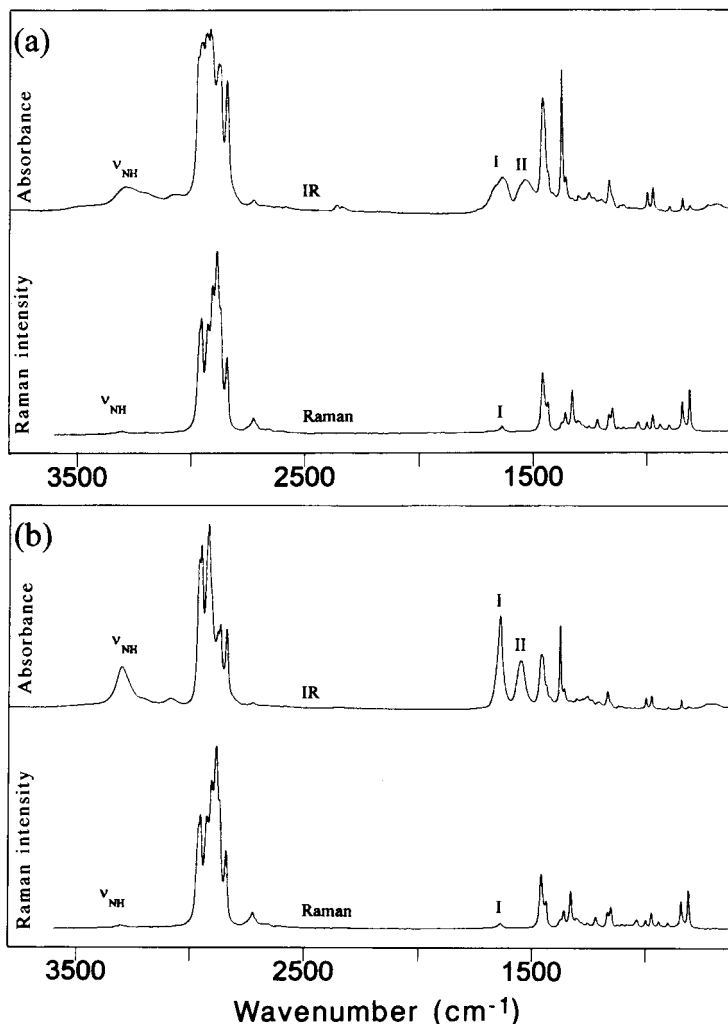


Figure 4 FTIR and FT-Raman spectra of iPP/N6 blends: (a) 70/30 and (b) 70/30 with 10% PP-*g*-MA.

tween the iPP and N6 phases³¹ and the high level of incompatibility between the components.^{12,32,33}

Figure 2 shows the variation of the melt viscosity as a function of shear rate for iPP/N6 blends with different weight percent of PP-*g*-MA, compared with the same blend composition without the compatibilizing agent. A progressive increase in the viscosity with the concentration of the compatibilizing agent can be observed, this being greater the lower the shear rate. The increase in melt viscosity of the compatibilized blends cannot be attributed to the PP incorporated in the PP-*g*-MA, since, as can be observed in Figure 3, its viscosity is much lower than that of pure iPP, and, as such, the effect that the presence of PP-*g*-MA should exert on the viscosity of the blend is the opposite. Thus, the increase in viscosity must originate from new and stronger interactions pro-

voked by the presence of the maleic groups^{8,11,17} and/or by the formation of new covalent bonds between these groups and the terminal amine groups of the polyamide.^{11,13,14}

To examine these hypotheses, a comparative vibrational spectroscopic study was made from the FTIR and FT-Raman spectra of each of the iPP/N6 blends and of the pure components, registered from films prepared under careful conditions so as to avoid as much as possible the oxidation and degradation of the components, particularly in the case of the polyamide.³⁴

The vibrational spectra of the individual components and the blends are typical. In the case of the polyamide, three bands characteristic of the amide group can be clearly seen: the N-H stretching mode $\nu_{\text{N-H}}$ at 3300 cm^{-1} , those corresponding to carbonyl stretching $\nu_{\text{C=O}}$ at 1636

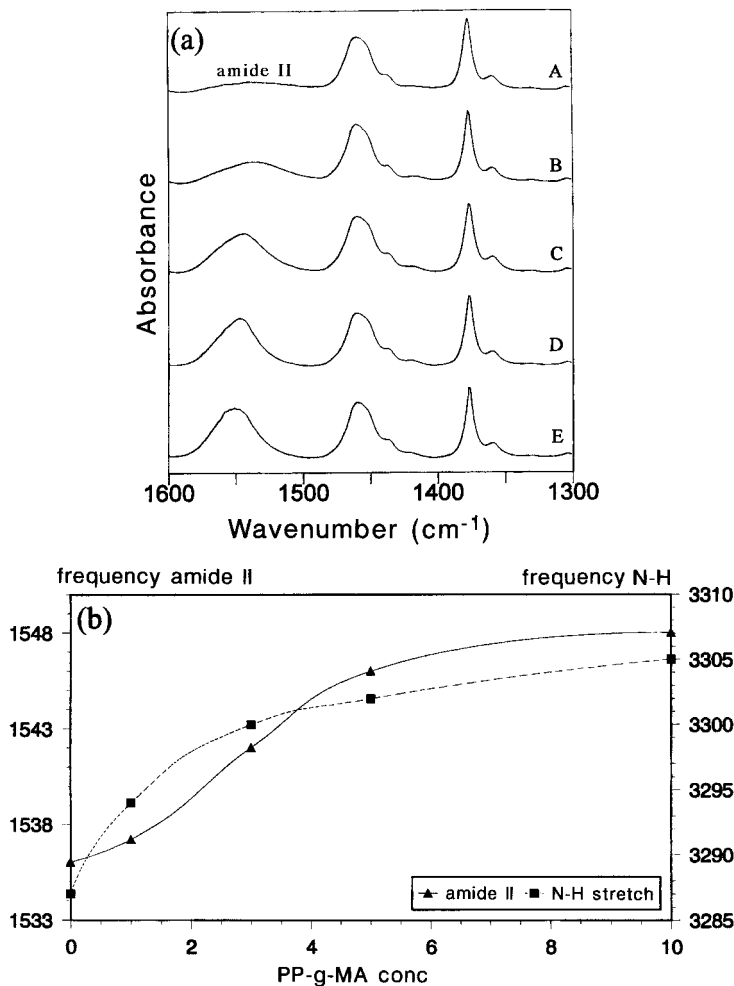


Figure 5 (a) Spectral variations in the amide II region versus concentration of PP-*g*-MA for 70/30 iPP/N6 blends: (A) 0% PP-*g*-MA; (B) 1%; (C) 3%; (D) 5%; (E) 10%. (b) Graphical representation of the variation in frequency associated with the amide II and N—H stretching modes versus concentration of PP-*g*-MA.

cm^{-1} , and the mixed mode $\nu_{\text{C}=\text{N}} + \delta_{\text{N}-\text{H}}$ at 1546 cm^{-1} (known as the amide I and amide II modes), respectively. The infrared spectrum of functionalized polypropylene PP-*g*-MA shows three bands at 1850, 1786, and 1715 cm^{-1} , characteristic of the maleic group, associated with the symmetric and antisymmetric stretching vibrations of the carbonyl group ($I_s/I_{as} = 0.17$) and the stretching mode of the residual acidic carbonyl, respectively.

Figure 4(a) shows the FTIR and FT-Raman spectra of the 70/30 iPP/N6 blend. It can be clearly observed that the most intense bands in both cases are due to the C—H stretching region of the iPP, although these bands between 3000 and 2800 cm^{-1} fall above the Beer limit and, as such, their intensity and appearance are not representative. It is important to note that in the present study the concentration of N6 in the

blends is lower than that employed on other occasions.^{8,25} However, the principal vibrations due to the amide group can be seen, as indicated in the figure, and these and the rest of the spectral details fall acceptably within the Beer limit in all cases. In the FT-Raman spectra, the amide bands are much weaker and, consequently, less useful in the analysis. Figure 4(b) shows the FTIR and FT-Raman spectra of the 70/30 iPP/N6 blend with the 10% PP-*g*-MA compatibilizing agent. The most important differences can be seen in the infrared spectra, where a considerable increase in the intensity of the amide modes can be appreciated. Although the difference in the intensity of these bands is surprisingly large, this observation, already indicated in previous studies,^{8,11,17} supports the idea of the formation of a graft copolymer between the polyamide and the function-

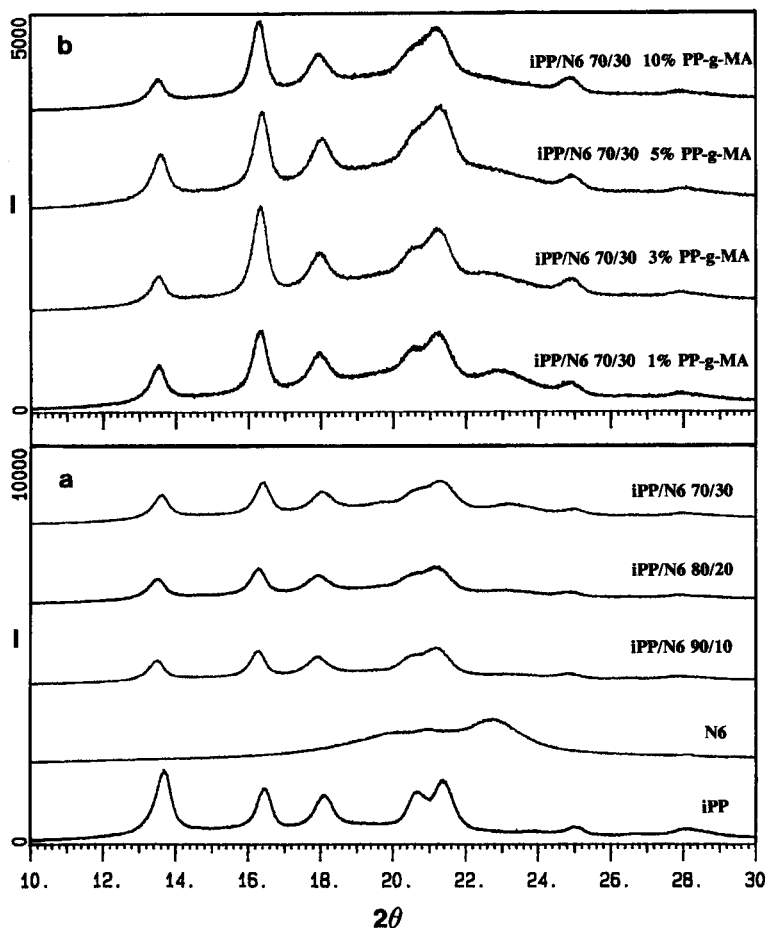


Figure 6 X-ray diffractograms of (a) noncompatibilized iPP/N6 blends and the pure components and (b) compatibilized 70/30 iPP/N6 blends.

alized polypropylene, via the acylation of the nucleophilic terminal amino group of the polyamide. However, the increase in intensity cannot be solely associated with new chemical bonds, given that the concentration in the blend is very low. However, as well as the changes in the relative intensity of the indicated bands, there are important variations in both the frequencies and the distribution of frequencies observed.

It is important to examine the relationship between the crystallinity of the polyamide in the blends and the spectral behavior. In Figure 5(a), the variation of the spectral region corresponding to the amide II mode with the concentration of PP-*g*-MA is shown, the spectra being normalized using the doublet at 973 and 998 cm^{-1} , characteristic of the PP component in the blend. A gradual change in the intensity of this mode vs. other bands is associated with CH_3 deformations in PP and CH_2 deformations in both PP and N6. Figure 5(b) shows the variation in the frequency associated with the modes $\nu_{\text{N-H}}$ and $\nu_{\text{C-N}} + \delta_{\text{N-H}}$ ver-

sus the concentration of PP-*g*-MA in the blend. Although the blends show a lower crystallinity with increasing concentration of the compatibilizing agent, as is discussed later, the bandwidth at half-height (FWHH) of the bands or features seems to decrease, which is not usual for semi-crystalline polymers, as can be seen in the case of the pure polyamide, where the typical sharpening of the spectral bands with increase in crystallinity can be observed.³⁵ Thus, it is clear that the changes observed in the spectra of the polyamide in the blend do not seem to be related to the crystallinity of the N6.

In both crystalline and amorphous linear aliphatic polyamides, one can assume that all the amide groups are hydrogen-bonded at low temperatures.^{36,37} In the case of amorphous polyamides, the increase in the concentration of "free" amide groups, characterized by the appearance of a band associated with the N—H stretching mode at 3444 cm^{-1} , is very weak. In our case, in the 70/30 iPP/N6 blend without a compatibilizing agent,

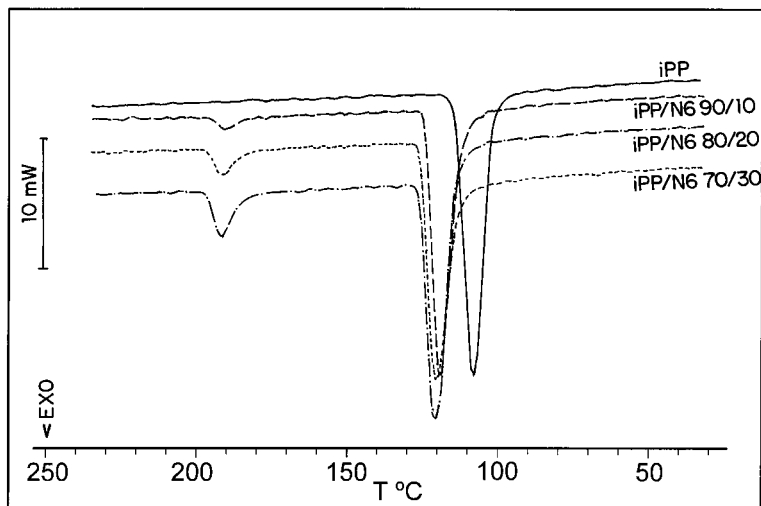


Figure 7 DSC thermograms corresponding to the cooling cycle from the melt in non-compatible iPP/N6 blends and pure iPP.

a slight increase in the absorption at around 3480 cm^{-1} and the appearance of a relatively intense shoulder at 1670 cm^{-1} can be observed. Both observations could be related to the presence of free N—H groups and free C=O groups, respectively. On the other hand, the reduction in the frequency of the amide II mode, from 1546 cm^{-1} in the pure polyamide to 1540 cm^{-1} in the blend, is consistent with reduced interactions. The increase in the intensity of a band at 1670 cm^{-1} has also been related to free carbonyl groups, as has the free N—H band at 3444 cm^{-1} in the case of a binary blend of a polyamide with poly(2-vinylpyridine).³⁸ On the other hand, in the case of the 70/30 iPP/N6 blend with a compatibilizing agent,

the shoulder at 1670 cm^{-1} is dramatically reduced and is accompanied by an important increase in the absorption of the amide modes.

It is clear that the formation of new chemical bonds in the compatibilization process, and the possibility of the generation of new interactions, are phenomena whose observation and interpretation are fundamental to the understanding of the nature of these changes. These observations are the object of a more profound study of the interactions between functionalized polypropylene and polyamides and will be reported at a later date.³⁹

To investigate whether the crystalline structure of each component in the blend is affected by

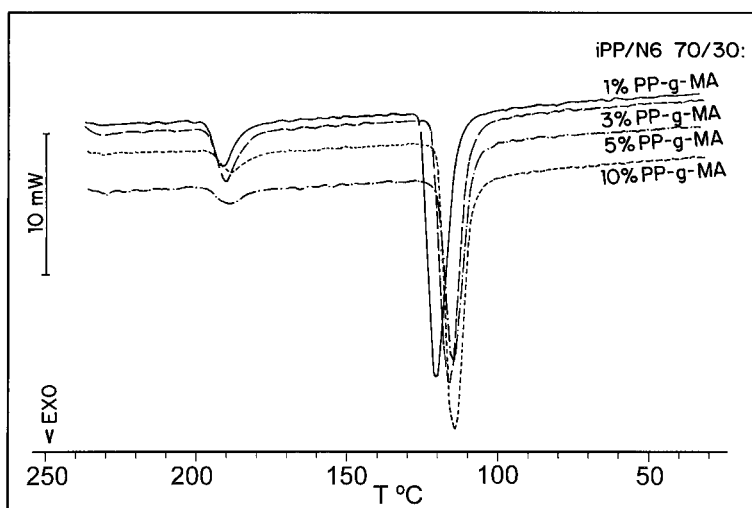


Figure 8 DSC thermograms corresponding to the cooling cycle from the melt in iPP/N6 blends compatibilized with PP-*g*-MA.

Table I Crystallization Temperatures and Degree of Crystallinity ($1 - \lambda$) of iPP/N6 Blends Obtained by DSC from Samples Cooled from the Melt at a Rate of 10°C/min

Sample	T_c (iPP) (°C)	$(1 - \lambda)_{iPP}$ $\times 100$	T_c (N6) (°C)	$(1 - \lambda)_{N6}$ $\times 100$
iPP	111.0	69.3	—	—
N6	—	—	193.7	37.6
90/10 iPP/N6	121.9	72.3	193.5	36.5
80/20 iPP/N6	123.6	75.5	192.6	36.4
70/30 iPP/N6	124.0	82.7	193.1	35.8
70/30 iPP/N6, 1% PP- <i>g</i> -MA	123.7	80.7	191.0	29.1
70/30 iPP/N6, 3% PP- <i>g</i> -MA	118.8	77.6	190.1	26.0
70/30 iPP/N6, 5% PP- <i>g</i> -MA	117.8	77.0	188.8	16.3
70/30 iPP/N6, 10% PP- <i>g</i> -MA	117.3	79.4	187.8	11.6

the crystallization of the other component or by the presence of the compatibilizing agent, the pure materials and the blends were analyzed by X-ray diffraction. It can be seen in Figure 6(a,b) that the reflections at $2\theta = 13.7^\circ$, 16.5° , 18.1° , 20.6° , 21.3° , 25.0° , and 28.1° , characteristic of iPP, remain unmodified in the presence of N6, whose crystalline reflections at $2\theta = 20.0^\circ$, 20.8° , and 22.6° are also unaffected by the presence of iPP. The only relevant observations are the increase in the relative intensity of the reflection at $2\theta = 22.6^\circ$, associated with the polyamide. In the noncompatibilized blends [Fig. 6(a)], this is caused by the increase in the concentration of the

N6 component. A reduction in the intensity of this reflection is observed in the PP-*g*-MA compatibilized blends with the same N6 concentration [Fig. 6(b)]. These results confirm that the blending process does not affect the crystalline structure of the components, which crystallize separately and show no evidence of cocrystallization.

Analysis of dynamic and isothermal crystallization by differential scanning calorimetry shows the existence of a crystal segregation process between the iPP and the N6, as shown in Figures 7 and 8. In Figure 7, the crystallization exotherms of the individual components in the noncompatibilized blend can be clearly distinguished. The values of the crystallization temperatures and crystallinities obtained are detailed in Table I. In the absence of a compatibilizing agent, the temperature of crystallization of pure iPP is lower than that of the same component in the blends, this effect being greater the higher the concentration of N6 (Fig. 7). On the other hand, the crystallinity associated with iPP increases with increasing N6 content. In the case of the polyamide, neither the temperature nor the degree of crystallinity is affected by the presence of the iPP matrix. These observations seem to indicate the existence of a nucleation effect in the crystallization of iPP provoked by the crystalline phase of the polyamide. On the other hand, in the presence of the compatibilizing agent PP-*g*-MA, although the nucleating effect of the N6 component on the crystallization of iPP can still be observed, its extent can be seen to diminish with increasing concentration of PP-*g*-MA in the blend (Fig. 8). This observation is reflected both in the values of the temperature of crystallization and in the crystallinity of the iPP in the blends, as shown in Table I. At the same time, the presence of the compatibilizing agent

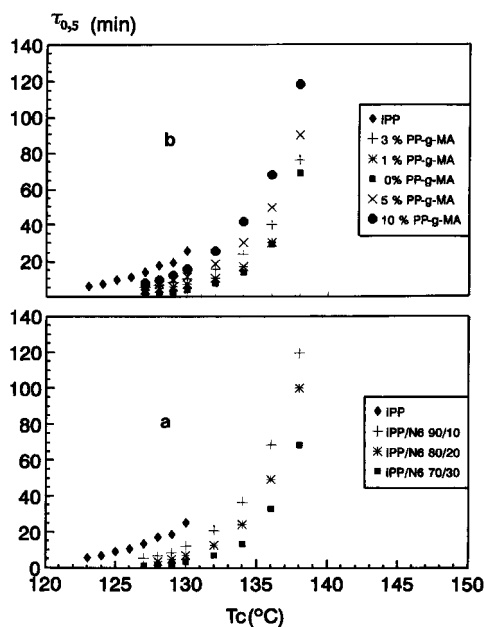


Figure 9 Representation of $\tau_{0.5}$ versus crystallization temperature for iPP in (a) noncompatibilized iPP/N6 blends and (b) blends compatibilized with PP-*g*-MA.

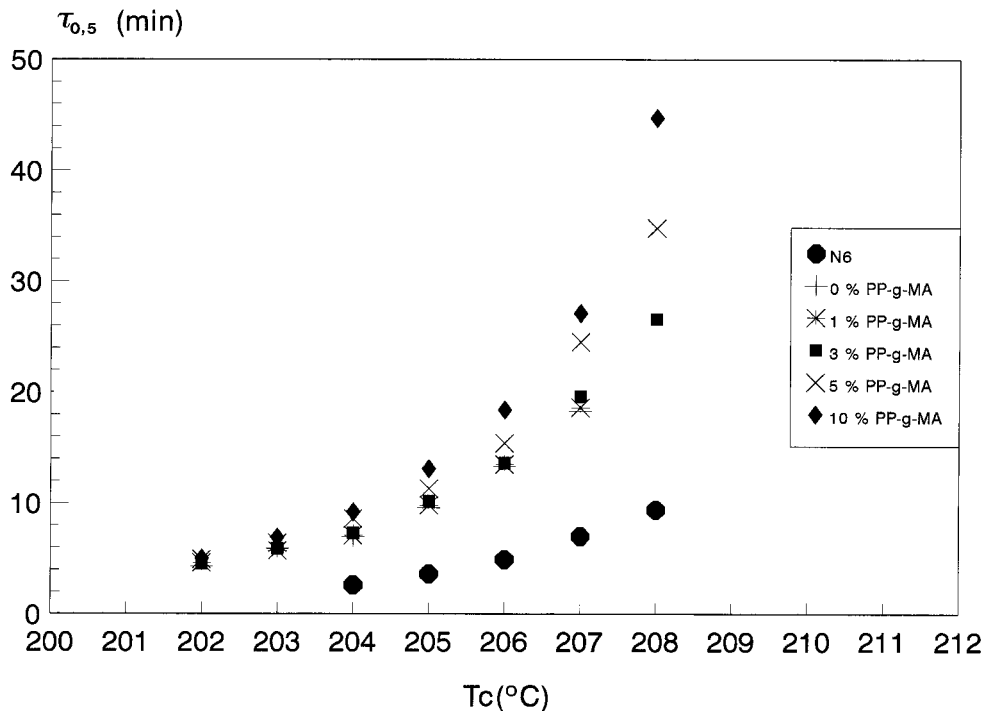


Figure 10 Representation of $\tau_{0.5}$ versus crystallization temperature for the N6 component in iPP/N6 (70/30) blends compatibilized with PP-*g*-MA.

provokes a reduction in both the crystallization temperature and the degree of crystallinity of N6 in the blends with respect to the pure polyamide, as a function of compatibilizing agent concentration. This behavior has been previously described for other blends among isotactic polypropylene, polyamide, and the compatibilizing agent and a number of different interpretations given.^{10,17–19,22,26} Some authors suggest that the crystallization of iPP and the polyamide take place separately but simultaneously,¹⁸ while others have described how the compatibilizing agent acts as a cocrystallizing agent between the components of the blend.²⁶ In another case, the structure and properties of fibers prepared from polypropylene and nylon-6, compatibilized with PP-*g*-MA, have been studied as a function of the concentration of PP-*g*-MA and the reduction in the crystallinity of the polyamide phase observed and justified by the reduction in segmental mobility of the polyamide chains due to grafting reactions.¹⁷ In our case, taking into account the melt viscosity and vibrational spectroscopy data previously described, the reduction in the crystallization temperature and the degree of crystallinity of the polyamide in the presence of PP-*g*-MA can be attributed to a compatibilization phenomenon generated by an increase in interactions between the components of the blend.

In a previous publication, the kinetics of crystallization was analyzed under isothermal conditions, for the same type of blends prepared in a Brabender torque rheometer.²⁷ In this case, the influence of both the method of preparation of the blends and the effect of the presence of each component on the crystallization of the other, in the presence or absence of a compatibilizing agent, was investigated. Figure 9 shows the variation of $\tau_{0.5}$, the time needed to reach 50% crystalline transformation, as a function of crystallization temperature, for the case of iPP and its blends, without and with a compatibilizing agent, in (a) and (b), respectively. In all cases, $\tau_{0.5}$ increases exponentially with the crystallization temperature, in a time scale analogous to that obtained in the case of blends prepared in the Brabender. In Figure 9(a), it can be observed that the crystallization rate of iPP is higher in the blends than in pure iPP, increasing correspondingly with the concentration of N6 because of its nucleating effect in the crystallization of the iPP matrix. Similar effects were found by Grof et al.⁴⁰ This effect is also found in the presence of the compatibilizing agent, as can be seen in Figure 9(b). However, in this case, it is reduced because of the presence of PP-*g*-MA, being lower the greater the concentration of the compatibilizing agent, as a consequence of a reduced nucleating activity of the N6 component in the crystallization of iPP. The

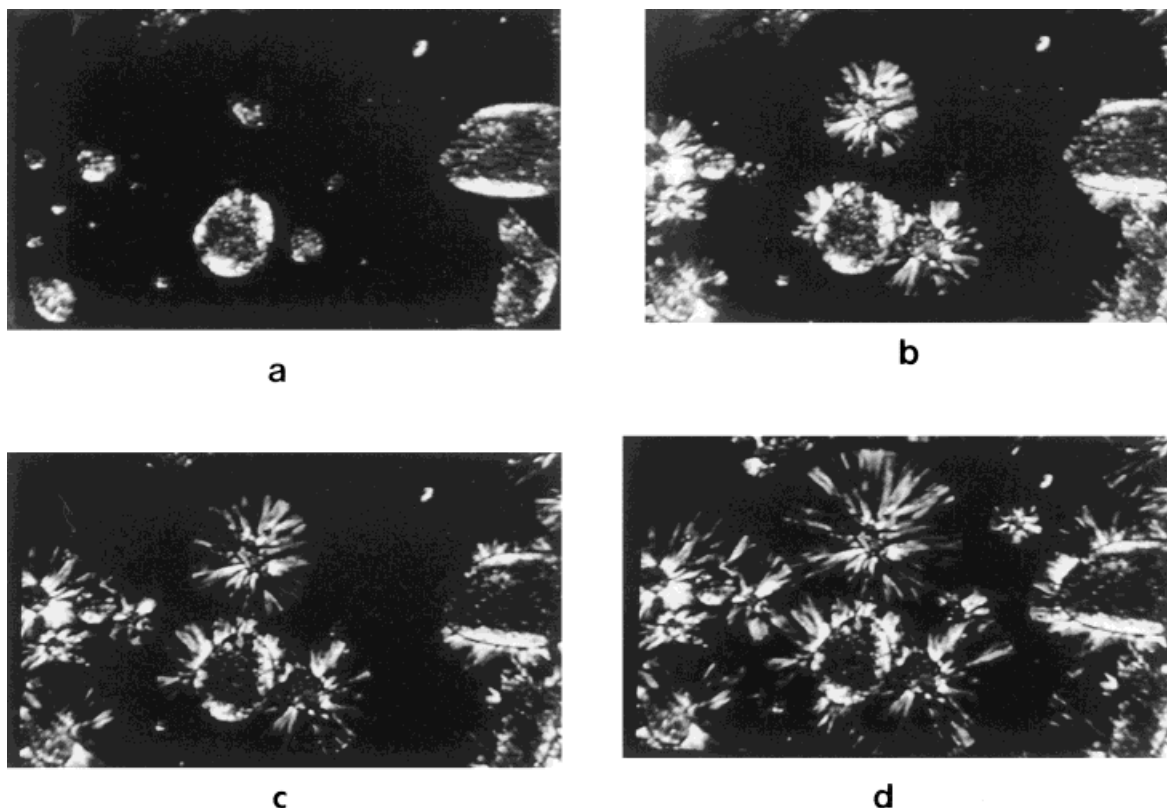


Figure 11 Polarized light microphotographs of the isothermal crystallization of the 70/30 iPP/N6 blend: (a) N6 at 205°C; (b) iPP at 128°C, 1 min; (c) iPP at 128°C, 2 min; (d) iPP at 128°C, 3 min.

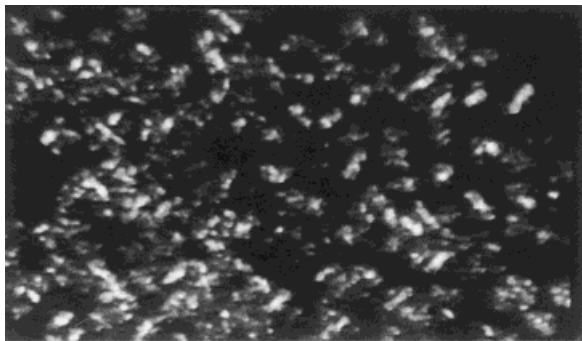
same occurs under dynamic crystallization conditions, as described previously.

Furthermore, the crystallization of the polyamide N6 is affected by the presence of the compatibilizing agent. Figure 10 shows that the values of $\tau_{0.5}$ for the N6 component in the (70/30) blends are much greater than are those corresponding to the pure polyamide, increasing with the concentration of PP-*g*-MA. This behavior may be related to the diluent effect of the molten iPP present in the blends when the polyamide crystallizes. These results are in agreement with those obtained from dynamic crystallization and clearly show that the presence of PP-*g*-MA increases the miscibility of the system.

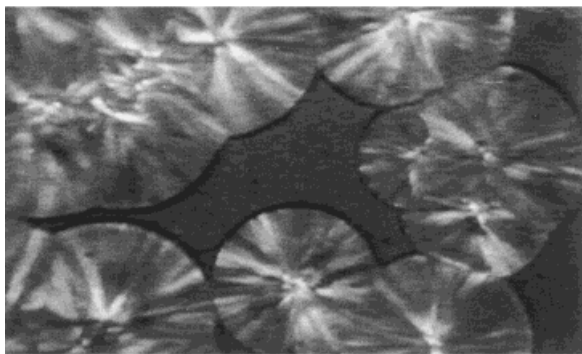
Following the same criteria as for previous work,²⁷ the experimental data were analyzed using the Avrami equation.^{41,42} The Avrami exponent is related to the type of nucleation and growth^{43,44} and is obtained from the slopes of the graph of $\log[\ln(1 - \theta)]$ versus $\log t$. A value of 3 was obtained for the pure iPP and iPP in noncompatibilized blends and blends with low compatibilizing agent concentrations, 1 and 3% PP-*g*-MA. A value of 4 was obtained for blends with 5 and 10%

PP-*g*-MA, which corresponds with that obtained for pure PP-*g*-MA. For the polyamide N6, a value of 3 was obtained in all cases, both with and without a compatibilizing agent. These results are analogous to those obtained previously for blends prepared in a Brabender²⁷ and demonstrate that in this case the method of preparation does not modify the crystallization mechanism.

To analyze the temperature coefficient of the crystallization process, the equilibrium melting temperatures employed were 238°C for the polyamide N6 (ref. 27) and 208°C for iPP.²⁹ For homogeneous nucleation and bidimensional growth,^{29,43} the product of the basal interfacial free energy, σ_e , and the lateral interfacial free energy, σ_u , is nearly constant for iPP, irrespective of the presence of PP-*g*-MA in the blends. However, the same product for the polyamide component increases progressively with the concentration of the compatibilizing agent, indicating that the process of compatibilization increases the interfacial free energy of the crystalline phase of the polyamide. Once again, these results coincide with those reported previously for the case of the blends prepared in the Brabender.²⁷



a



b

Figure 12 Polarized light microphotographs of the isothermal crystallization of (a) iPP in the 70/30 iPP/N6 blend with 10% PP-*g*-MA, at 128°C, 2 min, and (b) pure iPP at 128°C.

The analysis of the morphology of the superstructures grown during the isothermal crystallization process confirm the existence of the crystallization of iPP nucleated by the presence of previously crystallized polyamide. In this respect, Figure 11 shows the evolution of spherulites of polypropylene, nucleated at the interface of the drops of previously crystallized N6, for the 70/30 iPP/N6 blend. Further, in Figure 11(a), the N6 domains isothermally crystallized at 205°C clearly show a particulate morphology in the iPP matrix, which remains molten at this temperature. When the material is rapidly cooled to 128°C and held at this temperature, spherulitic structures of iPP develop at the surface of the polyamide domains, as shown in Figure 11(b–d). These iPP superstructures seem to correspond to monoclinic α -type spherulites, or “blended spherulites” according to the definition given by Norton and Keller,⁴⁵ providing evidence of the influence of crystallized N6 particles acting as nucleating

agents or heterogeneities in the crystallization of iPP. On the other hand, with an identical isothermal crystallization treatment in the presence of PP-*g*-MA, it is very difficult to distinguish a segregated N6 phase since the dispersion of the polymers is more intimate due to an improved interaction between the two phases. This is in agreement with the increase in viscosity observed in the compatibilized blends previously described. In this case, the nucleating effect of the polyamide in the crystallization of iPP was not observed, and very distorted spherulites of iPP were formed since the more homogeneously dispersed N6 perturbs the interlaminar aggregation of iPP spherulites [Fig. 12(a)] compared with those developed in pure iPP under the same thermal conditions [Fig. 12(b)].

CONCLUSIONS

The analysis of the melt viscosity as a function of shear rate reflects the existence of a process of compatibilization between iPP and N6, in the presence of maleic acid-modified polypropylene, PP-*g*-MA. The spectroscopic results seem to support the existence of a significant increase in the interactions of the polyamide, which are a function of the concentration of the compatibilizing agent. This fact substantially affects both the crystallinity and the rate of crystallization of both components of the blend. Neither parameter is affected by the method of preparation of the blends. The results obtained from X-ray diffraction, differential scanning calorimetry, and thermo-optical analysis demonstrate the separation of the crystallization processes associated with iPP and N6.

The presence of a compatibilizing agent leads to a reduction in the crystallinity of the polyamide and in its rate of crystallization, due to the diluent effect of the molten iPP. On the other hand, in the absence of PP-*g*-MA, while no substantial modification is observed for the N6, an increase in the crystallinity and crystallization rate of iPP and a modification of its spherulitic superstructure occurs, which is a function of the N6 concentration as a consequence of the nucleating effect of the crystallized component of N6. This effect is smaller in the presence of a compatibilizing agent.

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