

The Effect of Graft Copolymers of Maleic Anhydride and Epoxy Resin on the Mechanical Properties and Morphology of PP/ABS Blends

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ABSTRACT: In this work, maleic anhydride-grafted polypropylene (PP-g-MAH) and maleic anhydride-grafted poly(acrylonitrile-butadiene-styrene) (ABS-g-MAH) at 2 : 1 mass ratio were added as a compatibilizer in the PP/ABS blends. The compatibilizing effect was evaluated by adding the graft copolymers together with epoxy resin/imidazole curing agent (E51/2E4MZ). The reaction in reactive extrusion, morphological structure, and properties of PP and ABS blends were investigated by using infrared spectroscopy (FTIR), scanning electron microscopy (SEM), and X-ray spectrum, transmission electron microscope (TEM), dynamic thermomechanical analysis (DMA), differential scanning calorimetry (DSC), and mechanical properties tests. The results showed that the compatibilizing effect was greatly improved because of the addition of the graft copolymers together with epoxy resin/imidazole curing agent (E51/2E4MZ) because the link structure of PP-g-MAH and ABS-g-MAH was formed by the reaction of anhydride group with epoxy group catalyzed by the imidazole. The size of the dispersed phase decreased dramatically, the interfacial adhesion between ABS particles and PP matrix was improved, and the tensile strength and flexural modulus of the PP/ABS blends increased further. The optimizing properties were obtained at 3 phr E51/2E4MZ. © 2014 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* **2014**, *131*, 40898.

KEYWORDS: applications; blends; compatibilization

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INTRODUCTION

Polypropylene (PP) and poly(acrylonitrile-butadiene-styrene) (ABS) as two kinds of polymer materials have been widely used in many industrial sectors. PP's Vicat softening point is about 120–140°C, with remarkable thermal resistance. However, its flexural modulus is about 0.9–1.2 GPa, with low stiffness. Flexural modulus of ABS is higher than that of PP, about 2–2.5 GPa, with a certain ductility but poor thermal resistance (ABS' Vicat softening point is only about 90°C). The blends of PP with ABS could reduce their respective drawbacks and obtain a better comprehensive performance. The two polymers has bad compatibility.¹ The values of solubility parameter of PP and ABS have great difference. In addition, the compatibility of PP and ABS is further deteriorated due to the crystallization of PP, and the crystallization process will induce phase separation. It presents large particles of dispersed phase in the blend, as well as fragile interface and poor mechanical properties.^{2–4} A preparation of PP/ABS blend has always been difficulty to obtain good comprehensive properties. The most important route to enhance the compatibility between the two polymers is via the introduction of specific interactions between them. The polar

group of the graft copolymers is commonly used to improve the components compatibility by reducing the interfacial tension, which contributes to obtain smaller dispersed phase particle size and improve interfacial adhesion.^{5–7} Strength and modulus of the blend are improved by adding the rigid graft copolymers such as anhydride-grafted PP (PP-g-MAH),² PP-g-acrylic acid,⁵ and PP-g-2-HEMA⁶ etc. ABS produces toughening effect when the content is more than 80% in the blends.^{3,5} The toughness of the blend increases with the addition of soft graft copolymers such as styrene-butadiene-grafted-maleic anhydride copolymer⁸ because the good bonding soft interface can produce a certain stress relaxation, on the contrary, the strength, and rigidity of the material is decreased. When the blends of PP with ABS are modified by the inorganic nanoparticles, the strength and stiffness of blends increase, but toughness decreases.⁹ It is greatly significant to search new approaches for further improving the compatibility of the blends to obtain good comprehensive performances.

Modifying thermosetting resin by using thermoplastic resin to produce a good balance of strength and toughness has been normally described^{10–12} and the effect of epoxy resin improving

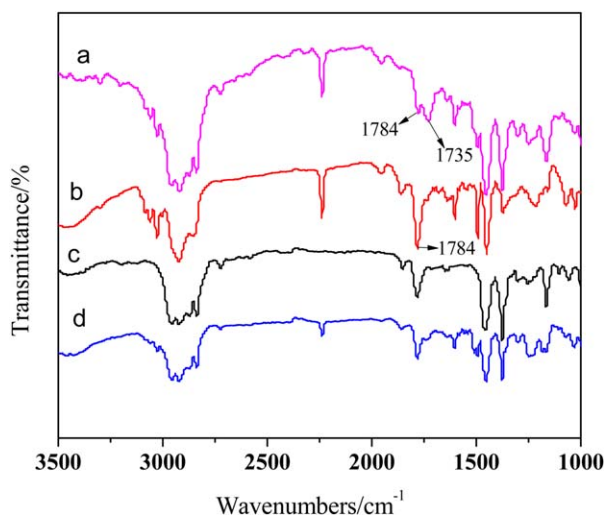


Figure 1. FTIR spectra: a. PP/ABS/M/ E51/2E4M, b. PP/ABS/M/E51, c. PP-g-MAH, and d. ABS-g-MAH. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

compatibilization of PP/inorganic fillers or PP/rubber was reported as well.^{13–17} As far as we known, the comprehensive compatibilizing effect of graft copolymers and epoxy resin on PP/ABS blend has not been reported. The epoxy group in epoxy resin molecule has high reaction activity with polar group of PP-g-MAH and ABS-g-MAH to form link structure, which may improve the compatibility of PP with ABS to increase the properties of the blend.

In this article, PP-g-MAH and ABS-g-MAH were incorporated in PP and ABS blend, as well as epoxy resin and imidazole. PP-g-MAH and ABS-g-MAH were linked by the reaction of epoxy groups with anhydride groups in twin-screw extruder blending process catalyzed by the imidazole.^{18,19} The compatibilizing effect by this method was enhanced as compared with the pure graft compatibilizer, and the mechanical properties were improved.

EXPERIMENTAL

Materials

PP (K9026, copolypropylene) was produced by Lanzhou Petrochemical, branch of China National Petroleum Corporation. ABS with 18.7% contents of butadiene (ABS, 747S) was produced by Chimei Industrial, Taiwan. PP-g-MAH (CM9801) with 1 wt % grafting degree was supplied by Rizhisheng New Technology Development, China. ABS-g-MAH (OY-02) with 1.5 wt % grafting degree was supplied by Cixi Ouyuan Trade, China.

The bisphenol A type epoxy resin (E51), with epoxy value 0.51, was produced by Shanghai resin Production, China. The imidazole fast medium temperature curing agent (2-ethyl-4-methyl-imidazole, 2E4MZ) was purchased from Shikoku Kasei, Japan.

The Preparation of the PP/ABS Blends

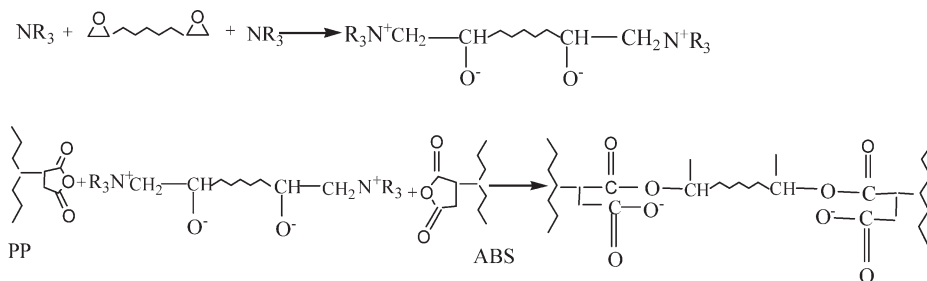
The mass ratios of PP/ABS were 90/10, 80/20, 70/30, 60/40, and 50/50, and the mass ratio of PP-g-MAH and ABS-g-MAH was kept constant in 2 : 1. The mixture of the MAH graft copolymers is named M in the following content. Different contents of M (0, 1, 3, and 5 phr) were added (number of mass parts per 100 mass parts of PP and ABS). Then, on the basis of above prescriptions, different contents of EP (E51) (0, 1, 3, and 5 phr) were added to investigate the comprehensive compatibilizing. The content of 2E4MZ (2-ethyl-4-methyl-imidazole, curing agent) was based on the EP content (4/100). The complexes of EP (E51) and 2E4MZ is named E in the following content. The PP and ABS were dried for 5 h at 90°C, and all the components were weighed carefully according the recipes, stirred in a blender for 3 min, extruded and granulated in a twin-screw extruder (TSE-40A, Ruiya, China). The barrel temperatures ranged from 175 to 220°C. Specimens for mechanical properties test were prepared by injection molding machine (CJ80m3V, Zhengde, China) at barrel temperatures between 190 and 230°C.

Characterization and Measurements

Mechanical Properties Tests. Tensile properties were measured using a material testing machine (WdW-10C, Shanghai Hualong Instrument Company, China) at a crosshead rate of 50 mm/min according to the ISO527-1993 standard. Flexural properties were determined on the same testing machine at crosshead displacement rate of 2 mm/min according to ISO178-2003 standard. Notched impact test was performed on an Izod impact experiments according to ISO180-2000 standard. The notched depth is 2 mm and notch tip radius is 0.25 mm. All the values were calculated as averages from five samples at least.

Infrared Spectroscopy. Fourier transform infrared (FTIR) spectrometer (Nexus 670, Nicolet, USA) was used to characterize the chemical absorption of the blends. The powders of the blends mixed with potassium bromide (KBr) were compressed into plates for FTIR spectra analysis. The samples were analyzed in the spectral range of 4000–400 cm^{-1} at room temperature.

Scanning Electron Microscopy (SEM). The samples were prepared by immersing in liquid nitrogen for 3 h until they occur brittle fracturing, and then they were covered with a layer of gold by sputtering in vacuum conditions to provide an electrical conduction and reduce the surface charging prior to SEM



Scheme 1. The accelerated effect of tertiary amine of ZE4MZ.

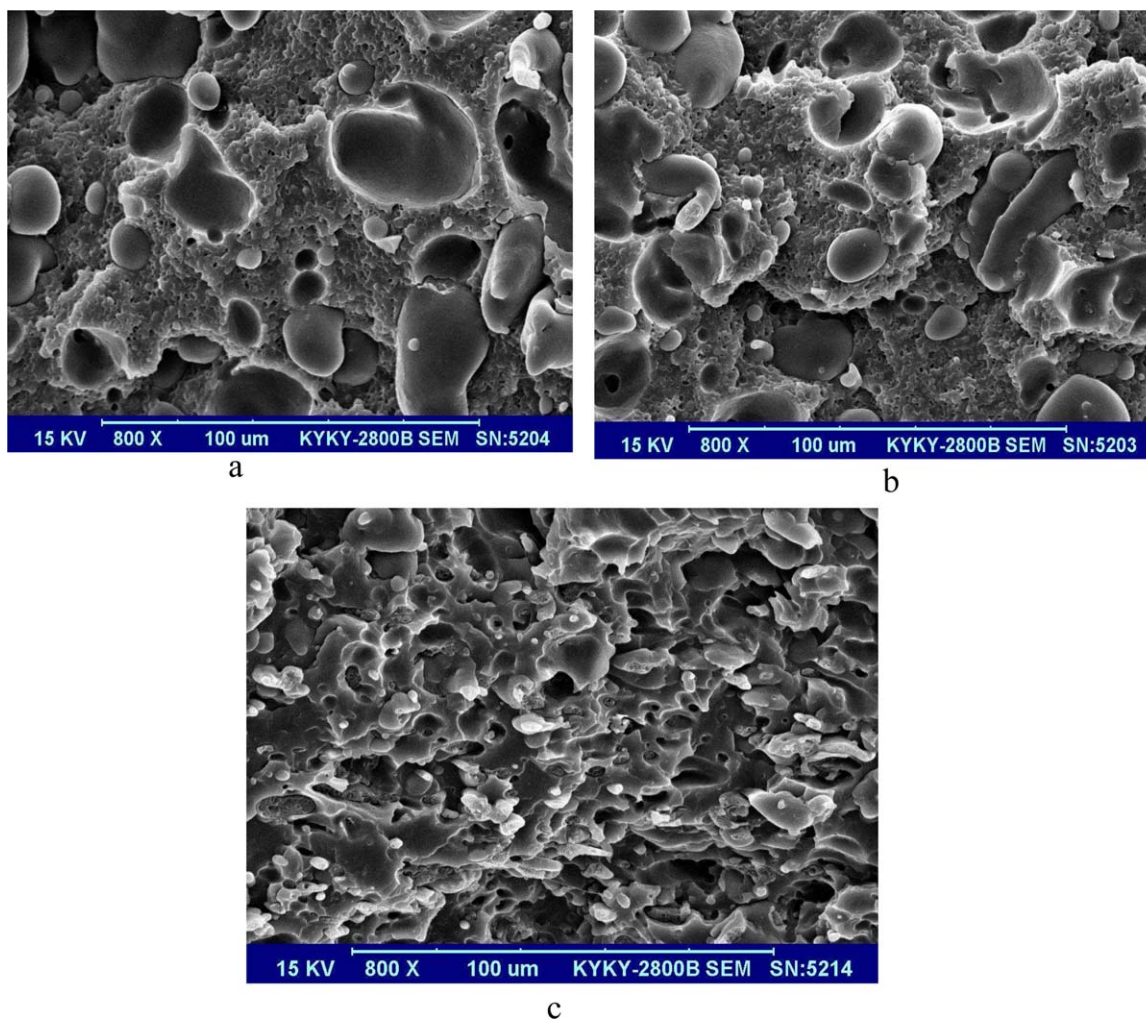


Figure 2. SEM photographs of PP/ABS blends: a. PP/ABS(70/30), b. PP/ABS/M (70/30/5), and c. PP/ABS/M/E(70/30/5/3). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

examination. The samples were observed by SEM (KYKY-AMRAY-2800, KYKY Technology Development, China) under the condition of 25 kV.

X-Ray Spectrum. With the help of (JSM-6490LV) scanning electron microscope and INCA-350 X-ray spectrometer (JEOL, Japan), various elements content in the samples were determined. Samples were covered with a layer of carbon by sputtering in vacuum conditions. The acceleration voltage was 20 kV.

Transmission Electron Microscopy (TEM). The ultramicrotomy of samples cut by freezing (about 150 nm), fumigated with osmium tetroxide staining in order to distinguish the ABS phase in deepen color, were observed with a TEM (JEM200CX, JEOL, Japan) at an accelerating voltage of 100 kV.

Dynamic Thermomechanical Analysis (DMA). Dynamic mechanical properties were valued by using a dynamic mechanical properties testing machine (Q800, TA, USA) to determine the glass transition temperature (T_g) in oscillatory torsional mode at 0.1% strain and 1 Hz frequency. Test data were collected from -50 to 150°C at a scanning rate of $5^\circ\text{C}/\text{min}$. Nitro-

gen gas was circulated in the environmental chamber to minimize degradation during testing.

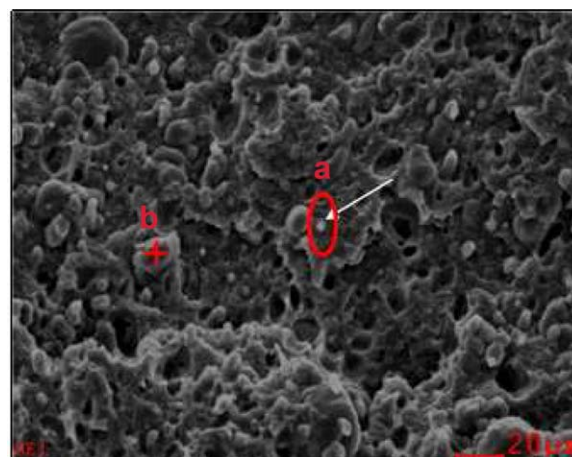


Figure 3. X-ray spectra probing sites in SEM photos of PP/ABS/M/E (70/30/5/3): a. on the tiny particle, b. on the ABS dispersed phase. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Table I. Content Percentage of Various Elements From X-Ray Spectra [PP/ABS/M/E(70/30/5/3)]

Element	Spectrum (a) (wt %)	Spectrum (b) (wt %)
C	80.82	92.53
N	12.41	4.45
O	6.67	3.02

Crystallization Behavior. Crystallization behavior of the sample was carried out in a differential scanning calorimeter (DSC) (Q10, TA, USA) under a nitrogen atmosphere (40 mL/min). Samples were taken about 5 mg and put in the aluminum pan. The samples were heated to 230°C with a heating rate of 10°C/min and kept 5 min and then cooled to room temperature at the rate of 10°C/min. Exothermal peak was calculated from the cooling curve of the experiment. The degree of crystallinity (X_c) of PP in the blends was corrected by the fraction of PP in each sample.

RESULTS AND DISCUSSION

FTIR Analysis

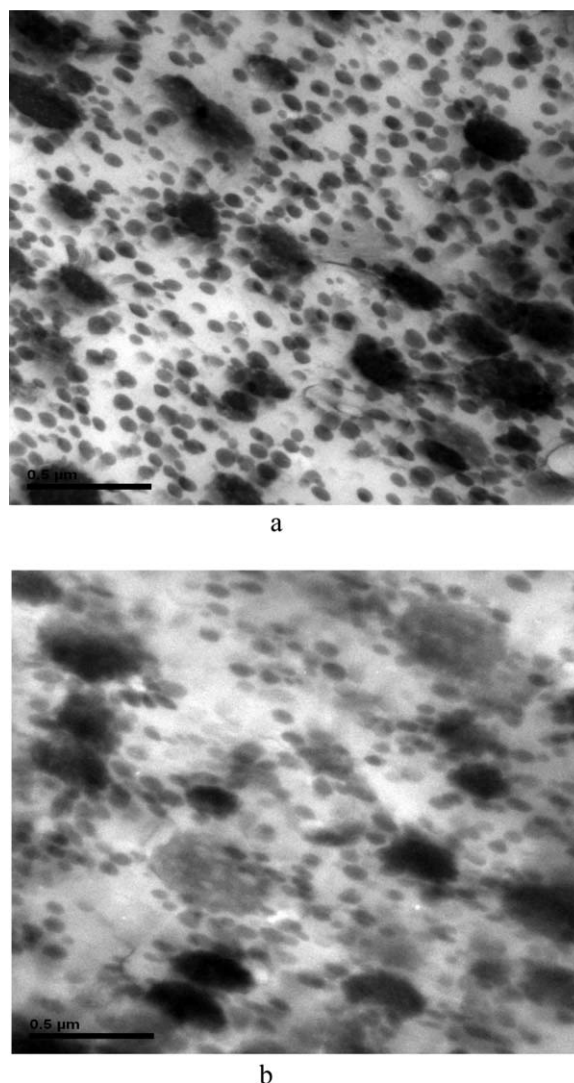
Epoxy groups present in epoxy resin are capable of strong specific interaction with various polar groups, for example, epoxidized soybean oil (ESO) was reacted with monomethyl maleate with AMC-2 catalyst to give the monomethyl maleic esters.²⁰ The product may give some special compatibilizing effect to improving properties of blends.

Because the contents of M and E were low in PP/ABS/M/E blends, and the infrared spectrum absorption peaks belonging to the reaction product would be diluted, the samples for infrared spectrum only contained PP-MAH, ABS-g-MAH, E51, and 2E4MZ. The mixture of PP-MAH and ABS-g-MAH at the mass ratio of 2 : 1 (M) with 3 phr E (E51/2E4MZ) was mixed in the twin-screw extruder. Another blends of PP-MAH/ABS-g-MAH at the mass ratio of 2 : 1 with EP but without 2E4MZ was prepared in the same way. Figure 1 shows FTIR spectra of the PP-MAH, ABS-g-MAH, and the above two blends. Absorption peak around 1784 cm^{-1} appeared, which corresponded to the conjugated carbonyl groups of MAH, and was in good agreement with the study of the Refs. 21,22. For the sample containing M and E, the absorption peak around 1735 cm^{-1} as a consequence of the formation of ester carbonyl group appeared. As shown in Figure 1(b), the absorption peak of ester carbonyl group (around 1735 cm^{-1}) was not been found in the FTIR spectra of the blend with E51 but without 2E4MZ, it could be concluded that reaction took place between epoxy group and anhydride group to form ester during the extrusion process catalyzed by the imidazole, which produced one kind of linked structure between PP-g-MAH and ABS-g-MAH (PP-g-MAH-EP-MAH-g-ABS). The accelerated effect of tertiary amine of 2E4MZ on the reaction can be described as Scheme 1.

SEM and X-Ray Spectrum Analysis

Figure 2 shows SEM images of the fracture surface of the samples. The mass ratios of PP/ABS, PP/ABS/M, and PP/ABS/M/E were 70/30, 70/30/5, and 70/30/5/3, respectively. It was shown

from Figure 2(a) that the poor compatibility between PP and ABS in blend of PP/ABS (70 : 30) resulted in large particles of ABS phase in PP matrix and smooth surface of the particles. The image of PP/ABS/M = 70 : 30 : 5 [as shown in Figure 2(b)] showed that the poor interfacial adhesion was still obvious though the dispersed phase particles were refined. This indicated that the addition of M produced certain compatibilizing effect; however, the effect was insufficient for the two poor compatibility components of PP and ABS. As shown in Figure 2(c), when 3 phr E was added, it was found that the dispersed phase particle diameter was dropped drastically and the particle surface became rough (the surface roughness of particles is usually expected in case of good interfacial adhesion between the particles and the matrix), which showed the interfacial adhesion was improved. Other kind of very tiny particles, whose morphology was different from the dispersed phase particles, were founded in SEM photos of PP/ABS/M/E (70/30/5/3) blend and analyzed by X-ray spectrum. The probing sites were on the tiny particle and the ABS dispersed phase (Figure 3). The test data was listed in Table I. It was clearly seen that the contents of

**Figure 4.** TEM photographs of PP/ABS blends: a. PP/ABS/M (60/40/5), b. PP/ABS/M/E (60/40/5/3).

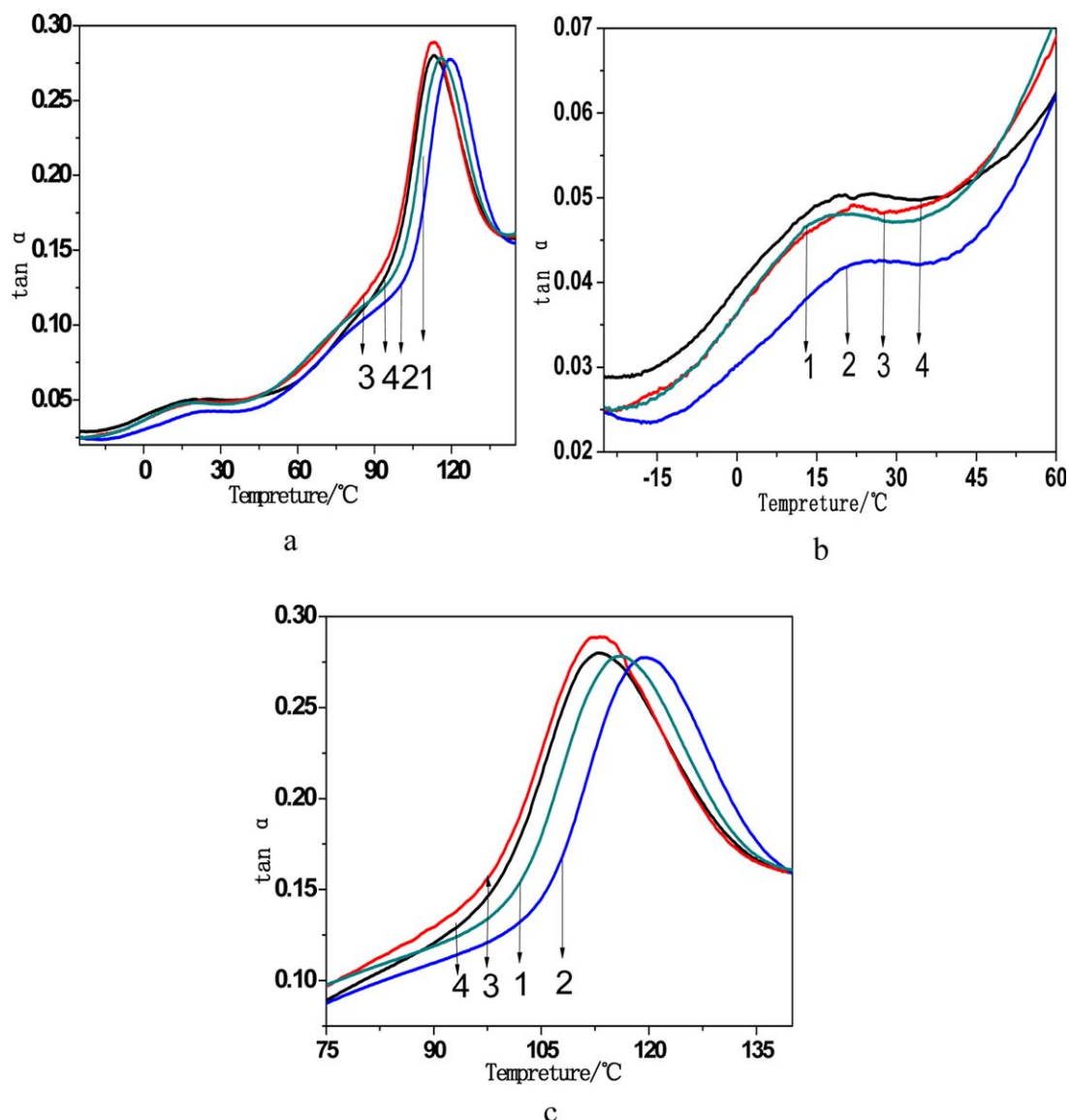


Figure 5. Variation of loss tangent ($\tan\delta$) with temperature [1- PP/ABS (70/30), 2-PP/ABS/M (70/30/5), 3- PP/ABS/M/E (70/30/5/3), 4- PP/ABS/M/E (70/30/5/5)]; a. plots of loss factor, b. glass transition of PP, and c. glass transition of ABS. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

oxygen and nitrogen in the tiny particle were evidently higher than that in the ABS dispersed phase. 2-ethyl-4-methyl-imidazole (2E4MZ) and ABS contain nitrogen element in the blend, and the nitrogen content of 2E4MZ is higher than that in ABS. On the other hand, epoxy resin and the MAH graft copolymers contain oxygen element in the blend, and the oxygen content of the graft

Table II. The Glass Transition Temperature of the Blends

Samples	T_g		ΔT_g
	PP	ABS	
PP/ABS (70/30)	17.5	116.2	98.7
PP/ABS/M (70/30/5)	23.5	119.0	95.5
PP/ABS/M/E (70/30/5/3)	19.7	113.0	93.3
PP/ABS/M/E (70/30/5/5)	19.2	113.5	94.3

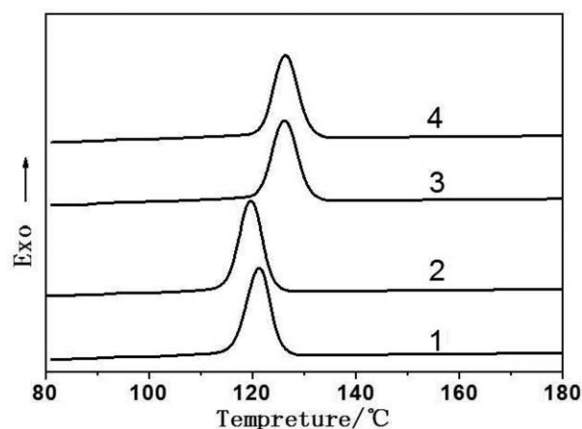


Figure 6. DSC cooling curves of the blends: 1. PP/ABS (70/30), 2. PP/ABS/M (70/30/5), 3. PP/ABS/M/E (70/30/5/3), and 4. PP/ABS/M/E (70/30/5/5).

Table III. The Crystallization Peak Temperature and Degree of Crystallinity of PP in the Blends

Samples	Crystallization peak temperature (°C)	Degree of crystallinity (%)
PP/ABS (70/30)	121.5	41.5
PP/ABS/M (70/30/5)	120.5	42.4
PP/ABS/M/E(70/30/5/3)	127.2	43.9
PP/ABS/M/E(70/30/5/5)	127.3	43.6

copolymers is little because of the low grafting degree. It could be inferred that some of these tiny particles were the aggregates of epoxy resin and imidazole curing agent. Therefore, a part of the epoxy resin was cured during extrusion process and formed tiny particles in matrix, which reduced the plasticizing effect of the addition of liquid epoxy resin.

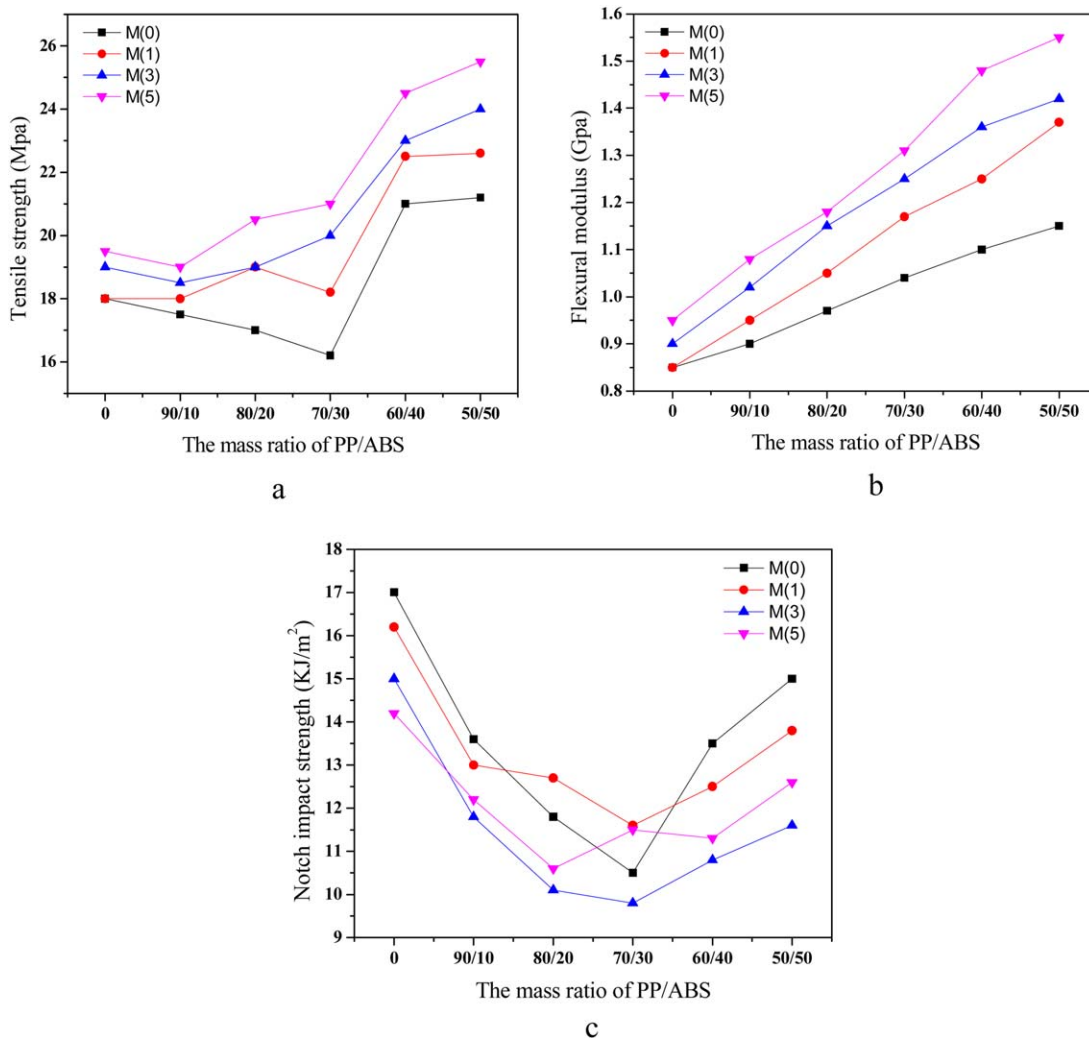
TEM Analysis

The TEM images of PP/ABS/M (60/40/5) and PP/ABS/M/E (60/40/5/3) blends were presented in Figure 4. The dark region was the microdomain of stained polybutadiene (PB) in ABS dispersed

phase. As shown in Figure 4(b), PB phase interface became blurred and the parcel structure appeared with the incorporation of E. In ABS copolymer, polystyrene and acrylonitrile (SAN), whose solubility parameter is about 18–20 $(\text{J}/\text{cm}^3)^{1/2}$,²³ formed continuous phase, and polybutadiene, whose solubility parameter is about 16.65 $(\text{J}/\text{cm}^3)^{1/2}$,²⁴ formed dispersed phase. PP, with about 16.77 $(\text{J}/\text{cm}^3)^{1/2}$ solubility parameter,²⁵ is most likely to have better compatibility with PB in the PP/ABS blend. Additionally, for ABS-g-MAH, all the anhydride groups were on the PB chains.²⁶ So, the PP-g-MAH molecular chains, which were linked with ABS-g-MAH by EP in the reactive extrusion would distributed in the boundary of PP and PB or entered into PB microdomain, which decreasing darkness of the stained PB phase and forming parcel structure. Comparing Figure 4(a,b), when E was added, the morphology of PB microdomain changed from uniform distribution to a certain degree of uneven distribution, which was related to the distribution of epoxy resin in matrix.

DMA Analysis

The curves of loss tangent ($\tan\delta$) versus temperature of several samples are shown in Figure 5. The transition around 20°C

**Figure 7.** Mechanical properties of PP/ABS/M as a function of the content of ABS: a. tensile strength, b. flexural modulus, and c. notched Izod impact strength. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

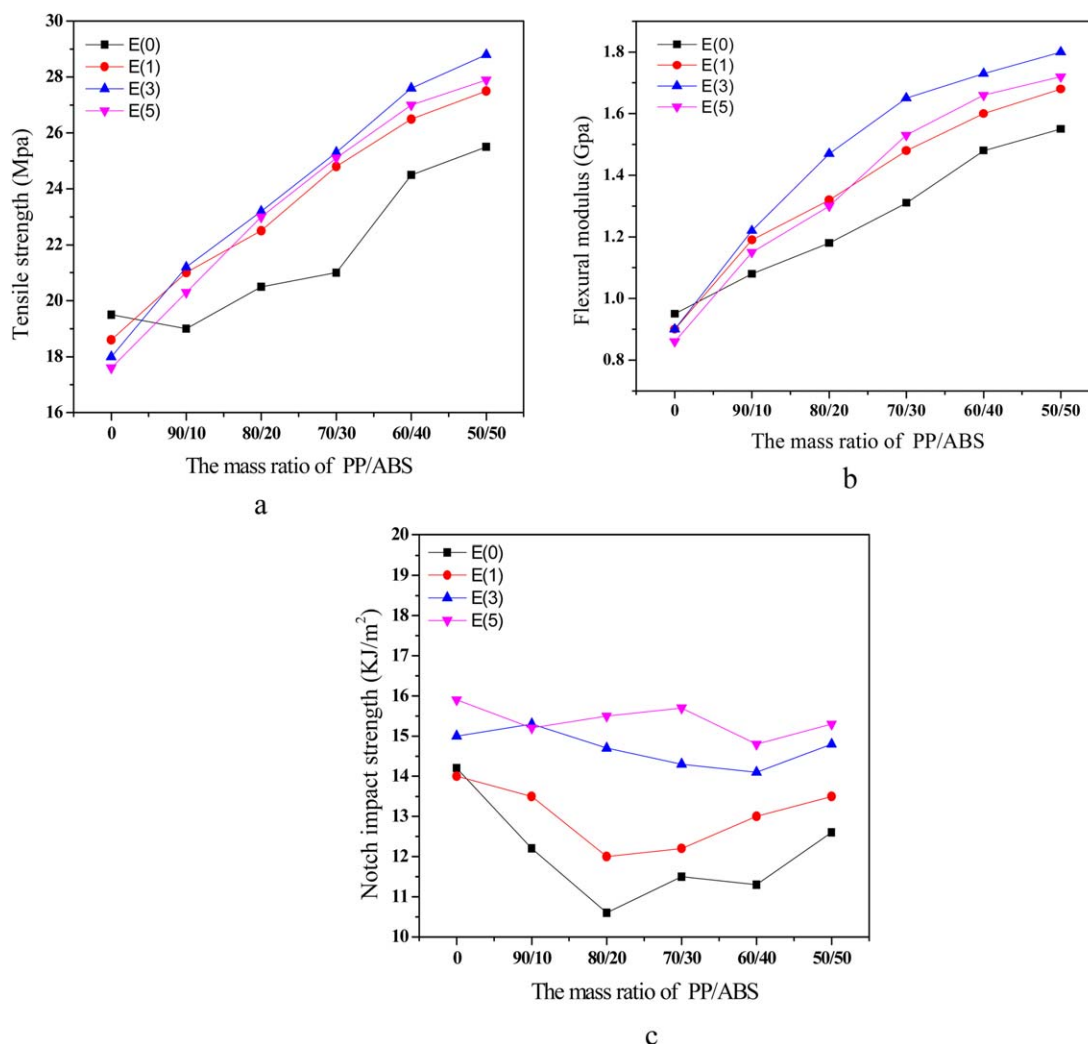


Figure 8. Mechanical properties of PP/ABS/M/E as a function of the content of ABS: a. tensile strength, b. flexural modulus, and c. notched Izod impact strength. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

appeared, corresponding to the glass transition temperature of partially crystalline PP, which is similar with other results.²⁷ The highest peak around 115°C in the curves corresponds to the glass transition temperature of ABS.²⁸ Two glass transition temperatures and the difference for each curve were listed in Table II, which indicated the change of intermolecular force and compatibility.

When M was added in PP/ABS/M blend, it was seen from Table II that the values of two glass transition temperatures increased, which related to the higher intermolecular force of the polar graft copolymers, but the difference between the two temperatures was not obviously reduced. As the result, compatibility was not greatly improved, which is in good agreement with the above SEM photos. Compared with PP/ABS/M (70/30/5), when 3 phr E was added in the PP/ABS/M/E (70/30/5/3) blend, the glass transition temperatures of ABS and PP decreased, and the ΔT_g (difference of glass transition temperatures of PP and ABS in the blend) was reduced by 5.4°C, i.e., the two glass transition temperatures closed to each other, which indicated the synergistic compatibilizing effect of M and E through reactive extrusion was more efficient. According to our research, epoxy resin could

not be cured completely for about 3 min in a twin-screw extruder, so the decrease of the glass transition temperatures can be attributed to the plasticizing effect of the part of uncured epoxy resin. However, with increasing the epoxy resin content further, the compatibilizing effect decreased. This phenomenon would be due to epoxy resin tends to aggregate within the matrix with increasing content.

DSC Analysis

The DSC cooling curves were shown in Figure 6. The degree of crystallinity corrected with the mass fraction of PP in the blends and the crystallization peak temperature were listed in Table III. Compared with PP/ABS (70/30) blend, the degree of crystallinity of PP slightly increased because of the addition of M in the PP/ABS/M (70/30/5) blend. The main reason may be the molecular weight of PP-g-MAH is lower than that of PP (K9026), which makes the ordered arrangement of macromolecules more easily in the crystallization process. On the other hand, the crystallization behavior was influenced by the epoxy resin. It is noted that the degree of crystallinity and the crystallization peak temperature of PP increased with the addition of epoxy

resin. As shown in Figure 3, epoxy resin was cured and formed those diffuse distribution tiny particles in matrix. The increase of the degree of crystallinity and crystallization peak temperature was due to the effect of heterogeneous nucleation. Jiang et al.¹⁴ have reported that epoxy particles in the PP/epoxy blends acted as effective nucleating agents, and accelerated the crystallization of the PP component in the PP/epoxy blends.

Mechanical Properties

The tensile strength, flexural modulus, and notched Izod impact strength of the PP/ABS/M blends with different contents of ABS and M were shown in Figure 7. The mass ratio of PP/ABS had a great effect on the mechanical properties of the blends. From Figure 7(a), except the tensile strength at 90/10 (PP/ABS), tensile strength of the blends increased with the increase of ABS content when the content of M was more than 3 phr. The curves of Figure 7(b) showed that the flexural modulus increased monotonically with increasing ABS content. Under the condition of the same mass ratio of PP/ABS, properties of the blends increased with increasing M content because the MAH graft copolymers played a role in increasing compatibility of PP and ABS. The tensile strength and flexural modulus of the blend with 50 : 50 mass ratio of PP/ABS and with 5 phr M were 25.5 Mpa and 1.55 Gpa, respectively, increased by 20% and 35% as compared with the blend without M. However, from Figure 7(c), the impact strength of the material was not improved with increasing ABS content, it could be seen that the impact strength went first sharply drops as ABS content increased from 0 to 30 phr, and then increased a little for further increasing ABS content. The effect of addition of M and its content on impact strength did not show obvious regularity. It is similar to the experimental results in Ref. 2 that the impact strength did not be obviously improved by adding PP-g-MAH.

On the basis of 5 phr M, E was added in the blends. The tensile strength, flexural modulus, and notched Izod impact strength of PP/ABS/M/E blends with different amounts of E (0–5 phr) and ABS content were shown in Figure 8. The mechanical properties of materials were further improved because of the addition of E and obtained optimum performance when the content of E was 3 phr. The tensile strength and flexural modulus of PP/ABS/M/E blend with 5 phr M and 3 phr E were 28.8 Mpa and 1.80 Gpa, respectively. The tensile strength and flexural modulus increased by 36 and 56% as compared with the blend without E and M, respectively. However, with increasing the content of E further, the mechanical properties of the blend decreased, which might be due to the decrease of the compatibilizing effect and the plasticizing effect of uncured epoxy resin. It was seen from Figure 8(c) that the impact strength increased with the content of E, and the impact strength changed little with the increase of ABS content when the content of E was more 3 phr. When compared with the blends without E, the impact strength was improved. Similar test results have been obtained in the same blend system that the toughness of PP/ABS blend could be obviously improved only when ABS content was more than 80%.^{3,5} The improvement of the mechanical properties of the material was mainly attributed to the compatibilizing effect of the link structure of PP-g-MAH and ABS-g-MAH. The result of DSC showed the degree of crystallinity of PP increased a little

due to the addition of E, which also was one of the reasons for an increase of strength and modulus.

CONCLUSION

The blends of PP and ABS were prepared in extrusion with a twin-screw extruder. Adding MAH graft copolymers alone could exhibit an extent of compatibilizing and made properties of the blends improved, but the size of the ABS dispersed phase did not obviously decrease, and the interfacial adhesion was still poor.

When introducing epoxy resin/imidazole (E51/2E4MZ, E) together with the graft copolymers, the PP-g-MAH-EP-g-ABS link structure was formed by the reaction of epoxy group with anhydride group in the process of reactive extrusion with imidazole catalyst. The particle size of the ABS dispersed phase was obviously reduced, interface became blurred and the interfacial adhesion was improved. The two glass transition temperatures of PP and ABS in the blend closed to each other, which indicated the link structures enhanced the compatibilizing effect of the MAH graft copolymers. The degree of crystallinity of PP increased a little due to the effect of heterogeneous nucleation produced by the dispersed tiny cured epoxy particles in matrix. The tensile strength and flexural modulus of the material increased further due to the combination of MAH graft copolymer and E. The notched impact strength of the blends containing E was enhanced as compared with the blends without E. The optimizing properties were obtained at 3 phr E. However, within the range of the formulation in this test, the impact toughness could not be improved with increasing the content of ABS.

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