Study of Dynamically Cured PP/MAH-*g*-PP/Talc/Epoxy Composites

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Received 17 September 2004; accepted 7 June 2005
DOI 10.1002/app.22967

ABSTRACT: In the present study, an epoxy resin was dynamically cured in a polypropylene (PP)/maleic anhydride–grafted PP (MAH-*g*-PP)/talc matrix to prepare dynamically cured PP/MAH-*g*-PP/talc/epoxy composites. An increase in the torque at equilibrium showed that epoxy resin in the PP/MAH-*g*-PP/talc composites had been cured by 2-ethylene-4-methane-imidazole. Scanning electron microscopy analysis showed that MAH-*g*-PP and an epoxy resin had effectively increased the interaction adhesion between PP and the talc in the PP/talc composites. Dynamic curing of the epoxy resin further increased the interaction adhesion. The dynamically cured PP/MAH-*g*-PP/talc/epoxy composites had higher crystallization peaks than did the PP/talc composites. Thermogravimetric analysis showed that the addition of MAH-*g*-PP and the epoxy resin into the PP/talc composites caused an obvious improvement in the thermal stability. The dynamically cured PP/MAH-*g*-PP/talc/epoxy composites had the best thermal stability of all the PP/talc composites. The PP/MAH-*g*-PP/talc/epoxy composites had better mechanical properties than did the PP/MAH-*g*-PP/talc composites, and the dynamically cured PP/MAH-*g*-PP/talc/epoxy composites had the best mechanical properties of all the PP/talc composites, which can be attributed to the better interaction adhesion between the PP and the talc. The suitable content of epoxy resin in the composites was about 5 wt %. J Appl Polym Sci 99: 2752–2758, 2006

Key words: polypropylene (PP); epoxy resin; talc; dynamic cure

INTRODUCTION

The addition of fillers to polymers is a fast and cheap method to modify the properties of the base materials.[1] In the last decade, polypropylene (PP) has been found to make interesting matrices for filled materials because of its combination of remarkable properties and low cost.[2–3] Of the fillers for PP, mica,[3–5] calcium carbonate,[2–3,6] and talc[7] are the most often used. Each filler brings its own characteristics to the PP and may also influence the crystallization of the PP and, as a consequence, the properties of the composites. As for talc, both modified[8,9] and unmodified PP/talc composites[10–12] have received attention.

The properties of the composites strongly depend on the interfacial adhesion between a filler and the polymer matrix. Good interfacial adhesion may improve the mechanical properties of the composites, whereas poor adhesion may decrease the mechanical properties.[13] To enhance the mechanical properties of the composites, coupling agents were used to modified the surface of the fillers. Ordinary coupling agents are silane and titanate coupling agents and other small molecular coupling agents. Another method often used is modification of the matrix. Grafting of different chemical molecules to a polyolefin chain is a frequently applied method for improving the compatibility of immiscible polymer blends and interfacial adhesion in composites. PP, a nonpolar polymer, is usually grafted with polar groups such as acrylic acid, maleic anhydride, and acrylic esters. Functionalized polyolefins have been used as a coupling agent in filler/polyolefin composites,[14–20] improving interaction adhesion between the matrix and the filler and enhancing the mechanical properties of the composites. Functionalized polyolefins can create strong bonds with the filler surface and form physical entanglements with the matrix. The effect of functionalized polyolefins on PP composites filled with mica[14–15] and calcium carbonate[16–18] and reinforced with glass fiber[19–21] have been investigated.

Our laboratory applied dynamic vulcanization to preparing a new type of polypropylene (PP)/epoxy blend.[22–24] The blends were prepared in the mixing chamber of a Haake RC90 rheometer by dynamic curing of an epoxy resin in molten PP. As the epoxy resin and PP were immiscible, maleic anhydride–grafted polypropylene (MAH-*g*-PP) was used as a compatibilizer. The dynamic curing of the epoxy resin led to

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Journal of Applied Polymer Science, Vol. 99, 2752–2758 (2006)

improvement in the modulus and stiffness of the PP/epoxy blends, and the addition of MAH-*g*-PP resulted in increased impact strength and tensile strength.

In the present study, an epoxy resin was dynamically cured in a PP/MAH-*g*-PP/talc matrix to prepare dynamically cured PP/MAH-*g*-PP/talc/epoxy composites. The aim of this work was to use dynamic curing of an epoxy resin to increase the interaction adhesion between PP and talc in the PP/MAH-*g*-PP/talc composites. The mechanical properties, morphology, crystallization behavior, and thermal stability of the dynamically cured PP/MAH-*g*-PP/talc/epoxy composites were studied.

EXPERIMENTAL

Raw materials

The polypropylene, F401, was produced by Yangzi Petrochemical Co., Ltd. (Nanjin, China). It had a melt flow index (MFI) of 1.9 dg/min (230°C, 2.16 kg). Diglycidyl ether of bisphenol A resin (DGEBA, M_w: 1120 g/mol) was supplied by Shanghai Resin Co., Ltd. (Shanghai, China). The curing agent, 2-ethylene-4-methane-imidazole (EMI-2,4), was produced by Shanghai Chemical Agent Company (Shanghai, China). Maleic anhydride–grafted polypropylene (MAH-*g*-PP) with a MAH content of 1% was prepared by Shanghai Sunny New Technology Development Co., Ltd. (China). Talc powder (1250 mesh) was supplied by Shanghai PLT Composite Materials Co., Ltd. (Shanghai, China).

Sample preparation

Before blending, the MAH-*g*-PP, talc, and epoxy resin were dried at 80°C under vacuum for about 8 h. The PP/talc composites were prepared in the mixing chamber of a Haake Rheometer RC90 at 190°C and 50 rpm. MAH-*g*-PP and PP were first mixed for 2 min, then the epoxy resin and talc were added, and 2 min later, the curing agent (EMI-2,4) was added with the mixing continuing. The total mixing process lasted about 10 min. The composites were moved out and compression-molded in a press at 195°C for 5 min and then were cold-pressed, producing samples for testing.

Measurements of mechanical properties

The tensile properties were measured using an Instron 4465 Tester (UK) at a crosshead speed of 20 mm/min according to ASTM D638. Notched Izod impact strength was tested using a Ray-Ran Universal Pendulum Impact Tester (UK) at an impacting pendulum speed of 3.5 m/s according to ASTM D256. Flexural properties were measured using the Instron 4465 Tester (UK) and a three-point-loading rig, and the central head was loaded on the specimen at a speed of 1.7 mm/min according to ASTM D790.

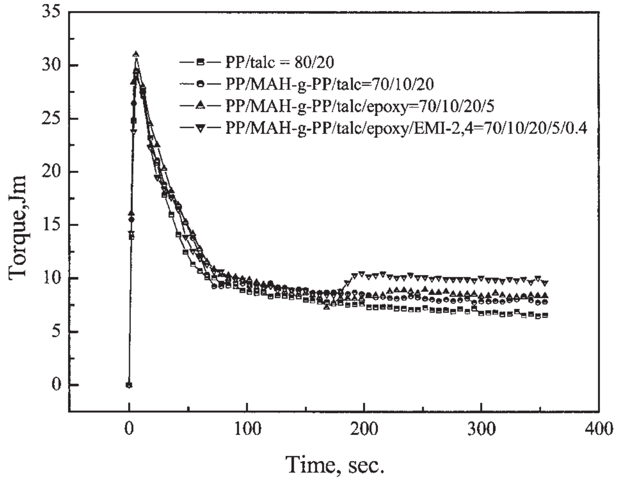


Figure 1 Plots of torque versus time for the PP/talc composites at 190°C.

Scanning electron microscopy

The samples were fractured under liquid nitrogen, coated with the gold, and observed using a Hitachi S-2150 scanning electron microscope (SEM).

DSC analysis

Crystallization behavior was analyzed using a Perkin-Elmer DSC (Paris 1; Wellesley, MA) in a nitrogen atmosphere. For each test, a 4- to 5-mg sample was first heated to 200°C at 50°C/min and then maintained at this temperature for 5 min in order to eliminate any previous thermal history. The specimen was subsequently cooled to 40°C at a rate of 10°C/min and then heated to 200°C at a rate of 10°C/min for data collection.

Thermogravimetric analysis

Thermogravimetric analysis (TGA) was carried out in a Perkin–Elmer TGA7 (Wellesley, MA). The samples were scanned from 30°C to 800°C at a heating rate of 20°C/min in nitrogen atmosphere.

RESULTS AND DISCUSSION

Torque versus time

Torque measurements can be used to give qualitative information concerning the chemical reactivity and the extent of reaction in the polymer blends.[25]

Figure 1 shows the relationship between torque and time for the PP/talc composites at 190°C. The torque at equilibrium of the PP/MAH-*g*-PP/talc (70 : 10 : 20) composite was slightly higher than that of the PP/talc (80 : 20) composite, and the addition of 5 wt % epoxy resin into PP/MAH-*g*-PP/talc (70 : 10 : 20) composite gave rise to an additional increase in the torque at equilibrium. The dynamically cured PP/MAH-*g*-PP/talc/epoxy (70 : 10 : 20 : 5) composite had the highest torque at equilibrium in all PP/talc composites. As MAH-*g*-PP has active maleic anhydride groups, the MAH-*g*-PP in the PP/MAH-*g*-PP/talc (70 : 10 : 20) composite could create with hydroxyl groups at the surface of the talc, resulting in greater viscosity than with the PP/talc (80 : 20) composite. For the PP/MAH-*g*-PP/talc/epoxy (70 : 10 : 20 : 5) composite, increased torque at equilibrium indicated that the epoxy resin in the composite involved the interaction of the MAH-*g*-PP with the talc. We believe that the epoxy resin reacted with the maleic anhydride groups of MAH-*g*-PP and the hydroxyl groups at the talc filler surface, leading to higher viscosity in the PP/MAH-*g*-PP/talc/epoxy (70 : 10 : 20 : 5) composite. It can be seen that the addition of the curing agent (EMI-2,4) with a mixing time of 4 min led to an obvious rise of torque. This shows that the epoxy resin had been cured by EMI-2,4 when it was being mixed with the molten PP and the talc.

SEM analysis

Figure 2 shows SEM micrographs of the fracture morphology of the PP/talc composites. The talc in the PP/talc (80 : 20) composite [Fig. 2(a)] was dispersed in the PP matrix as lamellae 3–8 μm in size, and the boundaries between the PP and the talc were distinct, although for the PP/MAH-*g*-PP/talc (70 : 10 : 20) composite [Fig. 2(b)], the boundaries became obscure. It can be concluded that MAH-*g*-PP can improve the interaction adhesion between the PP and the talc. Figure 2(c,d) shows the fracture morphology of the PP/MAH-*g*-PP/talc/epoxy (70 : 10 : 20 : 5) and dynamically cured PP/MAH-*g*-PP/talc/epoxy (75 : 5 : 20 : 5) composites. The boundaries become more obscure, showing better interaction adhesion between the PP and the talc. As the epoxide groups of epoxy resin can react not only with the active maleic anhydride groups of MAH-*g*-PP but also with the hydroxyl groups at the surface of the talc. We believe that the reactions occurred in the PP/MAH-*g*-PP/talc/epoxy composites, forming an anchor that promoted interfacial adhesion between the PP and the talc. Dynamic curing of the epoxy resin in the PP/MAH-*g*-PP/talc composites can further increase interfacial adhesion. The fracture morphology of the PP/talc/epoxy (80 : 20 : 5) composite [Fig. 2(e)] was similar to that of the PP/talc (80 : 20) composite, showing that the epoxy resin did not increase the interaction between the PP and the talc, which can be attributed to the different polarities of the epoxy resin and the PP.

Crystallization and melting behaviors

PP is a highly crystalline polymer that can crystallize rapidly, even under quenched conditions. Figures 3 and 4 show the first cooling and second heating DSC scans of PP and different PP/talc composites, and Table I gives a summary of the crystallization and melting data from the DSC thermograms. The crystallinity of the PP component in the composites was calculated with the following equation:

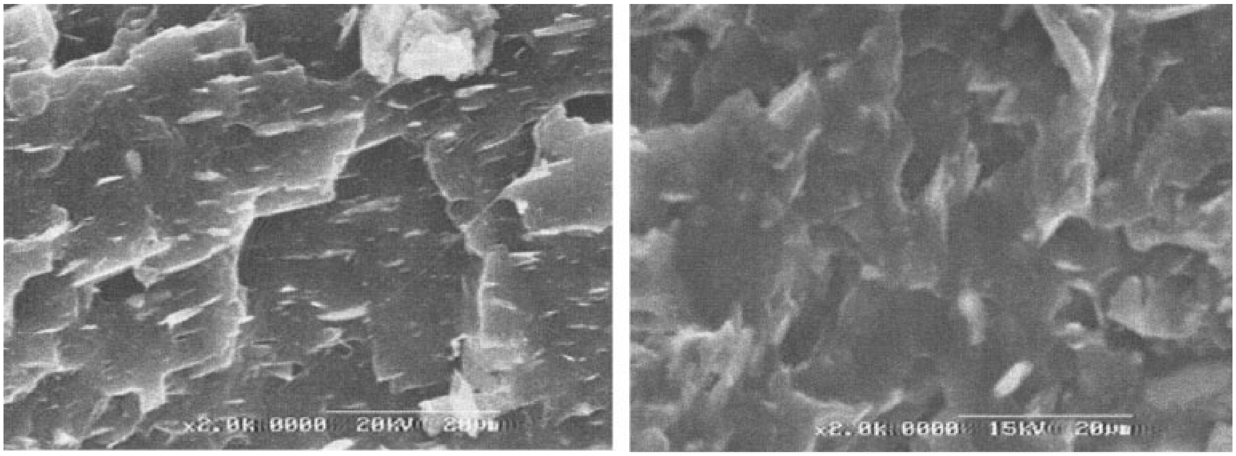
$$X_c\,(\%) = (\Delta H/\Delta H_0) \times (100/x) \tag{1}$$

where X_c is the crystallinity of PP, ΔH is the measured heat of crystallization of PP in the PP/talc composites, ΔH_0 (= 170 J/g) is the heat of crystallization of 100% crystalline isotactic polypropylene,[26] and x is the mass fraction of PP in different PP/talc composites.

For PP and all PP/talc composites, only a single crystallization peak can be seen. All the crystallization peak temperatures of the PP/talc composites were higher than the crystallization peak of PP (113.1°C). The crystallization peak temperature of PP in the PP/talc (80 : 20) composite was increased by approximately 13°C compared to that of the pure PP. A shift of the crystallization peak to a higher temperature suggests that talc can act as an effective nucleating agent, accelerating the crystallization of PP in the composites. Liu et al.[27] reported that the silicate layers can act as nucleating agents and accelerate the crystallization of PP in PP/clay composites. The crystallization peak temperatures of PP in the PP/MAH-*g*-PP/talc (70 : 10 : 20), PP/MAH-*g*-PP/talc/epoxy (70 : 10 : 20 : 5), and dynamically cured PP/MAH-*g*-PP/talc/epoxy (70 : 10 : 20 : 5) composites were slightly higher than that of PP in the PP/talc (80 : 20) composite. This may be a result of the increased interaction of the talc with the PP matrix, improving the nucleating activity of the filler and promoting crystallization during the cooling process. A similar result was reported by Qiu et al.[27], The crystallinity of the PP in all PP/talc composites was higher than that of the pure PP; the crystallinity of the PP in the PP/talc (80 : 20) was 61.2%, an increase of 5% compared with that of pure PP. The melting temperature of PP in all the PP/talc composites was slightly higher than that of pure PP (162.3°C). An increase in the melting temperature of the PP in the composites was directly related to the size of the PP crystals.[28]

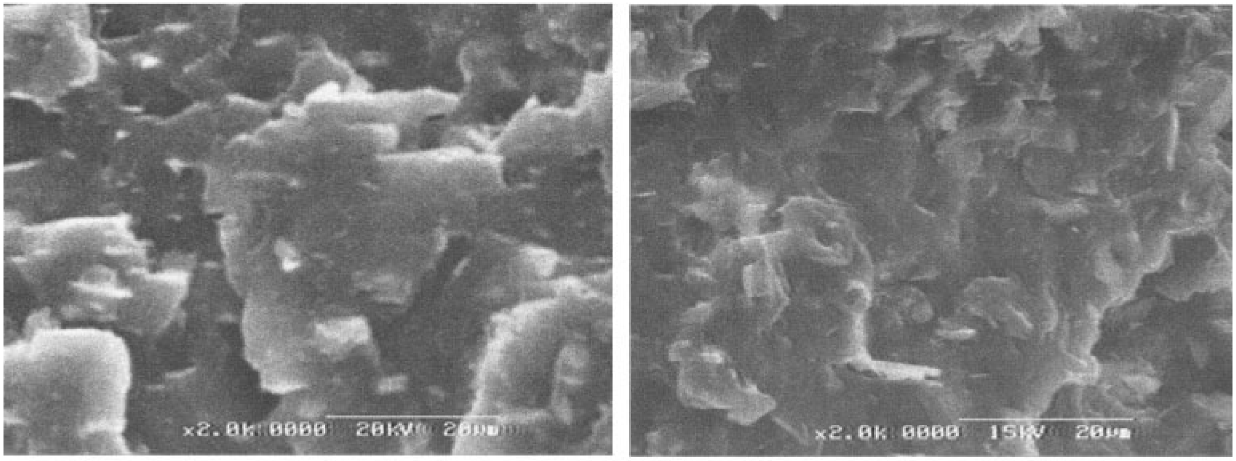
TGA analysis

Polymer degradation generally is an undesirable process that involves deterioration of polymer properties.



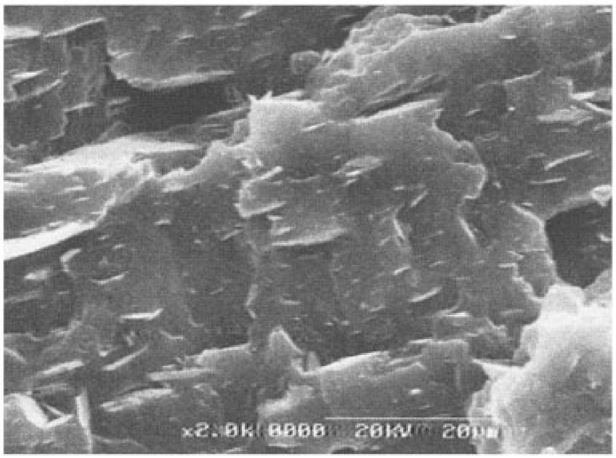
(a) PP/talc = 80/20

(c) PP/MAH-g-PP/talc/epoxy = 70/10/20/5



(b) PP/MAH-g-PP/talc = 70/10/20

(d) PP/MAH-g-PP/talc/epoxy/EMI-2,4 = 70/10/20/5/0.4



(e) PP/talc/epoxy = 80/20/5

Figure 2 SEM micrographs of the PP/talc composites.

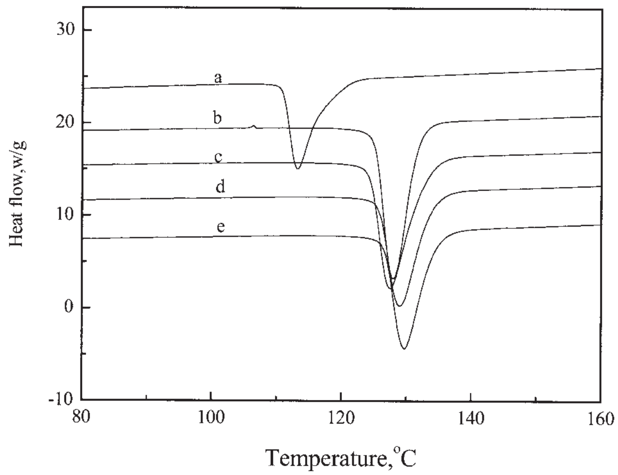


Figure 3 DSC thermograms at first cooling of: (a) PP; (b) PP/talc %equals; 80 : 20; (c) PP/MAH-*g*-PP/talc %equals; 70 : 10 : 20; (d) PP/MAH-*g*-PP/talc/epoxy %equals; 70 : 10 : 20 : 5; (e) PP/MAH-*g*-PP/talc/epoxy/EMI-2,4 %equals; 70 : 10 : 20 : 5 : 0.4.

TABLE I
Results of DSC Analysis PP and PP/Talc Composites

Composition	T_m (°C)	T_c (°C)	X_c (wt %)
PP	162.3	113.1	56.1
PP/talc (80:20)	166.4	126.5	61.2
PP/MAH-*g*-PP/talc (70:10:20)	166.1	128.0	62.1
PP/MAH-*g*-PP/talc/ epoxy (70:10:20:5)	166.2	129.0	58.9
PP/MAH-*g*-PP/talc/ epoxy/EMI-2,4 (70:10:20:5:0.4)	166.5	129.4	59.6

T_m: peak melting temperature; T_c: peak crystallization temperature; X_c: crystallinity of PP.

One of the most accepted methods of studying the thermal properties of polymeric materials is thermogravimetry, by means of which integral and derivative thermogravimetric curves can provide information about the nature and extent of degradation of the polymeric materials.[29] Thermograms of the PP and PP/talc composites are shown in Figure 5, and Table II gives a summary of the initial and final degradation temperatures, T_0 and T_f, respectively. The degradation of PP started at 354°C and finished at 424°C. The weight loss in this temperature region, 354°C–424°C, corresponded to the formation of volatile products that arose from random chain scission and intermolecular transfer involving tertiary hydrogen abstractions from the polymer by the primary radical.[30] The talc did not degrade in this temperature range. The incorporation of 20 wt % talc into the PP/talc (80 : 20) composite increased the T_0 from 354°C to 364°C; the T_0 of the PP/MAH-*g*-PP/talc (70 : 10 : 20) composite was slightly higher than that of the PP/talc (80 : 20) composite and similar to that of the PP/MAH-*g*-PP/talc/epoxy (70 : 10 : 20 : 5) composite. The dynamically cured PP/MAH-*g*-PP/talc/epoxy (70 : 10 : 20 : 5) composite had the highest initial degradation temperature (T_0) in all the PP/talc composites. All the degradation of PP/talc composites occurred in one step, similar to in PP. The results show that the incorporation of the talc into the PP could improve the thermal stability of PP and that the addition of MAH-*g*-PP and epoxy resin to the PP/talc composites obviously could further improve thermal stability. The dynamically cured PP/MAH-*g*-PP/talc/epoxy composite had the best thermal stability of all the PP/talc composites.

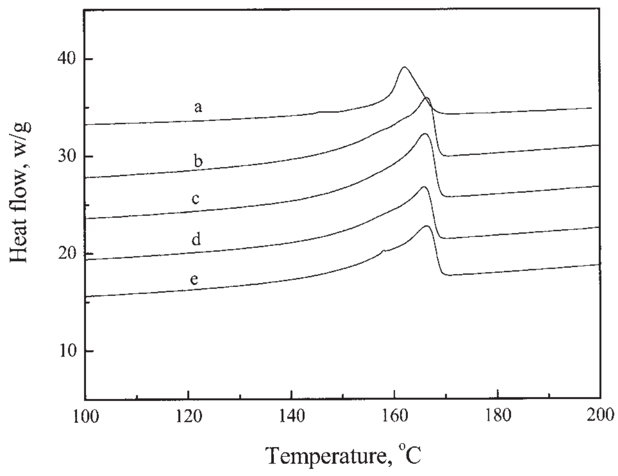


Figure 4 DSC thermograms at second heating of: (a) PP; (b) PP/talc %equals; 80 : 20; (c) PP/MAH-*g*-PP/talc %equals; 70 : 10 : 20; (d) PP/MAH-*g*-PP/talc/epoxy %equals; 70 : 10 : 20 : 5; (e) PP/MAH-*g*-PP/talc/epoxy/EMI-2,4 %equals; 70 : 10 : 20 : 5 : 0.4.

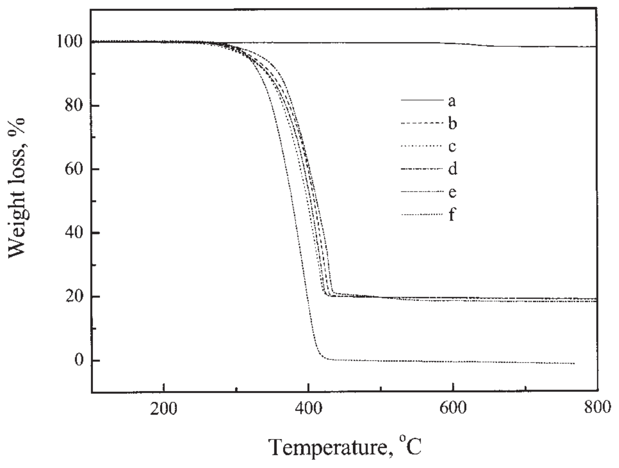


Figure 5 Thermograms of the PP and the PP/talc composites: (a) talc; (b) PP; (c) PP/talc %equals; 80 : 20; (d) PP/MAH-*g*-PP/talc %equals; 70 : 10 : 20; (e) PP/MAH-*g*-PP/talc/epoxy %equals; 70 : 10 : 20 : 5; (f) PP/MAH-*g*-PP/talc/epoxy/EMI-2,4 %equals; 70 : 10 : 20 : 5 : 0.4.

TABLE II
Results of TGA Analysis PP and PP/Talc Composites

Composition	T_0 (°C)	T_f (°C)
Talc	—	—
PP	354	424
PP/Talc (80 : 20)	364	431
PP/MAH-*g*-PP/talc (70 : 10 : 20)	369	436
PP/MAH-*g*-PP/epoxy (70 : 10 : 5)	357	428
PP/MAH-*g*-PP/talc/epoxy (70 : 10 : 20 : 5)	370	437
PP/MAH-*g*-PP/talc/epoxy/EMI-2,4 (70 : 10 : 20 : 5 : 0.4)	379	446

T_0: initial degradation; temperature; T_f: final degradation temperature.

George et al.[29] suggested that thermal degradation of polymer blends depends on not only the thermal stability of its components but also on the morphology and extent of interaction between the phases. The increases in the T_0 of the PP/MAH-*g*-PP/talc (70 : 10 : 20), PP/MAH-*g*-PP/talc/epoxy (70 : 10 : 20 : 5), and dynamically cured PP/MAH-*g*-PP/talc/epoxy (70 : 10 : 20 : 5) composites can be attributed to interaction adhesion that was better than that with the PP/talc (80 : 20) composite.

Mechanical properties

Mechanical properties including notched Izod impact strength, tensile properties, and flexural strength of different PP/talc composites are summarized in Table III. It can be seen that the addition of 20 wt % talc in the PP matrix gave rise to improved flexural modulus and tensile strength, but decreased impact strength and elongation at break because of poor interaction adhesion between the PP and the talc. With the addition of 10 wt % MAH-*g*-PP, the PP/MAH-g-PP/talc (70 : 10 : 20) composite had higher flexural modulus and tensile strength, but lower elongation at break compared with the PP. The PP/MAH-*g*-PP/talc/epoxy (70 : 10 : 20 : 5) composite had better mechanical properties than did the PP/MAH-*g*-PP/talc (70 : 10 : 20) composite, and the dynamically cured PP/MAH-*g*-PP/talc/epoxy (70 : 10 : 20 : 5) composite had the highest flexural modulus (2542 MPa) and impact strength (40 J/m) of all the PP/talc composites. We thought that the improvement in the mechanical properties of PP/MAH-*g*-PP/talc/epoxy was a result of the better interaction adhesion between the PP and the talc compared with that of PP/MAH-*g*-PP/talc, and dynamic cure of the epoxy resin was able to further the mechanical properties of the composites. The mechanical properties of the PP/talc/epoxy (80 : 20 : 5) were similar to those of the PP/talc (80 : 20), which shows that epoxy resin cannot improve the interaction between PP and talc in the absence of MAH-*g*-PP. Figure 6 shows the effect of epoxy resin content on the mechanical properties of the dynamically cured PP/MAH-*g*-PP/talc/epoxy composites. Tensile strength and flexural modulus increased with increasing epoxy resin content, and impact strength reached a maximum of 40 J/m at an epoxy resin content of 5 wt %. The elongation at break decreased rapidly when the epoxy resin content was above 5 wt %. The results show that the suitable content of epoxy resin in the composites was about 5 wt %. This also shows that the epoxy resin in dynamically cured PP/MAH-*g*-PP/talc/epoxy composites used as a coupling agent could increase the interaction between the PP and the talc in the presence of MAH-*g*-PP, and when used in the appropriate amounts, it led to obvious improvements in the mechanical properties of the composites, but additional increases in the amount of the epoxy resin did not improve the mechanical properties any more.

CONCLUSIONS

The dynamic curing of an epoxy resin in the PP/MAH-*g*-PP/talc composites was demonstrated to effectively increase the interaction adhesion between the PP and the talc. The torque at equilibrium of the PP/MAH-*g*-PP/talc/epoxy (70 : 10 : 20 : 5) composite was slightly higher than that of the PP/MAH-*g*-PP/talc (70 : 10 : 20) composite, indicating the epoxy resin was able to increase the interaction between the PP and the talc. Dynamic curing of the epoxy resin could

TABLE III
Mechanical Properties of PP and Different PP/Talc Composites

Composition	Impact strength (J/m)	Tensile strength (MPa)	Elongation at break (%)	Flexural strength (MPa)
PP	30	30.5	260	1250
PP/talc (80 : 20)	20	36.8	5.8	2250
PP/MAH-*g*-PP/talc (70 : 10 : 20)	32	40.7	13.6	2376
PP/talc/epoxy (80 : 20 : 5)	27	37.6	7.2	2283
PP/MAH-*g*-PP/talc/epoxy (70 : 10 : 20 : 5)	36	41.9	14.1	2435
PP/MAH-*g*-PP/talc/epoxy/EMI-2,4 (70 : 10 : 20 : 5 : 0.4)	40	43.2	14.9	2542

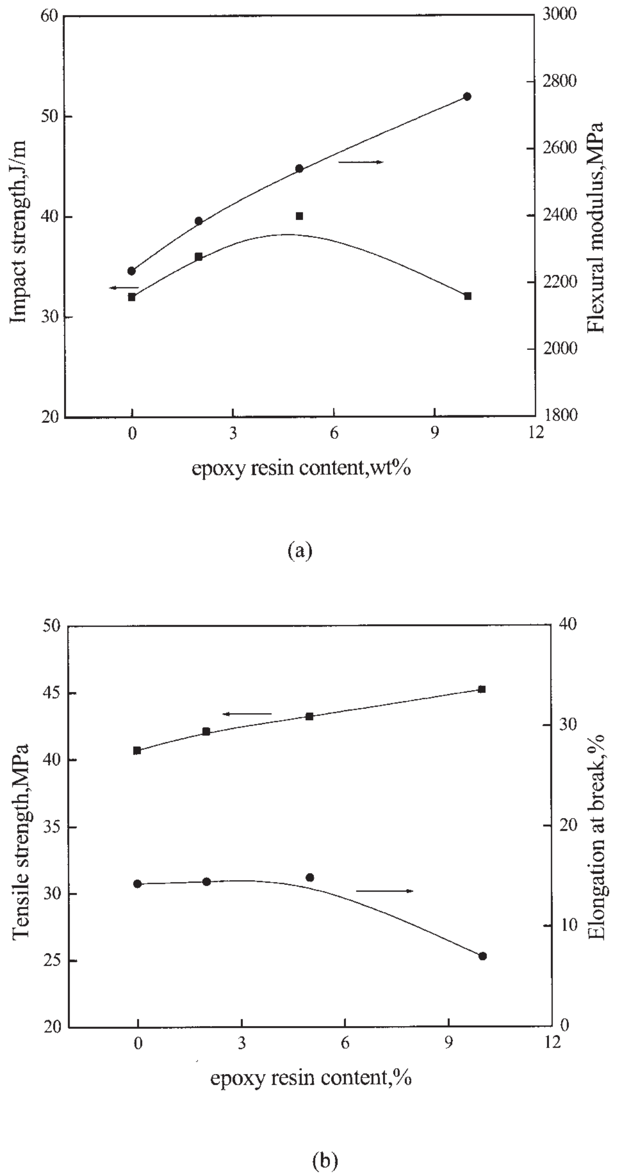


Figure 6 Effect of epoxy resin content on the mechanical properties of dynamically cured PP/MAH-*g*-PP/talc/epoxy composites.

further increase the interaction adhesion. SEM analysis showed that the epoxy resin did not increase the interaction adhesion between the PP and the talc for the PP/talc/epoxy composites without the presence of MAH-*g*-PP. DSC analysis showed that the talc in the PP/talc composites was able to act as a nucleating agent and that the dynamically cured PP/MAH-*g*-PP/talc/epoxy composites had higher crystallization peaks. TGA showed that the incorporation of the talc into the PP could improve the thermal stability of PP, and the addition of MAH-*g*-PP and the epoxy resin further improved the thermal stability of the PP/talc composites, with the dynamically cured PP/MAH-*g*-PP/talc/epoxy composites showing the best thermal stability. The PP/MAH-*g*-PP/talc/epoxy composites hade better mechanical properties than did the PP/MAH-*g*-PP/talc composites, and of all the PP/talc composites, the dynamically cured PP/MAH-*g*-PP/talc/epoxy composites had the best mechanical properties. The suitable content of epoxy resin in the dynamically cured PP/MAH-*g*-PP/talc/epoxy composites was about 5 wt %.

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