A Study of the Impact of Iron Content on the Thermal Response of the sPP/Fe Composites

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Abstract-A set of syndiotactic polypropylene/iron (sPP/Fe) composite samples were manufactured with the extrusion technique to study the impact of iron content on the thermal behavior of sPP/Fe composites in the melt phase. The dosage of iron contents varied from 0 to 8%. Melting point (T_m), crystallization temperature (T_c), and thermal degradation temperature (T_d) were measured by Differential Scanning Calorimetry (DSC) and Thermal Gravimetric Analysis (TGA) for each composite sample. Thermal temperatures (T_m, T_c, and T_d) increased with increasing the iron contents due to the enhancement of the strength and thermal stability of the sample. This investigation is a validated fact that fillers (iron) alter both the macroscopic and microscopic properties of the polymer composites.

Keywords-thermal properties; DSC; TGA; polymer composites; thermal degradation temperature

I. INTRODUCTION

Polymer composites have become one of the most important manufactured materials and can be industrialized by the incorporation of filler (natural or synthetic) into the polymer to improve its targeted properties [1-5]. Polymer composites with different types of fillers and combinations are widely used with improved mechanical, thermal, barrier and fire resistant properties for different applications [6-8]. However, the end desired microstructure and macrostructure properties of polymer composites are greatly dependent on the nature, amount, geometry and interfacial interactions of the components [9]. The thermal properties are highly dependent and sensitive to microstructure properties of polymer and polymer composites [10-14]. Therefore, the relationship of the thermal response and microstructure properties is an excellent key in both the process and quality control to realize and develop new materials with the desired properties. Thermal properties can be used in the understanding of molecular structure and the link with both microstructure and macrostructure properties [6, 7, 15-18]. Some of the recent scientific advances in the iron/polymer and its properties are mentioned below.

Authors in [19] investigated the thermal properties including thermal conductivity, thermal diffusivity, and specific heat of metal (copper, zinc, iron, and bronze) powder-filled high-density polyethylene composites experimentally in the range of filler content 0-24%. They found a region of low particle content (0-16%), where the particles are embedded homogeneously in the polymer matrix and do not interact with each other. Fillers at higher content regions, tend to accumulate and conductive chains result in a quiche enhancement in thermal conductivity. Authors in [20] studied the thermo-mechanical properties of polymer/metal composites. The Acrylonitrile Butadiene Styrene (ABS) was used as a matrix and copper and iron particles as fillers. The effect of metal powder was studied to confirm the effects of metal particles on the thermo-mechanical properties of the composites, such as tensile strength and thermal conductivity. It was found that the tensile strength of the composites decreases with increasing loading of the metal particles, while the thermal conductivity of the metal/polymer composite filament was found to improve by increasing metal content. More recently, authors in [21] examined the electrical and thermal conductivity of the ternary epoxy composites (CMs) with two-component fillers, multiwall carbon nanotubes (MWCNT)/TiO_2, and MWCNT/carbonyl iron (Fe). Both electrical and thermal properties were found sensitive to both fillers. However, MWCNT/TiO_2 or MWCNT/Fe into epoxy supports smaller changes in thermal conductivity as compared to the two-phase MWCNT/epoxy composites.

To the best of our knowledge, the understanding of the thermal properties of syndiotactic polypropylene/Iron (sPP/Fe) has not been established by now. Thus, in the present work, sPP/Fe composites with different iron dosages were investigated to study the influence of iron content on the thermal properties of the composites, including melting point temperature (T_m), crystallization temperature (T_c), and thermal degradation temperature (T_d).

II. METHEDOLOGY

A. Experimental Work

1) Materials

Polymer composites were prepared using Syndiotactic polypropylene (sPP) and iron (Fe) with content variety as a

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filler. Zhongwei Industrial Co. Ltd supplied the polymer with 60% degree of syndiotacticity, and Sigma Aldrich provided the iron in powder form. Five samples were prepared with Fe content varying from 0 to 8% with a step size of 2%.

2) Preparation of Polymer Composites

The extrusion technique was used to formulate a set of sPP/Fe composites with different percentages of Fe as shown in Table I. For each sample, at first the required amount of Fe was dried in an oven at 75°C for 24 hours, and then it was accurately weighed. After that, when the extruder reached 250°C, both polypropylene and the required amount of iron entered the extruder for mixing to prepare the composite samples. The size of the prepared polymer-iron composite sample was mechanically reduced to suitable size before completely dried. Then, a film of the composite sample was prepared by compression molding at 170°C and 7Psi pressure for the duration of 1 hour using Hot Press.

B. Analytical Work

Differential Scanning Calorimetry (DSC) and Thermal Gravimetric Analysis (TGA) were conducted to investigate the influence of iron content on the thermal behavior of the sPP/Fe composites.

1) Differential Scanning Calorimetry

The melting temperature (T_m) of all samples was determined by DSC using non-isothermal crystallization tests. Two main cycles (heating and cooling) were used in this technique. In the heating cycle, the sample having a weight of 4mg was heated up to 200°C at a constant heating rate of 10°C/min. After the melting point peak was obtained, the sample was kept under this annealing process for 5 minutes in order to eliminate the crystallinity and remove thermal history. During the cooling cycle, the DSC temperature was decreased up to -130°C with a cooling rate of -10°C/min. Therefore, the peaks of crystallinity and glassy behavior of the composites were observed. Evaluations were made using the instrument software TA-60 to estimate the melting temperature (T_m) and crystallization temperature (T_c) of all the samples.

2) Thermal Gravimetric Analysis

Thermal degradation temperature (T_d) shows the maximum temperature at which a polymer is useful. In this investigation, the thermal degradation temperature of all composites was measured using TGA. This analysis was conducted by observing the drop in the mass of the composite sample. In the beginning of the analysis, the sample was weighed, and then loaded to the equipment and heated up to 600°C. During the heating process, the polymer started to burn at a certain temperature where the fall in the mass of samples was observed. The obtained results were analyzed with TA-60 for each sample.

III. RESULTS AND DISCUSSION

Table I expresses the thermal information of all polymer composites extracted from DSC and TGA. The number shown in the sample ID represents the percentage of sPP and Fe in each composite sample.

TABLE I.   THERMAL TEMPERATURES OF THE COMPOSITE SAMPLES

Sample ID	Fe %	T_c (°C)	T_m(°C)	T_d (°C)
PP-Fe-(100-0)	0	113.96	146.72	360
PP-Fe-(98-02)	2	114.79	147.86	380
PP-Fe-(96-04)	4	115.76	148.62	395
PP-Fe-(94-06)	6	116.51	149.71	405
PP-Fe-(92-08)	8	117.69	150.62	418

A. The Effect of Iron Content on Crystallization Temperature

DSC determines the heat flow as a function of temperature, which is directly related to the latent heat of crystallization. In DSC measurements, the temperature at the peak of the endothermic heat flow represents the crystallization temperature of the composite sample. Fig. 1. Figure 1 illustrates the experimental results of DSC related to the crystallization temperature of each sample. Figure 2 shows the relation between the crystallization temperatures of a set of composite samples with different Fe content. It was found that the increase in Fe content increases the crystallization temperature and hence the crystallinity of the composites due to the increase of the crystalline phase.
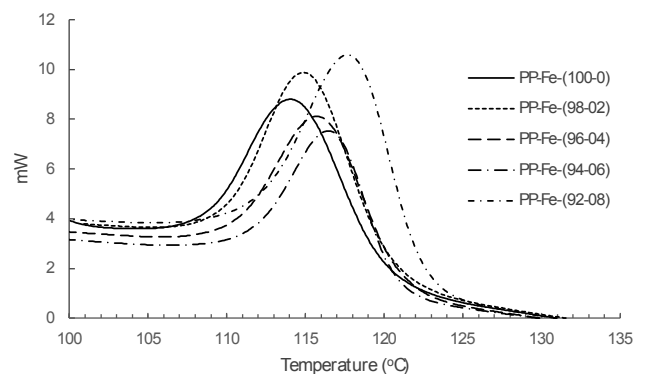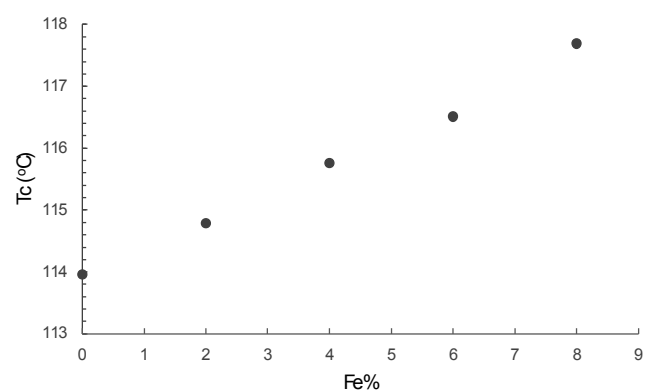

Fig. 1.   DSC results (Tc) of all composite samples.

Fig. 2.   Relationship between iron content and crystallization temperature.

B. The Effect of Iron Content on Melting Point Temperature

The melting point of all the samples was determined from the peak of DSC as displayed in Figure 3.
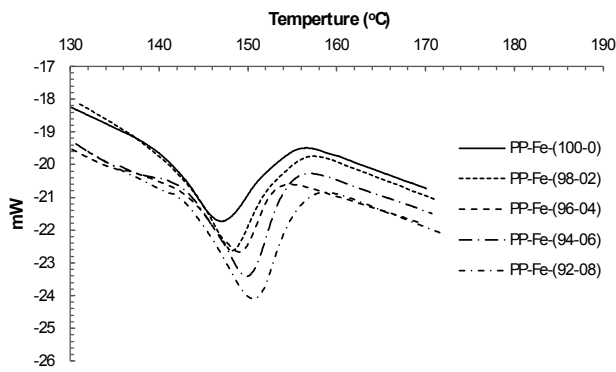
Fig. 3.   DSC results of all samples showing the melting point peak.

The influence of Fe content on the melting point of the composites was noted. The melting point was found to increase with the increase in the Fe content as illustrated in Figure 4. This can be related to the fact that an increase in the Fe content increases the adhesive properties of the composites and more energy is required to break the bonds of monomers. In other words, an increase in Fe content increases the latent heat of fusion of the composite samples.
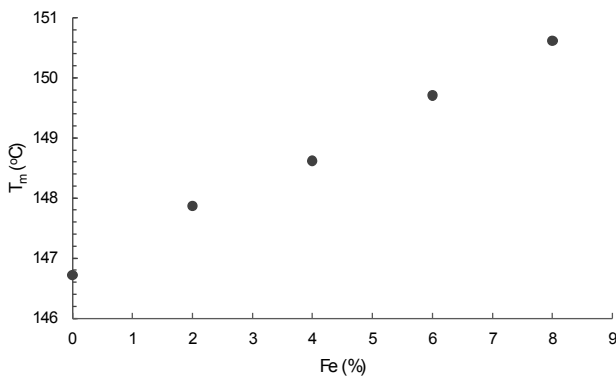

Fig. 4.   Relationship between the Fe content and the melting point of all composite samples.

C. The Effect of Iron Content on Thermal Degradation Temperature

The T_d of sPP/Fe was measured by TGA. Figure 5 displays that T_d changes with the Fe content of sPP/Fe composites. T_d is noted from the dropping point of the mass of the sample (the point at which the mass of the composites starts to drop). This is the maximum point of thermal tolerance of the sample. At this point, irreversible changes occur in the sample. We noted two drops in the mass of sPP/Fe samples. The first drop occurred at relatively low temperature due to the removal of moisture content from the sample. The second drop occurred at higher temperature, which shows the drop in the mass of the sample.

The relationship between thermal degradation temperature and Fe content is presented in Figure 6. The thermal degradation temperature increases when Fe content increases. The thermal degradation can be related to the fact that Fe content enhances the thermal stability of the sPP/Fe

composites. T_d shows the maximum temperature at which a polymer is useful. At higher temperatures, the components of the long chain of the polymer can disrupt (chain scission) and interact with one another (cross-link), thus altering the properties of the polymer.
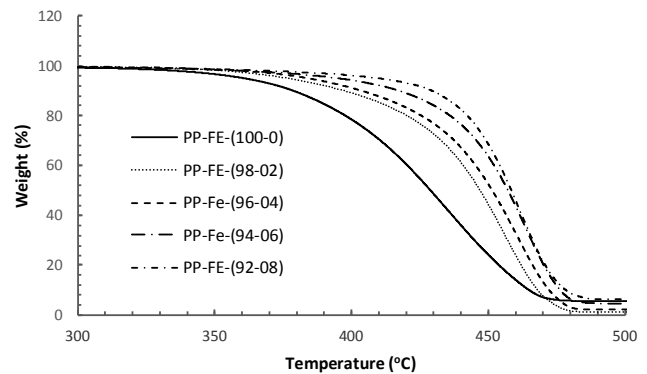

Fig. 5.   TGA results of the composite samples.
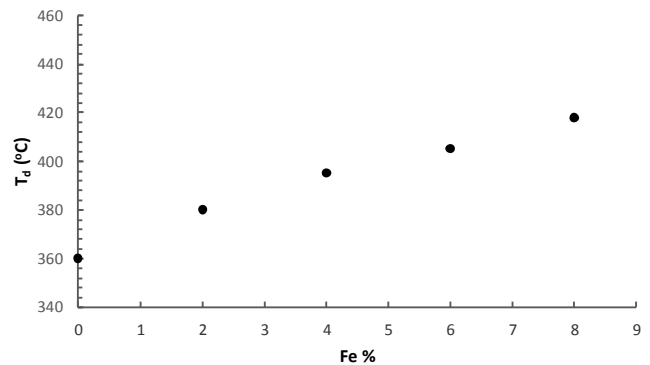

Fig. 6.   Relationship of Fe content and degradation temperature.

In the light of the above findings, it is clear that Fe as filler alters the thermal properties of sPP/FE composites. The same result was found in [17]. The authors found that rheological and thermal properties like melting point vary with varying filler content. To the best of our knowledge, the thermal properties of sPP/FE composites have not yet been studied.

IV.   CONCLUSION

Thermal properties including melting point, crystallization, and thermal degradation temperatures are used as a tool to predict not only thermal stability, but also other microstructure properties and polymer chain dimensions which affects overall polymer chain dynamics. The iron content alters the thermal properties of the polymer composites and hence the polymer chain dimensions. The experimental results established that thermal measurements performed on sPP/Fe composites of different iron contents show that melting point, crystallization temperature and thermal degradation temperature significantly depend on the iron loading. In other words, crystallization, melting point, and thermal degradation temperature increase with increase in the iron dosage due to the consequently

increased crystallinity, increased latent heat of fusion, and increased the thermal stability respectively.

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