Mechanical Properties of Mg(OH)₂/Polypropylene Composites Modified by Functionalized Polypropylene

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ABSTRACT: Modified Mg(OH)₂/polypropylene (PP) composites were prepared by the addition of functionalized polypropylene (FPP); and acrylic acid (AA) and by the formation of *in situ* FPP. The effects of the addition of FPP and AA and the formation of *in situ* FPP on the mechanical properties of Mg(OH)₂/PP composites were investigated. Experimental results indicated that the addition of Mg(OH)₂ markedly reduced the mechanical properties of PP. The extent of reduction in notch impact strength of PP was higher than that in flexural strength and tensile strength. However, tensile modulus and flexural modulus increased with increased Mg(OH)₂ content. The addition of FPP facilitated the improvement in the flexural strength and tensile strength of Mg(OH)₂/PP composites. The higher the Mg(OH)₂ content was, the more significant the effect of FPP was. The incorporation of AA resulted in further increased mechanical properties, in particular the flexural strength, tensile strength, and notch impact strength of Mg(OH)₂/PP composites containing high levels of Mg(OH)₂. It not only improved mechanical properties but also increased the flame retardance of Mg(OH)₂/PP composites. Although the mechanical properties of composites modified by the formation of *in situ* FPP were lower than those of composites modified by only the addition of AA in the absence of diamylperoxide, the mechanical properties did not decline with increased Mg(OH)₂ content. Moreover, the mechanical properties increased with increasing AA content. The addition of an oxidation resistant did not influence the mechanical properties of the modified Mg(OH)₂/PP composites. © 2003 Wiley Periodicals, Inc. J Appl Polym Sci 88: 2139–2147, 2003

Key words: poly(propylene) (PP); mechanical properties

INTRODUCTION

To improve the flame retardance of polypropylene (PP), the incorporation of a high level of Mg(OH)₂ is necessary, generally leading to reduced mechanical properties for PP.[1,2] The mechanical properties of the Mg(OH)₂/PP composites investigated by Hornsby and Watson[3] indicated that the addition of Mg(OH)₂ resulted in a decline in the mechanical properties of PP, in particular for impact strength (IS) due to the difference in surface polarity between Mg(OH)₂ and PP. Another investigation reported that although the addition of a small amount of Mg(OH)₂ increased IS, the IS of PP significantly decreased as the Mg(OH)₂ content increased from 30 to 60 wt %.[4] The effect of the

surface treatment of Mg(OH)₂ on the mechanical properties of Mg(OH)₂/PP composites has also been investigated. Although the surface treatment of Mg(OH)₂ resulted in an improvement in the mechanical properties of PP, different surface modifiers exhibited different effects. Hornsby and Watson[5] found that the surface treatment of Mg(OH)₂ by silane only gave rise to a slight improvement in flexural strength (FS) and flexural modulus (FM). Due to the absence of an initiator, ethylene of silane was unable to graft onto the matrix. No strong chemical reaction occurred, between the particle surface and the functional groups of silane, limiting the improvement in FS. The IS of PP filled by Mg(OH)₂ treated by a titanate coupling agent increased as the titanate content was increased. Through modification by fatty acids and their derivatives, a significant improvement in IS and FS of composites was obtained, which was attributed to a strong chemical interaction between the components, and which improved the surface adhesion. PP grafted with acrylic acid (AA) as a coupling agent also markedly improved the FS of Mg(OH)₂/PP composites.

The effect of Mg(OH)₂ morphology on the mechanical properties of Mg(OH)₂/PP composites has also been reported. Cook and Harper[6] revealed that an irregular spherical microporous particle benefitted the

surface interaction between the particle and the matrix to increase the FS. Platelet $Mg(OH)_2$ initiated crack more easily compared to spherical ones and resulted in a lower IS. Jancar and Kucera[7] found that the mechanical properties of PP filled by needle $Mg(OH)_2$ were higher than that of PP filled by platelet $Mg(OH)_2$. They suggested that the surface area of needle $Mg(OH)_2$ was larger, resulting in an increase in the effective contact of the $Mg(OH)_2$ surface with matrix, which reinforced interfacial interaction and improved the mechanical properties.

Similarly, the mechanical properties of $Mg(OH)_2/$ PP composites was also affected by the particle size of $Mg(OH)_2$. Cook and Harper[8] observed that the FM of PP filled with small particles was higher than that with larger particles. The surface treatment of filler increased the flexibility of the interface and reduced the flexural properties of the composites. However, a higher IS was obtained. Another investigation indicated that PP filled with particles 2 μm in size exhibited the best mechanical properties due to the best dispersion, and the mechanical properties decreased with increasing particle size. The dispersion of filler is another important factor affecting the mechanical properties of composites. Better dispersion produced an increase in tensile strength (TS), FS, and IS.[4]

To improve the impact properties of $Mg(OH)_2/PP$ composites, composites modified by the addition of elastomer have been studied. Wang et al.[9] found that the incorporation of 60 wt % $Mg(OH)_2$ into PP gave rise to a noticeable decrease in TS and IS. The addition of an elastomer, ethylene–propylene rubber grafted with maleic anhydride, raised the IS of PP filled with untreated $Mg(OH)_2$. Surface treatment of $Mg(OH)_2$ further increased the IS and moduli of the composites. EPR grafted with maleic anhydride may have encapsulated the $Mg(OH)_2$ surface with a layer of elastomer and reduced the surface free energy, which resulted in an increase in the degree of dispersion of the filler and improved the interfacial adhesion between the particle and the matrix, an improvement in the deformation ability of the surface layer under loading, which sequentially dissipated more energy. However, the addition of elastomer decreased the TS of the composites.

Although the surface treatment of $Mg(OH)_2$ by organic coupling agents can improve the mechanical properties of PP composites, in recent years, the use of functionalized polyolefins to improve the compatibility, interfacial adhesion, and mechanical properties of PP blends and PP composites has been widely investigated, such as the use of $CaCO_3/PP,$[7,10,11] mica/PP,[12] talc/PP,[13] halogen-antimony trioxide/PP,[14] $Al(OH)_3/$ PP,[15–20] glass fiber/PP,[21] and natural fiber/PP[22,23] composites. However, few studies[5,9] on the effect of functionalized polypropylene (FPP) in $Mg(OH)_2/PP$ composites have been reported, in particular on the

effect of the formation of *in situ* FPP on $Mg(OH)_2/PP$ composites. The physical and mechanical properties of crystalline polymers and their composites depend on their crystallization morphology, which is relative to crystallization behavior. In our laboratory, $Mg(OH)_2/$ PP composites modified by the addition of FPP and AA, and the formation of *in situ* FPP were prepared. Studies on crystallization and melting behavior and the crystallization morphologies of modified $Mg(OH)_2/PP$ composites indicated that the addition of FPP and AA accelerated crystallization, increased crystallization temperatures, and decreased the spherulite size of PP due to the heterogeneous nucleation effect of FPP and AA. $Mg(OH)_2$ exhibited a heterogeneous nucleation effect on PP and increased the crystallization temperature of PP, much like mica[24] and $CaCO_3$[25] FPP and AA activated the heterogeneous nucleation effect of the $Mg(OH)_2$ surface and caused further increase in the crystallization temperatures of PP. However, the crystallization temperatures of $Mg(OH)_2/PP$ composites modified by the formation of *in situ* FPP were lower than those of $Mg(OH)_2/PP$ composites modified by the addition of FPP and AA. In the modified $Mg(OH)_2/PP$ composites, the addition of FPP and AA and the formation of *in situ* FPP had no influence on the spherulite size of PP, which was attributed to a heterogeneous nucleation effect of $Mg(OH)_2$ that was higher than that of the modifiers.[26] Because the mechanical properties of $Mg(OH)_2/PP$ composites depend on crystallization behavior and morphology, in this study the mechanical properties of $Mg(OH)_2/PP$ composites modified by the addition FPP and AA and the formation of *in situ* FPP were emphatically investigated.

EXPERIMENTAL

Materials

Powered PP, F401, and pelletized PP, CTS-700, were commercial products of Guangzhou Yinzhu PP Ltd. (China). Chemical-grade AA was purchased from Shanghai Feida Industrial Trade Ltd. (China) and used without further purification. Chemical-grade dicumyl peroxide (DCP), which was used as an initiator for the AA grafting reaction, was obtained from Shanghai Chemical Reagent Stocking and Providing Station Pool Enterprise Central Factory (China). $Mg(OH)_2$ was a commercial product of Zhejiang Chemical Plant (China). The solvent, acetone, was obtained from Guangzhou Chemical Reagent Factory (China).

Preparation of composites

Preparation of FPP

DCP and AA were dissolved in acetone, totally blended with powered PP in a GH-10 high-speed mix-

ing machine (Beijing Plastic Machine Factory, China). After the acetone had completely evaporated, the mixture of AA and DCP was coated on the surface of the powdered PP, and the pretreated PP was prepared. The pretreated PP was then extruded with a SHJ-53 twin-screw extruder (Nanjing Aviation Institute Xinli Plastic Machine Factory, China) at temperatures of 185–190°C with the screw speed set at 60 rpm. The extruded products, the PP grafted by AA (FPP), were frozen inline in a water bath, dried, and granulated.

Preparation of Mg(OH)$_2$/PP composites modified by the formation of *in situ* FPP

Mg(OH)$_2$, DCP, AA, and pelletized PP were totally blended with a high-speed mixing machine, and the mixtures were then extruded by a twin-screw extruder at temperatures of 185–190°C with the screw speed set at 60 rpm. The extruded products were granulated and named as Mg(OH)$_2$/PP composites modified by *in situ* FPP.

Preparation of Mg(OH)$_2$/PP composites modified by the addition of FPP

Mg(OH)$_2$, premade FPP, and pelletized PP were totally blended with a high-speed mixing machine, and the mixtures were then extruded by a twin-screw extruder at temperatures of 185–190°C with the screw speed set at 60 rpm. The extruded products were granulated and named as Mg(OH)$_2$/PP composites modified by additive FPP.

Preparation of the test specimens of Mg(OH)$_2$/PP composites

The pelletized Mg(OH)$_2$/PP composites modified by different methods were injection molded into GB test specimens with a CJ150 ME-NC injection molding machine set with barrel temperature profile ranging from 190 to 210°C.

Determination of mechanical properties

Notched IS was measured on a XJJ-5 impact testing machine (Chengde Test Machine Factory, Hebei Province, China) with procedures given in GB/T1043-93. FS and FM were measured on a LWK-5 electronic universal tension testing machine (Guangzhou Test Machine Factory, China) after GB1040-92 with a crosshead speed of 10 mm/min. TS and tensile modulus (TM) were detected on a WD-5A electronic universal testing machine (Guangzhou Test Machine Factory) at a crosshead speed 10 mm/min in accordance with GB/T1040-92.

RESULTS AND DISCUSSION

Mechanical properties of Mg(OH)$_2$/PP composites

The relationship of TS, TM, FS, FM, notched IS, and Mg(OH)$_2$ content are shown in Figure 1. The addition of Mg(OH)$_2$ led to a pronounced drop in notched IS of PP, which was consistent with the results in some reports. The notched IS of PP filled with 20 wt % Mg(OH)$_2$ (M-2) was 83% that of pure PP (M-0). For sample M-6, containing 60 wt % Mg(OH)$_2$, notched IS was only 56% of that of pure PP. TS and FS of PP also decreased with increasing Mg(OH)$_2$ content. The addition of 20 wt % Mg(OH)$_2$ produced a drop in the TS and FS by 5% and a 25% reduction was observed for the PP filled with 60 wt % Mg(OH)$_2$. The incorporation of Mg(OH)$_2$ exhibited greater effect on the notched IS than on the FS and TS. However, the rigidity of the composites increased with the addition, and increasing the content of Mg(OH)$_2$ led to a double increase in the TM and FM of PP.

Mechanical properties of Mg(OH)$_2$/PP composites modified by the addition of FPP

Figure 2 shows the relationship of TS, TM, FS, FM, notched IS, and Mg(OH)$_2$ content. The mechanical properties of PP gradually reduced with increasing Mg(OH)$_2$ content. At the equivalent Mg(OH)$_2$ content, the notched IS of composites modified by the addition of FPP was lower than that of unmodified composites. However, the addition of FPP increased the TS. The TSs of PP composites modified by the addition of 20 wt % (MF-2), 40 wt % (MF-4), and 60 wt % (MF-6) Mg(OH)$_2$ increased by 5.5, 17.3, and 13.8%, respectively, relative to that of unmodified Mg(OH)$_2$/PP composites (MF-0). Apparently, FPP had a more significant effect on the improvement of TS at higher Mg(OH)$_2$ contents. At 60 wt % Mg(OH)$_2$, the TS of MF-6 modified by FPP was markedly higher than that of unmodified M-6. Compared with the TS of pure PP, the reduced extent of TS was lower than that of the unmodified composites. The TS of MF-6 merely decreased by 15%, reaching the TS of unmodified M-4 containing 40 wt % Mg(OH)$_2$, and the TS of MF-4 was reduced by 3.3%. A more pronounced effect of FPP on the FS of Mg(OH)$_2$/PP composites was observed. In unmodified Mg(OH)$_2$/PP composites, the FS of composites gradually dropped with the addition of and increase in Mg(OH)$_2$. For Mg(OH)$_2$/PP composites modified by the addition of FPP, the FS changed little with the addition of and increases in Mg(OH)$_2$. At equivalent Mg(OH)$_2$ content, the FS of composites modified by the addition of FPP were higher than that of the unmodified composites. At 20 wt % Mg(OH)$_2$, the addition of FPP led to a higher FS than that of pure PP. The FS of PP composites filled by 40 wt % Mg(OH)$_2$ and modified by the addition of FPP was the
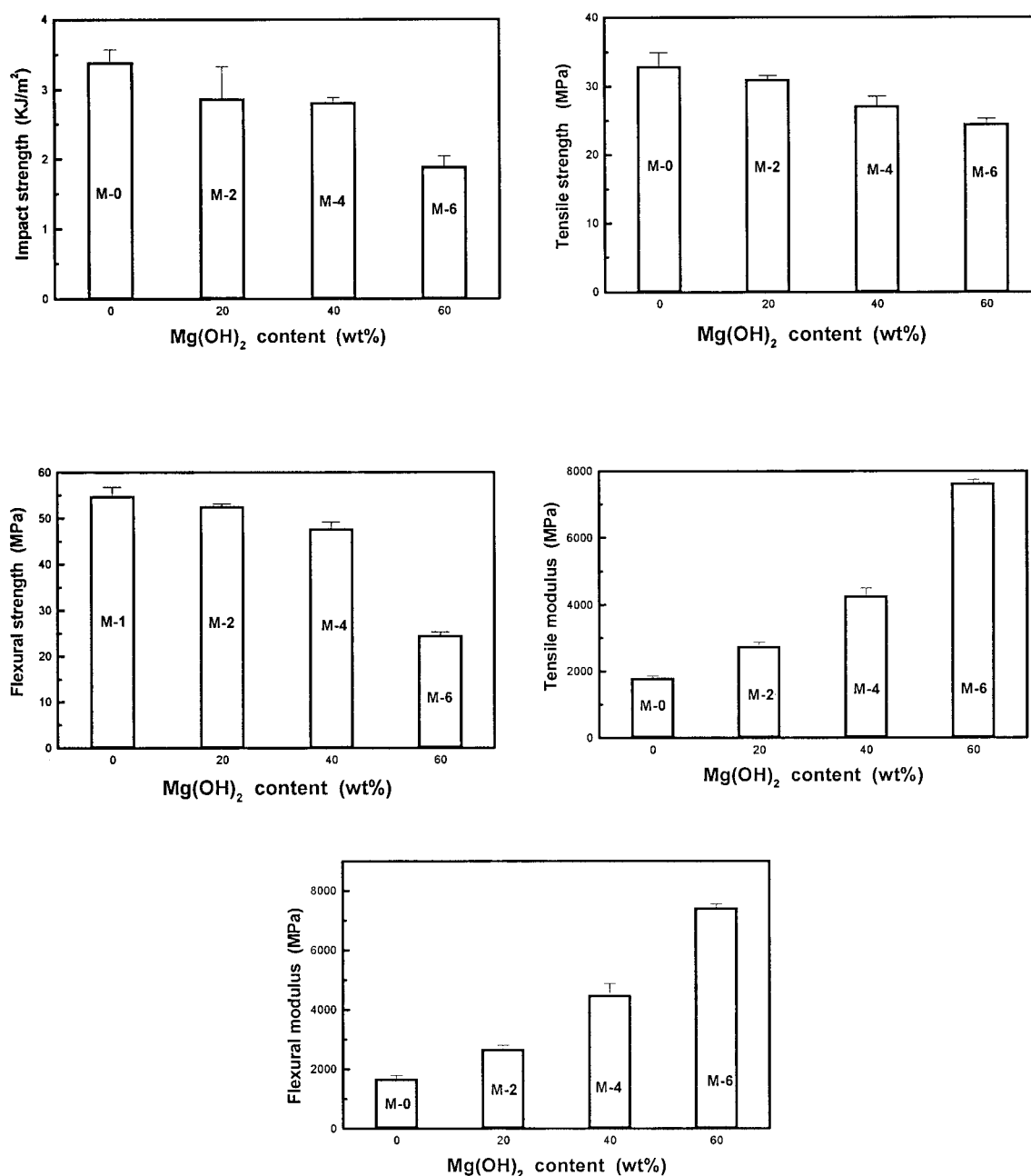
Figure 1 Mechanical properties of $Mg(OH)_2$/PP composites filled with different $Mg(OH)_2$ content.

same as that of pure PP. At 60 wt % $Mg(OH)_2$, the FS of modified composites was even higher than that of the unmodified $Mg(OH)_2$/PP composites. However, the addition of FPP decreased the modulus of composites, especially the composites containing high $Mg(OH)_2$ contents. These results indicated that addition of FPP facilitated the improvement in notched IS and FS of $Mg(OH)_2$/PP composites. The higher the $Mg(OH)_2$ content was the more significant was the effect of FPP and the higher was the TS; FS, and notched IS.

Mechanical properties of $Mg(OH)_2$/PP composites modified by the addition of AA

The relationship of TS, TM, FS, FM, notched IS, and $Mg(OH)_2$ content are shown in Figure 3. The notched IS of PP modified by the addition of 2 phr AA (MA-20) slightly declined compared to that of pure PP. The TS and FS remained the same, accompanying a small increase in modulus. For the composites filled with 60 wt % $Mg(OH)_2$ and modified by the addition of 2 phr AA (MA-26) the notched IS was higher than that of MA-20, and near to that of PP (M-0). The FS and TS
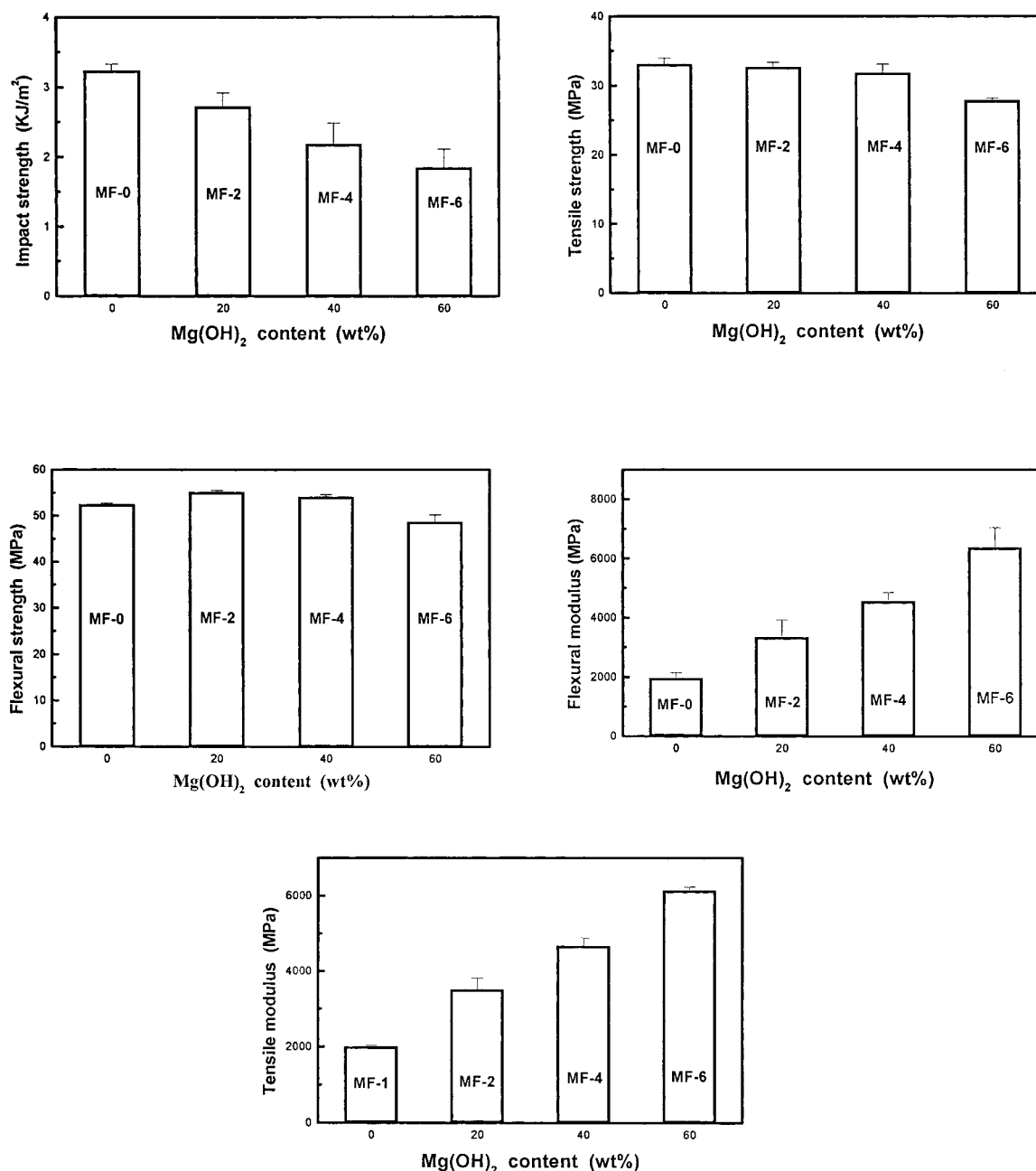
Figure 2 Mechanical properties of Mg(OH)$_2$/PP composites modified by the addition of FPP.

were significantly higher than that of pure PP and MA-20. Compared with the notched IS of M-6 and MF-6 composites filled with 60 wt % Mg(OH)$_2$, the notched IS of MA-26 increased by 70.3 and 74.0%, the TS rose by 49.8 and 31.5%, and the FS improved 54.5 and 33.0%, respectively. It is believed that the AA dispersed in the Mg(OH)$_2$–PP interface more readily during the melting extrusion. The polar part of AA easily interacted with Mg(OH)$_2$ by strong physical interactions, and the ethylene group of AA was ready to graft with the macroradial produced by PP thermal decomposition. This favored the improvement of the interface adhesion between Mg(OH)$_2$ and PP, leading to a improved strengths in the composites. However, when the AA content was up to 4 phr, the notched IS, TS, and FS of composites filled by 60 wt % Mg(OH)$_2$, (MA-46) were reduced but were still higher than that of M-6 and MF-6, and the TS was equal to that of MF-4 and M-2. These results suggested that only the addition of AA monomer into Mg(OH)$_2$/PP composites could cause an improvement in the strengths of composites. However, there existed a critical value of AA content. Excessive AA content led to a drop in the properties of compos-
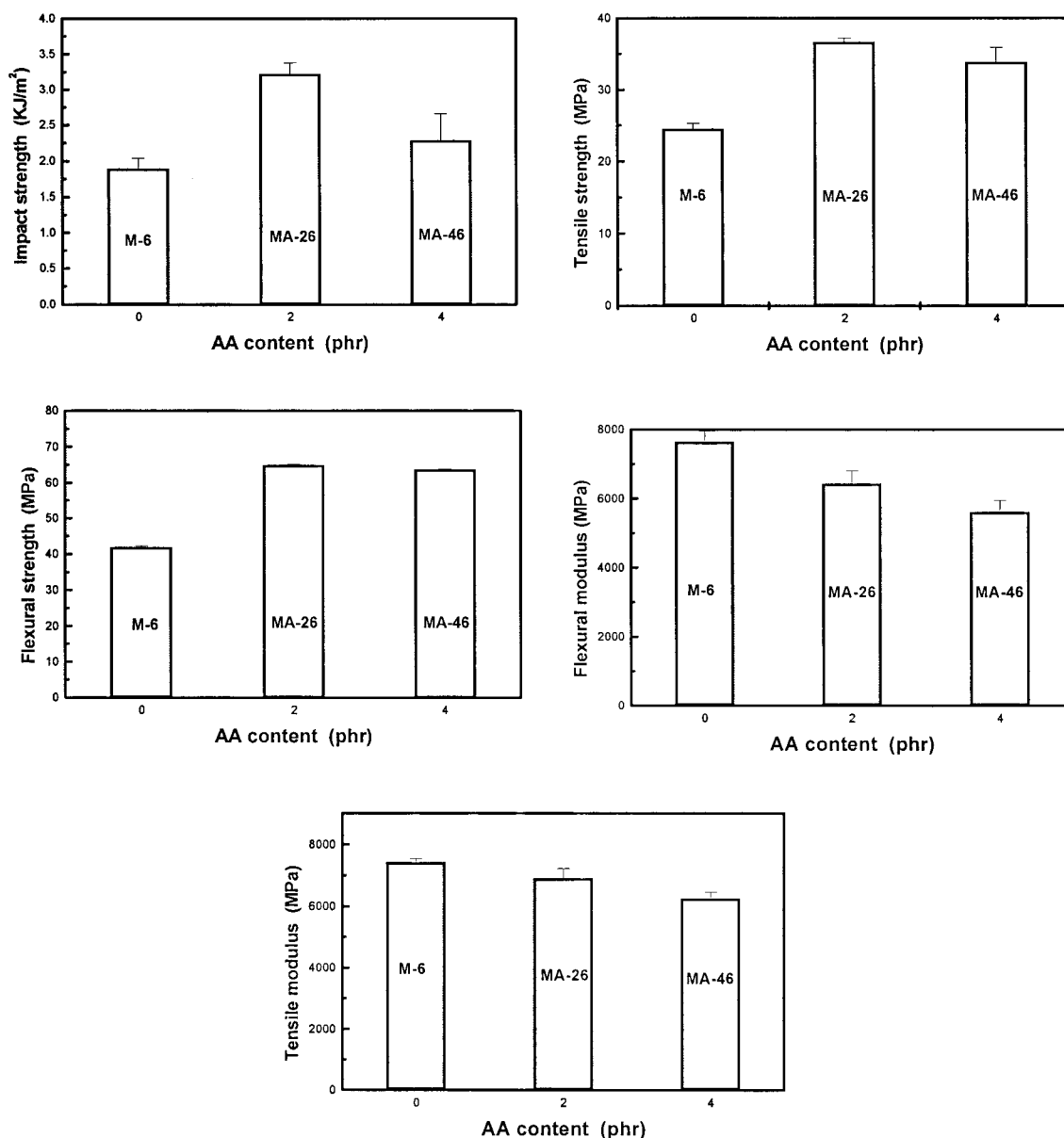
Figure 3 Mechanical properties of Mg(OH)$_2$/PP composites modified by different AA contents.

ites. Due to the absence of an initiator, during the preparation process of Mg(OH)$_2$/PP modified by the addition of AA, only a small quantity of free radials were produced by the thermal degradation of PP, and a small quantity of *in situ* FPP was formed at the interface between Mg(OH)$_2$ and PP. The excessive AA dispersed at the interface between Mg(OH)$_2$ and PP and homopolymerized to form a homopolymer to disperse at the Mg(OH)$_2$ and PP interface or in the matrix. The AA homopolymer lacked adhesion with PP, which led to a reduction in the mechanical properties of the composites. The addition of AA permitted an increase in the Mg(OH)$_2$ content, keeping on the equivalent prop-

erties, which not only improved flame retardance but also helped the composites retain their properties.

Mechanical properties of Mg(OH)$_2$/PP composites modified by the formation of *in situ* FPP

The earlier results indicated that the addition of AA resulted in an improvement in the strengths of the Mg(OH)$_2$/PP composites. This was attributed to the formation of FPP between the PP and Mg(OH)$_2$. To study the formation of FPP during the preparation processing of composites, Mg(OH)$_2$/PP composites modified by the addition of AA in the presence of an
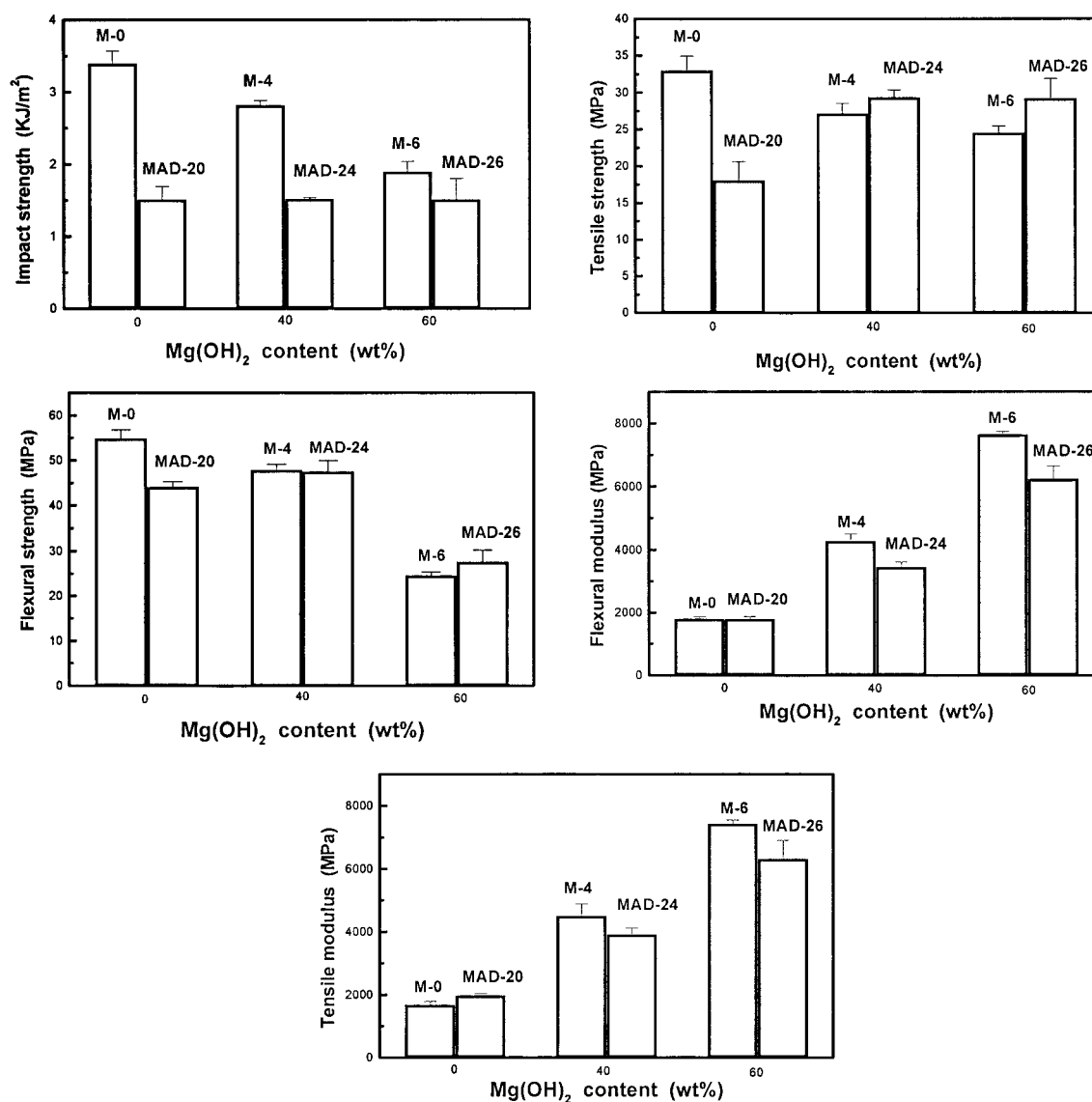
Figure 4 Mechanical properties of Mg(OH)$_2$/PP composites modified by the formation of *in situ* FPP.

initiator, DCP, and an oxidation resistant were prepared, and the mechanical properties of the modified composites were investigated.

Effect of DCP

Figure 4 shows the mechanical properties of the modified Mg(OH)$_2$/PP composites. It can be seen form Figure 4 that severe degradation of PP modified by 2 phr AA (MAD-20) was observed in the presence of DCP. The notched IS and TS were only half of that of pure PP (M-0). The FS was reduced by 20%. Compared with MAD-20, the notched IS remained the same for the composites filled with 40 wt % (MAD-24) and 60 wt % (MAD-26) Mg(OH)$_2$ and modified by AA in the presence of DCP. The TS and FS markedly increased. The TS of MAD-24 increased by 62.7%, TM by 93.4%, FS by 8.0% and FM by 97.9%. The TS of

MAD-26 was improved by 61.7%, TM by 250%, and FM by 220%. Although there was a great difference in mechanical properties between M-0 and MAD-20, the FS and TS of MAD-24 were close to that of M-4. We suggest that the thermal degradation of PP by the addition of DCP resulted in a drop in the molecular weight and mechanical properties of PP during melting extrusion. On the other hand, the free radial induced by DCP initiated the AA grafting onto PP, and the *in situ* FPP between PP and Mg(OH)$_2$ was formed. The carboxyl of FPP interacted with Mg(OH)$_2$, and the PP chain of FPP was compatible with the PP matrix and cocrystallized. These interfacial interactions improved the interface adhesion between components, leading to enhanced mechanical properties. The final properties of the composites depended on the balance of these interactions. In Mg(OH)$_2$/PP composites modified by the formation of *in situ* FPP, except the
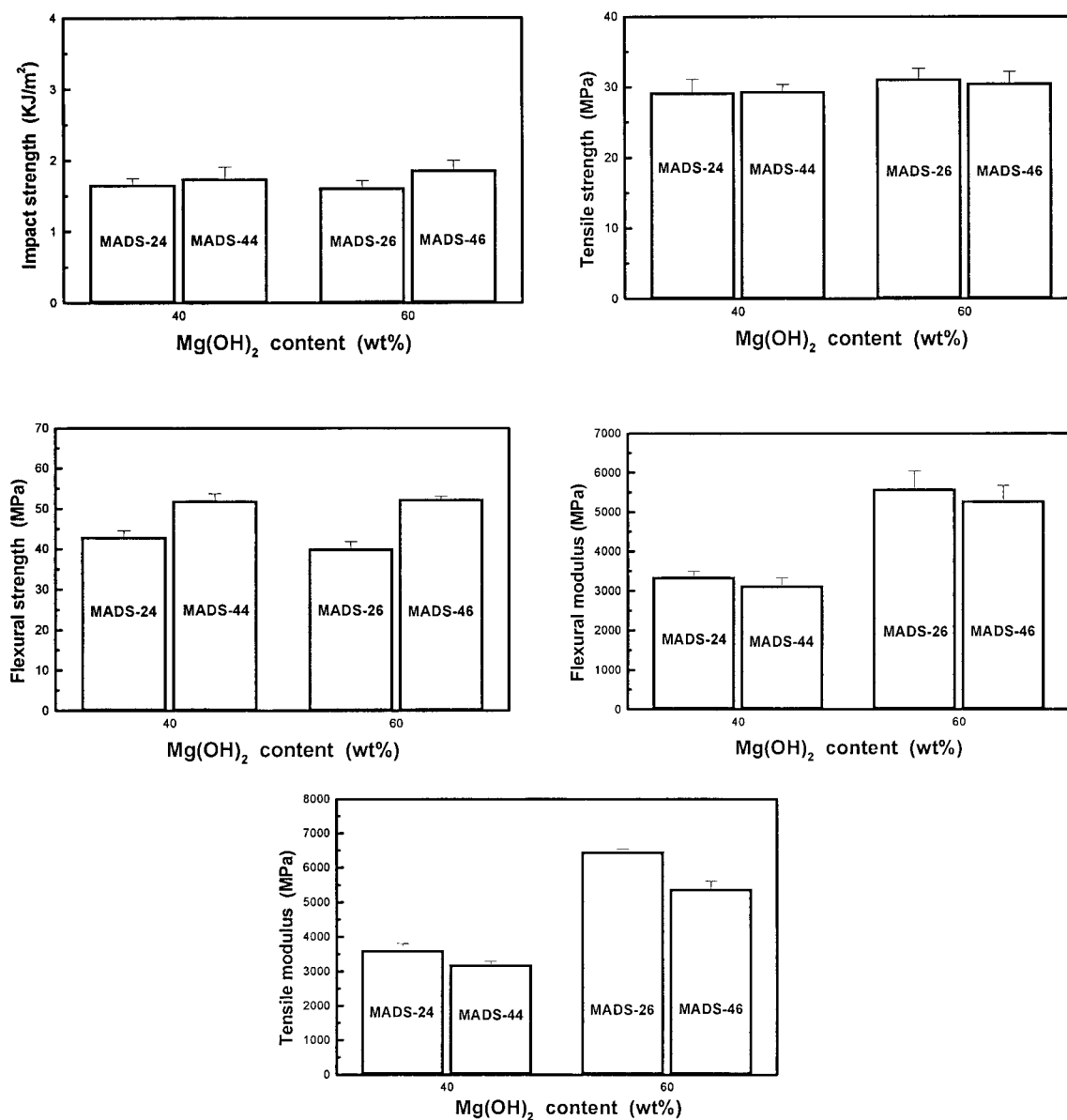
Figure 5 Mechanical properties of $Mg(OH)_2$/PP composites modified by the formation of *in situ* FPP in the presence of a stabilizer.

modulus which was determined by the $Mg(OH)_2$ content, increasing the $Mg(OH)_2$ content from 40 to 60 wt % hardly influenced the strength of composites due to the improvement of the interface adhesion between $Mg(OH)_2$ and PP by the formation of the *in situ* FPP.

When the AA content was increased to 4 phr, both the notched IS and TS of MAD-46, containing 60 wt % $Mg(OH)_2$, were higher than that of MAD-26. In the absence of DCP, the strength of composites declined with increasing AA content. In the presence of DCP, opposite results were obtained. We propose that in the absence of DCP, the formation of macroradials depended on thermal degradation of PP during the preparation process of composites. A little *in situ* FPP was formed. Excessive AA monomer homopolymer-

ized and existed at the interface between $Mg(OH)_2$ and PP. In the presence of DCP, a lot of macroradials were yielded. The increase in the AA content increased the formation of FPP in the interface between $Mg(OH)_2$ and PP, leading to enhanced interfacial interactions between the particle and matrix and an effective improvement in the strength of the composites.

From the FM and TM of the MA and MAD series, we could see that modulus decreased with increasing AA content. We suggest that the formation of *in situ* FPP in the interface between $Mg(OH)_2$ and PP produced a soft interface that was easy to deform and that dissipated the impact energy under high-speed impact. Therefore, the notched IS of MAD-46 was higher than that of MAD-26.

Effect of the oxidation resistant

Figure 5 shows the mechanical properties of Mg(OH)$_2$/PP composites modified by the formation of *in situ* FPP in the presence of an oxidation resistant. Figure 5 shows that compared with MAD-24 at an equivalent DCP content, the notched IS of Mg(OH)$_2$/PP composites (MADS-26) in the presence of oxidation resistant were slightly improved. However, no change in FS and TS was obtained. The notched IS, FS, and TS of MADS-26 all increased. However, for Mg(OH)$_2$/PP composites (MADS-46), the oxidation resistant exhibited no effect on mechanical properties.

The results indicated that at an equivalent DCP content, the effect of oxidation resistant on the mechanical properties of Mg(OH)$_2$/PP composites modified by 2 phr AA was larger than that by 4 phr AA. The thermal decomposition of DCP produced a free radial, whereas the oxidation resistant captured the free radial. PP grafted with AA depended on the amount of the free radial and caused a competition between the AA grafting into the macroradial of PP and the oxidation resistant capturing the free radial. In accord with these results, we suggest that AA reacting with PP macroradial was faster. The free radial produced by DCP induced the decomposition of PP to produce the macroradial of PP. The AA grafted into the macroraddial of PP and FPP was formed. The excess of free radial reacted with oxidation resistant. When the AA content was increased to 4 phr, it is possible that all the PP macroradial produced by DCP grafted with AA, and no excessive free radial existed. Hence, the addition of oxidation resistant produced no effect on the mechanical properties of Mg(OH)$_2$/PP composites modified by the formation of *in situ* FPP.

CONCLUSIONS

The addition of Mg(OH)$_2$ markedly reduced the mechanical properties of PP. The extent of reduction in the notched IS of PP was higher than that in FS and TS. However, TM and FM increased with increasing Mg(OH)$_2$ content. The addition of FPP facilitated the improvement in the FS and TS of Mg(OH)$_2$/PP composites. The higher the Mg(OH)$_2$ content was, the more significant the effect of FPP was. The incorporation of AA resulted in further increased mechanical properties, in particular the FS, TS, and notched IS of Mg(OH)$_2$/PP composites containing high levels of Mg(OH)$_2$. Hence we were able to increase the

Mg(OH)$_2$ content while retaining the mechanical properties of composites. This not only improved the mechanical properties but also increased the flame retardance of Mg(OH)$_2$/PP composites. Although the mechanical properties of composites modified by the formation of *in situ* FPP were lower than those of composites modified only by the addition of AA in the absence of DCP, the mechanical properties did not decline with increased Mg(OH)$_2$ content. Moreover, the mechanical properties increased with increasing AA content. The addition of an oxidation resistant did not produce much effect on the mechanical properties of the modified Mg(OH)$_2$/PP composites.

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