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# Effect of Functionalized Polypropylene on Thermal, Mechanical, and Morphological Properties of Polypropylene/Nylon-6 Blends

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# EFFECT OF FUNCTIONALIZED POLYPROPYLENE ON THERMAL, MECHANICAL, AND MORPHOLOGICAL PROPERTIES OF POLYPROPYLENE/NYLON-6 BLENDS

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#### ABSTRACT

The compatibilizing effect of polypropylene-graft-butyl acrylate (PP-g-BA) on the morphological, thermal, and mechanical properties of polypropylene and nylon-6 (Ny-6) blends was investigated. Addition of 2.4% (w/w) PP-g-BA compatibilizer in binary PP/Ny-6 blends results

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in more regular and finer dispersions of the two phases and improved mechanical properties. Various mathematical models have been used to explain the mechanical properties of these blends. Molau's test and MFI data support the formation of PP-g-Ny-6 due to chemical reaction between the terminal amino groups of Ny-6 and the ester groups of PPg-BA copolymer.

#### INTRODUCTION

Blending of polymers offers a means to obtain tailor-made products with a good range of properties at a low cost for specific applications. In the case of miscible blends, the method is straightforward. However, blending of two polymers usually leads to immiscibility, and the desired properties are not achieved without the presence of a third component, a compatibilizer, which is able to decrease the interfacial tension and increase the adhesion between the two phases.

Blends of polyolefins and polyamides [1–4] combine properties of both components and have great industrial importance. For example, by blending polypropylene and polyamide, one can combine the thermal and mechanical properties of polyamide with isotactic polypropylene's (iPP) insensitivity to moisture. Among polyolefins, iPP is a most important polymer for its intrinsic properties such as high melting temperature, low density, high chemical inertness, and capability of being produced with different morphological and molecular structures. It can be modified by grafting functional groups onto it so that it can be successfully blended with polar polymers such as polyamide [5], poly(ethylene terephthalate) [6], and polycarbonate [7].

Attempts have been made to increase the compatibility of polypropylenepolyamide-6 (PP/Ny-6) blends. Ide and Hasegawa [8] added maleic anhydride functionalized iPP graft copolymer to binary blends of PP/Ny-6 and obtained an almost homogeneous morphology. Similar observations were made by Koregava and Okada [9] when iPP-g-acrylic acid was used as the compatibilizer for the PP/ Ny-6 system.

Compatibilization through the addition of maleic anhydride or carboxylic acid functionalized PP copolymer has also been reported for polyolefin-polyamide blends [10-13] in particular. Takashi [14] reported excellent improvement in the impact strength of PP/Ny-6 by blending it with PP-g-acrylic acid.

However, the potential of PP-g-butyl acrylate (PP-g-BA) has not been exploited for PP/Ny-6 blend systems. In this paper we report investigations on PP/Ny-6 binary and PP/Ny-6/PP-g-BA ternary blends, particularly to

- 1. Correlate the morphology of the blend with the mechanical, mainly tensile and impact properties.
- 2. Establish a correlation between the experimental tensile modulus and theoretically calculated values derived from various proposed mathematical models.

#### THEORETICAL BACKGROUND

Many theories have been put forward for predicting the elastic modulus of heterogeneous blends. According to Dickie [15], there are three principal groups of models that can predict the modulus-composition dependence for blends.

- 1. Mechanical coupling model
- 2. Self-consistent model
- 3. Bounds on modulus model

#### Mechanical Coupling Model

The mechanical coupling model is an empirical expression containing an adjustable parameter. However, it is not a morphologically or mechanically realistic model for blend structures.

#### Self-Consistent Model

The self-consistent model is based on the assumptions that

- 1. Perfect adhesion exists between the matrix and the inclusion
- 2. Interinclusion interactions are negligible
- 3. The inclusions are spherical

According to Kerner's model [16] based on the above assumptions, when two polymeric systems have the same Poisson's ratio  $(\nu)$ , the tensile modulus of the blends is given by

$$E_{\rm b} = E_{\rm m} \left[ \frac{\phi_{\rm d} E_{\rm d} / [(7 - 5\nu_{\rm m}) E_{\rm m} + (8 - 10\nu_{\rm m}) E_{\rm d}] + \phi_{\rm m} / 15(1 - \nu_{\rm m})}{\phi_{\rm d} E_{\rm m} / [(7 - 5\nu_{\rm m}) E_{\rm m} + (8 - 10\nu_{\rm m}) E_{\rm d}] + \phi_{\rm m} / 15(1 - \nu_{\rm m})} \right]$$
(1)

The Poisson's ratios of Ny-6 and iPP are 0.4 and 0.35, respectively. These values are close enough to use Kerner's equation in the above form for PP/Ny-6 blends. The terms in Eq. (1) have standard notations: E is the tensile modulus,  $\phi$  is the volume fraction of the discrete phase, and  $\nu$  is Poisson's ratio. The subscripts b, m, and d refer to the blend, the matrix, and the dispersed phase, respectively.

For blends in which inclusions are loosely bound, they contribute little to the overall modulus of blends  $(E_b)$ . Hence the observed modulus of blends is due to the contribution of the matrix only, leading to the assumption  $E_d \cong 0$ . As a result, Eq. (1) is reduced to

$$E_{b}^{-1} = E_{m}^{-1} \left[ 1 + \frac{15(1 - \nu_{m})\phi_{d}}{(7 - 5\nu_{m})\phi_{m}} \right]$$
(2)

In Kerner's derivation only particle-matrix adhesion, but no particle-particle interaction, was assumed. Therefore, Kerner's model may not be applicable to polymer blend systems in which strong interactions between the inclusion and matrix exist. For such systems, Nielsen [17] suggested a modification of Kerner's model.

According to Nielsen, 1) for a rigid polymer dispersed in a rubber matrix:

$$\frac{E_{\rm b}}{E_{\rm m}} = \frac{1 + AB\phi_{\rm d}}{1 - B\psi\phi_{\rm d}} \tag{3}$$

in which

$$B = \left(\frac{E_{\rm d}}{E_{\rm m}} - 1\right) \quad \left(\frac{E_{\rm d}}{E_{\rm m}} + A\right)$$

and

$$\psi = 1 + \left(\frac{1 - \phi_{\max}}{\phi_{\max}^2}\right) \phi_d$$

2) For rubber inclusions in a rigid matrix:

$$\frac{E_{\rm m}}{E_{\rm b}} = \frac{1 + AB_{\rm i}\phi_{\rm d}}{1 - B_{\rm i}\psi\phi_{\rm d}} \tag{4}$$

in which

$$B_{\rm i} = \left(\frac{E_{\rm m}}{E_{\rm d}} - 1\right) \left/ \left(\frac{E_{\rm m}}{E_{\rm d}} + A\right)\right.$$

and

$$\psi = 1 + \left(\frac{1-\phi_{\max}}{\phi_{\max}^2}\right)\phi_d$$

 $\phi_{\text{max}}$  is the maximum packing volume which can be considered as a scale of the interaction between the two phases. The constant A is  $(7 - 5\nu_{\text{m}})/(8 - 10\nu_{\text{m}})$  for Eq. (3) and  $(8 - 10\nu_{\text{m}})/(7 - 5\nu_{\text{m}})$  for Eq. (4).

#### **Bounds on Modulus Model**

Paul [18] used the calculus of variation to bound the strain energy and set the limits on the modulus of composites. According to Paul, the upper bound is given by

$$E_{\rm b} = (1 - \phi_{\rm d})E_{\rm m} + \phi_{\rm d}E_{\rm d} \tag{5}$$

and the lower bound by

$$E_{\rm b} = 1 \left/ \left( \frac{1 - \phi_{\rm d}}{E_{\rm m}} + \frac{\phi_{\rm d}}{E_{\rm d}} \right)$$
(6)

#### MATERIALS AND METHODS

Isotactic polypropylene (Koylene MOO30, specific gravity 0.899, MFI 1  $g \cdot \min^{-1}$ ) was obtained from the Indian Petrochemical Co., Vadodara, India. Nylon-6 (Ny-6) (M28RC, specific gravity 1.127, MFI of Ny-6, 3.2  $g \cdot \min^{-1}$ ) was obtained from Gujarat State Fertilizer Co., Vadodara, India.

Details about the synthesis of PP-g-BA with a degree of grafting of 2.85% and its use as a compatibilizer in the preparation of blends were reported earlier [19].

#### Preparation of Blends

A Brabender Plasticorder system coupled with a single-screw extruder (L/D = 16) was used for the preparation of blends. The Ny-6 granules were dried at 105 °C for 3 hours prior to use. All the binary and ternary blends were prepared in a one-step mixing process. The temperatures of the three zones of the extruder were

kept at 190, 220, and 235 °C, and the die temperature at 230 °C. The mixing was carried out at 30 rpm. Binary PP/Ny-6 blends of 75/25, 60/40, 50/50, 40/60, and 25/75 composition and corresponding ternary blends of PP/Ny-6/PP-g-BA (2.4%) compositions were prepared for the study.

The compounded pellets were dried and injection molded to obtain test specimens for the measurement of tensile properties, Izod impact strength, and flexural strength.

Tensile measurements were carried out on an Instron 1195 machine at room temperature following the procedure described in ASTM D 683. A crosshead speed of 50 mm  $\cdot$  min<sup>-1</sup> was used in all measurements. The flexural strength measurement was carried out following the procedure described in ASTM D 790. A three-point bending system utilizing central loading on a doubly supported beam was used for the measurement. A crosshead speed of 2.8 mm  $\cdot$  min<sup>-1</sup> was used for all measurements.

Impact strength was measured following the procedure described in ASTM D 256. Specimens with a triangular notch of 2.5 mm depth at a 45° angle were used for the measurements. Rockwell hardness and heat deflection temperature measurements were carried out according to ASTM D 785 and ASTM D 648, respectively.

Thermal analysis was performed under nitrogen atmosphere using a Delta Series DSC-7. The temperature was raised from 25 to 250°C at a rate of  $10°C \cdot min^{-1}$ , and after 5 minutes it was swept back at  $-10°C \cdot min^{-1}$ . The second heating run was recorded at  $10°C \cdot min^{-1}$ . The melting temperature  $(T_m)$  and heat of fusion  $(\Delta H_f)$  of the samples were obtained from the peak maxima and the area under the melting peaks, respectively. The percent crystallinity of the PP and Ny-6 phases was calculated by using the following equations:

% crystallinity (PP) = 
$$\frac{\Delta H_{\rm f}^*(\rm PP)}{\Delta H_{\rm f}^{\rm o}(\rm PP)} \times 100$$

and

% crystallinity (Ny-6) = 
$$\frac{\Delta H_{\rm f}^*(\rm Ny-6)}{\Delta H_{\rm f}^*(\rm Ny-6)} \times 100$$

where  $\Delta H_{f}^{*}(PP)$  and  $\Delta H_{f}^{*}(PA6)$  are the heats of fusion of PP and Ny-6 in the blends, respectively.  $\Delta H_{f}^{\circ}(PP)$  and  $\Delta H_{f}^{\circ}(Ny-6)$  are the heats of fusion of 100% crystalline PP and Ny-6, respectively.

Blend morphology was studied using a JEOL JSM 35 C scanning electron microscope operated at 15 kV. The surfaces of the cryogenically fractured standard tensile bars were coated with gold to avoid charging under an electron beam.

Melt flow indices (MFI) of PP, Ny-6, and blends were determined using a Davenport rheometer. The capillary die length was 8 mm, the diameter 2 mm, and the driving weight 2.65 kg at 230°C.

#### **RESULTS AND DISCUSSION**

When PP-g-BA copolymer is added to PP/Ny-6 blends, the following reaction takes place between the ester group of butyl acrylate and the amino group of Ny-6 during melt mixing of Ny-6 and PP-g-BA at 235 °C [20, 21]:



Evidence supporting the formation of graft copolymers was obtained from Molau's solubility test [22]. When the binary blends were placed in formic acid, which is a solvent for Ny-6 and a nonsolvent for PP, Ny-6 was dissolved and PP phase was separated and floated on the solution within 1–2 hours. In blends containing PP-g-BA (2.4% w/w), a colloidal solution was formed in formic acid without separation of the PP phase, indicating the presence of PP-g-Ny-6 copolymer.

# Morphology

Figures 1-10 show SE micrographs of the cryogenically fractured standard bar surfaces of PP/Ny-6 blends. Particles in the matrix morphology are observed in Figs. 1 and 4 for PP/Ny-6 75/25 and 25/75 blends. This is clearly seen at higher magnification in Figs. 5 and 6. A typical morphology of an immiscible mixture, i.e.,



FIG. 1. SE micrograph of cryogenically fractured surface of PP/Ny-6 (75/25) blend  $(200 \times)$ .



FIG. 2. SE micrograph of cryogenically fractured surface of PP/Ny-6 (60/40) blend  $(200 \times)$ .

very large, and irregular domains is observed with relatively smaller contact areas with the matrix in these figures. The surfaces of particles are observed to be very smooth. A sign of poor adhesion between the two phases is noted from the phase boundaries and pull-outs of the dispersed phase during the fracture process. PP/Ny-6 blends of 60/40 and 50/50 compositions show a co-continuous two-phase interpenetrating morphology in Figs. 2 and 3.

Figures 7-10 show the morphological texture of PP/Ny-6/PP-g-BA ternary blends at two different magnifications. It can be observed from Figs. 7 and 8 that ternary PP/Ny-6/PP-g-BAs of 73.2/24.4/2.4 and 24.4/73.2/2.4 composition show a two-phase morphology, but the mixing of the phases has been greatly improved due to compatibilization. It is also seen from Figs. 9 and 10 (and later in Table 2) that the size of dispersed particles is decreased considerably and that particles show rough surfaces with larger contact areas with the matrix in compatibilized blends. These observations indicate that PP-g-BA acts as a compatibilizer for PP/Ny-6 blend systems. However, at blend compositions of  $\sim$  50:50 where a co-continuous



FIG. 3. SE micrograph of cryogenically fractured surface of PP/Ny-6 (50/50) blend  $(200 \times)$ .



FIG. 4. SE micrograph of cryogenically fractured surface of PP/Ny-6 (25/75) blend  $(200 \times)$ .

morphology is exhibited, not much difference in domain size is observed for PP/Ny-6 and PP/Ny-6/PP-g-BA blends (Table 1).

# **Tensile Properties**

The stress-strain curves for the PP/Ny-6 binary and PP/Ny-6/PP-g-BA ternary blend systems along with PP and Ny-6 are shown in Fig. 11. Both PP and Ny-6 show necking behavior, while the blends exhibit negligible necking. Addition of Ny-6 to PP increases the modulus and tensile strength progressively. It is observed from Fig. 12 that tensile modulus values of all ternary PP/Ny-6/PP-g-BA blends are higher than those of the corresponding binary PP/Ny-6 blends. The increase in the tensile modulus of binary blends with an increase in NY-6 content is due to the



FIG. 5. SE micrograph of cryogenically fractured surface of PP/Ny-6 (75/25) blend  $(1000 \times)$ .



FIG. 6. SE micrograph of cryogenically fractured surface of PP/Ny-6 (25/75) blend  $(1000 \times)$ .

higher tensile modulus of Ny-6, whereas the presence of compatibilizer (PP-g-BA) in the ternary blends improves the homogeneity of the corresponding blends, resulting in further improvement in tensile modulus.

Theoretically calculated tensile moduli of various ternary blends, based on the various theories discussed earlier, are given in Fig. 13, along with the experimental data. It is observed that for PP-rich and Ny-6-rich blends compatibilized with 2.4% (w/w) PP-g-BA, Kerner's model based on the assumption of perfect adhesion between the matrix and dispersed phase and Nielsen's model based on the assumption of perfect adhesion between the dispersed phase and matrix and the existence of particle-particle interaction predict the experimental data rather well. On the other hand, Kerner's model for loosely bound particles in the matrix predicts a trend completely opposite to the experimental data. This implies that the discrete Ny-6 or



FIG. 7. SE micrograph of cryogenically fractured surface of PP/Ny-6/PP-g-BA (73.2/24.4/2.4) blend  $(200 \times)$ .



FIG. 8. SE micrograph of cryogenically fractured surface of PP/Ny-6/PP-g-BA (24.4/73.2/2.4) blend  $(200 \times)$ .

PP phases are not loosely bound to the PP or Ny-6 matrix, and some adhesive force exists between PP and Ny-6 in PP/Ny-6/PP-g-BA blends. The tensile moduli values for a rigid polymer dispersed in a rubber matrix and vice versa were calculated by adjusting  $\phi_{max}$  values to 0.7 in the Nielsen's model Eqs. (3) and (4). From Fig. 13 it can be seen that these calculated values agree with the experimentally obtained values.

It is observed from Figs. 14 and 15 that the tensile and flexural strength of all compatibilized blends are higher than those of noncompatibilized blends due to improved homogenicity.

## Impact Properties

Figure 16 illustrates the impact strength of PP/Ny-6 and PP/Ny-6/PP-g-BA blends as a function of blend composition. It can be seen from Fig. 16 that compatibilized blends produce a higher impact strength than the corresponding noncompati-



FIG. 9. SE micrograph of cryogenically fractured surface of PP/Ny-6/PP-g-BA (73.2/24.4/2/4) blend  $(1000 \times)$ .



FIG. 10. SE micrograph of cryogenically fractured surface of PP/Ny-6/PP-g-BA (24.4/73.2/2.4) blend  $(1000 \times)$ .

bilized blends. This can be attributed to the larger particle sizes  $\sim 25-30 \ \mu m$  (Table 1) and little adhesion between the dispersed phase and matrix (Figs. 1, 4-6), resulting in poor impact strength of binary blends.

From SEM micrographs (Figs. 7-10) it can be observed that the addition of PP-g-BA (2.4% w/w) to PP/Ny-6 blends produces a drastic reduction in the average dimensions (Table 1) of the domains of the dispersed phase with large contact areas, indicating the adhesion between PP and Ny-6. As a result, the impact strength of all ternary blends is higher than that of binary blends.

## Melt Flow Index (MFI)

From Fig. 17 it can be observed that all binary and ternary blends show a lower melt flow index than does Ny-6. It is seen from Fig. 17 that ternary PP/Ny-6/ PP-g-BA blends have lower MFI values than do the corresponding binary PP/Ny-6

Blend composition	Mean diameter for 30 measurements, μm	Average deviation for 30 measurements	Standard deviation for 30 measurements	
PP/Ny-6:				
75/25	25.40	0.012	3.126	
40/60	30.21	0.014	2.590	
25/75	20.42	0.052	0.187	
PP/Ny-6/PP-g-BA:				
73.2/24.4/2.4	12.75	0.030	0.456	
39.1/58.5/2.4	25.02	0.002	0.565	
24.4/73.2/2.4	6.89	0.043	1.242	

TABLE 1. Accuracy and Precision of the SEM Results



FIG. 11. Stress-strain curves for (a) PP/Ny-6 blends: 1, 75/25; 2, 60/40; 3, 50/50; 4, 40/60; 5, 25/75. (b) PP/Ny-6/PP-g-BA blends: 1, 73.2/24.4/2.4; 2, 58.5/39.1/2.4; 3, 48.8/48.8/2.4; 4, 39.1/58.5/2.4; 5, 24.4/73.2/2.4.



FIG. 12. Effect of blend composition on tensile modulus for ( $\bullet$ ) PP/Ny-6 blends and ( $\blacksquare$ ) PP/Ny-6/PP-g-BA blends.



FIG. 13. Tensile modulus versus blend composition for PP/Ny-6/PP-g-BA: (---), Paul's upper and lower bounds model, (--) Kerner's model with perfect adhesion, (---) Kerner's model with loosely bound inclusion, (--) Nielsen's model,  $(\bullet)$  experimental data.



FIG. 14. Effect of blend composition on tensile strength for ( $\bullet$ ) PP/Ny-6 blends and ( $\blacksquare$ ) PP/Ny-6/PP-g-BA blends.



FIG. 15. Effect of blend composition on flexural strength for ( $\bullet$ ) PP/Ny-6 blends and ( $\blacksquare$ ) PP/Ny-6/PP-g-BA blends.

blends. This may be due to such structural changes as the formation of PP-g-Ny-6 copolymer during melt mixing, as discussed earlier.

Values of the heat distortion temperature (HDT) and Rockwell hardness for PP/Ny-6 blends and PP/Ny-6/PP-g-BA blends are given in Table 2. It can be inferred from Table 2 that all the ternary blends have slightly higher hardness values than the corresponding binary blends, but the variation is not significant.



FIG. 16. Effect of blend composition on impact strength for ( $\bullet$ ) PP/Ny-6 blends and ( $\blacksquare$ ) PP/Ny-6/PP-g-BA blends.



FIG. 17. Effect of blend composition on melt flow index for ( $\bullet$ ) PP/Ny-6 blends and ( $\blacksquare$ ) PP/Ny-6/PP-g-BA blends.

Blend composition (RHR)		HDT, °C	PP		Ny-6	
	Rockwell hardness		<u>т</u> •с	Crystallinity,	т °С	Crystallinity,
			1 <sub>m</sub> , C	/0	1 <sub>m</sub> , C	
PP/Ny-6:						
(PP)	80	67.0	164.5	49.2		—
75/25	84	66.0	164.5	49.1	225.1	43.4
60/40	90	66.2		_	_	
50/50	95	66.4	164.2	49.1	225.0	43.2
40/60	99	65.0	_	—	-	
25/75	104	64.1	164.3	49.2	225.0	43.3
(Ny-6)	115	62.2	_		225.1	43.4
PP/NY-6/PP-g-BA	<b>A</b> :					
73.2/24.4/2.4	86	70.2	163.8	47.5	224.3	39.1
58.5/39.1/2.4	91	70.2		-	_	_
48.8/48.8/2.4	95	71.3	162.1	46.2	223.8	39.8
39.1/58.5/2.4	101	67.4	_		_	
24.4/73.2/2.4	105	69.5	160.6	47.0	222.1	41.1

TABLE 2. Thermomechanical and Thermal Properties of the Various Blends

# **Thermal Properties**

The melting peak temperatures for PP/Ny-6 and PP/Ny-6/PP-g-BA blends as determined from DSC thermograms are given in Table 2. Variation in melting point did not occur for the PP or Ny-6 phases in PP/Ny-6 blends, but in compatibilized blends the melting peaks shifted to a little lower temperatures due to the formation of PP-g-Ny-6.

The percent crystallinity of PP and Ny-6 phases in blends is given in Table 2. The percent crystallinity of PP and Ny-6 in the ternary PP/Ny-6/PP-g-BA blends was observed to be lower than in the corresponding binary blends. As mentioned earlier, reaction between the terminal amino groups of Ny-6 and the ester groups of PP-g-BA takes place during melt mixing of PP/Ny-6/PP-g-BA blends, resulting in the formation of PP-g-Ny-6. These grafted side chains hinder polymer chain packing. As a result, a reduction in percent crystallinity is observed.

#### Percentage Water Absorption

As PP is insensitive to moisture, from Fig. 18 it is observed that all the blends have lower percentage water absorption values than does Ny-6. From Fig. 18 it can also be observed that all the ternary blends absorb less water than the corresponding binary blends. The water susceptibility of Ny-6 is mainly due to the presence of amino groups. In PP/Ny-6/PP-g-BA blends, the ester group of butyl acrylate interacts with the amino group. As a result, the number of free amino groups in Ny-6 is reduced, hence reducing the susceptibility of Ny-6 for water. This observation can also be taken as an additional indication of the formation of PP-g-Ny-6.



FIG. 18. Effect of blend composition on percent water absorption for ( $\bullet$ ) PP/Ny-6 blends and ( $\blacksquare$ ) PP/Ny-6/PP-g-BA blends.

#### CONCLUSION

Polypropylene-graft-butyl acrylate copolymer having a butyl acrylate content of 2.85% (w/w) acts as an interfacial agent for PP and Ny-6 blends. The addition of just 2.4% (w/w) of the graft copolymer results in a significant improvement in impact strength (~40%) and in tensile mechanical properties. A more regular and finer dispersion of phases is observed upon addition of a small amount of PP-g-BA copolymer in PP/Ny-6 blends. When the value for the maximum packing volume ( $\phi_{max}$ ) is adjusted, the elastic moduli values predicted by Neilsen's model and Kerner's models for a system with perfect adhesion between the dispersed phase and matrix were found to agree with the experimental values.

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