

# Non-isothermal crystallization behavior of PP/Mg(OH)<sub>2</sub> composites modified by different compatibilizers

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## Abstract

Polypropylene (PP) blends with three kinds of grafted macromolecules, PP/Mg(OH)<sub>2</sub> composites and its composites modified by different compatibilizers were prepared by melt extrusion in a twin screw extruder. The crystallization and melting behaviors of PP blends and PP/Mg(OH)<sub>2</sub> composites were investigated with differential scanning calorimeter (DSC). The results indicated that addition of PP-g-MA has no influence on the crystallization temperature of PP, but POE-g-MA and EVA-g-MA decrease the crystallization temperature of PP. For PP/Mg(OH)<sub>2</sub> composites, addition of Mg(OH)<sub>2</sub> increases the crystallization temperature of PP and induces the formation of  $\beta$ -crystal in PP matrix. For PP/Mg(OH)<sub>2</sub> composites modified by different compatibilizers, the synergism effect of heterogeneous nucleation of Mg(OH)<sub>2</sub> and PP-g-MA or POE-g-MA further increases the crystallization temperatures. For PP/Mg(OH)<sub>2</sub> composites modified by EVA-g-MA, the addition of EVA-g-MA to PP/Mg(OH)<sub>2</sub> composite has little effect on the crystallization and melting behavior of PP. It is suggested the synergism effect of heterogeneous nucleation of Mg(OH)<sub>2</sub> and compatibilizer depends upon the compatibilization between PP and compatibilizer. The increase in crystallization temperatures of PP in PP/Mg(OH)<sub>2</sub>/compatibilizer facilitates the formation of  $\alpha$ -PP, but weakens the formation of  $\beta$ -PP.

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**Keywords:** Polypropylene; Mg(OH)<sub>2</sub>; Compatibilizer; Crystallization and melting behaviors

## 1. Introduction

Polypropylene (PP) is a most common commodity plastic, which is of practical use in many areas, such as home appliances, automotive, construction, and other industrial applications. However, the poor flame resistance hinders its practical application in some fields. Compounding PP with flame retardants, such as magnesium hydroxide (Mg(OH)<sub>2</sub>), has been of wide interest and it has been an effect way to improve the flame resistance of PP. But the unfortunate consequence is a reduction in mechanical properties [1–5].

Interfacial adhesion is regarded as the most important factor responsible for deterioration in the mechanical properties of polymer composites. Mg(OH)<sub>2</sub> has highly polar hydrophilic surfaces, whereas the polymers (e.g., PP) are often non-polar and hydrophobic. When they compound each other, interfacial

adhesion between the filler surface and the matrix is poor. Many studies [6,7] have been focused on the modification of the filler surface through coating. Hornsby and Watson [7] studied the mechanical behavior of PP composites containing Mg(OH)<sub>2</sub>. They found that very significant improvements in toughness were obtained when high levels of magnesium stearate were used to ensure complete coverage of the Mg(OH)<sub>2</sub> surface. Modification of the filler surface through coating alters the properties of the surface, and the degree of interfacial adhesion may thereby be improved.

The interfacial adhesion can also influence the crystalline of PP in mineral-filled PP composites. Generally, mineral fillers can influence the crystallization process of the PP matrix by acting as heterogeneous nuclei. The heterogeneous nucleation leads to the increase in nucleation and crystallization rate. The improvement of the interfacial adhesion between polymer and filler can reduce the free energy of a nucleation process, which is favorable to the nucleation process at the phase boundary [8]. Lin et al. [9] studied the crystallization and melting behavior of PP/Mg(OH)<sub>2</sub> composites modified by acrylic acid grafting

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PP (PP-g-AA). Addition of PP-g-AA into PP/Mg(OH)<sub>2</sub> composites further enhanced the crystallization temperatures of PP. They suggested that there is an activation of PP-g-AA to the heterogeneous nucleation effect of Mg(OH)<sub>2</sub> surface. At the same time, there are many experiments that showed that the filler's nucleation action was reduced after surface modification, especially when modified by a coupling agent having a long fatty chain [10]. Velasco et al. [11] reported that the nucleation activity of Mg(OH)<sub>2</sub> particles on PP was reduced in coated grades of Mg(OH)<sub>2</sub>. The employed coatings worked isolating and preserving particles surface from direct interaction with polymer chains.

Above investigations indicated that the crystallization behavior of PP in PP/Mg(OH)<sub>2</sub> composite depends upon the interfacial interaction between Mg(OH)<sub>2</sub> and PP. In order to investigate the interfacial interaction between Mg(OH)<sub>2</sub> and PP, and between PP and modifier, on crystallization behavior of PP, a series of the compatibilizer/PP blends, PP/Mg(OH)<sub>2</sub> composites and modified PP/Mg(OH)<sub>2</sub> composites were prepared. The compatibilizers, including PP-g-MA, POE-g-MA and EVA-g-MA, have the same polar groups but different backbones. The same polar groups will result in the same interfacial interaction between Mg(OH)<sub>2</sub> and compatibilizer. Different compatibilizers with different backbones will result in different compatibility with PP and interfacial interaction between Mg(OH)<sub>2</sub> and PP. The different interfacial interaction among the PP, Mg(OH)<sub>2</sub> and compatibilizer will result in a change in the crystallization behavior of PP in PP/Mg(OH)<sub>2</sub> composite. In this paper, DSC and WAXD were used to investigate the structure and crystallization and melting behaviors of PP blends with different compatibilizers and PP/Mg(OH)<sub>2</sub> composites modified by different compatibilizers.

## 2. Experimental

### 2.1. Materials

Polypropylene was EPS.30R, supplied by Dushanzi Pcg (Xinjiang, China). This is a blend of isotactic polypropylene and propylene–ethylene rubber. Magnesium hydroxide, employed as filler, was a high purity untreated grade (1.8–2.4 μm) supplied by Foshan Jinge Firefighting materials Co. (Foshan, China). PP-g-

MA, POE-g-MA and EVA-g-MA were supplied by Guangzhou Lushan Chemical Materials Co. (Guangzhou, China). Characteristics of PP and compatibilizers are given in Table 1.

### 2.2. Sample preparation

All materials were dried in an oven at 60 °C for 12 h before melting extrusion. In the experiment, PP/Mg(OH)<sub>2</sub>/compatibilizer composites were prepared by melt-mixing all the components in a one-step procedure using a Berstoff ZE25A corotating twin-screw extruder ( $L/D=40$ ,  $D=35.5$  mm) at a rotating speed of 250 rpm. The blending temperature profile is 200/190/200/200/220/210/200 °C. A summary of the compositions for the different PP/Mg(OH)<sub>2</sub>/compatibilizers composites used in this study is given in Table 2.

### 2.3. DSC measurements

DSC measurements were made on a Perkin-Elmer DSC-7 thermal system (Perkin-Elmer Cetus Instruments, Norwalk, CT) in nitrogen atmosphere with 10 °C/min heating and cooling rate. Calibration was performed using pure indium at the same heating rate. Each sample weighed about 4–6 mg. The samples were heated from 50 to 220 °C, melted at 220 °C for 3 min, and then cooled to 50 °C, followed by reheating to 220 °C for the second heating run. The crystallization and melting parameters were recorded from the cooling and reheating scans.

The percentage of (β-phase,  $\Phi_\beta$ , can be obtained from the crystallinities of the α-phase and β-phase according to Ref. [12]

$$\Phi_\beta (\%) = \frac{X_\beta}{X_\alpha + X_\beta} \times 100 \quad (1)$$

$$X_i (\%) = \frac{\Delta H_i}{\Delta H_i^\theta} \times 100 \quad (2)$$

where  $X_\alpha$  and  $X_\beta$  are the crystallinities of the α- and β-phase, respectively, which can be calculated separately according to Eq. (2), where  $\Delta H_i$  is the calibrated specific fusion heat of either the α- or the β-form,  $\Delta H_i^\theta$  is the standard fusion heat of the α- and the β-crystals of iPP, being 178 J/g and 170 J/g, respectively [13]. Because the DSC curves of some samples exhibited both α- and β-fusion peaks, the specific fusion heats for α- and β-phase were determined according to the following calibration

Table 1  
Characteristics of PP and compatibilizers

Materials	Mark	Properties
Polypropylene (EPS.30R)	PP	ethylene content 2.87%, $M_n=52,000$ , $M_w/M_n=9.2$ , $MFI=0.88$ g/10 min <sup>a</sup> , and $T_c^p=114.5$ °C <sup>b</sup>
Isotactic polypropylene grafted with maleic anhydride	PP-g-MA	MA Grafting ratio 1.0%, $T_c^p=110.6$ °C <sup>b</sup> $MFI>15$ g/10 min <sup>a</sup>
Ethylene-octene copolymer grafted with maleic anhydride	POE-g-MA	MA Grafting ratio 1.1%, octene content 24–26% $T_g=-54.6$ °C <sup>c</sup> , and $MFI=0.72$ g/10 min <sup>a</sup>
Ethylene-vinyl acetate copolymer grafted with maleic anhydride	EVA-g-MA	MA grafting ratio 1.0%, VA content 28%, $T_g=-28.8$ °C <sup>c</sup> , $T_c^p=52.2$ °C <sup>b</sup> , $MFI=2.46$ g/10 min <sup>a</sup>

<sup>a</sup> 190 °C, 2.160 kgf.

<sup>b</sup> Cooling from 220 °C to room temperature (–10 °C/min).

<sup>c</sup> Melting from –90 to 220 °C.

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