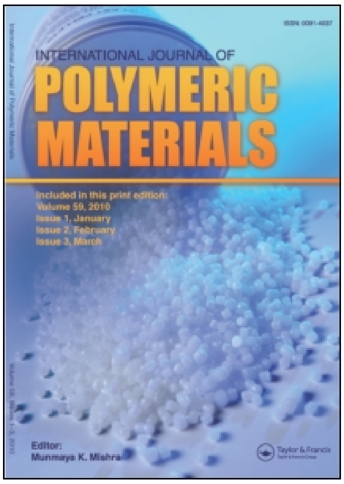

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Morphology, Thermal and Mechanical Properties of PA1010/PP-g-ATBS/PP Blends

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The modification of polypropylene (more precisely, a propylene/ethylene random copolymer containing 10% ethylene) has been accomplished by melt grafting of acrylamide tertiary butyl sulfonic acid(ATBS) initiated with a radical initiator. The resulting PP-g-ATBS was used to prepare ternary blends of PA1010/PP-g-ATBS/PP and binary blends of PA1010/PP. The size of domains of PP in ternary blends is much smaller than that in binary blends. It was found that mechanical properties of ternary blends obviously surpassed that of binary blends. These behavior could be contributed to chemical interactions between sulfonic acid groups of PP-g-ATBS and end amino group of PA1010. Thermal and rheological analysis were performed to confirm the possible chemical reactions taken place during the blending process.

Keywords: Polypropylene; polypropylene-ethylene copolymer acrylamide; grafting; polyamide 1010; blends

INTRODUCTION

Multicomponent polymer materials are frequently made by blending two or more miscible or immiscible polymers. It is very difficult to obtain good dispersion in polymer blends which components are insoluble each other, particularly for combinations of polar polymers with nonpolar polymers. Due to their difference of polarities, the system usually separates into two distinct phases. The major component forms a matrix and the minor component forms domains, whose size and shape greatly depends upon several factors, such as melt vicosities of components, interfacial tension and adhesion, processing condition, and so on. The final properties of these blends are strongly affected by the volume fraction of the two components, the size and shape of domains, and the interfacial tension and adhesion between the two phase.

Generally, simple blends of polymers exhibit unstable phase morphologies and poor properties. In typical immiscible polyblending system, a satisfactory physicomechanical behavior critically depends on the proper interfacial tension to generate a small phase size and strong interfacial adhesion to transmit force effectively between two phase. Methods to reduce interfacial tension and improve phase adhesion between immiscible components have been one of the main subjects in past two decades. Many articles [1–4] have reported that the presence of a block or graft copolymer having appropriate chemical structure can provide lowering of the interfacial energy and improvement of the interfacial adhesion between two phases, and the final morphological effect is a reduction of dispersed particle in the blends.

Polypropylene is often blended with other polymers to improve their performances for some special applications. Functionalized PP compatibilizers are popular third component that is added to aid both adhesion and mixing in PA/PP system. Previous studies have shown PP-g-MAH is a effective compatibilizer for PA6/PP blends [5–9]. Compatibilization is considered to occur through chemical linkage of the anhydride on the compatibilizer chain and end amino groups of PA6. The graft copolymers preferentially reside at the interface. Several authors also modified PP with acrylic acid, this kind of grafted PP has been used to compatibilize PA/PP blends [10].

In our previous work [11], the preparation and characterization of PP-g-ATBS have been reported. The main purpose of this work is to study the morphologies of PA1010/PP and PA1010/PP-g-ATBS/PP blends. In particularly PA1010 rich blends were investigated.

EXPERIMENTAL

Materials

The polyamide-1010 (PA1010) was purchased from JIlin Shijingguo Union Chemical Co. (China). Its relative viscosity is 2.1, and melt flow index is 1.0 g/10min. The polypropylene (PP) was supplied by LiaoNing PanJin Gas Chemical Co. (China) with melt flow index 10 g/10 min, C2-content of this PP sample is 10% (weight). PP grafted with acrylamide tertiary butyl sulfonic acid (ATBS) was prepared by reaction extruding initiated with dicumyl peroxide as reported in previous article [11]. The content of ATBS in PP-g-ATBS used in this work was 0.46% (in mole).

Preparation of PA1010/PP and PA1010/EP-g-ATBS/EP Blends

Binary PA1010/EP and ternary PA1010/PP-g-ATBS/PP blends were prepared by melt mixing the polymers in a twin-screw extruder (L/D = 30) at the temperature 195 $\sim$ 215°C, composition of blends were listed in Table I. Testing specimens for mechanical properties were prepared by using JSWF17SA injector with barrel temperature of 195 $\sim$ 215°C, and injection pressure of 60 MPa.

Thermal Analysis

The thermal behaviors of blending samples was determined on Perkin-Elmer DSC-2. The thermograms was obtained from 50 $\sim$ 250°C at a heating rate of 10 C/min. All measurements were performed under a nitrogen atmosphere. Samples used for DSC experiments were extracted with xylene to remove EP component from related binary blends.

TABLE I The mechanical properties of PA1010/PP and PA1010/PP-g-ATBS/PP blends

PA1010 (%)	*PP-g-ATBS* (%)	*PP* (%)	*Tensile strength* *MPs*(σ_b)	*Young's modulus* *MPs*(E_y)	*Elongation at break* (%)	*Flexural strength* *MPs*(σ_f)	*Flexural modulus* *MPs*(E_f)
85	0	15	38.7	986	22	64	1345
85	5	10	44.8	1123	69	68	1439
75	0	25	36.5	941	39	61	1269
75	5	20	42.7	1049	78	66	1379
65	0	35	35.2	878	43	58	1178
65	2.5	32.5	38.9	997	69	63	1231
65	5	30	41.6	1104	73	65	1276
65	10	25	45.8	1189	95	70.2	1346
65	20	15	46.2	1203	97	69.8	1327
55	0	45	35.7	876	40	52	1092
55	5	40	39.7	1034	78	60	1199

Rheological Properties

Melt viscosities of blends were measured at 215°C with a cone-plate viscometer, Contraves Rheomat 120. The diameter of the cone-plate is 20 mm and cone angle is 0.5°. The thickness of the testing specimens obtained by using a molding method is about 0.2 mm. Experiments were carried out under nitrogen atmosphere, with shear rate $5\ S^{-1}$.

Measurements of Mechanical Properties

Measurements of tensile properties of specimens were performed by means of INSTRON 1121 on ISO/R527-1966E. Measurements of flexural modulus and strength were also performed with INSTRON 1121 on the basis of ISO178-1975E. Izod impact strength were carried out with XJU-22 impact testing machine on basis of ISO/R180-1961. Dried specimens were prepared in a vacuum oven at 80°C for 18 hours.

Morphological Observation

A scanning electron microscope, JXA-840 was used to observe the morphologies of the blends. Before observation, the examined sections were etched with xylene for 8 hours to increase the contrast.

RESULTS AND DISCUSSION

Thermal and Rheological Properties

Thermograms of PA1010/PP and PA1010/PP-g-A-ATBS/PP blends after extracted with xylene were shown in Figure 1. In the thermogram(a) which corresponds to the extracted residue of PA1010/PP, only one peak of fusion at nearly 199.7°C appeared. This peak can be attributed to PA1010, since PP phase was extracted by xylene from the PA1010/PP blend. However, three peaks can be observed in thermogram(b) of the extracted residue of PA1010/PP-g-ATBS/PP. The peak at 199.7°C is attributed to PA1010. The other peaks at 175°C and 160°C can be considered as contributions of crystals of propylene and ethylene sequences of PP. This feature suggested that some chemical reactions

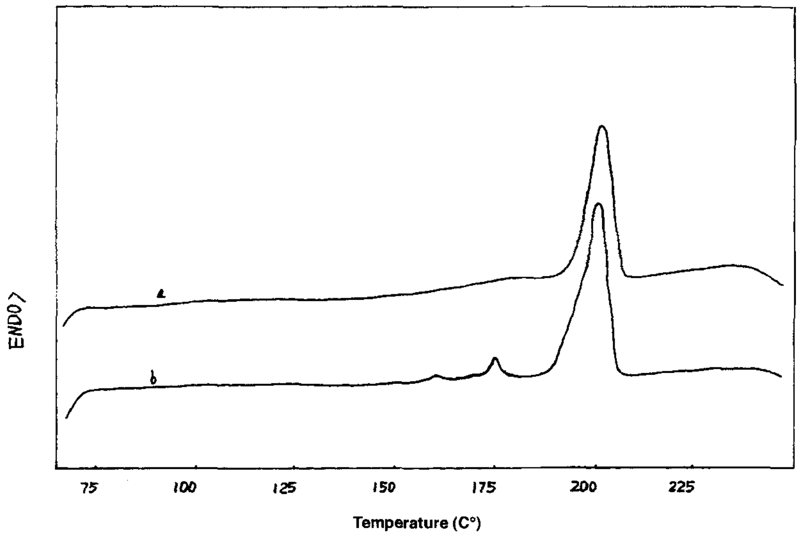
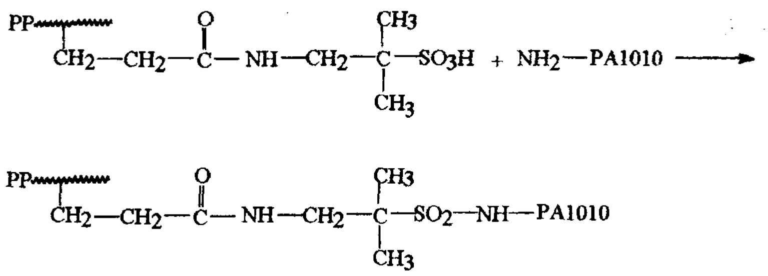


FIGURE 1 The insoluble residue thermograms of PA1010/PP (a) and PA1010/PP-g-ATBS/PP (b) blends after extracted with xylene.

occurred between ATBS in PP-g-ATBS blend and terminal amino groups of PA1010 in the PA1010/PP-g-ATBS/PP blend as shown as follows:



$$\mathrm{PP}\sim\sim\mathrm{CH_2{-}CH_2{-}\overset{\displaystyle O}{\overset{\|}{C}}{-}NH{-}CH_2{-}\underset{\displaystyle CH_3}{\overset{\displaystyle CH_3}{C}}{-}SO_3H} + \mathrm{NH_2{-}PA1010} \longrightarrow$$

$$\mathrm{PP}\sim\sim\mathrm{CH_2{-}CH_2{-}\overset{\displaystyle O}{\overset{\|}{C}}{-}NH{-}CH_2{-}\underset{\displaystyle CH_3}{\overset{\displaystyle CH_3}{C}}{-}SO_2{-}NH{-}PA1010}$$

The coupling reaction between two components made it impossible to extract all the PP phase from the ternary blend by using xylene.

The possible coupling interaction between PA1010 and PP-g-ATBS was confirmed again by comparing their apparent viscosity of binary and ternary blends. An increase of apparent viscosity of ternary blend could be attributed to the enhancement of molecular weight of blending components [12–13]. The effective of compositions on apparent viscosities of PA1010/PP and PA1010/PP-g-ATBS/PP blends was shown in Figure 2. An obvious increase is viscosity at same shear rate was observed for PA1010/PP and PA1010/PP-g-ATBS/PP blends. For example, the apparent viscosity of binary blend is 150 Pa.s lower than that of ternary blend. This result indicated that the molecular weight of ternary blend is higher than that of binary blend, which is induced by coupling reactions happened between sulfonic acid groups of PP-g-ATBS and terminal amino group of PA1010.

Morphology

Figure 3 shows SEM micrographs of PA1010/PP and PA1010/PP-g-ATBS/PP fracture surface samples etched with xylene. The matrix is PA1010 and the dispersed phase were PP domains. Several features can be observed from these pictures: (1) sizes of PP domains in ternary blends

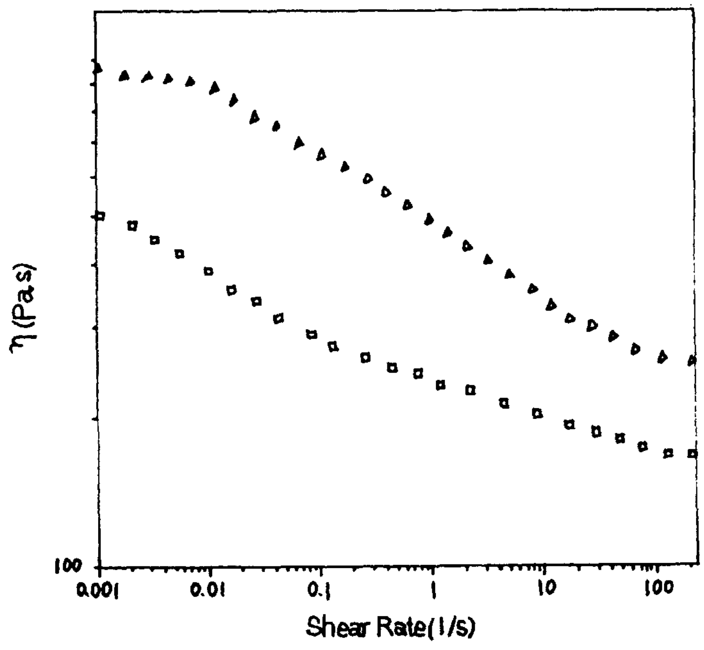


FIGURE 2 The apparent viscosities of PA1010/PP (□) and PA1010/PP-g-ATBS/PP (Δ).

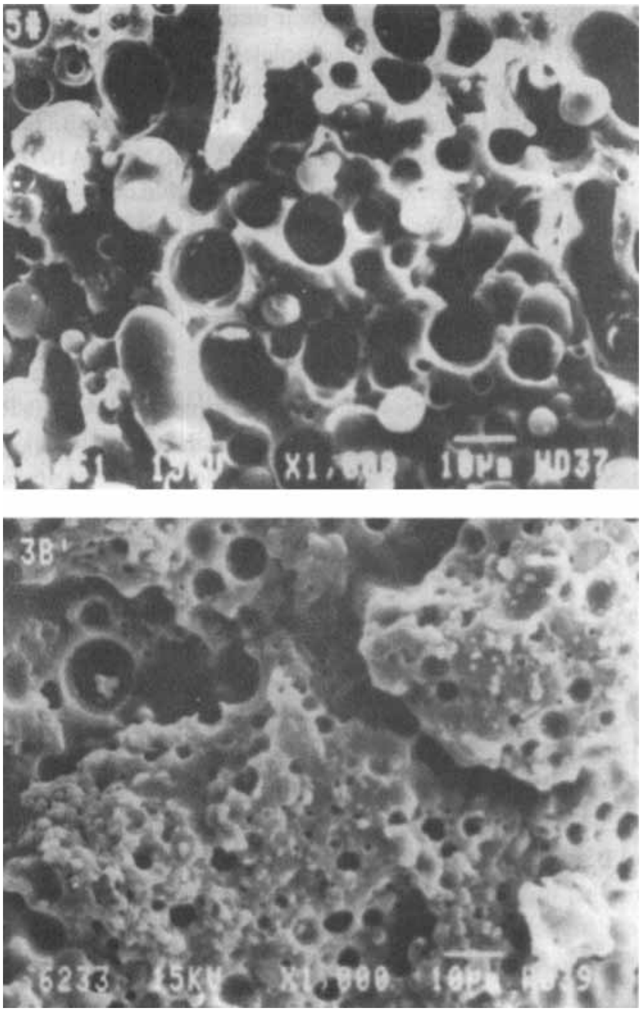


FIGURE 3 SEM microsgraphs of PA1010/PP (a) and PA1010/PP-g-ATBS/PP (b) blends etched with xylene.

are much smaller than that of PP in binary blends. As shown in Figure 3a, diameters of holes left by PP domains in binary blends are about 5 ~ 10 μm, and related values of PP domains in ternary blends are about 2 ~ 7 μm. (2) Homogeneous dispersion of PP domains in ternary blends can be observed in Figure 3b, (3) Interfaces between PA1010 matrix and PP domains are much clear and smooth for binary blends. This indicated that PP domains completely ripped off during etching with xylene. However, interfaces between PA1010 matrix and PP domains in ternary blends are rough and not so smooth. The above results strongly suggest that miscibility of PA1010 and PA1010 and PP phases in ternary blends was improved by possible coupling reactions between sulfonic acid group of PP-g-ATBS and terminal amino groups PA1010.

Mechanical Properties

Main mechanical properties of dried sample of PA1010/PP and PA1010/PP-g-ATBS/PP blends, such as tensile strength (σ_b), young's modulus (E_y), elongation at break (%) m flexural strength (σ_f) and flexural modulus (E_f) are listed Table I. It can be note that when the contents of PA1010 are same, the tensile strength values of PA1010/PP-g-ATBS/PP were 10–30% higher than that of PA1010/PP. Similar results are also observed for other parameters. This features can be tentatively explained as the improvement of interfacial adhesion and homogeneous dispersion of PP domains in ternary blends, which is due to inter-coupling reactions between PP-g-ATBS and PA1010 components. Izod impact strength of dried specimens of PA1010/PP and PA1010/PP-g-GMA/PP blends with different compositions ar shown in Figure 4. They increased monotonically with the addition of PP or PP-g-ATBS. It suggested that both PP and PP-g-ATBS materials are good toughening agents for PA1010. The more content PP or PP-g-ATBS, the higher the Izod impact strength is. However, the toughening effect of PP-g-ATBS is much better than PP. For example, values of PA1010/PP-g-ATBS/PP blends is about 10 J/m higher than that of PA1010/PP blends. This results can be also attributed to the homogeneous morphological dispersion and improved interfacial adhesion of PA1010/PP-g-ATBS/PP blends.

CONCLUSIONS

1. Coupling reactions happened between sulfonic acid group of PP-g-ATBS and amino group of PA1010, which could be verified by the

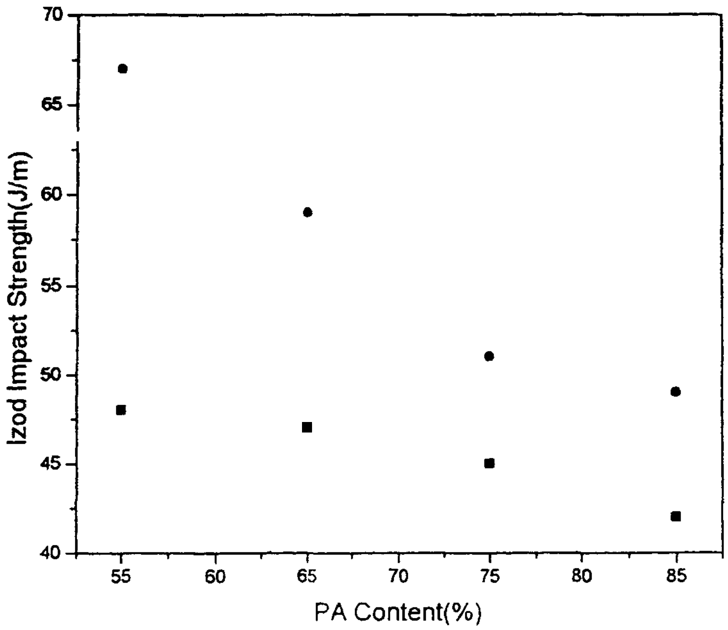


FIGURE 4 The relationship of Izod Impact Strength versus PA content of PA1010/PP (■) and PA1010/PP-g-ATBS (5 wt% content)/PP (●) blends.

appearance of melt peaks of polypropylene in PA1010/PP-g-ATBS/PP blends extracted with xylene and the enhancement of apparent viscosities of PA1010/PP-g-ATBS/PP.

2. As contrasted with morphologies of PA1010/PP and PA1010/PP-g-ATBS/PP blends, more smaller and homogeneous dispersion PP domains in PA1010/PP-g-ATBS/PP blends were formed.
3. Mechanical properties of PA1010/PP-g-ATBS/PP blends are superior to PA1010/PP blends. This can be attributed to the homogeneous morphological dispersion and the improved interfacial adhesion between PA1010 and PP in PA1010/PP-g-ATBS/PP blends.

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