Thermal Post Processing of Munja Fiber Reinforced Polymer Composites

M K Lila*, U K Komal, and I Singh
Department of Mechanical and Industrial Engineering, Indian Institute of Technology Roorkee, Roorkee, India
* manish.lila@gmail.com

ABSTRACT

In metals, different intermediate processes (forging, annealing, quenching etc.) are employed to enhance their mechanical as well as structural properties, but the same has not been exploited fully for polymer matrix composites. In the current experimental investigation, *Saccharum Munja* fibers (10, 20, 30% w/w) reinforced polypropylene composites were fabricated using extrusion injection molding process and subjected to elevated temperature for different time durations and subsequently characterized for their mechanical properties. A significant increase was observed in tensile as well as flexural properties in developed composites with lower fiber content with respect to the duration of thermal cycle. The increase in mechanical properties may be attributed to the strengthening of the interface between fibers and the matrix, as was confirmed by morphological examination of the fractured specimens. The obtained results revealed that intermediate processes can be successfully employed to natural fiber based polymer composites in order to enhance their mechanical and structural properties.

KEYWORDS: *Natural fibers; Composites; Thermal post processing*

1 INTRODUCTION

Metals are subjected to various intermediate process after fabrication to alter their mechanical and other properties, these methods includes, annealing, quenching, carburising etc. Therefore, similar methods needs to be evaluated in context of polymer and polymer composites to study their effect on the mechanical and other properties. Some studies has been carried out by researchers on annealing of pure polymers and its effect on mechanical properties (Yeh et al, 1976; Peterlin, 1978). Annealing refers to heating of material up to a certain temperature for a specific time and then cooling at slower rate, which result in change in its structural as well as mechanical properties. Polymers are considered as semi-crystalline in nature and possess lower thermal coefficients, therefore the effect of annealing is dissimilar in case of pure polymers as compared to metals. The macroscopic properties of semi-crystalline polymers are determined by structural morphology at the molecular level, including crystallinity fraction and crystallite size and structure (Vishwanath et al., 2013). It is reported that semi-crystalline polymers change their physical properties when they are heated to elevated temperatures below their melting point. Also, the selection of annealing temperature, annealing time and cooling rate are some of the crucial factors due to variation in molecular chain structure of similar type of polymers. Most of the thermoplastic polymers exhibits three different transitions when heated from -100°C to +150°C, namely α-transition, β- transition and γ-transition (Mofokeng et al., 2011). The temperature at which β- transition

takes place is considered as its glass transition temperature, while γ-transition is exhibited at the softening temperature of thermoplastic polymers. Due to this, different type of transformations are reported when the polymers are subjected to different temperatures, which are related to alterations of the morphological structure (Bai et al., 2011).

Natural fiber reinforced composites are now being used as an alternate of pure polymer as well as synthetic fiber reinforced polymers, due to their specific properties. A variety of articles are available exhibiting the effect of natural fiber reinforcement on the mechanical properties of short fiber reinforced composites. Various fibers have been incorporated in different matrices as well as different fabrication methods have been employed and already reported. To increase interfacial adhesion among fiber and matrix, various treatments, as well as compatibilizers, can be used while fabricating composites, but at the same time, this may lead to an increase in cost, carbon emissions and fabrication time. The mechanical properties of composites are also governed by the cooling rate, as at slow cooling rate the crystalline properties increase, which further leads to better mechanical properties as well as better elongation of the composites. While fabricating polymer matrix composites in closed mold process, the mold temperature can be kept high for proper solidification of the part at a uniform rate, which leads to slow cooling of the part. But at the same time, this slow cooling leads to lower productivity due to higher cooling time. Therefore, similar to metals and pure polymers, intermediate thermal processes can also be employed on natural fiber reinforced polymer composites to enhance their mechanical properties, after fabrication.

Short fiber reinforced polymer composites are anisotropic in nature mainly due to variation in fiber concentration and orientations at different locations, which makes the analysis of their thermal constant as a difficult task. Therefore, the term thermal post processing is used instead of annealing in the present manuscript. A few articles are available on annealing of neat thermoplastic polymer, but scarcity of articles in context of natural fiber reinforced composites necessitated the investigation of thermal post-processing on their mechanical properties.

2 MATERIALS AND METHOD

A homopolymer of polypropylene (PP-1110MAS) has been selected to be used as matrix material and procured from M/s Indian Oil Corporation Ltd., India, based on the its availability, use and suitability of the process. The properties of selected matrix material are given in Table 1.

Table 1: Basic properties of Polypropylene used

Property/ Material	Standard	PP (1110MA)
Density (g/cm^3)	ASTM D1505	0.91
MFI (g/10min)	ASTM D1238	11 (I-2)
Tensile Strength (MPa)	ASTM D3039	34
Flexural Modulus (MPa)	ASTM D7264	1550
Elongation at yield (%)	ASTM D3039	10
Melting Point (°C)	-	130- 167
Vicat Softening Point (°C)	ASTM D1525	154

Munja fibers ropes has been procured from local market, cut in the required size, subsequently washed in lukewarm water to remove the fiber entanglement and dried before processing.

2.1 Fabrication of composites

The neat polymer and munja fibers are weighed and mixed in different fiber fraction (10%, 20% and 30%) to fabricate different composites. The mixtures are separately fed in a single screw extruder (Make: Sai Extrumech, Model: SAI25) to produce a composite wire of 4mm diameter at a processing temperature of 180°C. The extrudate is subsequently pelletized and dried at 90°C for 6 hours to remove any moisture content during cooling. Individual lot of pellets are then fed into the hopper of injection molding machine (Make: Electronica, Model: Endure-60), with a barrel temperature profile as 200-195-

190-180 (Nozzle). The mold of the machine is designed in such a way that each injection shot fabricates samples for both tensile and flexural testing as per ASTM D-3039 and ASTM D-7264, respectively. The fabricated specimens were further subjected to thermal post processing. The nomenclature used for 10%, 20% and 30% fiber reinforced polypropylene composite specimen are 10MP, 20MP and 30MP, respectively.

2.2 Thermal analysis

Before thermal post processing, the neat polymeric samples were analyzed for their thermal properties, which includes the analysis of softening point as well as thermal constants. Differential Scanning calorimetry of polypropylene was performed on NETZSCH Model (DSC 200 F3) in a Nitrogen environment at 10°C/ min heating rate. The temperature cycle of the study was taken as 100°C-200°C-100°C and the obtained curve is shown in Fig 1.

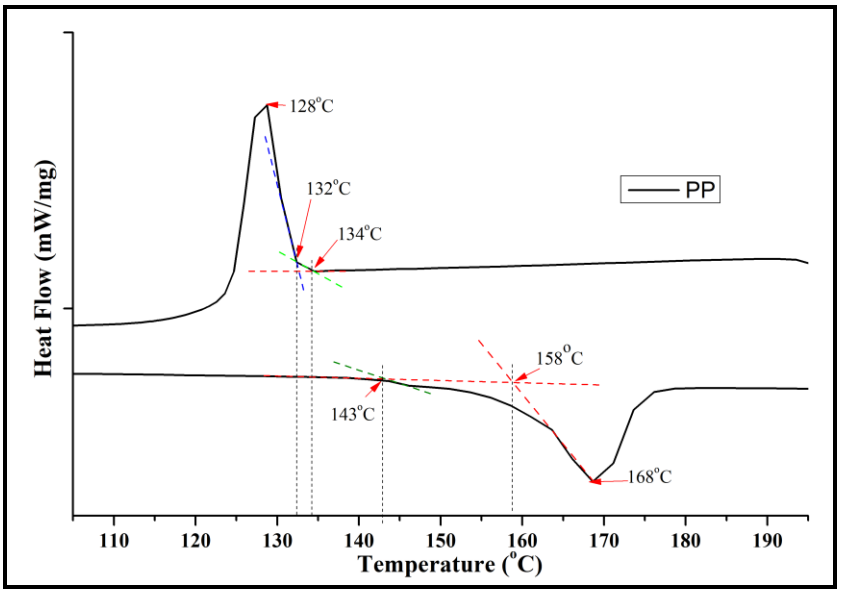

Fig 1: DSC curve obtained for polypropylene

The thermal constants (thermal conductivity, diffusivity and specific heat capacity) of polypropylene were evaluated using thermal constant analyser (Make: Hot disk, Model: TPS 2500S) with Kaptan insulated sensor (Design: 5501, Radius 6.4mm) and the results obtained are given in Table 2. The thermal coefficients of the developed composite specimen were hard to analyse due to limitations of the apparatus available, as it is designed for isotropic materials only.

Table 2: Thermal constants of polypropylene

Thermal Conductivity	Thermal Diffusivity	Specific Heat Capacity
0.1792 W/m.K	0.0997 m^2/s	1.796 kJ/kg.K

2.3 Experimental details

To get the exact time duration to completely heat up the specimen, energy balance equation is used, in which, the heating of mateiral is considered as a lumped system where the temperature remains uniform within the body and changes with time only. According to this, at time $t = 0$, when an object is placed in a medium at temperature T_x, the heat transfer takes place between the body and the environment, with a heat transfer coefficient h. During a differential time interval dt, the temperature of the body rises by a differential amount dT.

An energy balance of the solid for the time interval dt can be expressed as

Heat transfer into the object during dt = The increase in the energy of the object

This expression can be written in mathematical terms as

$$h.A_s(T_x - T\ ) = m.C_p.dT \quad (1)$$

where h is the heat transfer coefficient of the medium (air), As is the surface area of the object to be heated, T_x is the temperature of the medium, m is the mass of the object, Cp is the specific heat of the object and dT can be written as d(T - T_x) as T_x is constant.

$$h.\ A_s.\ dt = -m.\ C_p.\frac{d(T-T_x)}{T-T_x} \quad (2)$$

$$\ln\left[\frac{T_t-T_\infty}{T_i-T_\infty}\right] = -\frac{hA_s}{mC_p}.t \quad (3)$$

Where *h* is the heat transfer coefficient of the medium (air), A_s is the surface area of the specimen to be heated, *m* is the mass of the specimen, C_p is the specific heat of the specimen material, T_i is the initital temperature of the specimen, $T\infty$ is the temperature of the medium, and T_t is the temperature of the specimen after time *t*. The selected final temperature (T_∞) was kept 1°C lower than the softening point of the matrix (based on DSC curves) by considering the maximum possibility of any structural change, if occurs.

Based on the results obtained, the time required to heat up the matrix specimen was evaluated based on steady state heat flow equation (Eq 1) using the data given below

- h= 10 J/m^{2}sK for air,
- T_∞ = 141°C, T_t= 140°C, T_i= 21°C,
- A_s= 11720mm^2,
- m= 15.7g (PP),
- C_p= 1.796 kJ/kg K (PP)

By using the above parameters, the time required to completely heat up the specimen up to the oven temperature was calculated as 19min (1151 s) for neat PP specimens. The time duration for thermal processing at was selected as 0.5 hour (A), 1hour (B), 1.5 hours (C), 3 hours (D), 4.5 hours (E) and 6 hours (F). 5 specimen of each sample were selected for thermal processing of neat polymer as well as its composite.

Fig 2: Placement of specimens in hot air oven.

Initially, hot air oven was preheated at desired temperature, and first lot (F) is placed in the oven, as shown in Fig 2. After 1.5 hours, 2nd lot (E), and subsequently other lots were placed in the oven at specified time durations, selected as per the experimental design. After a total of 6 hours, the oven was switched off, for slow cooling of the specimen kept inside. The specimen were removed from the over

after 10 hours and kept at ambient condition to remove any thermal stress. After that, the thermally treated specimens were characterized for their mechanical properties.

After placing all the specimen as specified time and completion of heat treatment process, the oven is switched off for homogeneous cooling of all the specimen. The specimens are removed from the oven after 4 hour of cooling at normal temperature and kept in ambient environment for 2 days to remove any thermal stresses developed due to the process. The specimens were then characterized for their mechanical properties on universal testing machine (Model: INSTRON-5982, Make: INSTRON INC, USA)

2.4 Analysis of mechanical properties

Tensile and flexural tests of neat polymer and its composites have been performed on Universal Testing Machine (Model: INSTRON -5982, Make: Instron Inc., USA). Tensile testing has been performed at a crosshead speed of 2 mm/minute with a gauge length of 50 mm. Flexural tests has been conducted at a crosshead speed of 2 mm/ minute with a span length of 60 mm.

3 RESULTS AND DISCUSSION

Initially, neat polymeric specimens were tested for their mechanical properties to evaluate the effect of thermal post processing on their mechanical properties. Typical stress strain curves obtained in tensile and flexural testing of pure polymeric specimens are shown in Fig 3.

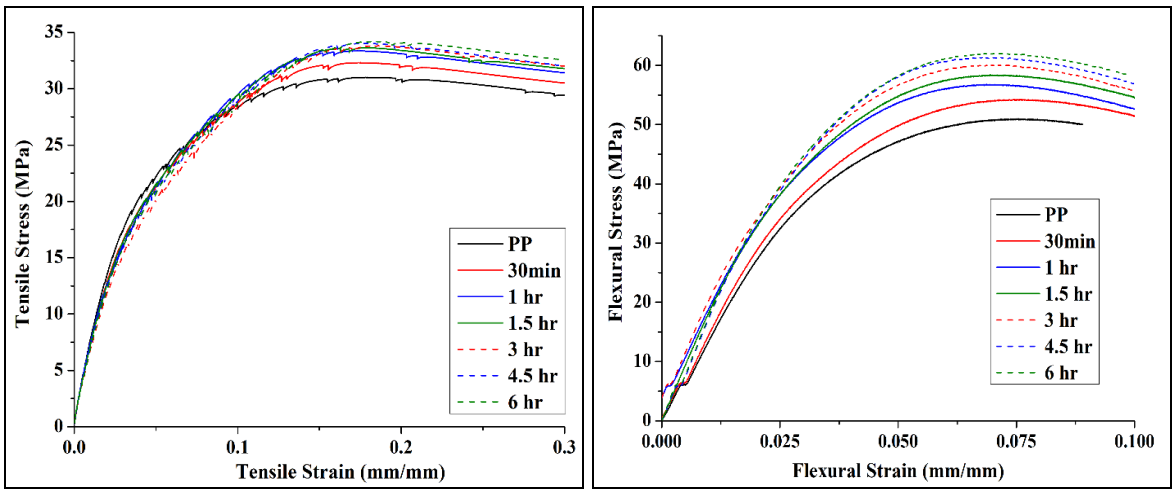

Fig 3: Tensile and flexural stress-strain curve for neat polypropylene specimen

An increase in tensile and flexural strength was observed when neat polypropylene specimens were subjected to elevated temperature for different time durations. In polypropylene, as compared to untreated specimen, an increase of 4.2%, 7.6%, 8.4%, 9.0%, 9.7% and 10.2% in tensile strength was observed for a time duration of 30min, 1 hr, 1.5 hr, 3 hr, 4.5 hr and 6 hr, respectively. The increase in flexural properties was observed as 6.5%, 11.37%, 14.51%, 17.86%, 20.3% and 21.6% for increasing time duration of thermal post processing. An insignificant reduction in elongation at maximum load during tensile testing as well as deflection at maximum load during flexural testing was observed. The modulus properties were also found to increase with processing time, which shows the increase in stiffness of the treated specimens.

Subsequently, the mechanical properties of developed composite specimens were also analyzed, which are shown in Fig 4. Similar to neat PP specimens, an increase was observed in mechanical properties of the composites with increased time duration of heating. This increase in properties can be attributed to enhanced interfacial adhesion between fiber and the matrix, which can be further confirmed with morphological study of the fractured specimen, as shown in Fig 5 for PP based composites. Less

fiber pullout and fiber fracture was observed, when composites were subjected to thermal post processing, this also confirms the enhanced interfacial bonding between fibers and the matrix.

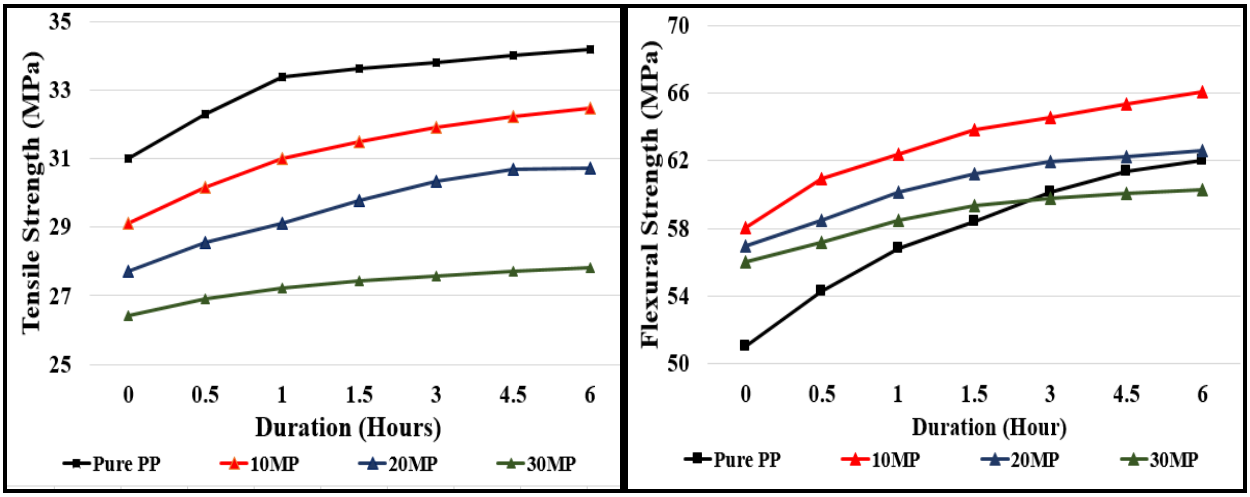

Fig 4: Tensile and flexural properties obtained after thermal post processing

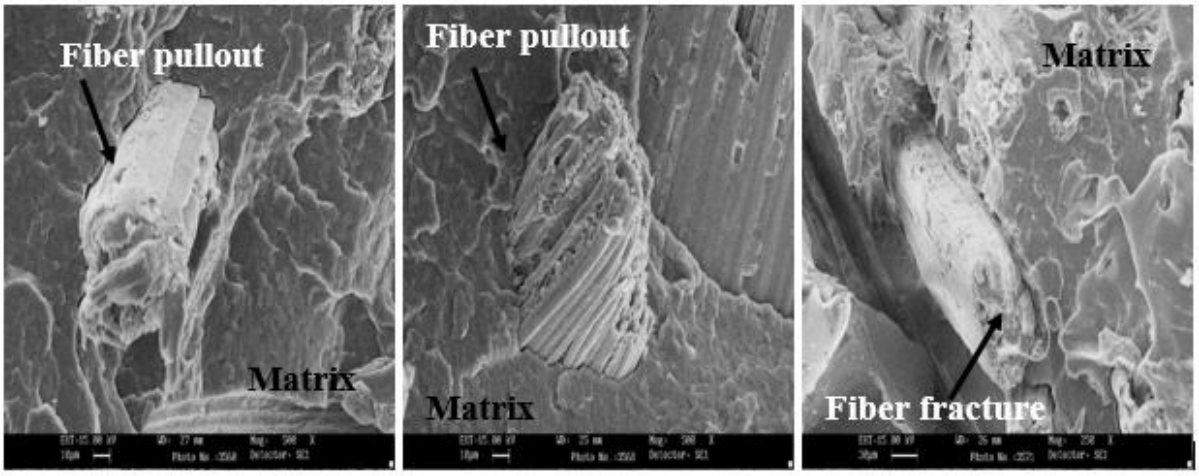

Fig 5: SEM images of fractured 20MP specimen (a) untreated (b) 1 hr (c) 3 hr

In composites, the increase in mechanical properties was found less than for pure polymeric specimens, which, subsequently further reduced with increasing fiber loading. This can be attributed to the thermal insulating behaviour of natural fibers due to their low thermal coefficients as compared to polymers. This behaviour may lead to non-uniform heat transfer within the composite specimen, thereby, exhibiting less effect of thermal post processing on the mechanical properties of the fabricated composites (Hadesiu et al., 2007; Bai et al., 2011).

4 CONCLUSION

Thermal post processing has been employed for the developed composites in order to investigate its effect on their mechanical and crystalline behaviour. The specimens were exposed to a temperature lower than their softening temperature for different time durations. Neat polymers exhibit a significant increase

in mechanical properties with respect to post-processing duration, which is attributed to reformation in molecular chain arrangement. The effect of thermal post-processing was found to reduce with increase in fiber loading, which was attributed to thermal insulating behaviour of incorporated natural fibers, which restrict the heat flow in developed composite. It is established that thermal post-processing definitely affects the mechanical properties and hence can be employed for natural fiber reinforced polymer composites.

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