Dynamic Mechanical Properties of Hemp Nanofibre Reinforced Epoxy Composite

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Abstract. In present study, dynamic mechanical properties of hemp nanofibre reinforced epoxy composite were carried out. The hemp nano composites were fabricated by hand lay-up technique followed by static compression using varying wt. % (1 to 5 wt. %) of nanofibres. Dynamic mechanical properties were studied in terms of storage modulus ($E'$), loss modulus ($E''$), damping ($\tan\delta$) and glass transition temperature ($T_g$). In addition, thermal stability of prepared nano composites was also analyzed using thermogravimetric analysis (TGA). The results suggested that composite with 2 wt. % nanofibres had overall better thermal and dynamic mechanical properties than other all composites.

1. Introduction

Bio composites are the most demanding materials owing to advantages such as low density, and high specific strength and modulus [1-8]. Nano technology has been received attention in field of bio composites by researchers because of improved surface area to volume ratio offered by nano particles. The homogeneous dispersion of nanosized fibre into polymer matrices can affect significantly their thermal properties as well as dynamic mechanical properties. Besides, biodegradability and renewability, fibres in nano dimensions have high thermal stability and improved glass transition temperature [9]. Recently, nanocellulose fibres have been used in (i) biomedical applications i.e. artificial blood vessels and dental tissue regeneration, (ii) electronic industry i.e. head phones, (iii) digital displays i.e. electronic paper and (iv) packaging industry [10]. Therefore, many researchers have reported studies on production of nanofibre and properties of nanofibre reinforced polymer composite. Khalil et al. [11] studied the methods of the production of nano sized fibres. Padal et al. [12] investigated the thermogravimetric analysis of jute nano fibre with varying percentage weight (1% to 5%) in epoxy resin based composite and found that the rate of degradation was slightly lower in case of 5% composite. Alemdar et al. [13] studied the properties of biocomposites of wheat straw of nanofibre and found that the degradation temperature of the nanofibres reached beyond 290 °C and also observed that the thermal stability of the nano fibres increases after chemical treatment.

The high surface to volume ratio, homogeneity in dynamic mechanical properties, thermal properties and mechanical properties are motivated for using fibres in the form of nano sized into polymer composites.
2. Materials and Method

2.1. Materials
In present work, Hemp nanofibres are used as reinforcement and epoxy resin as matrix. Hemp fibres were purchased from Bamboo Fibre Development Board, Dehradun, India. Hemp fibres were treated by alkali treatment (5% NaOH). The treated hemp fibres were converted into nanosized by using Planetary Ball mill at the speed of 750 rpm for 90 hrs. The Hemp nanofibres were mixed into epoxy with the help of Magnetic stirrer instrument at the speed of 450 rpm for 30 minutes. Epoxy resin (Lapox ultra) and corresponding curing agent (HardnerLapox ultra) were mixed in ratio 10:8 by weight to make the matrix.

2.2. Fabrication of composites
Hemp nano fibres were reinforced into epoxy resin with different wt. % (0, 1, 2, 3, 4, and 5%) to prepare the composites. The epoxy and corresponding hardener were mixed in a ratio of 10:8 by weight as recommended by suppliers. The mixture was stirred by the magnetic stirrer to disperse the hemp nano fibres and polymer matrix. A stainless steel mould having dimensions of 300 × 200 × 3 mm³ was used. The composite sheets were made by conventional Hand lay-up method followed by light compression moulding technique. Paraffin wax was used as releasing agent to facilitate easy removal of the composite from the mould after curing. Each composite sheet was cured under a load of 50 kg for 24 hours before it was removed from the mould. The dimension of specimens were cut as per as ASTM standard using a diamond cutter for dynamic mechanical analysis. The nomenclature of prepared samples of hemp nanofibres reinforced epoxy composites are given as follows:

| Nomenclature | Description |
|--------------|-------------|
| Epoxy        | Neat Epoxy  |
| H1           | Epoxy based composite having 1 wt. % of Hemp nano fibre |
| H2           | Epoxy based composite having 2 wt. % of Hemp nano fibre |
| H3           | Epoxy based composite having 3 wt. % of Hemp nano fibre |
| H4           | Epoxy based composite having 4 wt. % of Hemp nano fibre |
| H5           | Epoxy based composite having 5 wt. % of Hemp nano fibre |

2.3. Dynamic mechanical analysis
The Dynamic Mechanical Analyzer (Seiko Instruments DMA 6100) instrument was used to measure the dynamic mechanical properties at mode of 3 point bending test as a function of temperature. The size of samples of hemp nano fibre reinforced epoxy composites were cut into dimension 50 mm×13 mm×3 mm according to ASTM D 5023. Experiment was carried out in the temperature range 25-200 °C at the heating rate 2 °C per minute and at 5 Hz frequency. The dynamic mechanical properties such as storage modulus, loss modulus and damping factor of prepared samples were studied.

2.4. Thermogravimetric Analysis
The thermal degradation behaviour of neat epoxy resin and hemp nano fibre reinforced composites was investigated in TGA instrument. TGA experiment was carried out within temperature range 30-600°C with heating rate 10°C per minute.
3. Result and Discussions

3.1. Dynamic mechanical analysis at 5 Hz frequency

3.1.1 Storage modulus (E')

Fig. 1 shows the variation of storage modulus of hemp nano composites as a function of temperature at 5 Hz frequency. On comparing the storage moduli of epoxy and hemp nano composites, it was found that the values of $E'$ continuously decreases on increasing in temperature due to loss in stiffness. In the glassy region, the maximum value of $E'$ was found for hemp composite H2. There were a steady fall in values of $E'$ for hemp composite but a sudden fall in epoxy resin.

![Figure 1. Variation of storage modulus with temperature of epoxy and hemp nano composites](image)

3.1.2 Loss modulus (E'')

At 5 Hz frequency, the variation of loss modulus of epoxy and hemp nano composites as a function of temperature is shown in Fig. 2. It was found that the loss modulus increases up to glass transition temperature then decreases on increasing the temperature for epoxy and hemp nano composites. The maximum value of loss modulus was seen for hemp nano composite H2. It was also observed that the glass transition temperature was improved on increasing fibre loading up to 2 wt. %. The values of glass transition temperature for epoxy and hemp nano composites are shown in Table 1. From Table 1, it can be observed that hemp composite H2 had better glass transition temperature as compared to epoxy and other hemp composites.
3.1.3. Damping (Tanδ)
Damping is the ratio of loss modulus to the storage modulus which can be calculated by taking tangent of phase difference between sinusoidal stress and strain. The variation of damping of epoxy and hemp nano composites as a function of temperature at 5 Hz is shown in Fig. 3. The maximum and minimum values of Tanδ were observed for hemp nano composites H1 and H2 respectively. The values of glass transition temperature of epoxy and hemp nano composites are given in Table 2. From Table 2, it can be seen that hemp nano composite H2 had the maximum value of glass transition temperature.

3.2. Thermo Gravimetric Analysis (TGA)
The thermal degradation behaviour of epoxy and hemp nano composites is shown in Fig.4. Initial weight loss about 5 wt.% was observed up to 300 °C, due to removal of moistures from the composites. After 300 °C, a sudden drop in the weight loss about 50 wt.% of composites were observed, due to degradation and volatization of epoxy along with nano fibres present in composites. Percentage weight losses and their corresponding temperatures epoxy and hemp nano composites are given in Table 2.
Figure 4. TGA curves of epoxy and hemp nano composites

Table 1. Glass transition temperature (\( T_g \)) from loss modulus and tan delta curve

| Composite | \( T_g \) (°C) from loss modulus curve | \( T_g \) (°C) from Tan\( \delta \) curve |
|-----------|--------------------------------------|--------------------------------------|
| Epoxy     | 38.14                                | 52.81                                |
| H1        | 40.69                                | 54.64                                |
| H2        | 43.93                                | 57.13                                |
| H3        | 42.76                                | 52.33                                |
| H4        | 42.00                                | 54.22                                |
| H5        | 40.69                                | 56.50                                |

Table 2. Weight loss events with temperatures of epoxy and hemp nano composites

| Temperature | Weight loss (%) |
|-------------|-----------------|
|             | Epoxy | H1    | H2    | H3    | H4    | H5    |
| 100         | 0.013  | 0.0137| 0.169 | 0.137 | 0.17  | 0.264 |
| 200         | 0.57   | 0.764 | 1.03  | 0.90  | 0.92  | 1.24  |
| 300         | 2.66   | 4.70  | 3.48  | 3.50  | 3.80  | 5.60  |
| 400         | 48.13  | 48.53 | 50.00 | 49.50 | 48.6  | 53.07 |
| 500         | 85.07  | 85.52 | 84.20 | 84.20 | 84.03 | 82.85 |
| 600         | 96.45  | 97.30 | 98.65 | 97.70 | 98.29 | 97.7  |

4. Conclusions
Hemp cellulose nanofibres were successfully extracted by chemical and mechanical method, and dynamic mechanical and thermal properties of their epoxy based composites are investigated. On
increasing the hemp nanofibre loading up to 2 wt.%, dynamic mechanical properties were seen improved. The maximum value of glass transition temperature was found for hemp nano composite H2. Moreover, thermal stability of epoxy was enhanced due to incorporation of hemp nanofibres.

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