

Thermal properties of hybrid PP composites

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Abstract

Modified polypropylene composites were obtained by incorporating both chopped glass and mineral fillers. The compositions prepared were characterized. Studies on melting and crystallisation behaviour of PP composites were carried out with the help of differential scanning calorimetry. Studies revealed that peak melting temperature of PP were slightly affected by the presence of chopped glass fibers and wollastonite in the entire composition range. Unlike the crystalline/amorphous systems, the peak melting temperature remained more or less unaffected in the composites. Wollastonite acted as nucleating agent and facilitated crystallization/ nucleating effect.

Keywords: Polypropylene; Hybrid composites; Inorganic fillers; Thermal studies; DSC; Crystallinity

1. Introduction

Polypropylene (PP) is a widely utilized polymer, particularly known for its simple structure and versatility in injection molding applications [1, 2]. However, pure PP is seldom used due to its limited mechanical and thermal properties. To enhance its performance, it is often combined with various mineral fillers. These fillers are essential in industries to improve the mechanical strength, thermal stability, and overall dimensional integrity of the polymer [3-8]. Glass fibers have proven to meet the demands in automobiles because of their excellent strength, durability, thermal stability, and resistance to impact, friction, and wear. [9]. However, because of the inherent lower thermal conductivity and stability of these polymeric materials, their wide range of applications is restricted, especially where excellent heat dissipation and low thermal expansion are necessarily required [10]. In 2004, year Hadal performed DSC measurements on PE wollastonite hybrid composites and reported that reinforcement with wollastonite increases the starting crystallisation temperature and induces a shorter processing time in injection molding, and consequently affects the extent of crystallinity of the composite [11]. Thermal properties and mechanical properties are interdependent. As the crystallinity of PP is increased, modulus increased but strength and deformability decreased [12]. The effect of surface treatment of filler on nucleation is unpredictable [13-16].

It has been observed that fillers with smaller particle size usually act as nucleating agent possibly due to aggregate formation. Crystallinity of PP significantly affects the mechanical properties of its inorganic filler composite. Crystallinity makes a material strong but brittle. It is the indication of amount of crystalline region in polymer w.r.t amorphous region.

However, the nucleation effect depends strongly upon filler type, its surface modification and surface area. [17,18]. The most prominent effect of particulate fillers on the crystalline structure of semi-crystalline thermoplastics is their ability to work as a nucleating agent [19]. By modifying the surface chemistry of the filler nucleation effect may be unaffected, increased or decreased [12-16]. On the other hand, smaller particle size has been observed to increase the activity of

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CaCO₃ as a nucleating agent, possibly due to aggregate formation [13]. The mechanical properties of PP/CaSiO₃ composites are significantly dependent on the crystal form and crystallinity of PP [20-27]. However, the nucleation effect differs strongly by filler type, particle size (i.e. surface area) and filler surface treatment. Presence of short fibers in a polymer matrix directly influences the morphological and thermal characteristics of the matrix. Addition of wollastonite to PBT enhances crystallization process in PBT-wollastonite composite [28]. A.S Luyt concluded that wollastonite particles promote β crystallization of PP matrix and the results were well documented for wollastonite and other mineral fillers as well [29-32].

Particulate fillers have a prominent effect on the crystalline structure of semi crystalline thermo plastics. They have the ability to act as nucleating agents. By increasing crystallinity, modulus of PP increases and deformability decreases [33, 34]. It shortens processing time in injection molding by acting as a nucleating agent [35-41].

2. Experimental

2.1. Materials

The materials used in this research included polypropylene (Repol grade 11MA) from Reliance Petrochemicals, characterized by a melt flow index of 11 g/10 min. The chopped E-glass fibers (T-480 grade) were provided by Nippon Electric Glass Co. Ltd., Malaysia. Wollastonite, specifically Fillex-11AB3 (surface treated), was supplied by Wolkem India Limited. The grafted polypropylene (OPTIM grade 425) used in the study was procured from Pluss Polymers, Gurgaon, while the impact modifier (Engage 8200) came from Polmann India Ltd., Bahadurgarh, Haryana.

2.2. Preparation of Composites

Hybrid composites of PP reinforced with chopped glass fibers and wollastonite were prepared by using melt mixing technique in a single-screw extruder. The composites were prepared by one step process technique of melt compounding. The PP composites with gPP and with both gPP and impact modifier were prepared as per the compositions given in Table 1.

2.3. Differential Scanning Calorimetry

Differential scanning calorimetry (DSC) analysis were performed on a Mettler Toledo DSC 1 Star^e thermal analyser in nitrogen atmosphere. The samples (approximately 2-5 mg each) were heated from 0 to 300°C at a heating rate of 10°C/min. After that, they were rapidly cooled to 30°C at a rate of 10°C/min. The samples were further heated from 30 to 300°C at a rate of 10°C/min in order to obtain the melting endotherms.

From the DSC curves, the melting (T_m) and crystallization (T_c) temperatures were determined as the maxima of the corresponding fusion and crystallization peaks. ΔT_c , crystallisation peak width gives overall crystallisation. ΔT_m is the melting peak width explains the overall fusion. ΔH_c , heat of crystallisation explains the extent of crystallization. ΔH_m is the heat of fusion. The reported values of the melting and the crystallization enthalpies (ΔH_m and ΔH_c respectively) were estimated by dividing the areas under the peaks with the mass fraction of the PP in the composites. $t_{1/2}$ is half the time for crystallisation. Smaller the $t_{1/2}$, faster crystallisation becomes.

Table 1 Compositions

Composite name	Composite code PP/GF/WF/gPP/IM	PP (% by weight)	GF (% by weight)	WF(% by weight)	gPP (% by weight)	IM (% by weight)
A2	70/30/0	70	30	0	-	-
A3	70/20/10	70	20	10	-	-
A4	70/15/15	70	15	15	-	-
A5	70/10/20	70	10	20	-	-
A6	70/0/30	70	0	30	-	-
B2	65/30/0/5	65	30	0	5	-
B3	65/20/10/5	65	20	10	5	-
B4	65/15/15/5	65	15	15	5	-

B5	65/10/20/5	65	10	20	5	-
B6	65/0/30/5	65	0	30	5	-
C2	60/30/0/5/5	60	30	0	5	5
C3	60/20/10/5/5	60	20	10	5	5
C4	60/15/15/5/5	60	15	15	5	5
C5	60/10/20/5/5	60	10	20	5	5
C6	60/0/30/5/5	60	0	30	5	5

3. Results and Discussion

3.1. Melting studies of PP/GF/WF, PP/GF/WF/gPP and PP/GF/WF/gPP/IM hybrid composites

The melting endotherms and cooling exotherms of pure PP, PP/GF/WF, PP/GF/WF/gPP and PP/GF/WF/gPP/IM hybrid composites are presented in figures 1-3 respectively. The results obtained from the analysis of these thermograms are given in table 2 for their melting characteristics. The melting endotherms of pure PP, PP/GF/WF hybrid composites with A2 (30% GF), A3 (20% GF 10% WF), A4 (15% GF 15% WF), A5 (10% GF 20% WF) and A6 (30% WF) showed one peak melting temperature of 165.4, 160.5, 160.2, 158.7, 161.5, 159.9 degree Celsius respectively each corresponding to that of the pure component. Thus, we can conclude that the single melting point peak and approximately same peak melting temperatures of all hybrid composites suggest no intermolecular bonding.

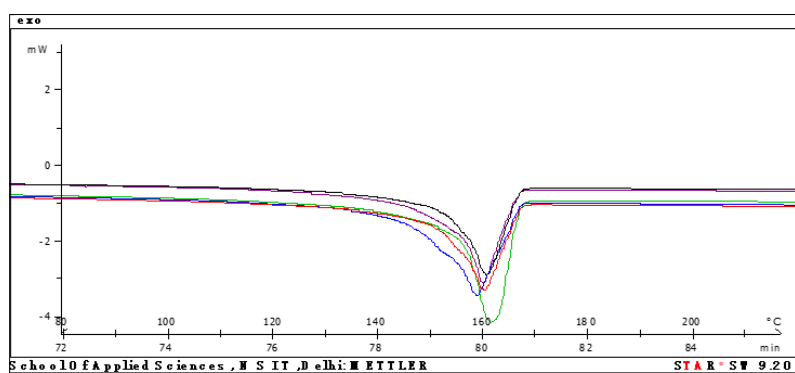


Figure 1 DSC Heating Scans for PP/GF/WF hybrid composites

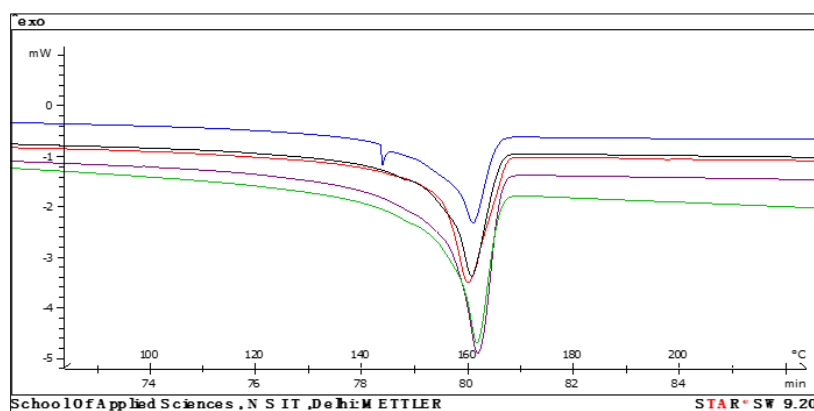


Figure 2 DSC Heating Scans for PP/GF/WF/gPP hybrid composites

From the melting characteristics of PP as observed from the heating scans, the peak melting temperature of PP is only slightly affected by the presence of chopped glass fibers and wollastonite in the entire composition range. Unlike the crystalline/amorphous systems, the peak melting temperature remains more or less unaffected in the composites. Even the addition of grafted PP or both matrix modifier and impact modifier have no effect on the peak melting temperature of the hybrid composites.

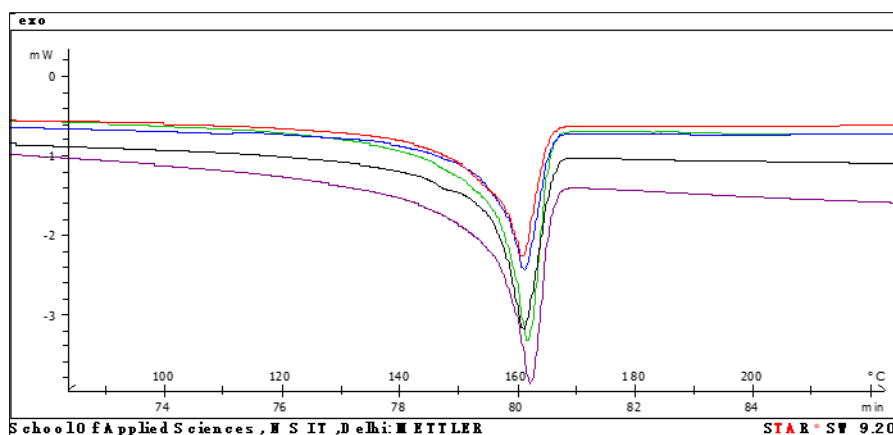


Figure 3 DSC Heating Scans for PP/GF/WF/gPP/IM hybrid composites

Table 2 Melting Characteristics of PP in the Hybrid composites

Composite code PP/GF/WF/gPP/IM	Onset of melting	Peak Melting temperature T_m	Endset of melting	ΔT_m	ΔH_f	% X_c	Increase or decrease in % X_c
PP	154.5	165.5	164.9	10.4	74.6	35.7	
70/30/0/0/0	154.6	160.5	159.9	5.3	71.7	34.3	-1.4
70/20/10/0/0	152.9	160.2	157.9	4.7	68.4	32.7	-3.0
70/15/15/0/0	150.4	158.7	154.8	4.3	73.8	35.3	-0.4
70/10/20/0/0	155.1	161.5	160.0	4.9	76.1	36.4	+0.7
70/0/30/0/0	153.9	159.9	158.4	4.6	79.1	37.8	+2.14
65/30/0/5/0	155.0	160.5	159.6	4.6	72.2	34.5	-1.2
65/20/10/5/0	154.2	159.9	158.8	4.6	80.1	38.3	+2.6
65/15/15/5/0	154.8	160.7	159.5	4.7	79.6	38.1	+2.4
65/10/20/5/0	155.1	161.5	159.5	4.3	70.6	33.8	-1.9
65/0/30/5/0	155.7	161.6	160.5	4.8	61.2	29.3	-6.4
60/30/0/5/5	155.4	160.9	159.7	4.3	68.2	32.6	-3.1
60/20/10/5/5	154.5	160.6	158.9	4.4	79.9	38.2	+2.5
60/15/15/5/5	155.3	161.1	159.6	4.3	66.3	31.7	-4.0
60/10/20/5/5	156.1	161.5	160.3	4.1	63.1	30.2	-5.5
60/0/30/5/5	156.3	162.0	160.5	4.3	68.4	32.7	-3.0

As observed in the table 2 blending affects slightly, the peak melting temperature of PP. However, the percent crystallinity of PP was affected and increased as percentage of wollastonite increased. At 30% wollastonite, a slight improvement in % crystallinity of PP has been observed. In addition, the melting temperature range (ΔT_m) of pure PP was affected to a large extent with addition of fillers.

Pure PP exhibits the widest melting temperature range of 10.4°C, which decreases with the addition of fillers. A narrow range of melting of PP indicates a narrow crystallite size distribution in presence of wollastonite. The slight increase in crystallinity of PP when 30% wollastonite is added, explains the nucleation effect of wollastonite on Polypropylene.

Furthermore, heat of fusion of composites increases from that of their respective pure components. This is indicative of some interaction of the components. Zhu and Reinsch have also reported that smaller particle size of filler usually acts as nucleating agent possibly due to aggregate formation [11, 12].

Values for heat of fusion are least for the C set of compositions with both grafted PP and impact modifier. Furthermore, when we compare the compositions of hybrid composites PP/GF/WF/gPP and PP/GF/WF/gPP/IM. The addition of grafted PP and impact modifier has not resulted in any major alterations in the peak melting temperature, melting temperature range and heat of fusion. For compositions having grafted PP, % crystallisation decreases. This is attributed to the increase in molecular mass, which results in lesser mobility and as a result, lesser polymeric chains available for crystallisation. This means that degree of mixing has no effect on melting behaviour of PP.

3.2. Crystallisation studies of PP/GF/WF, PP/GF/WF/gPP and PP/GF/WF/gPP/IM hybrid composites

Table 4 and Figures 4-6 present the cooling characteristics of pure PP and the crystallization exotherms of hybrid composites of PP/GF/WF, PP/GF/WF/gPP and PP/GF/WF/gPP/IM. Pure PP exhibits a prominent crystallisation exotherm at approximately 116°C. When cooled from the melt, the hybrid composites PP/GF/WF showed one exothermic peak corresponding to PP. Therefore, it is inferred that an increase in crystallization temperature of approximately 2-3°C for PP has been observed on addition of glass fibers and wollastonite.

Moreover, the peak crystallisation increases further approximately 5 degrees more when grafted PP and impact modifier are added to the compositions. The heat of crystallization value of PP decreases significantly in the presence of fillers. The ΔT_c of PP increased in the presence of chopped glass fibers and wollastonite fillers indicating a wider crystallite size distribution. However, the behaviour is different for the A4 composition (15% wollastonite, 15% glass fibers), which also exhibits a co-continuous morphology. The largest value of ΔT_c has been observed in this case.

The cooling characteristics of PP hybrid composites (Table 4) show an increase in peak crystallization temperature by approximately 3°C which is further increased by another 5°C for addition of only grafted PP and grafted PP + impact modifier

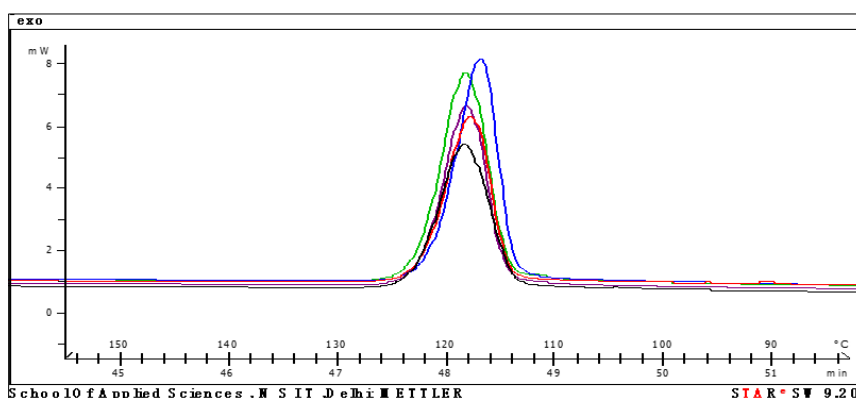


Figure 4 DSC Cooling Scans for PP/GF/WF hybrid composites

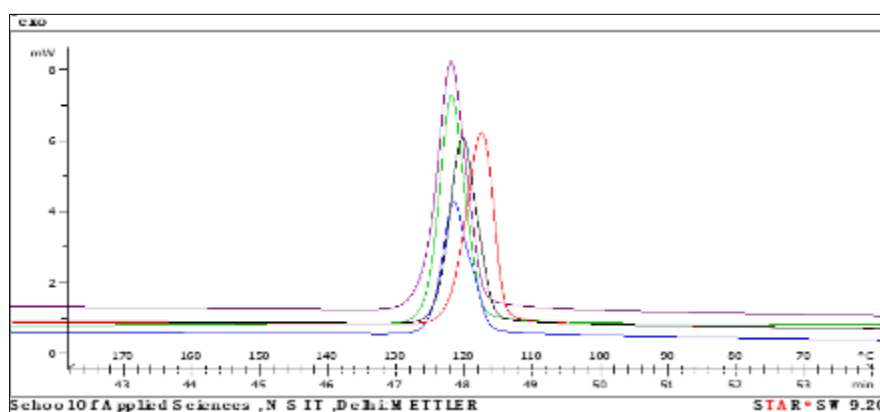


Figure 5 DSC Cooling Scans for PP/GF/WF/gPP hybrid composites

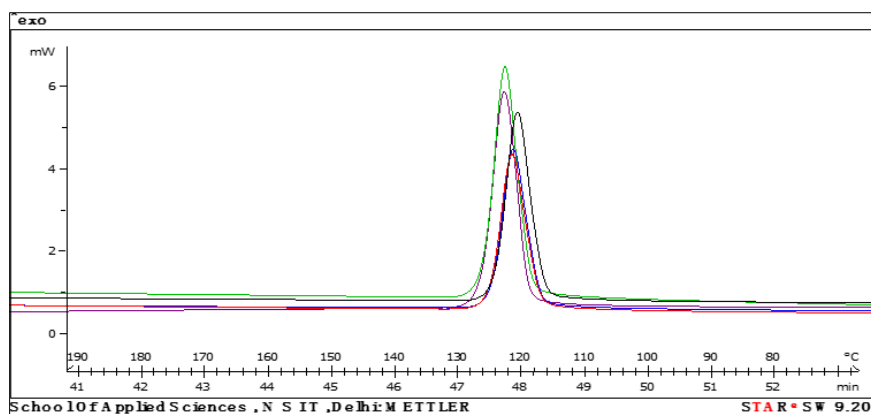


Figure 6 DSC Cooling Scans for PP/GF/WF/gPP/IM hybrid composites

Table 4 Cooling Characteristics of PP in the Hybrid composites

Composite code PP/GF/WF/gPP/IM	Onset of crystallisation	Peak Crystallisation temperature T_c	Endset of crystallisation	ΔT_c	ΔH_c	$t_{1/2}$	$\Delta T = (T_M - T_c)$
PP	120.2	116.0	124.9	4.7	97.7	0.4	49.5
70/30/0/0/0	123.1	118.5	131.1	8.0	78.5	0.5	42.0
70/20/10/0/0	122.5	118.1	131.5	9.1	70.9	0.4	41.7
70/15/15/0/0	121.5	117.3	133.0	11.5	77.1	0.4	41.4
70/10/20/0/0	122.8	118.6	130.0	7.2	78.2	0.4	42.9
70/0/30/0/0	122.6	118.4	130.3	7.8	71.8	0.4	41.4
65/30/0/5/0	124.2	120.6	131.7	7.5	70.5	0.4	40.0
65/20/10/5/0	122.2	117.7	129.5	7.3	82.1	0.4	42.1
65/15/15/5/0	125.1	121.7	133.1	8.0	75.1	0.3	39.1
65/10/20/5/0	125.5	122.3	132.2	6.7	70.2	0.3	39.2
65/0/30/5/0	126.1	122.3	132.7	6.7	63.9	0.4	39.3
60/30/0/5/5	124.4	120.8	130.9	6.5	68.8	0.4	40.0
60/20/10/5/5	125.1	121.8	133.0	7.9	79.8	0.3	38.7
60/15/15/5/5	124.9	121.5	131.8	7.0	77.6	0.3	39.6
60/10/20/5/5	126.2	122.9	132.1	5.9	72.3	0.3	38.6
60/0/30/5/5	126.5	123.1	132.5	6.1	68.5	0.3	38.9

The ΔT_c is reduced significantly when both impact modifier and grafted PP are added to the compositions.

In case of A (PP/GF/WF) compositions, the $t_{1/2}$ is decreasing slightly with the increase in volume fraction of wollastonite, indicating increase in rate of crystallisation. Degree of supercooling also decreases in this favour. Like the ΔH_f is maximum when relative wollastonite fraction is maximum. Similar trend is observed in case of crystallization which explains that wollastonite is acting as nucleating agent because an increase in peak crystallization temperature due to any of the components indicates a facilitation of crystallization/nucleating effect of the second component. T_c remains almost same for all compositions. $t_{1/2}$ is decreasing slightly with increase in wollastonite, means that wollastonite enhances the crystallisation rate. In case of B compositions (with grafted PP) and C compositions (with grafted PP and

impact modifier) crystallinity decreases. This might be attributed to decreased mobility of polymer chains as a result of increased molecular mass.

4. Conclusions

Wollastonite fibers were found to enhance the percentage crystallinity in PP thus wollastonite fibers have nucleation effect on PP. Addition of both grafted PP and impact modifier reduces ΔT_c and crystallinity due to decrease in the mobility of polymer chains.

Compliance with ethical standards

Disclosure of conflict of interest

The authors declare that they have no conflicts of interest.

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