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ORIGINAL PAPER

Thermal, structural and dynamical mechanical properties of hollow glass sphere-reinforced polypropylene composites

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Abstract

In the present study, the effect of maleic anhydride grafted polypropylene (MAPP) ratio on the thermal and dynamic mechanical properties of polypropylene/hollow glass spheres (PP/HGS) composites was investigated. Hollow glass spheres content was constant at 20 wt % and MA-g-PP was set at four different levels: 1, 5, 10 and 15 wt % in PP/HGS composites. The mechanical properties of polymer composites as a function of temperature were measured using dynamic mechanic analyzer. The oxidation induction times tests and thermal properties were carried out in thermogravimetric differential thermal analyzer in order to determine thermo-oxidative performance and thermal stability of the composites. Moreover, the x-ray diffraction (XRD), the Fourier transform infrared spectrophotometry and the scanning electron microscopy were used to analyze the structural characteristics of the polymer composites. The results demonstrated that the increased MAPP content enhanced dynamic mechanical and thermal oxidative aging properties of PP /HGS composites. The addition of MAPP did not alter the crystal structure according to XRD results.

Keywords Polypropylene · Glass spheres · TGA · DMA · FT-IR · XRD · SEM · MAPP

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Introduction

In recent years, hollow glass spheres (HGS) have been widely used in the field of composite production due to their lightweight property, high stiffness, low thermal conductivity, and electrical properties. Moreover, addition of HGS as fillers into polymers has been an accepted way of the enhancing the properties of the composites and reduce the cost. Glass microsphere filled polymer composites are utilized for electronic devices, subsea pipeline insulation and sporting goods. In addition, these composites are applied in the aviation, naval and automotive industries in order to meet the competing performance requirements of low weight and high strength. Lightweightness is important because light-weight components provide superior carrying capacity, better fuel efficiency and faster travel rates for vehicles. Many manufacturers have utilized polymer composites for body panels, seat backs, headliners, package trays, dashboards, and interior parts in automotive industries [1–4].

The properties of composites are also effected by the nature of bonding between the filler and the matrix. MAPP is one of the most common compatibilizers in the polymer composites production. In this study, MA-g- PP was used as a compatibilizer in order to achieve a strong interfacial bonding between HGS and PP and homogenous dispersion of hollow glass spheres in PP. This brought enhanced thermal and dynamic mechanical properties of PP/HGS composites.

In the literature, there are several studies on polymer composites by the addition of HGS at varying concentrations. Many researchers generally focused on the mechanical properties of HGS-reinforced polymers. Some of them investigated the influence of HGS reducing fire hazard of thermoplastic composites and the thermal conductivity [5–8]. Only a few of them investigated the thermal and mechanical properties of thermoplastic composites reinforced with HGS in the absence and presence of compatibilizer. Although a few studies have been conducted on the effect of MAPP in polymer composites [9–14], there have not been any studies reporting a direct comparison among PP/HGS/MAPP composites with respect to thermomechanical properties by means of OIT.

Celebi studied the effects of hollow glass microspheres (HGM) content and silane modification on the performance of PP composites. She found out that the use of silane influences the mechanical properties of composites; however, it has little or no effect on the thermal conductivity values. She used 3-(Trimethoxysilyl) propyl methacrylate (TMSM) silane coupling agent. She also revealed that the surface modification of HGM enhances the interfacial region between HGM and polymer matrix [9].

Sosiati et al. added various amounts (5, 10 and 15 wt%) of MAPP to the untreated sisal/PP and alkali-treated sisal/PP composites. According to their observations, tensile strength of the untreated sisal/PP composite containing 50 wt% of fiber was higher than that of 30 wt% fiber content. They also found out best interfacial bonding between sisal fiber and the PP matrix was observed at 5 wt% MAPP loading [10].

Kulkarni et al. investigated the effect of variation in melt flow index (MFI) of maleic anhydride-grafted polypropylene (PP-g-MAH) on various properties of fly

ash (FA)-filled polypropylene (PP) composites. They found out that the composites prepared using three polymeric compatibilizing agents, PP-g-MAH (3 wt%), with different MFI and very high MAH content showed better dispersion characteristics. They also observed that the use of PP-g-MAH (3 wt%) as polymeric coupling agent provides improvement in mechanical, thermal and morphological properties of filled polymers [11].

Kubat et al. investigated the dynamic-mechanical properties of high density polyethylene filled with 20% by volume of untreated glass spheres or glass spheres treated with a silane-based coupling agent as a function of temperature and imposed tensile deformation. They found out that surface treatment of filler particles influences the mechanical loss factor [12].

Patankar et al. studied on the mechanical properties of composites which consisted of sodium borosilicate hollow glass microspheres (HGM) as filler in high density polyethylene matrix with and without polyethylene-graft-maleic anhydride (PE-g-MAH). According to tension tests, they observed addition of PE-g-MAH enhanced the mechanical properties of HDPE/ HGM composites [13]. In another study by Patankar et al., they found out that PE-g-MAH didn't influence the relaxation behavior, crystallinity and thermal conductivity of the HDPE/ HGM composites significantly [14].

This paper describes an attempt to determine the effect of compatibilizer amount on the thermal and dynamic mechanical properties of PP/HGS composites. MAPP was used for the production of polymer composites in 1 wt.%, 5 wt.%, 10 wt.% and 15 wt.%. The produced polymer composites were structurally defined by performing the XRD and FTIR characterizations. The oxidation induction times tests (OIT) were carried out in order to predict thermal oxidative aging properties. In addition, the composites were characterized by the dynamic mechanical analyses in order to observe the mechanical properties depending on the temperature of the composites. Moreover, SEM was used to examine the morphology of the composites. This study is the first that OIT tests were performed on glass microsphere filled polypropylene composites.

Experimental

Composites of hollow glass microsphere filled polypropylene matrix were prepared. Five different groups were obtained. Compositions of PP/HGS/MAPP composites are given in Table 1. PP (Moplen EP 3307) supplied by Lyondell Basell. Its density is 0.900 g/cm^3, MFI value is 15 g/10 min (230 °C, 2,16 kg) and its heat deflection temperature (0.45 MPa, unannealed) is 95.0 °C. Glass spheres (MinTron 7) were supplied by RockTron International (Bristol, United Kingdom). Its moisture content and particle specific gravity are < 0.5% and 2.2–2.4 g/cm^3, respectively. Particle size distribution (d50) is 20–35 μ. Maleic anhydride modified polypropylene (MAPP) under the trade name Bondyram 1001 CN procured from Polyram Plastic Industries LTD, Israel, is used as the coupling agent. Bondyram 1001CN is a maleic anhydride modified co-polymer polypropylene recommended for coupling of polypropylene

Table 1 Composition of the PP/GS/MAPP polymer composites formulations

Groups	Polypropylene (wt%)	Glass spheres (wt%)	Maleic Anhydride-g-PP
1	80	20	–
2	79	20	1
3	75	20	5
4	70	20	10
5	65	20	15

composites. Its density is 0.90 g/cm^3, MFI value is 100 g/10 min (D-1238, 190 °C/2.16 kg) and its melting point is 160 °C.

Polypropylene, glass spheres and maleic anhydride were dried overnight at 105 °C for 24 h in a vacuum oven prior to melt blending. Mechanical premixing of solid compositions was done using a LB-5601 liquid–solids blender (The Patterson-Kelley Co., Inc. USA) brand batch blender for 20 min. Samples with various proportions of PP/HGS/MAPP composites were produced between 190 and 210 °C at 15 bar pressure, and a rotation rate of 20 rpm, with a Microsan extruder (Microsan Instrument Inc. Turkey). Polymer composites were also dried in vacuum oven at 105 °C for 24 h after extrusion. Subsequently, test samples were manufactured by injection molding. Injection temperature was 190–210 °C; pressure was 90–100 bar and screw speed was 20 rpm.

Oxidation induction times tests (OIT)

OIT tests were performed using the EXSTAR TG/DTA 6300 (Seiko, Tokyo, Japan) in a nitrogen atmosphere from ambient temperature to 200 °C at a heating rate of 5 °C/min and then in an oxygen atmosphere at 200 °C for 100 min.

Fourier transform Infrared Spectroscopy (FTIR)

Fourier transform infrared (FTIR) spectroscopy was performed using a FT-IR Spectrum 100 (PerkinElmer, Waltham, MA). The samples were scanned over the frequency range of 650–4000 cm^{-1} wave number range in the transmittance mode at room temperature.

Dynamic mechanical analysis (DMA)

Dynamic mechanical properties of the polymer composites were observed by a DMA 8000 (PerkinElmer, USA). The tests were carried out by using a frequency of 1 Hz and a heating rate of 5 °C/min from room temperature to 150 °C. Tests were performed using dual cantilever bending deformation mode.

Surface morphology

An EVO LS 10 SEM (Zeiss, Oberkochen, Germany) was used at 10 kV in order to examine the effect of filler content and interfacial bond strength in the different products. SEM images were taken in order to investigate the distribution of microstructure.

X-ray diffraction analysis (XRD)

The structural characterizations of composites were investigated by using XRD. A Panalytical X'Pert PRO X-ray diffractometer (Malvern, United Kingdom) was used for the measurements utilized monochromatic CuKα radiation operated at 45 kV and 40 mA at room temperature.

Thermogravimetric analysis (TGA)

The thermal properties of the composites were characterized by thermogravimetric analysis (TGA) technique in the temperature range of 25–800 °C. TGA held by a TG/DTA 6300 **(Seiko Exstar, Japan)** in nitrogen stream at a heating rate of 10 °C/min.

Results and discussion

FTIR analysis

In this study, FTIR is used to identify the prepared samples and observe influence of MAPP content on structural properties. The FTIR transmittance spectra of the composites with different concentrations (1.0, 5.0, 10.0, and 15.0 wt%) and without MAPP are shown in Fig. 1. There are some changes in spectra of PP/HGS composites visible with increased ratio of MAPP. The bands at 1375 cm^{-1}, 1451 cm^{-1}, 1166 cm^{-1} and 998 cm^{-1} are characteristic peaks of polypropylene which exhibit the -CH_3 bending vibration, –CH_2 bending vibration, –CH_3 symmetric deformation vibration and –CH_3 rocking vibration, respectively [15]. In the presence of MAPP, transmittance band at 1748 cm^{-1} observed by FTIR spectra corresponds to the carbonyl groups (C = O) of maleic anhydride (MA). For all samples, the bands at 804, 998 and 1166 cm^{-1} are observed due to vibrations of silica lattice in glass sphere. A broad band in the region of 840 cm^{-1} to 1166 cm^{-1} is observed for PP/HGS composites with the addition of MAPP. This band can be assigned to symmetric stretching vibrations of the hydrogen atoms with the oxygen atom increased. FTIR spectrum is the most important indicator of the presence of PP, MAPP and HGS.

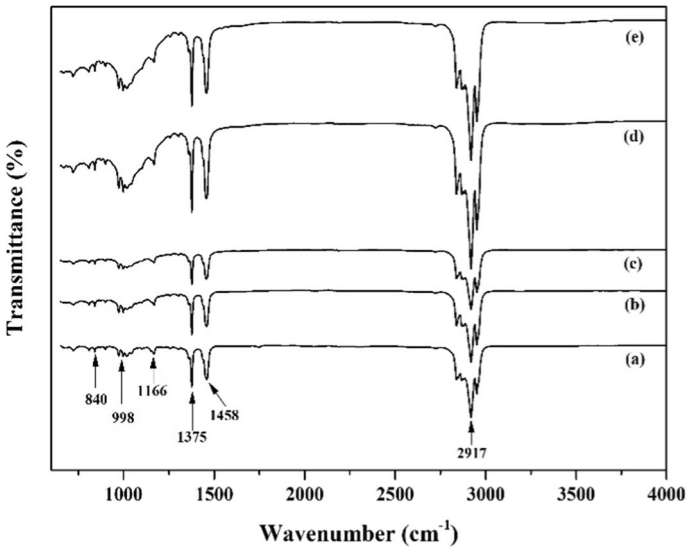

Fig. 1 Comparison of the FTIR spectra of **a** PP/HGS; **b** PP/HGS with 1% MAPP; **c** PP/HGS with 5% MAPP **d** PP/HGS with 10% MAPP **e** PP/HGS with 15% MAPP

Morphological characterization

Figure 2 shows the interaction between HGS and the PP matrix with the addition of MAPP. As can be seen in Fig. 2a, in the absence of MAPP, the dispersion of HGS is not homogeneous and there are voids. However, as the ratio of MAPP increases, the distribution is more homogeneous and uniform. With increasing MAPP content, the interaction between HGS and PP matrix becomes stronger. It is concluded that the interaction between them is improved. Also, most of the microspheres were observed to be embedded in the PP matrix with a strong interface adhesion. This result further confirmed that MAPP can improve the interfacial compatibility of PP/HGS composites. XRD results showed that the addition of MAPP did not alter the crystal structure. SEM images illustrated the enhancement in the morphology of the composites and pointed out that the improvement in the results of OIT and DMA analyses was depend on morphology [16].

Crystalline structure of the samples

The crystalline structure of the mixture components greatly affects the different mechanical properties and fracture toughness of the mixtures. Because of this, studying the effect of the blending process and the dispersed phase compatibilization process on the crystalline structure of the matrix material (PP) is very important.

As shown in Fig. 3, X-ray diffraction (XRD) patterns of the PP / HGS composites were recorded by monitoring the diffraction angles (2θ) from 5° to 50° on

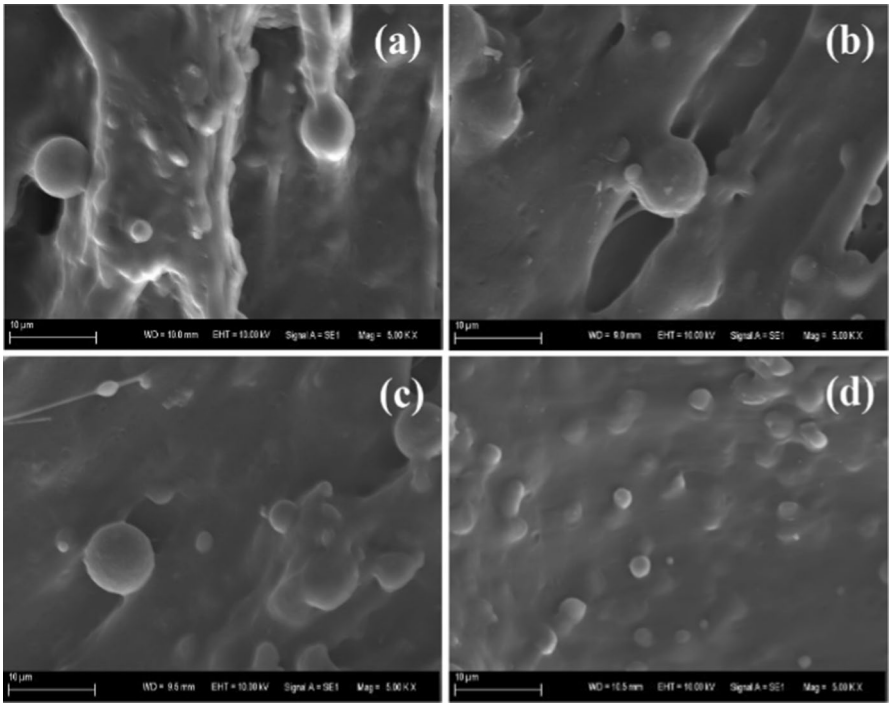

Fig. 2 SEM of the surface of **a** PP/HGS; **b** PP/HGS with 1% MAPP; **c** PP/HGS with 5% MAPP (d) PP/HGS with 15% MAPP

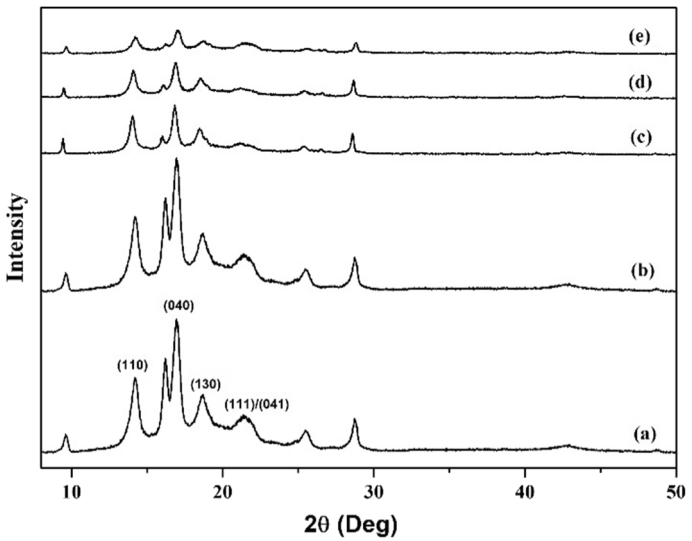

Fig. 3 XRD patterns of **a** PP/HGS; **b** PP/HGS with 1% MAPP; **c** PP/HGS with 5% MAPP **d** PP/HGS with 10% MAPP **e** PP/HGS with 15% MAPP

the diffractometer. Five different peaks at 2Θ of 14.27°, 16.73°, 18.73°, 21.25° and 21.7° corresponding to the (110), (040), (130), (131) and (041) planes were observed in the XRD spectrum of the PP/ HGS composites [17]. The diffraction peaks of PP / HGS composites were gradually weakened when MAPP content increased. The intensity of these peaks in PP/HGS with MAPP is lower than those in the PP /HGS composites without MAPP. New phases do not appear with addition of MAPP. This means that the crystal structure does not change, also indicating that the MA was homogeneously dispersed in the PP/HGS matrix [18]. When the crystal structure does not change, the differences in mechanical properties and/or fracture behavior are due to the variation in morphology. In recent years, there have been studies proving this [19].

Crystallinity significantly affects mechanical and physical properties of materials. XRD is routinely used to determine phase identification as well as the percent crystallinity of materials. Percent crystallinity is represented by the following equation:

$$\text{Percent Crystallinity} = \frac{I_{\text{crystalline}}}{I_{\text{crystalline}} + I_{\text{amorphous}}} \times 100 \tag{1}$$

The percent crystallinity decreases with increasing amount of MAPP due to the results obtained from Eq. 1 [20]. As the PP ratio decreased from 80 to 65%, the percent crystallinity values decreased from 99.28% to 91.89%, respectively (Fig. 4). As increasing amount of low crystalline MA in the matrix, the amount of high crystalline PP decreased. The decrease of the crystalline ratio of the matrix can be explained by the decrease of PP amount in the matrix. The obtained values are compatible with the decrease in the intensity of the peaks in the XRD spectrum [21, 22].

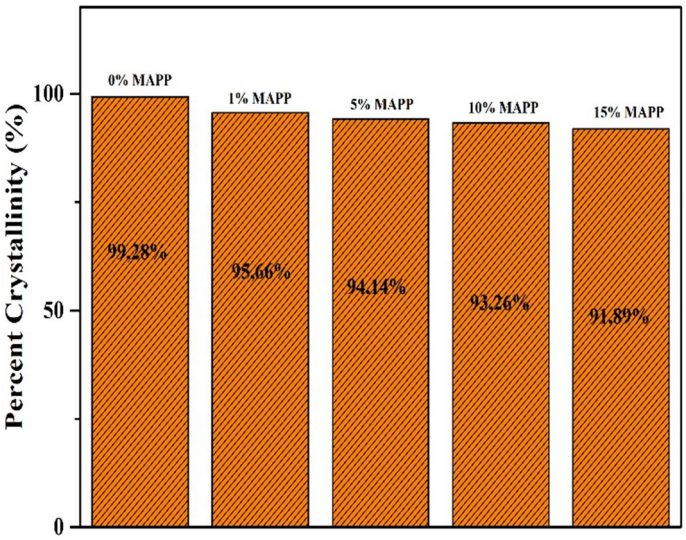

Fig. 4 Percent crystallinity of PP/HGS composites which contains MAPP with different percentages (0, 1, 5, 10 and 15 wt%)

DMA

Storage modulus (E') informs the elastic response of a material. The measurements of storage modulus carried out under 1 Hz as a function of temperature of fabricated composites are shown in Fig. 5. The highest storage modulus, 802.2 MPa, was obtained for composites with 15 wt% MAPP. An increase of 69% was seen compared to 474.5 MPa as the highest storage modulus of PP/HGS composites without MAPP. Since the addition of MAPP improves interface adhesion between PP matrix and HGS, load transfer at the interface has improved. The storage modulus of all composites decreased with the increase in temperature. The DMA results showed that the addition of MAPP increased the storage modulus of the composites resulted in higher stress transfer at the interface.

OIT

OIT tests have been commonly used as an effective and easy method to determine the thermal oxidative aging properties of polymer composites. Generally, the higher OIT value of the composite is, the better its resistance to oxidative degradation. Figure 6 shows the resistance to thermal oxidative aging of PP/HGS composites with varying wt.% MAPP. Although the PP thermal oxidation stability was lower (OIT = 37.2 min), the anti-oxidative stability was greatly increased with the increased MAPP ratio. Notably, MAPP had a positive effect on anti-thermal oxidative aging properties of the composites and increased the OIT values. This can be explained by the strengthening of bonds between glass spheres and PP and increasing homogeneity of the surface. Thus, it is possible

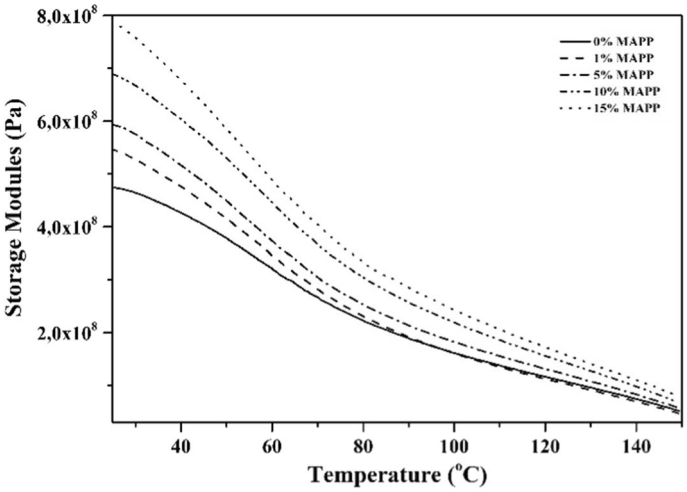

Fig. 5 Temperature dependence of storage modulus of PP/HGS composites which contains MAPP with different percentages (0, 1, 5, 10 and 15 wt%)

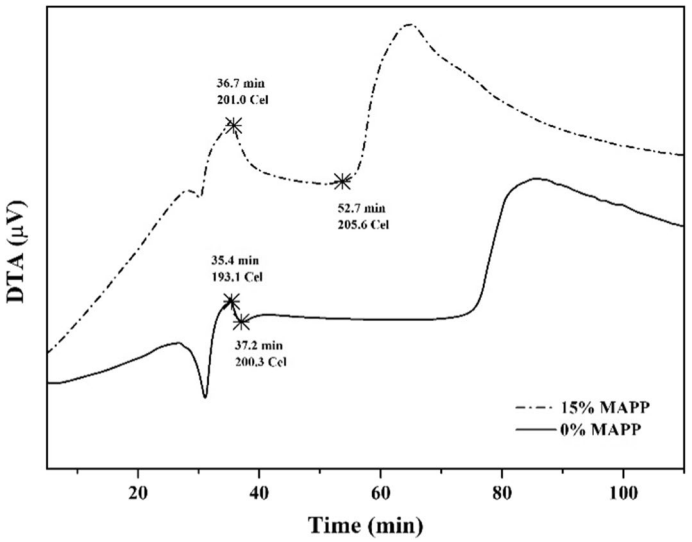

Fig. 6 OIT test results of PP/HGS composites which contains MAPP with different percentages (0–15 wt%)

to prepare the oxygen-resistant material by blending PP and glass spheres with suitable amount of compatibilizer in order to provide lightweightness [23, 24].

TGA

Thermal stability of materials is quite an important matter for so many applications. Materials with high thermal stability can maintain their mechanical integrity when they are exposed to high temperatures. In this study, thermal properties of PP/HGS composites with different concentrations (0.0, 5.0 and 15.0 wt%) of MAPP were evaluated in detail by TGA. The effect of the MAPP on the degradation temperature of PP/HGS composites is presented in Fig. 7. As seen from Fig. 7, MAPP has an effect on the thermal stability of PP/HGS composites. The ratio of decomposition at 300–500 °C decreases with increasing content of MAPP. With the increasing temperature, weight losses were 85.13% for PP/HGS, 80.39% at 5 wt% of MAPP loading and 79.35% at 15wt % of MAPP loading. Thus, results of the TGA experiments suggest that MAPP enhances the thermal stability of PP/HGS composites compared to untreated PP. Thus, results of the TGA experiments suggest that MAPP enhances the thermal stability of PP/HGS composites in comparison with untreated PP. The above observations are in good agreement with the study of López et al. MAPP increases thermal stability of composites. It may be caused by good interfacial bonding between HGS and the PP matrix [25].

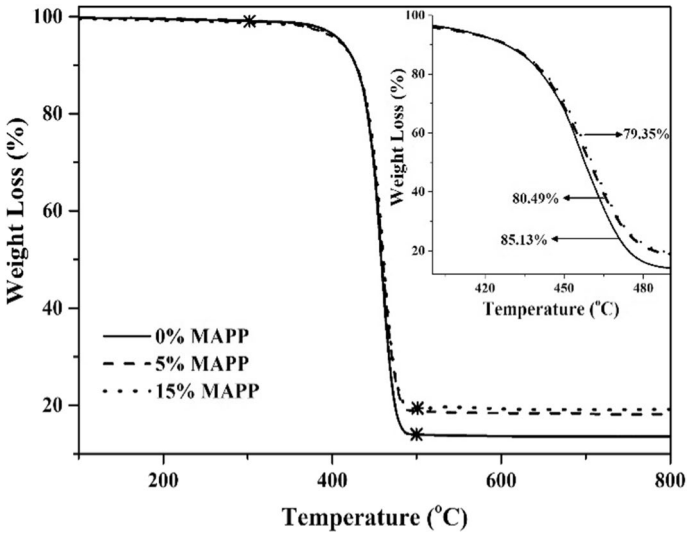

Fig. 7 TGA thermograms of PP/HGS composites which contains MAPP with different percentages (0, 5 and 15 wt%)

Conclusion

In this study the effects of MAPP on the performance of PP/HGS composites are investigated. The results show that MAPP content influences the mechanical and thermal oxidative aging properties of composites. In the absence of MAPP, interfacial bonding between the reinforcement and the matrix is not strong enough so cavities and nonhomogeneous distribution are present. The use of MAPP has positive impact on the dynamical mechanical and thermal oxidative aging properties of composites. However, it has no effect on the crystal structure. XRD patterns show that the crystallinity of the PP/HGS blends decreases with increasing MA content. This change is due to the decrease in the amount of high crystalline PP in the matrix. SEM analyses show that a stronger interaction exists between HGS and polymer matrix with increasing MAPP content. This state is resulted in most microspheres being embedded more into the PP matrix. Also, MA has a positive effect on anti-thermal oxidative aging properties of the composites and increases the OIT values. Besides, MAPP allows composites with better thermal stability to be formed. By enhancement of interfacial adhesion between PP and HGS, coupling agent increases thermal stability. As a result, the improvement in dynamic mechanical and thermal properties of PP /HGS composites by MAPP addition were not derived from crystal structure. In fact, this is related to morphological enhancement. Obviously, these composites can be widely used in the development of next generation automotive, naval and spacecraft materials. This work will throw light on researchers for developing new materials in application areas where keeping weight to a minimum is important.

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