

Dynamic Mechanical Analysis of Jute Nano Fibre Reinforced Composite

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Abstract- Polymer based nanocomposites have been subject of investigation by the dispersion of jute nanofibres within epoxy polymer matrix. Thermal properties of a composite play a vital role in evaluating the product performance as well as predicting the processibility characteristics in polymers in specific applications. The thermal behaviour of the interface between nanofibres reinforcement have been studied by thermogravimetric analysis and Differential scanning calorimetry. The addition of nanofibres with different weight percentages has been (1wt. % to 5wt. %) studied and compared with base composites. The thermal stability of nanocomposite is significantly improved due to incorporation of jute nanofibres. These nanofibre reinforcement between the matrix resin molecules offered some resistance towards the thermal degradation. DMA measurements are made using a single frequency and constant deformation amplitude while varying the temperature. Measurements, where the amplitude of deformation is varied or where multiple frequencies are used, provide further information. The dynamic properties are expressed in terms storage modulus, loss modulus and damping factors which are dependent on temperature, time and frequency. The dispersion of nanosized fibre within polymer polymer matrices can affect significantly their physical properties. The main source of modification is due to the interface macromolecular chains-nanofibre and to the huge area of the nanofibres.

I. INTRODUCTION

Nanocomposites the physical properties of the interface become dominant over the bulk properties of the polymer matrices. The addition of nanosized fibres to polymer matrix typically enhances the thermal and thermo-oxidative degradation of the polymer, the young modulus, and the strength of the polymer matrix and affects the crystallization process. In most cases, the effect of nanofibres consists in a rather modest increase of the temperature at which the mass loss of the polymer is highest. This parameter easily obtained by thermogravimetric analysis (TGA) and Differential scanning calorimetry (DSC) analysis can be considered a fingerprint of the formation of a polymer nanofibre interface. The degradation of polymer composites may be aggravated due to the interaction of a nanofibre with polymer molecules in the presence of thermal energy from working environments. These have been a considerable amount of work on the thermal degradation of polymers in nanocomposites. Dynamic mechanical that measures the properties of materials as they are deformed under periodic stress resin (DGEBA) along with a Triethylene Tetramine hardner (HY956) was used for extremes. Work.

Bisphenol- A epoxy the investigations. Glass fibre woven plain exhibits a phase difference between those fabrics were supplied by M/s Ecmas Pvt Limited, Visakhapatnam. The average fibre area weight (FAW) of glass fibre was 90°. However, most real world materials including

polymers are viscoelastic and 410g/m². Both structural and functional properties of coatings can be modified by filling with nanomaterials. is purely elastic, the phase difference between the stress and strain sine waves is 0° (i.e., they are in phase). If the material is purely viscous, the phase difference is The aim of the work is to study the thermal degradation behaviour of nanofibre reinforcement in epoxy polymer matrix. In DMA a variable sinusoidal stress is applied, and the resultant sinusoidal strain is measured. If the material being evaluated and to compare the thermal degradation of different weight percentage reinforcement with base composite. analysis is a thermal analysis technique.

II. EXPERIMENTAL WORK

A. Materials

Nanofibres were extracted from natural fibre Jute by mechanical milling and chemical treatment. The structural morphology and size was analysed by Scanning electron microscope (SEM) and X-ray diffraction. Epoxy, one of the most commonly used materials is the base polymer material for the current research.

B. Characterization of Nano fibres

X-Ray diffractometer (Phillips made X Pert Pro Diffractometer model) analyzed the nanofibres of jute at a scanning rate 4°/min with Cu, K_α radiation at 45 kv and 40mA. The size of the jute fibres were determined by using Scherrer formulae. The Scanning Electronic microscope (SEM) images of jute fibres and microfibrils were taken with JEOL model Scanning Electronic microscope. It is observed that the obtained jute fibres are micro to nano scale at different milling hours.

C. Preparation of Jute nanofibre composites

The Jute nanofibres with varying percentage weight (1wt.% to 5wt.%) reinforced in epoxy resins to prepare nanofibre composites by hand lay-up technique. The composites were prepared by using glass fibre woven mat and epoxy resin with 50 wt.% / 50 wt.% fraction. The epoxy resin is reinforced with different weight percentage

of Jute nanofibre reinforcing (0, 1, 2, 3, 4 and 5 wt. %) was mixed by using a mechanical stirrer at 750 rpm for 30 minutes at room temperature. Then, for each 100 gm of epoxy resin, 12% of curing agent TETA was added to the mixture by weight and thoroughly mixed until it became uniform. Finally, the composite is allowed to fully cure at room temperature for 24 hours. The finished laminate was used to prepare samples for investigating the thermal properties as per ASTM standards.

D. Thermal analysis of nanofibre composites.

The thermogravimetric analysis (TGA) is commonly employed in research and testing to determine characteristics of polymer nanofibre composites. This analysis is used to study the degradation temperatures, absorbed moisture content of materials, the level of inorganic and organic components in materials, decomposition points of explosives and solvent residues. It is also often used to estimate the corrosion kinetics in high temperature oxidation. The specimens were heated from room temperature to 600°C at heating rate of 10 °C /min. For comparing the effect of different weight percentages (1-5 wt. %) of jute nanofibre composites were also oven aged in hot air at various temperatures for 10 min.

Differential scanning calorimetry (DSC) of base composite and nanofibre reinforced with different weights were carried out by using Pyris Diamond DSC model Perkin Elmer apparatus. The present investigation 5-10 mg of samples at scanning rate of 20°C/min and temperature of 30-300°C under nitrogen atmosphere. Subsequently, the samples were held at 300°C for 5 min and then cooled from 300° to 30°C at the rate of 20°C/min. Corresponding melting temperature; heat of fusion and crystallization temperature were recorded.

In DMA the Jute nanofibre composites of different weight percentages (base composite and with 1 to 5 wt.% JNF/epoxy composite) specimen samples were prepared as rectangular bars of size 40x10x3 mm³ as per ASTM standards .The specimens are subjected to the three point bending test method as shown in Fig.1

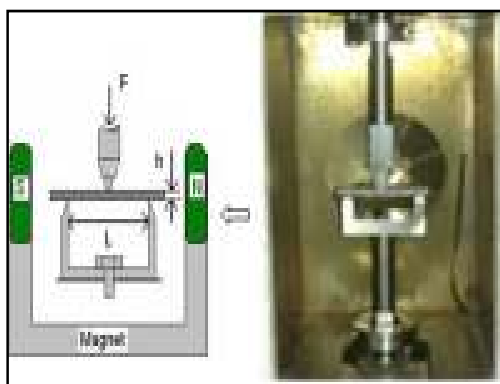


Fig.1. Three point Bending Test set up

III. RESULTS AND DISCUSSION

A. Differential scanning calorimetry:

DSC monitors heat effects associated with phase transitions and chemical reactions as a function of temperature. Crystallization is a typical exothermic process and melting a typical endothermic process. DSC curves are used to obtain thermal information such as the glass transition temperature, crystallization temperature and melt temperature. The DSC curves of base composite and 1 wt.% to 5 wt.% jute nanofibre reinforced composites were compared in the temperature range of 30° c to 300°C to determine the thermal transitions as shown in the figure.2. It can be seen that the glass transition temperature of the nanocomposites did not change significantly due to the addition of nano jute fibres, however the addition of jute nanofibres did affect the crystallization behavior of the polymer composite.

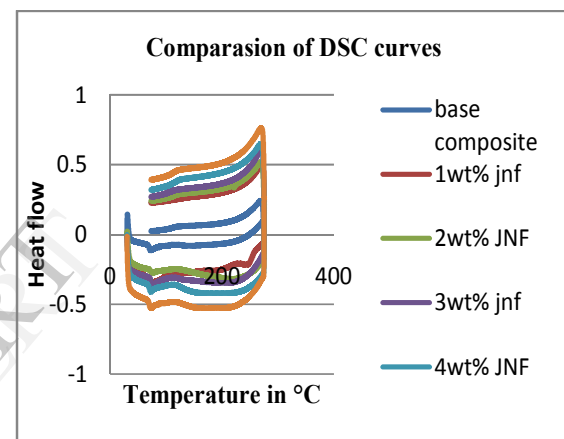


Fig.2. Comparison DSC curves of base composite and nanofibre reinforced composites

The crystallization began at a higher temperature 71°C for pure epoxy composite where as for all jute nanofibre reinforced composites 72°C to 75°C. The addition of nano jute fibres increased the crystallization temperature T_c by up to 1°C- 4°C compared to the pure epoxy composites. This result indicates that the nucleating effect of jute nanofibres composites was strengthened. The jute nanofibres played the role of nucleating agent and facilitated crystallization. This is due to stronger interaction between nano jute fibre surface and chains. The nucleating effect of jute nanofibre could also explain the increase of crystallinity. The nano scale dispersion of the filler and its orientation in the matrix are among these factors. All hybrid composites had a higher melting temperature compared to pure epoxy composites.

B. Thermogravimetric analysis

The thermal degradation behaviour of pure epoxy composite and Jute nano fibre reinforced polymer composites with 1-5wt% Jute nanofibre loading has been employing by TGA curves as shown in figure. A sudden drop in the mass of the sample indicates the thermal

degradation of the materials, however adding the jute nanofibre in the matrix increased the degradation temperature onset of the composites and also increased the decomposition temperature.

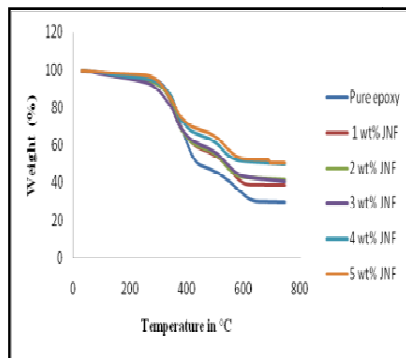


Fig.3. Comparison TGA curves of base composite and nanofibre reinforced composites

From the Fig.3 It is evident that the thermal degradation of base composite is started at 344.2°C and 100 % degradation was noticed at 560°C. However with the incorporation of jute nanofibres, there was substantial enhancement in the thermal stability of the nanocomposites with an initial degradation temperature at 370°C and final decomposition at 583°C. This indicates that a significant increase in the jute nanofibre content of fibre reinforced composite play an important role in controlling its rate of thermal degradation. The major source of thermal stability improvement may be due to the fact that a highly cross linked multilayer epoxy matrix which produces additional intermolecular bonding between fibre and matrix allowing more thermal energy distributed over these bonds within the interface.

C. Dynamic Mechanical analysis

DMA results of Jute nanofibre reinforcement with pure epoxy Polymer composite. The influence of percentage weight of 1 to 5% nanofibre reinforcement has been studied by Dynamic mechanical analysis. The base composite and nanofibre composite samples were subjected to dynamic mechanical analysis in order to understand the nanofibre interaction in the composite with the increase in percentage weight (1-5wt. %) nanofibre. DMA results show that the changes in a composite material, i.e storage modulus, loss modulus and tan delta with increasing temperature may be able to reveal a great deal about its thermal transition.

1) Storage Modulus

The Jute nanofibre composites with different weight percentages were tested by the three point bending method and the storage modulus comparison graphs were shown in Fig.4

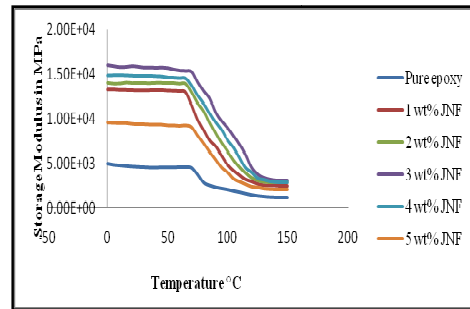


Fig.4 Comparison of Storage Modulus vs Temperature of base/ Jute nanofibre Composites

It is observed that the nanocomposites show a slightly higher storage modulus with the addition of nanofibres compared to pure epoxy composites. The storage modulus of all the composite samples dropped drastically between 65–80 °C which was their glass transition region. The glass transition temperature of pure composite has been found to be 65 °C, whereas the transition temperature of Jute nanofibre composites were at 70 – 75 °C. The storage modulus of Jute nanofibre reinforced composite increases with an increased fibre content in the glassy as well as rubbery region. This observation is most likely related to the interaction between the hydroxyl group of epoxy and hydroxyl groups that are known to exist on damaged sites on the Jute nanofibre surface or as a result of atomic scale defects formed along Jute nanofibre composites. The increase in glass transition also suggests that the degree of interaction between the polymer chains and the surface of the Jute nanofibre increase. The storage modulus increases in the presence of the Jute nanofibre which could be concluded as a combined effect of the nanofibres embedded in a viscoelastic matrix and the mechanical limitations. At high concentrations the fibre reduces the mobility and deformation of matrix, and in this case, the stress can be transferred from the epoxy matrix to the Jute nanofibre reinforcement.

2) Loss Modulus

The loss modulus value which reflects the amount of viscous dissipation in the composites revealed some interesting behaviour. The loss modulus is a function of temperature (E'') for both the pure epoxy and 1 to 5 wt. % of Jute nanofibre composites.

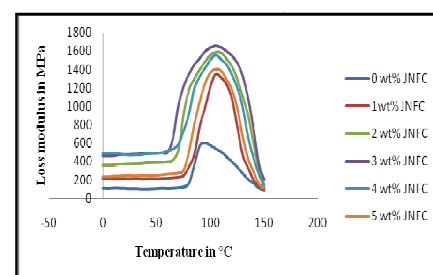


Fig.5 Comparison of Loss modulus curves of base/ Jute nanofibre composites

Fig.5. shows the comparison between the loss modulus vs temperature plots of the base/nanofibre composite samples. The T_g of the Jute nanofibre composites are slightly shifted to a higher temperature with a broader range of the transition region than the pure epoxy composite sample. As the loss factors are sensitive to molecular motions it could mean that the mobility of the polymer molecular chains decreases as the chains were hindered by the nanofibre reinforcement leading to shift of T_g . Good adhesion of Jute nanofibre with the surrounding polymer matrix would additionally benefit the dynamic modulus by hindering the molecular motion to some extent. The loss modulus increases with an increase in the percentage of Jute nanofiber. This may be due to energy losses caused by the rearrangements of the molecules and Jute nanofibre as well as the internal friction between the Jute nanofibre and the epoxy polymer matrix.

3) Tan Delta

The peak of the $\tan \delta$ curve is a popular measurement point for the T_g and it is usually easier to isolate than to determine the onset of the drop in the storage modulus. The $\tan \delta$ verses temperature

curve can be used to determine much more about a system than just its T_g . The width of the $\tan \delta$ peak can indicate how homogeneous a system. A system has very broad peaks which are generally composed of different polymer chain lengths or structures which gives rise to a boarder temperature range, that initiate significant viscous chain motions for the various components. On the other hand, systems with narrow peaks generally have a more narrow distribution of chain types and molecular weights. The height or amplitude of the $\tan \delta$ curve is directly related to a materials ability to dissipate energy through segmental motion. Systems with tall $\tan \delta$ peaks have higher ratios of energy absorbing viscous motions and are therefore generally tougher systems than those with low $\tan \delta$ amplitudes.

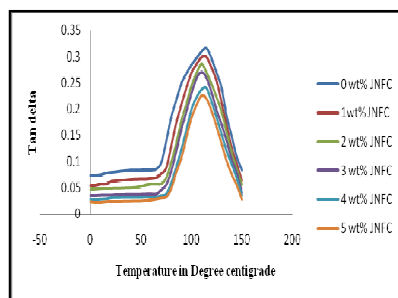


Fig.6 Comparison curves of Tan delta for base/ Jute nanofibre composites

From the Fig.6 it is noticed that the intensity of $\tan \delta$ peak decreased with an increased in Jute nanofibre reinforcement compared to the pure epoxy composite. The maximum damping parameter was observed as 0.220 of 5 wt% JNF composite and its improvement is 36 % when compare with pure epoxy composite. This might be due to

the decrement of the mobility of polymer molecular chains as hindered by

reinforcement which led to a reduction of height and sharpness of the peak in the curves. The increase in modulus together with positive shift in $\tan \delta$ peak position can be attributed to the physical interaction between the polymer and reinforcements that restrict the segmental mobility of the polymer chain in the vicinity of the nanofibre reinforcement. The decline in the elastic properties combined with the increase in damping at higher temperatures is attributed to the damage of the polymer chain structure.

IV Conclusions

The thermal properties of epoxy polymer filled with Jute nanofibre composites under nitrogen were investigated by TGA, DSC and DMA. The experimental data is used to compared the base/nanofibre composites. The nanofibre composites show some stabilization destabilization interaction according to the temperature region. The nanofibre reinforcement improves the crystallization temperature and thermal degradation temperature. Jute nanofibre stabilizes the polymer molecules and delays the occurrence of major cracking in the primary weight loss stage. A greater stabilization or destabilization effect was observed increasing with the amount of nanofibre composites. However nanofibre composites of smaller particle size have greater effect on the thermal properties of nanofibre composites. The dynamic properties of the polymer epoxy resins are temperature dependent. The storage modulus' decreases with increasing temperature, while the loss modulus increases (the fact which should facilitate internal damping). The DMA study indicates that the increased modulus, together with the positive shift in $\tan \delta$ peak position, is attributed to the physical interaction between the

polymer and nanofibres that restrict the segmental mobility of the polymer chains in the vicinity of the Jute nanofibres.

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