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Thermal Characterization of Ramie – Sisal Fiber Reinforced Hybrid Polypropylene Composites

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ABSTRACT

The effect of natural fiber loading on thermal behavior of Polypropylene based hybrid composites is reported. Four composite systems were considered for investigation: Neat Polypropylene (PP), PP/5 wt. % sisal fiber/5 wt. % Ramie fiber (PP/10), PP/10 wt. % sisal fiber/10 wt. % Ramie fiber (PP/20) and PP/15 wt. % sisal fiber/15 wt. % Ramie fiber (PP/30). These natural hybrid composites were processed and developed using melt mix method with the help of twin screw extruder followed by injection molding. The thermal response of these hybrid composites have been studied using Differential scanning calorimetric studies (DSC) and thermo gravimetric analysis (TGA). The experimentation results through DSC studies showed that the degree of crystallization has been enhanced due to improvement in the enthalpy of melting. Further, it was observed from TGA that the weight loss of composites at higher temperatures has been decreased due to influence of addition of hybrid fibers. This indicates the presence of hybrid fibers restrict the molecular movement thereby resisting the weight loss even at high temperatures. Further, the degradation of these composites has been studied in order to evaluate the thermal behavior. Among the composites studied, PP/30 composites exhibit the appreciable thermal behavior.

Keywords: Polypropylene, Ramie fiber, Thermal, Hybrid composites

I. INTRODUCTION

Polymer composites are very much susceptible to thermal conditions. The performance evaluation of all types of polymers depends on their thermal load tolerance at elevated temperatures and loading conditions. Thermoplastic composites are processed using melt mix method with the help of twin screw extrusion technique [1]. Under this condition, study of these polymer melt at different temperature decides the compatibility strength of associated filler of composites with base matrix. The effect of filler and / fiber

reinforcement has significantly proved to be the best modification method for improving the thermal behaviour of polymer composites [2]. The weight loss of polymers at various temperatures needs to be discussed in detail before modification of these composites. The viscous nature of polymer and their representation to enhance the strength and thermal behaviour adds to the potential of polymer composites. Both synthetic and natural fibers have exhibited their compatibility strength with different polymer materials. But their bonding strength with the thermoplastic base materials may differ from fiber to fiber. This strength defines the thermal nature of composites. The potential fibres such short glass, short carbon, short basalt and short kevlar fibers proved to be the best fibers for both mechanical and thermal behaviour of thermoplastic composites. In addition to the above fibers, natural fibers such as sisal fibers, Bamboo fibers, Banana fibers and others after proper sizing exhibited the better thermal behaviour [3]. Natural fibres will take a major role in the emerging "green" economy based on energy efficiency, the use of renewable materials in polymer products, industrial processes that reduce carbon emissions and recyclable materials that minimize waste [4]. The thermal behaviour of natural fibre reinforced polymer composites requires lot of attention in order to enhance the strength properties of these polymer composites to develop ecofriendly polymer composites. Further, the degradation of polymers at particular thermal load along with their associate fillers must be accountable in order to enhance their reinforcement composition. Lot of work has been carried out on the thermal behaviour of natural fiber reinforced polymer composites. The hybrid effect of Glass – Basalt fiber reinforcement on the thermal behaviour of PA66/PTFE composites was reported by Rudresh et al [4]. They studied the thermal behaviour of these hybrid composites using DSC and TGA technique at suitable thermal conditions. They showed from their study that the effect of fiber reinforcement improved the thermal stability of PA66/PTFE hybrid composites. Further, the weight loss of these composites was declined with increase in temperature. The thermal storage modulus of these composites has been enhanced due to fiber reinforcement. The experimental study on the thermal behaviour of maize fiber and polyester resin coated maize fibers samples were reported by Saravana Bavan et al [5]. These natural fiber composites were processed by vacuum assisted resin transfer molding technique. They investigated using Thermo gravimetric analysis and differential scanning calorimetric test. They found that the initial degradation temperature was around 200°C. But the maximum temperature for raw fiber was around 330°C and polyester coated maize fiber, 410°C. This showed the increase in thermal stability.
The hybrid effect of Jute fiber and Carbon finer on the thermal characterization of High density polyethylene (HDPE) composites have been studied and reported by Anshu Anjali Singh et al [6]. They conclude that the maximum degradation temperature of all the composite compositions are higher than that of matrix and increase with increasing Jute fiber content. The temperatures of highest rate of degradation of 10/90, 20/80 and 30/70 Jute fiber/ carbon fiber -HDPE composites were 483°C, 485°C and 488°C respectively and at 550°C the composite compositions show the residual mass of 4.3%, 5.8% and 7% respectively. Fouzi et al [7] investigated the thermal behaviour of natural fiber reinforced epoxy composites using TGA and DSC techniques. They used pineapple leaf fibre, kenaf fibre and mengkuang fibres. The samples for both analysis were subjected to maximum temperature of 600°C at the heating rate of 10°C/min. The results showed that the treated fibres show higher maximum peak temperature as compared to the untreated fibres. Additionally, the glass transition temperature showed a lower value for all treated fibre. It can

be concluded that investigation of thermal properties of these natural fibres could improve the utilization of natural fibre composites in various applications. Sreekumar et al [8] investigated the thermal stability of chemical treated and untreated sisal fiber reinforced polyester composites fabricated by resin transfer method.
They found that chemical treatments found to increase the thermal stability which has been attributed to the resultant physical and chemical changes. In the composites, as the fiber content increases, the thermal stability of the matrix decreases. The treated fiber reinforced composites have been found to be thermally more stable than the untreated derivatives. The thermal behaviour of Glass- basalt hybrid glass - carbon hybrid Polyamide and Polytetrafluroethylene (PA66/PTFE) thermoplastic composites have been studied and presented by Rudresh et al [9]. They studied the thermal behaviour through DSC and TGA technique. It is proved that thermal storage modulus have been improved after fiber reinforcement. Further, they stated that the weight loss of composites was declined even at higher temperatures due to fiber reinforcement. Lingesh et al [10] reported the thermal behaviour of Glass – Basalt hybrid fiber reinforced Polyamide and Polypropylene (PA66/PP) Blend composites. They investigated the thermal behaviour through DSC and TGA Technique. They reported that degree of crystallization has been improved due to fiber reinforcement. Further, they stated that effect fiber reinforcement could enhance the thermal resistance of these composites against weight loss.
In the journey of the above literature survey, it is observed that the thermal behaviour of both natural and synthetic fiber reinforcement could effectively improve the thermal stability of composites. But the hybrid effect of Ramie fiber and sisal fiber reinforcement on polypropylene based composites seemed to be the research gap. In this view, Ramie fiber and Sisil fiber with different proportions have been selected and their reinforcement effect on the thermal behaviour of Polypropylene thermoplastic composites has been studied. Ramie fiber and sisil fibers are good natural fibers for processing with high performance polymers like Polypropylene. Further, the thermal stability of these composites can explored using the concept of Differential scanning calorimetric studies and also Thermograviemetric analysis.

II. MATERIALS, METHODS, PROCESSING AND TESTING OF COMPOSITES

The following sections presents the detailed study of materials used for the development of polymer hybrid composites, their processing technique and thermal testing of polymer hybrid composites. The suppliers data used in the development of these Polypropylene based hybrid composites is detailed in the table 1.

TABLE I Data of the materials used in the process

Materials	**Form and size**	**Melting temperature**	**Density (gr/cc)**
Polypropylene	Granules (10 to 12) (μm)	160	0.9
Maleic grafted anhydride	Powder (10 to 12 (μm)	53	1.5
Ramie fiber	5 to 10 mm	---	1.5
Sisal fiber	5 to 10 mm	----	1.5

The polymer materials and natural fibers used for the development of hybrid natural composites are shown in table 1. The physical data of the materials as supplied are in the same table. The weight fraction formulation of the materials used for the production of composites is shown in the table 2.

TABLE II Weight fraction formulation of Polypropylene based hybrid natural composites

Materials	**Design-ation**	**Weight fraction percentage**			
		PP	**Ma-gH**	**SF**	**RF**
Polypropylene	PP	97	3	---	---
Polypropylene/5 wt.% sisal fiber/ 5 wt.% Ramie fiber	PP/10	87	3	5	5
Polypropylene/20 wt.% sisal fiber/ 10 wt.% Ramie fiber	PP/20	77	3	10	10
Polypropylene/15 wt.% sisal fiber/ 15 wt.% Ramie fiber	PP/30	67	3	15	15

A. Processing of Composites

The materials used for the fabrication process such as Polypropylene were dried at a temperature of 80⁰C for 48 hours before mixing to avoid plasticization and hydrolyzing effects. To maintain the homogeneity of the mixture, these mixtures were mixed in the mixer. The mixture was extruded using Barbender co-rotating twin-screw extruder (Make: CMEI, Model: 16 CME, SPL, chamber size 70 cm^3). The extruder consists of five heating zones where the temperature maintained in these zones were zone1 (220 ºC), zone2 (235 ºC), zone3 (240 ºC), zone4 (265 ºC) and zone5 (270 ºC) respectively and the temperature at the die was set at 220 ⁰C. The extruder screw speed was set at 100 rpm to yield a feed rate of 5 kg/h. The extrudates obtained was in the form of cylindrical rods which were quenched in cold water and then palletized using Palletizing machine. During initial stage, around 1 to 1.5 kg of initial extrudate was removed to get the pure blend and to remove impurities of extrudate of previous stroke of the extrusion. Before injection moulding, all polymer blended pallets were dried at 100⁰C in vacuum oven for 24 hours. All test specimens were injection molded from the pelletized polymer material obtained from co-rotating extruder. The temperature maintained in the two zones of the barrel were zone1 (265 ºC) and zone 2 (290 ºC) and mold temperature was maintained at 65 ⁰C. The screw speed was set at 10 – 15 rpm followed by 700-800 bar injection pressure. The injection time, cooling time and ejection time maintained during injection molding were 10, 35 and 2 s, respectively. All the molded specimens as per ASTM were inspected and tested visually and those found defect were discarded from testing.

B. Testing of Composites

Differential scanning calorimetric studies (DSC) Polypropylene based hybrid composites were carried out using universal V4.7 TA instrument DSC set up. 30 to 300 ⁰C temperature range has been used for this analysis and 4.8 mg of sample weight was considered for the analysis. The polymer sample was heated to 300⁰c at a rate of 10 ⁰c per minute under a controlled nitrogen atmosphere and the corresponding scans are recorded. To remove the previous thermal history the heated sample is held for 5 to 6 minutes at the same temperature. Later it was cooled back to 0⁰c and held for a period of 5 to 6 minutes with the same condition. Further for a temperature of

300 ^{0}C at the same heating rate is continued. Melt crystallization and other related parameters have been recorded. The enthalpy and degree of crystallinity of composite is determined. The degree of crystallization 'Xc' was calculated using the following formula:

$$Xc = \frac{\Delta Hm}{\Delta Hm^*} \text{ x } 100 \qquad (1)$$

Where ΔHm = Enthalpy of composite and ΔHm* = Enthalpy of pure crystalline Polypropylene.

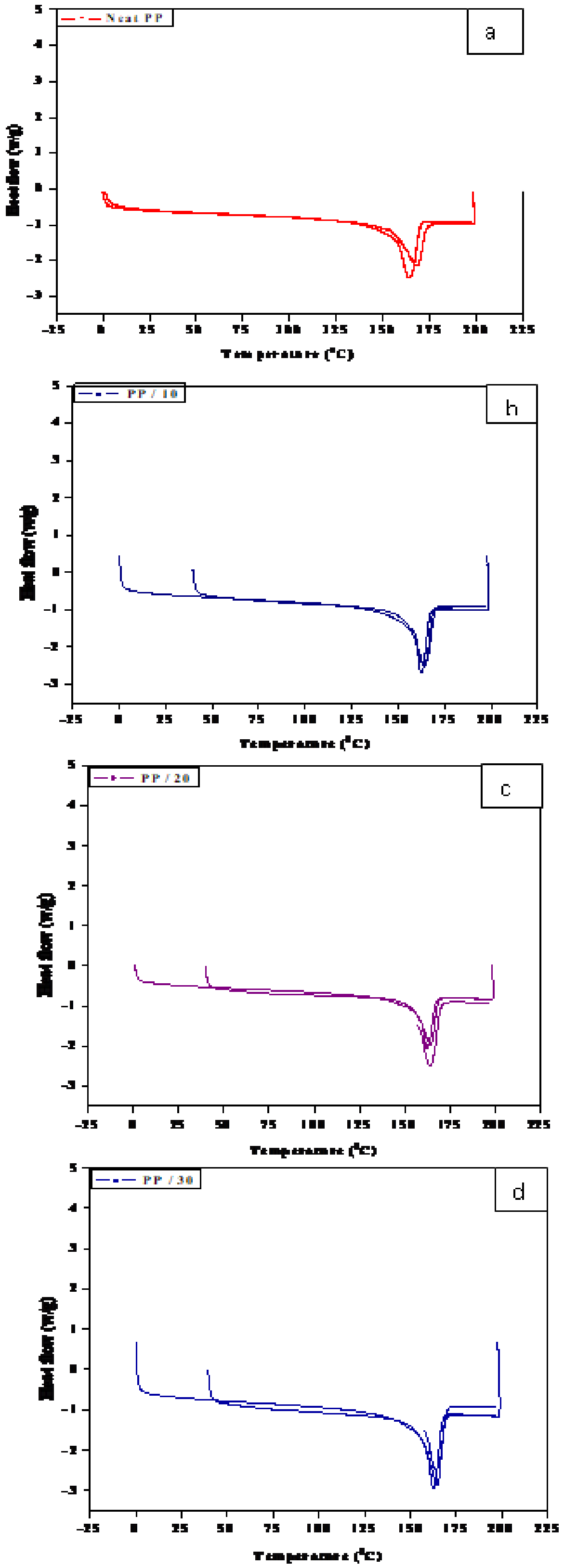
The thermal stability of polymer composites was evaluated through thermo gravimetric analysis (TGA). The temperature range of 30-800 ^{0}C under the influence of nitrogen atmosphere with a heating rate of 20 ^{0}C per minute was used for this study using Universal Instruments (TGA Q50 V 20.13). Thermograms were used to record the percentage weight loss at different temperatures. The derivative weight loss at different temperatures and degradation stage temperature has been recorded and analysed using derivative TGA thermo grams.

III. RESULTS AND DISCUSSION

A. Crystallization and Melting Behaviour of Polypropylene Hybrid composites

The effect of hybrid fiber reinforcement such as Ramie and Sisil fibre on the thermal behaviour of Polypropylene hybrid composites was studied using DSC and TGA Techniques. The DSC studies were used to determine the enthalpy of fusion and degree of crystallization of Polypropylene based hybrid composites. The onset temperature (T_o), the melting temperature (T_m), crystallization temperature (Tc), enthalpy of melting (ΔH_m) and degree of crystallinity (X_c) of hybrid fiber composites was estimated using DSC technique. The thermal data related to DSC studies are tabulated in the Table III.

The influence of fiber reinforcement on different thermal phases of polypropylene based composites was studied using DSC technique and the response against the thermal loading is depicted in figure 1 (a - d). The degree of crystallinity (Xc) is calculated using the equation 1[16]. The thermal response of these composites was affected by the type of fibers used.



igure 1: DSC Thermograms: a) PP, b) PP/10, c) PP/20 and d) PP/30 hybrid composites

Figure exhibits both downward and upward trends as an effect of exothermic and endothermic responses from the composites [17]. The different thermal phase transitions such as glass transition, crystallization and melting phase indicates that the composites under study were semi crystalline in nature. The onset temperature (T_o) of the neat blend was started at a temperature of 139.73 ^{0}C. Reinforcement effect of hybrid fibers enhanced the onset temperature of PP/10 Composites to 139.81 ^{0}C, PP/20 to 138.28 ^{0}C and 134.71 ^{0}C has been exhibited by PP/30 Composites. The peak temperature of neat PP was 157.68 ^{0}C. But the effect of adding these hybrid fibers could slightly decline the temperature. This is due slight porosity created due to higher reinforcement [10, 11]. Similar observations were made with the crystallization temperature. But the enthalpy of fusion has been enhanced due to the addition of hybrid fibers. The enthalpy of 106 J/g has been exhibited by PP/30 composites over neat PP of 87.26 J/g which is 17.67% increase. This may be due to the good compatibility between fibers and matrix which resist the movement of heat due to high dense interphase. The molecular momentum transfer due to reinforcement effect of natural hybrid fibers was less [14]. Here Ramie fiber forms good interphase with the thermoplastic matrix which may results in improvement in thermal storage modulus. Hence, high degree of enthalpy. Among the composites studied, PP/30 composites exhibit the good thermal stability.

B. Thermo gravimetric analysis

TABLE III Thermal data obtained from DSC Thermo grams

Composites	**Temperature** (^{0}C)			**ΔHm (J/g)**	**Crystall-anity (%) (Xc)**
	T_0	T_M	T_C		
Polypropylene (PP)	139	157	183	87.26	42.15
PP/10	139	154	173	88	42.51
PP/20	138	154	175	66.48	32.11
PP/30	134	154	177	106	51.20
The heat of fusion value for 100% crystalline PP is 207 J/g					

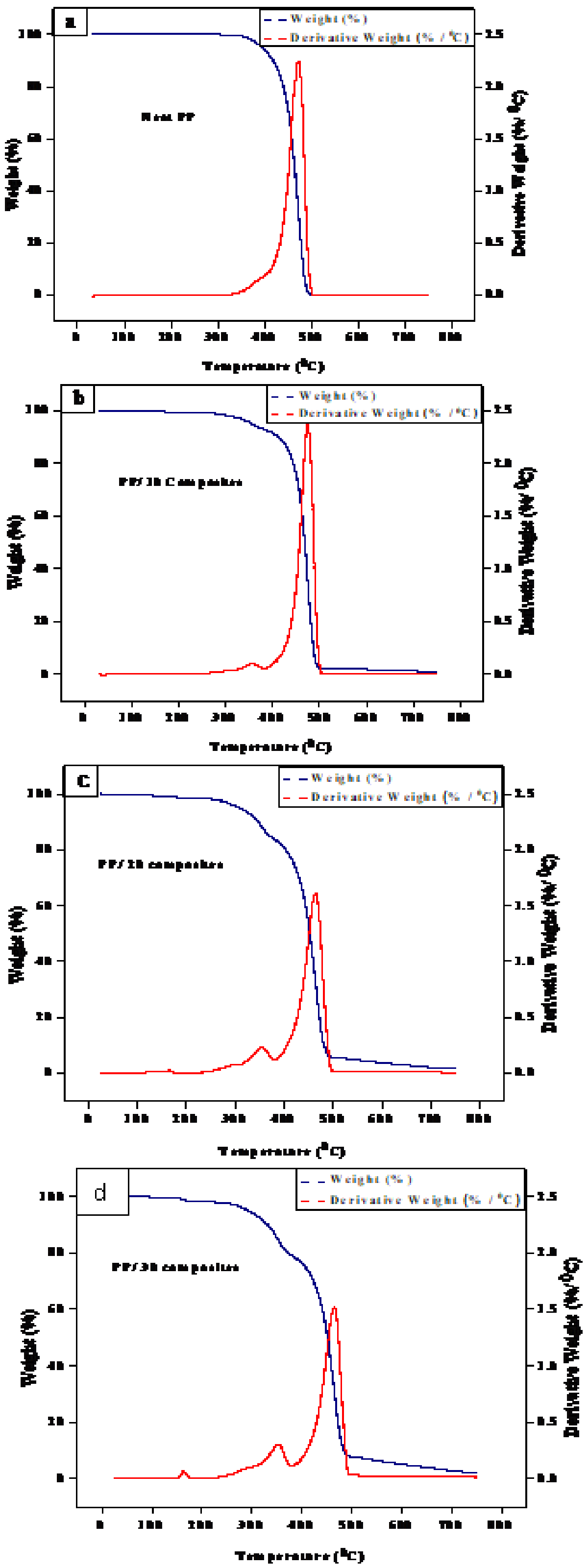


Figure 2: TGA Thermograms: a) PP, b) PP/10, c) PP/20 and d) PP/30 hybrid composites

The thermo gravimetric analysis of Polypropylene based hybrid natural composites were studied and the obtained thermal data from the analysis is recorded in the Table IV. Figure 2 (a - d) shows the plots of TGA and their derivative curves of Polypropylene based natural composites.

TABLE IV Thermal data obtained from TGA Thermo grams

Composites	**Temperatures at different weight loss (±1°C)**				
	10%	20%	30%	50%	Maxm.
Polypropylene (PP)	412	440	451	463	523
PP/10	413	441	456	468	748
PP/20	346	403	430	456	749
PP/30	331	371	423	451	749

The weight loss at different temperatures when the hybrid composites were subjected to thermal load is recorded in the table IV. TGA Study suggests that 10% weight loss occurs at a temperature of 412.6 ^{0}C for neat polypropylene. But PP/10, PP/20 and PP/30 composites exhibits the same percentage of weight loss at slightly lesser temperature than neat PP. Similar observations were noticed at weight loss of 20, 30 and 50%. But the maximum weight loss is exhibited by the composites at very high temperature. This clearly shows that the thermal resistance of composites has been enhanced due to the effect of fiber reinforcement [12, 13]. Maximum weight loss for neat PP at a temperature of 523.18 ^{0}C whereas for PP/30 composites, it was 749.36 ^{0}C. This showed the potential natural fibers which could improve the thermal stability of polymer composites. The resistance in weight loss against the thermal load is due to effective balancing of thermal resin across the network of hybrid natural fibers. The thermal network designed by PP/30 composites is such that it creates a permeable boundary with in the composite shell. This may avoid the loss of material during porosity [15]. This may avoid the over loss of weight at peak thermal regime. Among the composites studied, PP/30 exhibits the better thermal resistance.

IV. CONCLUSION

The thermal behavior of natural hybrid polymer composites was studied through DSC and TGA technique. The melting temperature and crystallization temperature was slightly deteriorated due to addition of hybrid fibers. The enthalpy of composites was significantly improved by the addition of hybrid fibers. PP/30 composites exhibited the better thermal stability among the composites studied. TGA Studies showed that the percentage weight loss of composites is little more at lower temperature. But the thermal resistance of composites was increased due to the addition of hybrid fibers. PP/30 composites exhibited the better thermal resistance among the composites studied

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