



The natural fibre and nano talc based hybrid composite of polypropylene: Thermal performance

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Hybrid composite are the latest development in the field of engineering materials and due to environmental concern we are employing natural fibre in place of synthetic fibres to save energy and environment as these hybrid composite are biodegradable. Reinforcement of nano talc in hybrid composite also enhances the properties of hybrid composite. Processing of hybrid composite is easy and economical. The hybrid composite produced by melt blending via co-rotating twin screw extruder. The thermal and morphological properties of hybrid composite have been investigated. The chemical structure has been observed by FTIR and morphology of hybrid composite by SEM has shown the uniform dispersion of natural fibres and talc in the polypropylene matrix. The thermal properties have been analysed by DSC, HDT, VST tests and gives positive results.

Keywords: Hybrid composite, Natural fibre, Nano talc, Polypropylene, FTIR, DSC, HDT, VSP, SEM

1 Introduction

Due to the increased environmental concern natural fibres reinforced polymeric nano composites are entering the global market of composite materials. Natural fibre filled composite consist of biodegradable and easily recyclable polymer matrices^{1,2}. Due to continuous depletion of global resources there is a trend and demands to use materials from renewable sources, in this background use of light weight, low cost, easily available natural fibres can replace synthetic fillers in polymeric composite^{3,4}.

Natural plant fibres such as flax, hemp, kenaf, jute, which are renewable alternative finds applications to replace traditional glass fibre reinforcement, due to increased concern about energy saving attitude. Natural fibres have many advantageous over synthetic fibres like biodegradability, easy availability, low cost, low specific weight, with superior mechanical properties such as flexibility, stiffness, tensile and compressive strength. It has been demonstrated that the PP/natural fibre composite have the potential to replace PP/glass fibre composite⁵⁻⁸. Natural fibre composite uses 45% less energy as compared to glass fibre composite and emissions like carbon dioxide, methane, sulphur dioxide, carbon monoxide are also

on lower side⁹. Natural fibre reinforced composite are 20-30% are lighter than glass fibre reinforced composite which leads to more use in automobile and aircraft components which leads to higher efficiency and reduces emissions subsequently^{10,11}

Polymeric nano composites incorporates nano particles into a matrix of polymer established as new engineering materials. The composite in which dispersed particles are of nanometer size, at least one dimension are defined as nano composite. Nano composites are prepared by adding sufficient nano particles to polymer matrix to improve its performance. This produces high performance composites when good dispersion of the particles is achieved then the properties of the nano scale particles are better than those of the polymer matrix¹².

Talc qualifies as good reinforcement material as it is layered mineral with a high aspect ratio, this is due to its plate like structure having micron size dimensions on length and width with nano metric thickness. The effects of talc on polymer have been well studied. It was demonstrated that talc improves mechanical properties and macromolecular orientation of polymer¹³.

Hybrid composite reinforced with more than one filler is an easy way to achieve suitable improved mechanical properties¹⁴⁻¹⁶. Popularity of hybrid

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composite is uniformly increasing due to improved properties which cannot be attained in composite system containing only single reinforcement¹⁷. Use of natural fibre in hybrid composite is less expensive as compared to conventional reinforcement hence reduced the price of hybrid composites¹⁸.

Polypropylene is one of the important thermoplastic polymers used for the preparation of hybrid composite. As it is light weight, excellent mechanical properties good heat and chemical resistance. It is easily process able and economical in production. However polypropylene has low resistance to crack propagation, resulting in mechanical failure. In recent years this drawback has been overcome through the preparation of nano composite¹⁹.

In the present work authors selected *Agave Americana* leaf fibres as natural fibre. *Agave Americana* leaf fibres have properties of low density and high tenacity. These fibres are long and biodegradable. It is cultivated world wide as an ornamental plant. It grows well even in neglected conditions. It can grow on dry soils unfit for cultivations. This fibre is 100 % biodegradable. The water requirement for *Agave Americana* harvesting is 4/5 times less as compared to sugarcane. The colour of *Agave Americana* fibre ranges from milky white to golden yellow²⁰. The others fillers for the hybrid composites are nano talc, starch, in polypropylene matrix. The aim of the present work is to study the morphology and thermal properties of the prepared hybrid nano composite through FTIR, SEM, DSC, HDT and VSP.

2 Experimental

2.1 Materials

The polypropylene homo polymer of 35 MFR (grade PP H 350FG) was purchased from Reliance Industries. The nano talc was obtained from Soapstone Pvt. Ltd., Jaipur (Rajasthan, India). The potato starch having molecular weight 162.14 was procured from Loba Chemie, Mumbai. The maleic

anhydride grafted polypropylene (grade Epolene G-3015) was purchased by Eastmann (USA). Natural fibre extracted from *Agave Americana* plant by wetting process.

2.2 Preparation of hybrid composite

As polypropylene and starch does not react chemically, hence to improve compatibility between these the PP-g-MA is used as a coupling agent, which forms hydrogen bonding between the polymer matrix and starch²¹⁻²⁴. In this research work the content of compatibilizer was kept constant at 10%. Compatibilizer is a compound which has a polar head and a non polar hydrocarbon head. The compatibilizer should be miscible with the polypropylene matrix²⁵⁻²⁸. The polypropylene, nano talc, thermo plastic starch, and *Agave Americana* fibres were mixed homogeneously in the high speed mixer for 2 min as per formulation given in Table 1, hybrid composites were prepared by melt blending with the help of co-rotating twin screw extruder.

The barrel temperature of twin screw extruder was between 170 to 190 °C from barrel no 1 to 10, and melt temperature of the extruder was around 175 °C, the screw rpm was kept around 275 and melt pressure of the extruder maintained at 25 MPa during melt extrusion process.

3 Characterization

3.1 Fourier transmission infrared spectroscopy (FTIR)

The chemical structure analysed by FTIR spectroscopy technique. It shows the interaction between the polymer and other reinforcement materials of hybrid composites. The all hybrid composites have been scanned from 4000 to 550 cm⁻¹ range. The test was conducted according to ASTM E 1252:2013 with ATR (Attenuated Total Reflection).

3.2 Scanning electron microscopy (SEM)

The scanning electron microscopy is used to study the morphologies of the compounds. The specimens were fractured in liquid nitrogen. The samples were coated by 12 micron layer of gold to avoid electrical

Table 1 — Formulation of polypropylene based hybrid composites.

S.No.	Sample	Polypropylene (PP) %	Starch (ST) %	Nano Talc (TL) %	Natural Fibre (NF) %
1	PP	100	00	00	00
2	PPST20TL01NF05	100	20	01	05
3	PPST20TL03NF03	100	20	03	03
4	PPST20TL05NF01	100	20	05	01
5	PPST00TL05NF00	100	00	05	00

charging. Imaging of the samples, were carried out under high vacuum with operating at 15 KV, with Hitachi-Japan Model No S-3700N.

3.3. Differential scanning calorimetry (DSC)

The differential scanning calorimetry (DSC) test method used to study crystallization and melting behaviour of PP and hybrid composites. DSC test conducted according to test method ASTM D 3418:2015.

3.4 Heat deflection temperature (HDT)

The heat deflection temperature is a single point measurement and does not indicate long term heat resistance of composite material. This test is performed under ASTM D 648:2016.

3.5 Vicat softening point (VSP)

Vicat softening point (VSP) used in comparing the heat softening quality of composite material. This test is conducted as per ASTM D 1525:2009.

4 Results and Discussion

4.1 Fourier transmission infrared spectroscopy (FTIR)

Figure 1 illustrates FTIR of hybrid composite of polypropylene reinforced with potato starch, natural fibre and nano talc.

FTIR spectroscopy employed to prove the formation of hybrid composites and compatibilization among various constituents of hybrid composite. Several strong peaks were observed in $2850\text{-}2950\text{cm}^{-1}$ which indicates asymmetric vibration of C-H bond,

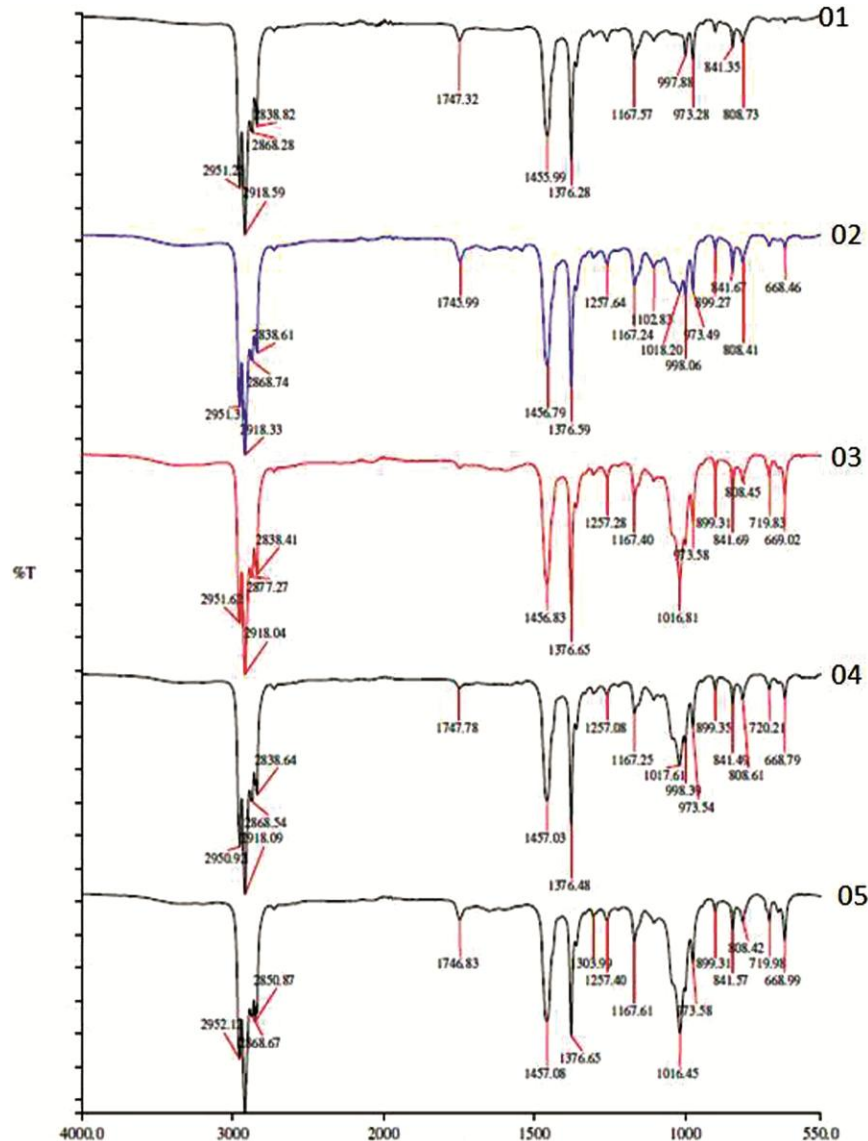


Fig. 1 — The FTIR spectra of polypropylene based hybrid composites.

1747cm^{-1} (C=O bond formation between starch and anhydride group), 1456cm^{-1} and 1376cm^{-1} (indicates CH_3 group), peaks at 1167 , 1016 , 973cm^{-1} indicated that polypropylene is present in all hybrid composites. The peak observed at 1747cm^{-1} indicating that anhydride group is present as compatibilizer. The peaks at 609 , 708 and 841cm^{-1} confirms the presence of saccharides structure, which confirm the present of *Agave Americana* natural fibre.

4.2 Morphological analysis by SEM

Figure 2 shows scanning electron micrographs (SEM) of polypropylene based hybrid composites of natural fibre, nano talc and starch.

These images show that the thermoplastic starch and natural fibres are being continuously and uniformly dispersed in polypropylene and form a finely dispersed homogeneous phase of the composite. The PPST20TL01NF05 (sample no 2) having maximum content of natural fibre, this image clearly shows that the natural fibre *Agave Americana* were well embedded in the polymer matrix, and this improved the compatibility and adhesion among the filler-matrix polymer of hybrid composites.

4.3 Differential scanning calorimetry (DSC)

DSC thermograms in Figs (3 & 4) show the heating and cooling curves between temperature 50°C to 200°C at a standard rate of cooling and heating. The readings

show that melting temperature does not modified with different composites, the crystallization temperature slightly increase in case of sample 3 as shown in Fig. 4.

It is also observed that enthalpy of melting is also maximum for sample 3 at value 104.9 J/g . In this study it is observed that talc's nucleating property is subdued in the presence of natural fibre and starch. However the result shows uniformity in hybrid composite which indicates proper and uniform adhesion and stability of the composite. The details of test summarize in Table 2.

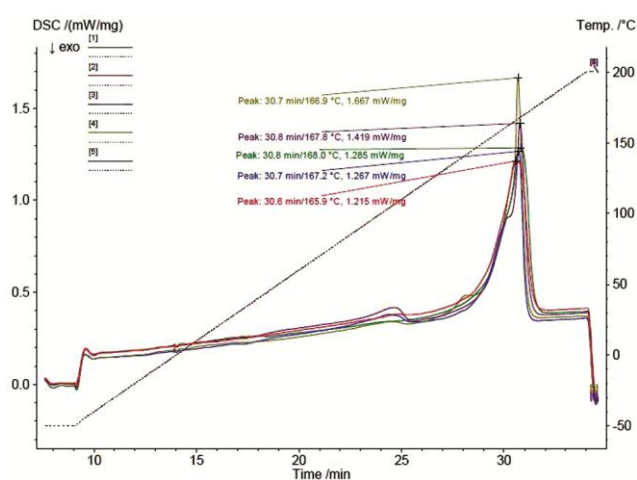


Fig. 3 —The melting point temperatures of polypropylene based hybrid composites.

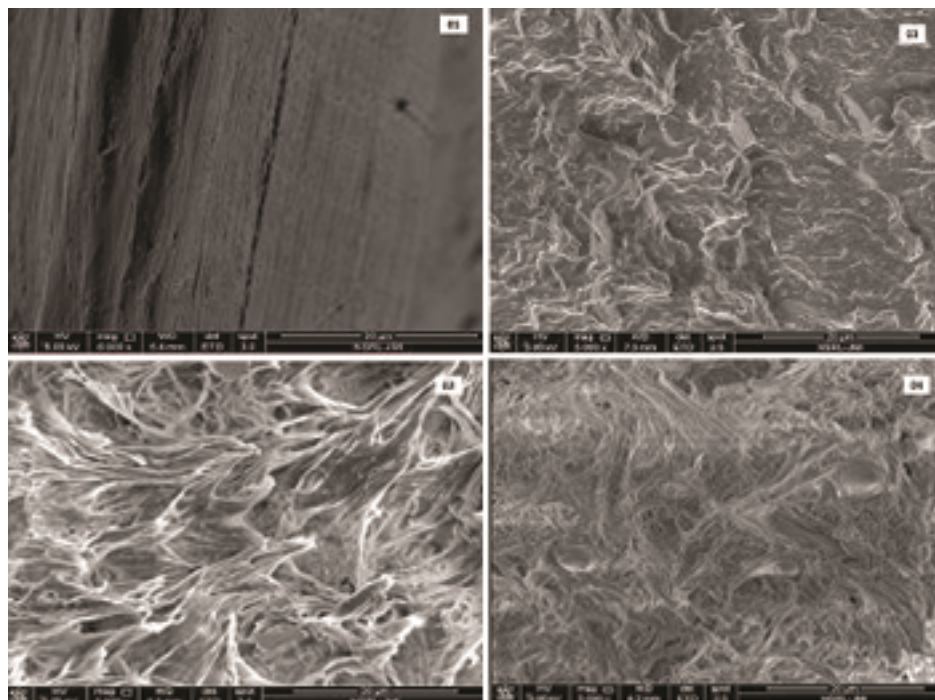


Fig. 2 —The SEM micrograph of polypropylene based hybrid composites.

Table 2 — The melting, crystallization and enthalpy of polypropylene based hybrid composites.

S.No.	Sample	Melting Point (°C)	Crystallization Temp(°C)	ΔH melting(J/g)	ΔH of crystallization(J/g)
1	PP	168.0	123.8	97.33	114.7
2	PPST20TL01NF05	165.9	119.2	93.59	106.4
3	PPST20TL03NF03	167.5	124.4	104.9	101.2
4	PPST20TL05NF01	166.9	122.4	90.62	102.6
5	PPST00TL05NF00	167.8	122.2	99.32	103.4

Table 3 — The heat deflection temperatures of polypropylene based hybrid composites.

S.No.	Sample	HDT(°C)	Deflection(mm)
1	PP	123.95	0.250
2	PPST20TL01NF05	107.25	0.250
3	PPST20TL03NF03	109.50	0.250
4	PPST20TL05NF01	117.05	0.250
5	PPST00TL05NF00	115.60	0.250

Table 4 —The vicat softening temperature of polypropylene based hybrid composites.

S.No	Sample	VST(°C)	Deflection(mm)
1	PP	149.35	1
2	PPST20TL01NF05	147.80	1
3	PPST20TL03NF03	142.80	1
4	PPST20TL05NF01	145.50	1
5	PPST00TL05NF00	142.80	1

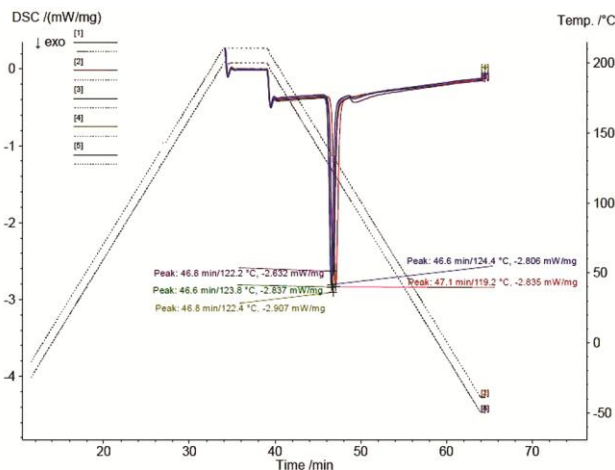


Fig. 4 — The Crystallization temperatures of polypropylene based hybrid composites.

4.4 Heat deflection temperature (HDT)

The Heat deflection temperature of all sample are preconditioned at 23 +/- 2°C and 50 +/- 10% RH for 40 hrs. The stress for each sample is kept constant at 0.45 MPa and employing heating rate of 120°C/hr. For HDT calculation deflection is 0.25 mm for each sample for each reading we took the average of two samples for more reliability. The values for each sample illustrated in Table 3. The incorporation of

nano talc increased the hardness of hybrid composites and hence sample no. 4 and 5 shows highest level of heat deflection temperatures.

4.5 Vicat softening temperature (VST)

For VST all samples are preconditioned at 23 +/- 2°C and 50 +/- 10% RH for 40 hrs. Load during testing kept constant at 10-0 +/- 2N and heating rate employed is 50 +/- 5°C/hr. Deflection during test kept constant at 1mm. VST temperature for sample 3 and 5 is constant at 142.80 °C, maximum VST temperature for pure PP is 149.35 °C . For each reading we took average of two samples. Values for each sample were shown in Table 4.

5 Conclusions

The FTIR, morphological, and thermal properties of Polypropylene Hybrid composites were studied. The morphological characterization based on scanning electron microscopy (SEM) has confirmed that natural fibres, starch and nano talc were distributed uniformly, indicating a good dispersion of natural fibres, nano talc and thermoplastic potato starch in the PP/PP-g-MA matrix.

Vicat softening point has been utilised to indicate the service temperature of polymer based hybrid composites. The VSP was increase with fibre loading and effect is proven in sample no. 2 having highest content of natural fibres *i.e.* agave Americana.

Heat deflection temperature (HDT) confirms that with increase of talc (nano level inorganic filler) content, the thermal stability improved for the hybrid composite. The presence of low level of natural fibres in sample 4 with 5% nano talc content shows highest level of HDT. DSC test shows that crystallization temperature slightly improved. In hybrid composite talc's nucleating property subdued due to natural fibre and starch, whereas melting temperature does not modified for the composite.

Homogeneous mixing of the filler is a positive indication which will help to replace the synthetic fibres. This hybrid composite can further be tested for various mechanical properties in future. This will help

to produce light weight, thermal resistant and toughdurable environmental friendly material in future.

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