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Crystal phase and crystallinity of polyamide 6/functionalized polyolefin blends

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Abstract

Blends of Nylon 6 (Ny6) and polyolefins functionalized with acrylic acid (polyethylene—PE–AA, polypropylene—PP–AA) were investigated in terms of crystallization behavior and resulting Ny6 crystalline structure. Thermal analysis showed that in the case of blends with functionalized polyolefin as a matrix: (a) Ny6 crystallization is spread and dramatically shifted toward lower temperatures, approaching that of the polyolefin component $125-132^{\circ}\text{C}$; (b) Ny6 γ crystal polymorph is the major phase present; confirmed and quantitatively evaluated by use of deconvolution computations performed on WAXS spectra of the blends.

When Ny6 is dispersed in functionalized polyolefin matrix, the weight content of Ny6 γ crystals increases up to three times with respect to analogous, non-compatibilized blends and up to \approx 16 times with respect to Ny6 homopolymer. These phenomena are explained by the reduction of size of Ny6 dispersed particles, caused by the interactions between the functional groups of polyolefin and the polar groups in polyamide chain. The nucleation mechanism is changed due to the lack of heterogeneous nuclei in most small Ny6 droplets, which results in the enhanced γ crystal formation. \oslash 2000 Elsevier Science Ltd. All rights reserved.

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1. Introduction

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The physical and mechanical properties of incompatible heterogeneous polymer blends may be enhanced by compatibilization through the introduction of physical or (and) chemical interactions between the components. The presence of functional groups along polymer chains, giving rise to specific interactions with the other blend constituent, can enhance the interfacial adhesion and phase dispersion, so improving the overall characteristics of these systems.

The compatibilization of polyamide–polyolefin blends has gained attention in recent years [1–10] mainly due to the technological applications of these materials. It has been shown that the presence of functional reactive groups (maleic anhydride, acrylic acid, diethylmaleate) on the polyolefin chains has a strong effect on the phase morphology, physical and mechanical features of the blends.

The subject of this work is a structural and morphological study of binary blends of Nylon 6 (Ny6) and polyolefins functionalized with acrylic acid, obtained by different mixing processes. The main aim was to analyze the effect of the presence of functional groups on the phase interactions,

the morphological features, the crystallization behavior and the structure of the components in the blends, as a function of composition and processing conditions [11].

The crystallization behavior of a polymer in a blend is affected by many factors, such as composition, thermal history, interfacial interactions, size of dispersed particles, and size distribution. If the dispersion is fine enough, other equilibrium and non-equilibrium phenomena should be considered. In the presence of very small particles the character of nucleation changes: different types of heterogeneous nuclei become activated or even homogenous nucleation occurs [12]. The change in nucleation type induces variations in undercoolings at which crystals start to grow—the crystallization may be shifted to lower temperatures, resulting in different crystalline phases and forms. It has been shown that for a number of polymers the nucleation type of polymer droplets can change from heterogeneous to homogeneous. In particular, for Ny6 dispersed droplets the crystals can start growing at a temperature of about 60° C lower than that observed for heterogeneous nucleation [13]. The crystalline structure of Ny6 is temperature dependent, specifically the contributions of α and γ crystals change with the undercooling [14].

Here, we focus the attention on the crystalline structure

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Fig. 1. Apparent viscosity vs. shear rate of polyolefins used in the studies.

and phase morphology of blends of Ny6 with low-density polyethylene (LDPE) and with polypropylene, both functionalized with acrylic acid. Using two different equipments for mixing: internal mixer and mixing extruder, varied the blending conditions.

2. Experimental

2.1. Materials

Two compatibilized systems were examined: blends of commercial Ny6 ADS 40, SNIA, $(M_w = 62000,$ density = 1.14 g/cm³, MFI = 4.6 g/10 min, $[-NH_2]$ end groups = 32 meg/kg) with ethylene–acrylic acid copolymer (PE– AA), Polysciences, containing 8 wt.% of acrylic acid and with acrylic acid grafted isotactic polypropylene (PP–AA), Polybond 1001, BP Chem, MFI = 40 g/10 min, density = 0.91 g/cm³, containing 6 wt.% of AA. Reference blends were prepared of the same grade of Ny6 with LDPE, Riblene FC 39 and with isotactic polypropylene (iPP) Moplen F30, Himont, $M_w = 270\,000$, density = 0.90 g/cm³, MFI = $12 \frac{\text{g}}{10}$ min. The viscosities of polyolefins used in blending at 250° C are presented in Fig. 1. The viscosities of polyolefins were measured on a Capillary Rheometer CEAST, after vacuum drying at 90° C. The range of shear rate corresponding to the mixing conditions in melt mixer and extruder (20–40 rpm) is between 7.5 and 150 s^{-1} . It is seen that the viscosities of iPP and PP–AA are quite similar at this temperature and shear rates are not very different from the viscosity of Ny6 (melt viscosity of Ny6 at 250° C, measured in a cone and plate Rheometrix RDS is about 800 Pa s at a shear rate of $100 s^{-1}$) so, in the differenciation of dispersion during the blending only the change in the interfacial energies due to the presence of acrylic acid groups in PP–AA should play an important role. In the case of Ny6/PE blends the low-density polyethylene was chosen as the reference polyolefin. Its viscosity at 250° C and a shear rate of 100 s⁻¹ (710 Pa s) is very similar to the viscosity of Ny6 (800 Pa s), hence the dispersion achieved should be optimal. Any better dispersion for the system Ny6/PE–AA (with lower viscosity of PE–AA) as compared to Ny6/LDPE should be attributed to the compatibilizing role of acrylic acid groups present in PE–AA.

2.2. Blending

Before blending, all components were dried under vacuum at 70 for polyolefins and at 110° C for Ny6, for 24 h. Two methods of blending were employed:

- 1. mixing in a Brabender Plasticorder internal mixer, operating at a roller speed from 20 to 40 rpm, at 250° C, under nitrogen flow, for 10 min; and
- 2. extruding in a Brabender extruder PL 2000, equipped with a 19 mm, $L/D = 25$ mixing screw and a slot capillary die, operating at 40 rpm, the barrel being at temperatures from 234 to 253 $^{\circ}$ C, the head at 256 $^{\circ}$ C. The samples were in both cases premixed before blending.

After blending the strands were cooled in air to ambient temperature. Blend compositions were in a range from 10 to 80 wt.% of Ny6. The same preparation procedure was also applied to pure components.

2.3. SEM

Melt quenched samples, fractured in liquid nitrogen and gold coated, were investigated using a Jeol T300 scanning electron microscope.

2.4. Calorimetry

DSC runs on 6–10 mg samples of obtained blends were performed under nitrogen flow using a Perkin–Elmer DSC 2C equipped with Data Station 3006, applying the following procedure: (a) first heating run, at $10^{\circ}C/\text{min}$, up to $250^{\circ}C$; (b) maintaining the temperature at 250° C for 5 min; (c) cooling to room temperature at 10° C/min; and (d) second heating run at 10° C/min, up to 250° C.

2.5. X-ray diffraction

Wide-angle X-ray (WAXS) diffraction spectra for samples obtained by mixing and extrusion, heated at 10° C/min to 250° C and cooled at 10° C/min to room temperature, were collected using a Siemens Diffractometer, CuK_{α} radiation at 40 kV, 20 mA and DRON 2.0, CuK_{α} , at 30 kV, 10 mA filtered electronically and with Ni filter to the wavelength of 0.1542 nm.

2.6. Deconvolution analysis

The method of separation of overlapping X-ray diffraction peaks consists on a least square minimization procedure for a sum of several Gaussian and Lorentzian curves $(A_i \exp[-0.5(2\theta_i - 2\theta)^2/\sigma_i^2]$ and $A_i/[1 + (2\theta_i - 2\theta)^2/\sigma_i^2]$, respectively), corrected for absorption, polarization and

Fig. 2. Scanning electron micrographs of fractured surfaces of Ny6 blends containing: (a) 80% PE–AA (5000×, melt mixed blend); (b) 80% LDPE (500×, melt mixed blend); (c) 80% PE-AA (3500×, extruded blend); (d) 60% PE–AA (3500×, extruded blend); (e) 80% PP–AA (3500×, extruded blend); and (f) 80% iPP (1000×, extruded blend).

background. To start the calculations, a set of initial parameters: postulated positions, widths and heights of peaks corresponding to reflections from various crystallographic planes, as well as those of amorphous halo, are usually required. The program, for each variable (position, height and width of every peak), performs a search of a surrounding *n*-dimensional grid of points (where *n* is the number of variables), to find points in which a function formed of the Gaussian–Lorentzian profiles decreases. When no such a point is found, the search increment factor

is decreased and the procedure is repeated, until the set accuracy of fitting is achieved.

This method, described in a more detailed form in [15] (see also [14]), has been mostly used for analysis of homopolymer samples [14–17]. In case of blends the problem is more complex. Besides the parameters mentioned above, some additional constraints must be set for the calculations, referring to known blend composition, correlations between integral intensities of reflections from different crystallographic planes in a given crystalline form, based on atomic

Fig. 3. The number-average particle diameters of the dispersed component as a function of the blend composition for Ny6/PP and Ny6/PP–AA blends (based on measurements of more than 100 particles in each case).

scattering factors, and also crystallinity levels of the components. While the first condition is obvious, the other two need discussion, which is given in Section 3.

3. Results

WAXS analysis followed comprehensive morphological and thermal investigations of described systems [11]. SEM micrographs (Fig. 2) revealed that the phase morphology strongly depends on the concentration of the functionalized polyolefins. The number-average particle diameters of the dispersed component are reported in Fig. 3 as a function of the blend composition for Ny6/PP and Ny6/PP–AA blends (based on measurements of more than 100 particles in each case). It is found that the average size of Ny6 particles are close to $1 \mu m$ and seem to be only little affected by the blend composition. In the same composition range the homopolymer blends Ny6/PP display much larger sizes of Ny6 droplets, between 5 and 16 μ m. For Ny6 concentrations above 50 wt.%, i.e. if Ny6 is a continuous phase, the sizes of PP inclusions are almost constant with increasing concentration, at approx. $4 \mu m$.

A finer dispersion of the polyamide phase was observed for melt mixing process which leads to the formation of apparently smaller dispersed Ny6 droplets than for extruding, due to better blending conditions. This effect is illustrated by the number average diameters (based on measurements of more than 100 particles in each case) of Ny6 dispersed phase for the blend Ny6/PE–AA 20/80. Average Ny6 particle sizes measured for melt mixed Ny6/LDPE blends with LDPE matrix is in the range of $5-5.5$ µm, while 0.51 µm for Ny6/ PE-AA melt mixed blend and 0.9 μ m for extruded blend.

A fine dispersion of Ny6 particles in functionalized polyolefin matrix was found to influence the crystallization behavior of Ny6 in the blends. Thermograms obtained

Fig. 4. DSC thermograms of: (a) Ny6/PP-AA; and (b) Ny6/PE–AA blends obtained by extrusion (cooling run– 10° C/min).

from DSC runs (Fig. 4) showed that in blends with PE–AA, dispersed polyamide crystallizes in a wide temperature range below the usual crystallization temperature of pure Ny6, down to crystallization temperature of PE–AA. In blends with PP–AA, the dispersed Ny6 crystallizes almost

Fig. 5. Temperature of melting peaks for samples of 50/50 Ny6/PP and Ny6/PP–AA vs. isothermal crystallization temperature.

coincidentally with the polyolefin. This effect was explained in terms of fractionated crystallization process [11]. If the number density of the dispersed polymer particles in the blend is larger than the number of heterogeneities which usually induce nucleation in this component, the crystallization of the dispersed phase may take place in several steps at higher undercoolings (at which other types of heterogeneities are activated) and, eventually, homogenous nucleation may take place [12]. The phase transition processes of the functionalized blends were also examined by means of simultaneous SAXS and WAXS real-time experiments (with synchrotron radiation source) in the range $80-245^{\circ}$ C at heating–cooling rate of 10° C/min [18]. For Ny6/PP–AA 60/40 and Ny6/PE–AA 60/40 blends the WAXS data pointed out the formation of Ny6 crystals at 132 and 125° C, respectively, about 60° C lower than the crystallization temperature of the pure Ny6. A detailed description of the crystallization and melting behavior of investigated blends, both under dynamic and isothermal conditions will be given elsewhere.

Melting thermograms of Ny6/polyolefin blends isothermally crystallized from the melt, in the range of $100-180^{\circ}$ C, (see Fig. 5 for the plot of T_m vs. T_c for isothermally crystallized 50/50 blend of Ny6/PP and Ny6/PP–AA) revealed also the presence of melting peaks at temperatures lower than the usual melting temperature of Ny6 α form $(T_{\rm m}^{\alpha} =$ 221° C), indicating the existence of a considerable amount of γ crystals ($T_{\text{m}}^{\gamma} = 215^{\circ}\text{C}$) in polyamide as a dispersed phase [19]. The melting peak of γ phase for non-functionalized blends of Ny6 and iPP does not appear at any Ny6 content.

WAXS analysis was performed in order to relate the crystalline structure of blends to their compositions. WAXS patterns and resolved separate peaks for pure components are presented in Fig. 6, scans and fits for a selection of investigated Ny6/PE–AA blends are shown in Fig. 7 and for Ny6/PP–AA blends in Fig. 8. For comparison, fits for blends of Ny6 with non-functionalized homopolymers iPP and LDPE were also obtained and are also shown in

Fig. 6. WAXS spectra and resolved peaks of the following mixer processed neat blend components: (a) Ny6 cooled from the melt at $10^{\circ}/\text{min}$; (b) Ny6 melt quenched in liquid nitrogen; (c) PE–AA; (d) LDPE; (e) PP–AA; and (f) iPP. Samples (c)–(f) cooled from the melt at 10° C/min. — experimental profile, \cdots calculated profile and resolved peaks. Peaks correspond to the α and γ phases of Ny6 in (a) and (b), the orthorhombic and triclinic phases of PE in (c) and (d) and the α and γ phases of PP in (e) and (f). The crystallographic planes are indexed in parentheses, the amorphous halo of each sample is labeled with δ .

respective figures. WAXS spectra of blends are composed of a considerable number of reflections (for Ny6/PP–AA blends at least 11 crystalline peaks and two amorphous halos) (Fig. 8), some of these reflections are located at very close 2θ -positions [Tables 1–3]. If two or more peaks occur at nearly the same positions, the fitting process results in

some cases in reducing one of such peaks, or even clearing it. It is then necessary to ensure that calculation results comply with a range of realistic physical conditions.

In general, relations between peaks characteristic for pure polymers should also be retained in their blends. In particular, the integral intensity ratios for pairs of crystalline reflections in each component should be confined within reasonable range (see also Ref. [14] for discussion concerning Ny6). Integral intensity (peak area) ratios were calculated from deconvoluted spectra of pure components and included, with allowed variability ranges, as the limiting conditions in the calculations for blends. The only exception was the reflection from γ crystalline form of Ny6 in the

Fig. 8. WAXS spectra and resolved peaks of mixer processed Ny6 blends containing: (a) 75% PP–AA; (b) 25% PP–AA; (c) 75% iPP; and (d) 25% iPP. — experimental profile, ··· calculated profile and resolved peaks for PP–AA and iPP, ---- resolved peaks for Ny6.

blends (only a (200) plane diffraction maximum was taken into account), which—as it was pointed out above—was supposed to change in the blends. For this reflection no constraints were set.

It was not always possible to fulfill all these conditions in fits satisfactory close to experimental scans, although fits comprising at least the most prominent features of the crystalline structures of the components were obtained.

Physical limitation that should be taken into account is the crystallinity level reached by the components in the blends. The tendency of the computations for some blends was to increase the crystallinities of the components, which

Table 1

| Homopolymer, wt. crystallinity | Fitted peaks' positions and widths (°) | | | | | | | | |
|------------------------------------------------------------------------------------------|----------------------------------------|--------------|------------|-----------|----------|----------|-------|-------|----------|
| | δ | α_1 | α_2 | γ | | | | | |
| (a) Ny6, cooled from the melt at 10° C/min and melt quenched in liquid nitrogen | | | | | | | | | |
| Nylon 6 36% | 21.7 | 20.2 | 23.9 | 21.5 | | | | | |
| | 2.60 | 0.43 | 0.53 | 0.60 | | | | | |
| Nylon 6 quenched 36% | 20.7 | 20.2 | 23.1 | 21.5 | | | | | |
| | 3.47 | 0.83 | 0.74 | 0.54 | | | | | |
| (b) PE-AA and LDPE, cooled from the melt at 10° C/min | | | | | | | | | |
| | δ | orthorhombic | | triclinic | | | | | |
| | | (110) | (200) | (010) | | | | | |
| PE-AA 36% | 20.0 | 21.5 | 23.8 | 20.0 | | | | | |
| | 3.20 | 0.33 | 0.40 | 0.86 | | | | | |
| LDPE 37% | 20.4 | 21.5 | 23.8 | 19.5 | | | | | |
| | 2.33 | 0.23 | 0.34 | 0.13 | | | | | |
| (c) PP-AA and iPP, cooled from the melt at 10° C/min | | | | | | | | | |
| | δ | | | | α | | | | γ |
| | | (110) | 040 | (130) | (111) | (041) | (060) | (220) | |
| | | | | | | (-131) | | | |
| $PP-AA 60%$ | 17.2 | 14.1 | 16.9 | 18.7 | 21.2 | 22.0 | 25.5 | 28.8 | 20.0 |
| | 3.45 | 0.32 | 0.24 | 0.28 | 0.29 | 0.28 | 0.21 | 0.35 | 0.45 |
| iPP 56% | 18.9 | 14.1 | 16.9 | 18.6 | 21.2 | 22.0 | 25.6 | 28.9 | 19.9 |
| | 3.45 | 0.30 | 0.23 | 0.26 | 0.30 | 0.29 | 0.29 | 0.55 | 0.35 |

Results of calculations for mixer processed neat blend components: (a) Ny6, cooled from the melt at 10°/min and melt quenched in liquid nitrogen, (b) PE-AA and LDPE and (c) PP-AA and iPP; samples (b) and (c) cooled from the melt at $10^{\circ}/\text{min}$

increased the mathematical quality of the fits. In order to assess the crystallinity levels for investigated blends the enthalpies of fusion were calculated from the DSC runs. Crystallinity levels obtained—up to 36% for Ny6, 37% for LDPE, 36% for PE–AA, 56% for iPP and 60% for PP–AA were consistent with the literature data [15,20,21].

Upper limits for crystallinity levels of the components were incorporated in the deconvolution program. Again, in some cases it was very difficult to find an "equilibrium" between these limits and quality of the fit. Results of deconvolution calculations are summarized in Tables $1-3.$

The contribution of the γ phase in Ny6 crystal structure can be assessed as the ratio of the integral intensity of the (200) γ crystal plane reflection to the total polyamide share. The deconvolution routine employed allows calculating the areas of all separated peaks. The fraction of Ny6 γ phase in blends with different polyamide concentration is plotted against Ny6 content in Fig. 9. As the Ny6 amount in blend decreases, the γ phase contribution raises markedly—from 1.6 ± 0.9 wt.% in mixer-processed Ny6 homopolymer, through 4.4 \pm 2.3 wt.% in Ny6/PP–AA 75/25 blend, 5.5 \pm 3:5 wt:% in Ny6/PE–AA 80/20 blend (Ny6 in continuous phase), to 29.1 \pm 5.7 wt.% in Ny6/PE–AA 40/60 blend and

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to 31.1 \pm 2.3 wt.% in Ny6/PP–AA 25/75 blend (all blends as obtained by the mixing procedure). Similar effects were observed in blends with functionalized polyolefins prepared by extrusion. Reference measurements for non-functionalized Ny6/LDPE and Ny6/iPP systems also revealed an increase in the γ phase fraction (as compared to the Ny6 homopolymer sample) but the effect is about two–three times smaller $(9.0 \pm 2.5 \text{ wt.}\%$ in Ny6/LDPE 20/80 blend and 6.2 ± 3.5 wt.% in Ny6/iPP 25/75 blend) and is fairly constant with blend composition. The tendency of remarkable growth of Ny6 γ phase contribution is apparent for all functionalized systems investigated, despite different accuracies of the fits obtained. (Also in case of more difficult WAXS scans, for which a number of trials had to be performed in search of fits complying given requirements, in all events the γ phase fraction was at comparable level).

4. Discussion

The effect of the influence of compatibilization on the polyamide γ phase content has been also observed in other blends based on this polymer [3,9,22–24]. However, only thermal data have been generally supplied and qualitative evaluation of X-ray experiments, giving no direct information on the blends structure. The deconvolution analysis employed in this work gives the possibility of a more straightforward investigation of this problem.

It should be noted that X-ray diffraction contour produced by a blend is strongly dependent on residual stress arising in a sample during crystallization. The shape and position of each separate peak of the spectrum is influenced by the value and the sign of the stress. These, in turn, result from a complex tangle of crystallization conditions (mainly temperature, cooling rate, heat removal), which influence morphology of the blend, and also from thermal expansion coefficients of the components, thermal shrinkage during crystallization, temperatures of crystallization of the components and their dispersion (phase inversion and degree of dispersion).

In all investigated blends of Ny6 with functionalized polyolefin as a matrix a tendency of the γ phase raising contribution with increasing functionalized polyolefin content in the blend was observed. This effect should be attributed to the size of dispersed Nylon particles which is markedly reduced by the interactions between the functional groups of polyolefin and the polar groups in polyamide chain. The occurrence of interactions between blend components has been reported [2,3], giving rise to the formation of a copolymer which can act as a compatibilizer for the blend, thus decreasing the interfacial tension and enhancing the phase dispersion. Fine dispersion of Ny6 particles (the particle size in a range of $1 \mu m$ —as it was stated before—strongly affects the

Fig. 9. The Ny6 α (O) and γ (\bullet) crystalline phase weight fraction of the total Ny6 content in blends with: (a) PE–AA; (b) LDPE; (c) PP–AA; and (d) iPP. $I_{\text{total}} = (I_{\text{amorph}} + I_{\gamma} + I_{\alpha})$; I_{amorph} , I_{α} , I_{γ} —the integral intensities of the Ny6 amorphous phase halo and the α and γ polymorph reflections, respectively.

nucleation process. Since heterogeneous nucleation of Ny6 is not increased in the blends and the number density of the dispersed polyamide particles exceeds the number of heterogeneities active in bulk polyamide at usual crystallization temperature, a large fraction of particles cannot crystallize at ordinary T_c . Therefore, a shift of the crystallization of Ny6 to lower temperatures occurs, eventually reaching the homogenous nucleation temperature, which is low (about 130° C) and is even lower in the boundary region, where Ny6 macromolecules are strongly interacting with the functional groups of the polyolefin. As it is seen from the resolved X-ray spectrum for melt quenched Ny6 sample (Fig. 6.) and has been reported for quenched Ny6 samples [25] (see also Ref. [18]), the γ crystal phase is more likely to form at such crystallization temperatures.

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