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ORIGINAL ARTICLE

Effect of nanoclay addition and chemical treatment on static and dynamic mechanical analysis of jute fibre composites

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Abstract

In this article, the influence of alkali treatment and addition of montmorillonite nanoclay as filler on mechanical and visco-elastic behaviour of jute fibre polymer composite were investigated. The composites are fabricated using 5wt% of nanoclay, untreated and chemically treated jute fibre of various percentage by handlayup method. The static mechanical properties like tensile, flexural, impact and inter laminar shear strength are studied as per respective ASTM standard. The dynamic mechanical analysis was carried out to evaluate storage modulus and damping factor of the prepared composite. The composition and structure of the functional groups of modified fibres were examined by Fourier transform infrared spectroscopy. The results showed that the interaction of filler addition and NaOH+$KMnO_4$ treatment of fibres have significantly improved the tensile, flexural and impact properties to 47.12, 201.13, 172.61MPa respectively. Dynamic mechanical analysis results revealed that the incorporation of filler increases the storage modulus and glass transition temperature. The incorporation of 5wt% clay and 25wt% jute fiber increase the glass transition temperature of the composite material from 109 to 115 °C.

Keywords: *chemical treatment, glass transition temperature, mechanical properties, nanoclay, natural fibre.*

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1. Introduction

Natural fibre reinforced polymer composites offer more advantage over conventional material owing to low cost, low density and high specific properties and these hybrid composites are one of the promising fields in polymer science that makes attention for application in a variety of sectors ranging from aircraft to the building industry[1,2]. Renewable nanomaterials are used as reinforcements in the field of nanoscience and nanotechnology in order to have advanced materials with novel properties[3]. Nanoclay/natural fiber hybrid composites have great attention recently due to their wide variety of properties in automotive, biomedical, food packaging and other consumer applications with better mechanical, thermal, optical and barrier properties[4]. Natural fibres like jute, bamboo and coir with varying span length subjected to mechanical and physical properties are studied and it was found that the young's modulus value increases gradually on increasing the span length. On investigating the SEM images of fiber, jute shows smoother surface when compared to bamboo and coir. This smooth surface with less porosity provides higher tensile strength when compared to coir fiber which has higher porosity[5]. A comprehensive literature review on various aspects of biocomposites and natural fibres with reference to physical modifications such as corona, plasma treatment and chemical modifications like alkaline, acetylation, silane, maleated coupling, enzyme treatment are discussed[6,7]. The jute fabrics reinforced with polypropylene matrix with various chemical treatments like hydroxyethyl methacrylate, urea, and $KMnO_4$ were studied and it was found that the improved mechanical properties are achieved in the combinations of all the above mentioned treatments rather than the individual treatment[8]. Fibres like hemp and banana are treated with chemicals like acetyl, silane, alkali with various concentrations and are examined by using SEM, FTIR spectroscopy. Treatment with chemical decreases the loosely linked hydroxyl group of hemicellulose, lignin and breakdown the covering surface which exposes the cellulose surface for better adhesion. This makes fiber less hydrophilic and improved mechanical properties[9,10]. The mechanical and thermal properties of nanocomposite i.e., epoxy resins filled with nanosilica are investigated and it was found that the decrease in the glass transition temperature was noted with higher silica loading due to decrease in cross-link density of high mobility regions but enhanced mechanical property with increasing filler content[11,12]. Addition of organo modified and montmorillonite clay as filler in the matrix and fiber significantly improves the mechanical properties that encourage to use of structural purposes[13,14]. The interlaminar shear strength and free vibration characteristics of hybrid nanocomposite plates by reinforcing glass fibre mat, coconut sheath and organically modified nanoclay in the polymer matrix was analysed. They observed that the second phase nanoscale filler in the matrix and fibre considerably improves the damping of the composites and shear strength property[15,16]. The dynamic mechanical analysis of particulate filled fibre reinforced polymer composites were investigated and it was found that the addition of filler shows better viscoelastic behaviour and higher glass transition temperature. The heterogeneity of the composite samples was analysed by using cole-cole plot[17-21]. The effect of silane, nano hybrid coatings on mechanical properties of basalt fibres are studied and it was found that the treated cum coated fibre shows 23% higher tensile strength than non-coated fibres[22]. The effect of water absorption on the mechanical properties of the hybrid composites was studied and it was found that the tensile and flexural properties were significantly reduced under wet condition. Similarly, in the clay filled hybrid composites the rate of moisture absorption decreases because the dispersal of nanoclay in the composites acts as an obstacle and restricts the flow of water in all direction of the composites[23,24]. The woven kevlar- kenaf hybrid composites with various volume fractions are prepared and subjected to ballistic impact properties. From the results, it is found that the ballistic properties of the composites increase with the increase in thickness and areal density of the hybrid composites[25]. The nanomechanical properties of different loading levels of clay filled polyester composites through Vickers hardness test was studied and it was found that inclusion of 5wt.% clay into the polymer matrix results in an improvement in hardness of 26.52% and the fracture toughness depends on the montmorillonite clay content[26]. From the above literatures, it is found that addition of fillers, fibre parameters and chemical treatments improves the mechanical properties of the composite to a greater extend. Hence, in this work, optimum jute fibre parameters are predicted by carrying out mechanical tests. Further, to this an optimum clay percent is added with it to form hybrid composite. Finally, fibres are treated with alkalis, the combined effect of chemical treatment, filler addition on mechanical and dynamic mechanical properties are analysed.

2. Materials and Methods

2.1 Materials

Jute fibres of yarn type was used as natural fibre reinforcement and it was procured from National Jute Board, Chennai, India. The montmorillonite nanoclay was obtained from Sigma Aldrich, Bangalore and India, it was used as filler in the polyester resin. The unsaturated polyester resin along with Cobalt napthanate as accelerator and Methyl Ethyl Ketone Peroxide are act as a catalyst, which are purchased from Vasavibala Resins, Chennai, India.

2.2 Nanocomposite preparation

Jute fibres of varying fibre content (i.e.,5%,10%,15%,20%,25% & 30wt.%) are used as primary reinforcement whereas montmorillonite nanoclay is used as secondary reinforcement in the polyester resin. Initially, jute fibre of 20 mm length with various percent are mixed with resin mixer (i.e., polyester and optimum clay) to fabricate composite. The hand layup method is followed for it. The optimum weight percentage of nanoclay (5wt %) obtained earlier[27] was mixed with unsaturated polyester resin by sonification technique at the optimal frequency of 10 kHz and 60 minutes duration. This nanocomposite laminates are allowed to cure for one day at room temperature. Then the specimens are cut from the laminate according to ASTM standards for different tests.

2.3 Short beam shear testing

The short beam shear test was conducted to find out the interlaminar shear strength (ILSS) by using Instron universal testing machine. The test was carried out according to ASTM standard D-2344 with a test speed of 2 mm/min. Composite plates were placed on two supports of diameter 3 mm and it was made free to rotate in order to have free lateral motion. Load was applied at the centre of the plate by using steel dowel of diameter 6 mm. The plate was loaded until fracture, and the fractured load was used to determine the apparent shear strength of the material. The ILSS was calculated for composite samples based on the Equation 1.

$$F^{sbs} = 0.75\ X\ P_{max}\ /\ (b\ X\ h) \quad (1)$$

where, F^{sbs} = short beam strength, MPa; P_{max} = max. load observed during the test, N; b = specimen width, mm; h = specimen thickness, mm.

2.4 Tensile, flexural and impact testing

Tensile and flexural tests (three-point bending) are carried out by using the Instron universal testing machine while impact test is carried out by using an Izod impact test set-up in Tinius Olsen machine. The Tensile test was carried out with a loading speed of 2 mm/min as per ASTM standard D-638. Three point bending test is carried out by using ASTM standard D-790. Impact test is carried out according to ASTM standard D-256 without notch. Each test is carried out on five samples and the average value is taken.

2.5 Fibre surface treatments

Untreated & chemically treated jute fibers were used for preparing the composites. Chopped Jute fibers (20 mm) were immersed in a vessel separately which contains 2% of NaOH aqueous solution, 2% of $KMnO_4$ and NaOH+$KMnO_4$ each for 1 hour at room temperature. The fibers were then washed with distilled water to remove the excess of sodium hydroxide and $KMnO_4$ on the fibers. Afterward, the fibers were dried out at 50 °C in an oven for 3hrs. Chemically treated jute fibres are then reinforced in polyester/ montmorillonite nanocomposites with optimum fibre (i.e., 15 wt. % obtained earlier[27]) content by using hand lay-up process. The reaction of jute fibre with potassium permanganate in the presence of sodium hydroxide is shown below (2).

$$Cellulose - OH + KMnO_4 \xrightarrow{OH^-} Cellulose - H - O - \underset{\underset{O}{\vdots}}{\overset{\overset{O}{\vdots}}{Mn}} - OK^+ \quad (2)$$

2.6 Fourier Transform Infrared (FTIR) spectroscopy

The FTIR analysis was carried out by using a JASCO FT/IR-6300 spectrometer for the analysis of functional group. About 5 mg of untreated and treated fibers were milled into a tiny particle and mixed with potassium bromide then placed on a disc. An average scanning speed of 2 mm/sec was recorded for each spectrum at the wave numbers which ranging from 399.19 to 4000.6 cm^{-1} with a resolution of 4 cm^{-1}.

2.7 Dynamic Mechanical Analysis (DMA) testing

Dynamic mechanical analyser (SEIKO, Model DMAI/DMSC 6100) was used for find out the dynamic properties such as storage modulus, loss modulus and tan δ of the nanocomposites with the specimen size of 50 mm × 50 mm × 3 mm. DMA test was carried out at a temperature range of 30-175 °C at 5 °C/minute with different frequencies of 0.1 Hz, 1 Hz, 2 Hz, 5 Hz and 10Hz under the tensile mode.

3. Results and Discussions

3.1 Interlaminar Shear Strength (ILSS)

The short beam test specimens are cut into required dimensions as per ASTM standards D-2344. Figure 1 shows the ILSS properties of the various composite tested. From the figure, it is found that the ILSS value for 5% nanoclay along with 5wt% jute fibre is 18.15MPa. On further addition of jute fibre i.e. 10, 15, 20wt% along with the 5wt% clay, the ILSS value increases by 10.3, 30.47, 40.72% respectively. From the Figure 1, it is found that the addition of 5% nanoclay with 25wt% jute fibre hybrid nanocomposite have enhanced the shear strength to 28.03 MPa with 54.44% increase when compared with 5% clay and 5wt% jute fibre and it was achieved due to enhanced bond characteristics, better interfacial areas and distinctive phase morphology of the fibre and nanoclay[28]. Further, it is clear that the addition of jute fibre in polyester matrix reduces the shear strength due to clustering of fibre in the polymer composite which attributing the non-uniform distribution of stresses[29].

3.2 FTIR analysis

From the FTIR spectroscopy the chemical structure of the cellulose, hemicellulose and lignin constituents for the untreated and chemically treated jute fibres were studied and shown in the Figure 2. From the figure, the peaks at 902 cm^{-1} and 1440 cm^{-1} were designated as C-H bending of amorphous and crystalline cellulose[9]. These peaks remain unchanged for NaOH and $KMnO_4$ treated fibres. This shows that the chemical treatments didn't affect the cellulose arrangement of the fibre. The peak near 1742 cm^{-1} was due to C-O stretching of the acetyl and carboxyl groups in hemicelluloses of the raw jute fibre. This peak was not present for alkali treated fibres. This can be due to the elimination of acetyl group present in hemicelluloses after chemical treatment. The peak near 1440 cm^{-1} was assigned to CH_3 deformation in lignin. The loss of lignin was found after alkali treatment and this is the reason for decrease in the absorption intensity ratio[30]. The absence of peaks at 1250 and 1550 cm^{-1} shows the removal of lignin and hemicellulose[31]. After potassium permanganate treatment, the aromatic band of lignin and cellulose intensity decreases and increase in the peak at 1720 cm^{-1} was found, which confirms the quinine formation on the surface of the fibre. Increase in the peak at 1026 cm^{-1} shows that the alkali treatment increases the hydroxyl group on the fibre surface[32]. The alkali and $KMnO_4$ treatment reduce the hydrophilic nature of the fibre and increases the fiber matrix adhesion[33].

3.3 Mechanical properties of composites with surface treated fibre

The chemically treated Jute fibre reinforced polyester/ montmorillonite hybrid nanocomposites fabricated are cut into required dimension as per ASTM standards and is tested for various mechanical properties. The results are shown in the Figures 3 to 5. From the results, it is found that the composite reinforced with both NaOH and $KMnO_4$

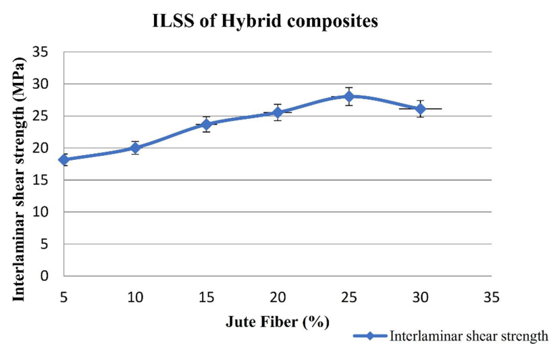

Figure 1. Effect of nanoclay content on interlaminar shear strength of Jute fibre reinforced polyester/ montmorillonite clay nanocomposites.

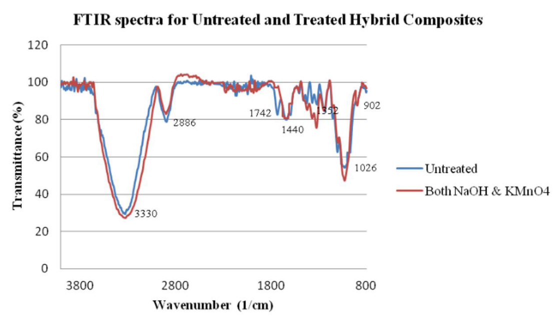

Figure 2. FTIR spectra curve for untreated and chemically treated NaOH and $KMnO_4$ fibre.

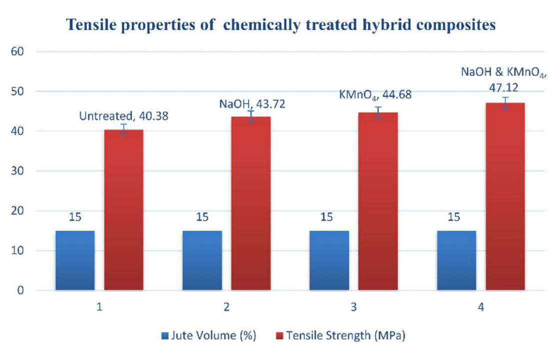

Figure 3. Comparison of the tensile properties of chemically treated jute fibre reinforced polymer composites with 5wt% montmorillonite clay as filler.

treated fiber shows an increase on the tensile strength from 40.38 to 47.12 MPa with respect to the composite made with the untreated fibers[30] as shown in Table 1. Similarly, the flexural and impact strength raises from 166.23 to 201.13 MPa and 151.58 to 172.61 MPa respectively with respect to untreated fibres. The fibres modified with sodium hydroxide aqueous solution along with the treatment of $KMnO_4$ enhances the property of the hybrid composite material as compared with the fibres without surface treatment. The results show that the chemical treatment improves the crystallinity of the fibres, which improves the adhesion between the polymer matrix and the fibre surface thereby increases the mechanical properties of the composite materials[33].

3.4 Dynamic mechanical analysis test result

3.4.1 Effect of clay and fibre content on storage modulus (E')

The stiffness property of the composite material can be measured by using storage modulus value[34]. Figure 6 to 10 shows the effect of temperature and montmorillonite nanoclay loading on the storage modulus of polyester hybrid nanocomposite at various frequencies. The Figure 7 depicts that the storage modulus value at 1Hz frequency for 5wt % clay and 5, 10, 15, 20, 25wt % of jute fibres added hybrid composites are 2.5, 3.3, 4.1, 4.8 and 5.2 GPa respectively. The result shows that the energy accumulation capability of the nanocomposites can be enhanced by the addition of fibre content. The storage modulus drops near the glass transition temperature (Tg), which was due to the softness of the composite. The storage modulus of the composite material can be improved by the incorporation of nanoclay as filler material in the composites. These filler act as a stiffening agent by reducing the movement of the polymeric molecules[35].

From Figure 7 it is found that addition of nanoclay enriched the modulus considerably because of better interaction between polyester, nanoclay and fibre. Elevated storage modulus is observed in both the regions (i.e., glassy and rubbery) and also the jute fibre addition increases the Tg value of composite material from 109 to 115 °C. This is considerable increment of storage modulus of the hybrid

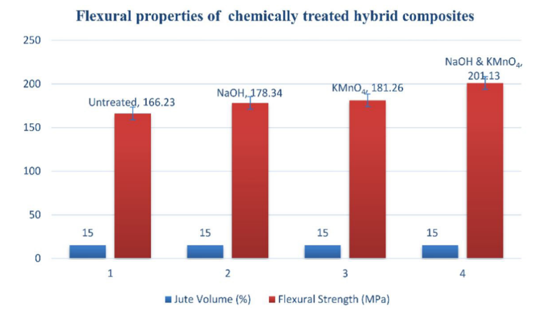

Figure 4. Comparison of the flexural properties of chemically treated jute fibre reinforced polymer composites with 5wt% montmorillonite clay as filler.

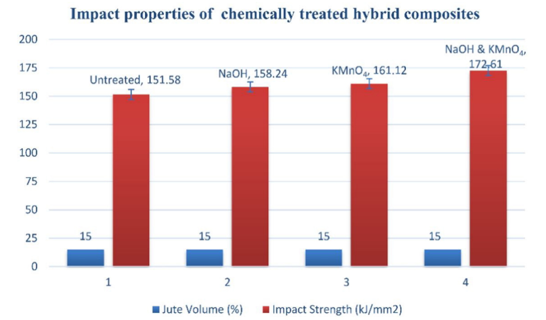

Figure 5. Comparison of the impact properties of chemically treated jute fibre reinforced polymer composites with 5wt% montmorillonite clay as filler.

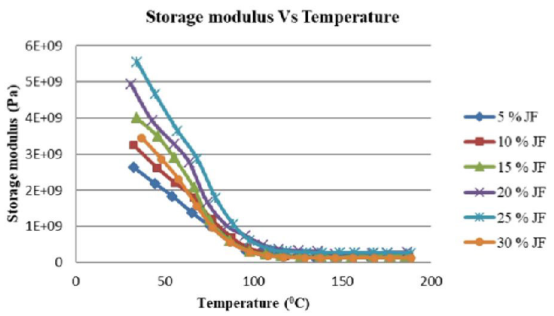

Figure 6. Storage Modulus of Jute fibre reinforced polyester/clay nanocomposites at 0.5Hz frequency.

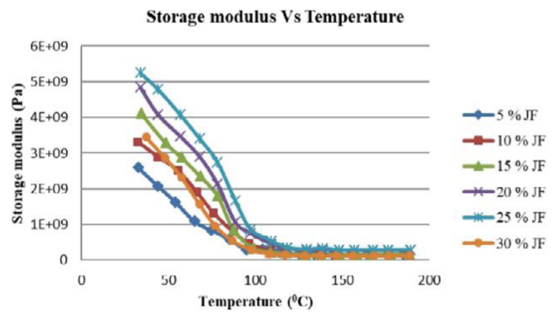

Figure 7. Storage Modulus of Jute fibre reinforced polyester/clay nanocomposites at 1Hz frequency.

Table 1. Experimental mechanical properties of untreated jute reinforced polymer composites with 5wt% montmorillonite clay as filler.

Serial No.	Jute Fiber Content (%)	Tensile Strength (MPa)	Flexural Strength (MPa)	Impact Strength (kJ/mm^2)
1	5	30.30	91.77	41.41
2	10	32.91	121.39	100.00
3	15	40.38	166.23	151.58
4	20	31.15	203.10	130.30
5	25	26.56	234.93	103.03
6	30	20.55	212.46	84.85

Mechanial properties data from reference[27].

composite material due to the incorporation of nanoclay in a polymer which enhances the stress transfer by acting as a secondary reinforcement in the composite[16]. Another observation observed from the figure is, nanoclay up to 5wt% in the polyester along with 25wt% jute fibre content increases the storage modulus extremely than the other weight percentage of montmorillonite nanoclay. This is because of increasing nanoclay content in the composite forms cluster formation. Due to this it forms heterogeneity property and it creates weak bonding between matrix and fibre.

3.4.2 Effect of clay and fibre content on damping factor (tan δ)

The ratio of loss modulus to storage modulus is called as damping factor and it measures energy dissipation through material when it is loading. The variation of Tan δ peak is associated with molecular movement and amount of energy dissipation. In a composite material the energy dissipation is dependent on the molecular movement in the polymer chain, fibre/matrix interaction, strength of the fibre, fibre breakage and crack propagation[35]. From the Figures 11 to 15 it is found that composite with 5wt% nanoclay and 25wt% jute content has lower Tan δ peak, which is due to better fibre/matrix adhesion, decrease in molecular mobility and enhanced load carrying capacity. These are due to the addition of jute fibre as primary reinforcement and nanoclay as secondary produces positive hybrid effect in the composite. This hybrid effect carries more loads and observes large amount of energy and thus will increase the performance of composite material. The inclusion of nanoclay allows a lesser amount of energy at the interface

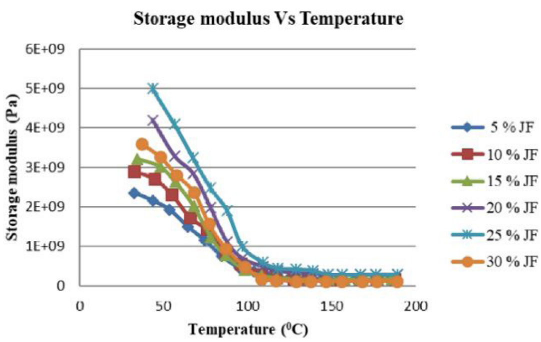

Figure 8. Storage Modulus of Jute fibre reinforced polyester/clay nanocomposites at 2Hz frequency.

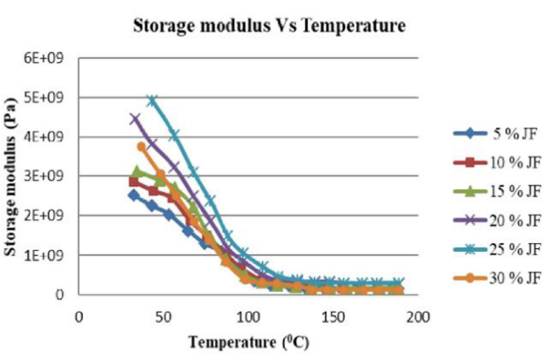

Figure 9. Storage Modulus of Jute fibre reinforced polyester/clay nanocomposites at 5Hz frequency.

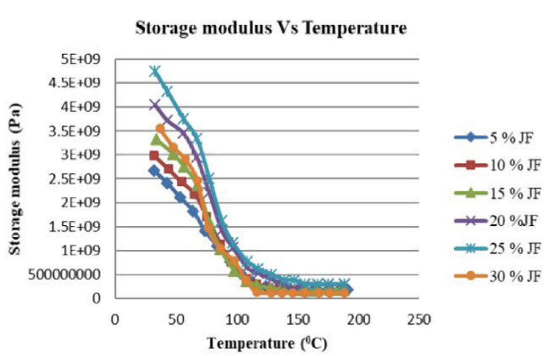

Figure 10. Storage Modulus of Jute fibre reinforced polyester/clay nanocomposites at 10Hz frequency.

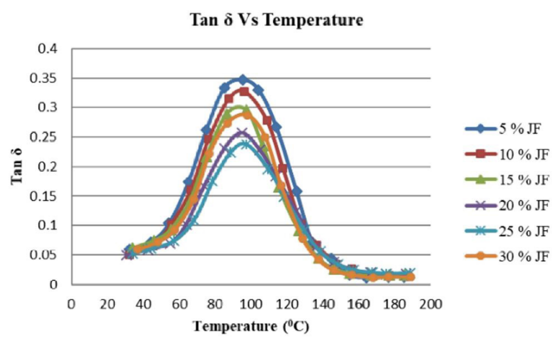

Figure 11. Effect of Tanδ at 0.5Hz frequency on Jute fibre reinforced polyester/clay nanocomposites.

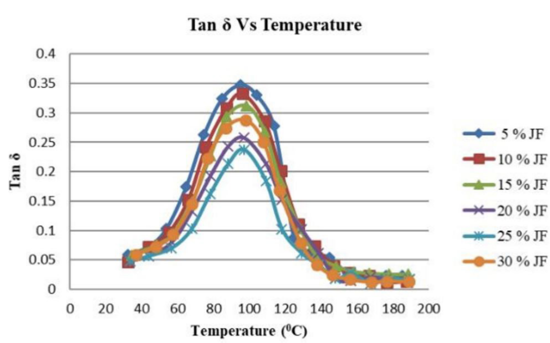

Figure 12. Effect of Tanδ at 1Hz frequency on Jute fibre reinforced polyester/clay nanocomposites.

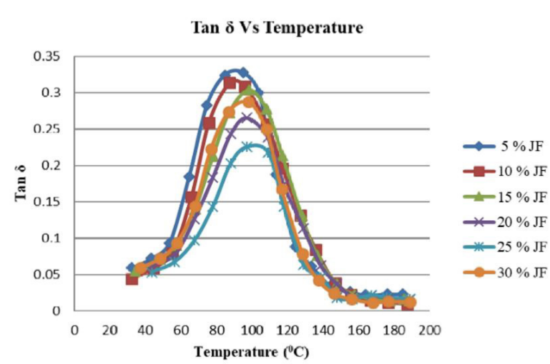

Figure 13. Effect of Tanδ at 2Hz frequency on Jute fibre reinforced polyester/clay nanocomposites.

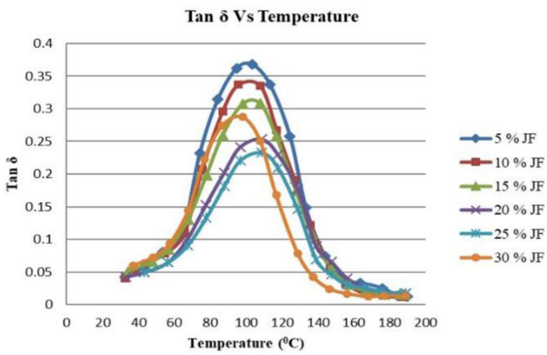

Figure 14. Effect of Tanδ at 5Hz frequency on Jute fibre reinforced polyester/clay nanocomposites.

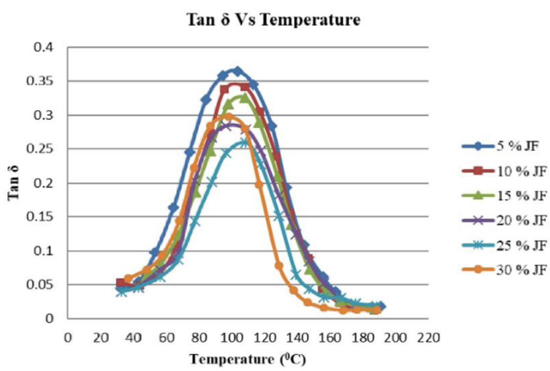

Figure 15. Effect of Tanδ at 10Hz frequency on Jute fibre reinforced polyester/clay nanocomposites.

which means better interface will dissipate lower energy and hence it will enhance the stiffness of the material and also the Tg value of composite material.

Table 2 shows the data's of Tg, peak height and peak width at half height obtained from tan δ curve at 1 Hz frequency. The peak height data is associated with the energy dissipation at the composite interface and peak width at half height relates the relaxation time. The higher value of peak height indicates the higher dissipation energy and from Table 2 it is found it for 5wt % jute fibre reinforced nanoclay filled composite material. The higher value of peak width is obtained for 30wt % jute fibre reinforced nanoclay filled composite material. It indicates that the higher dynamic heterogeneity is associated with that composite as mentioned by Saiter et al.[36]. Table 3 shows the tan delta of chemically treated with jute fibre composite study.

3.4.3 Cole-Cole plot

Figure 16 shows the Cole-Cole plot of 5, 25 and 30 weight percentage of Jute fibre reinforced polyester/montmorillonite nanocomposites. The homogeneity property of the composite material can be indicated by cole-cole plot[20] and it is plotted as loss modulus (E") vs. storage modulus (E') at a particular frequency. From the Figure 16, it can be noted that the addition of 5wt% clay and 25wt% jute fibre in the polymer shows the perfect semi-circle which indicates the uniform distribution of clay and fibre with polyester and thus maintain the homogeneity nature. On further addition of fibre i.e. 30wt%, the smooth and perfect semi circle changes to imperfect shape. This indicates that the material behaviour changes from homogeneous to heterogeneous[37]. From the results, it is concluded that the optimum percentage of clay and fiber enhances the dynamic mechanical properties of the composite material.

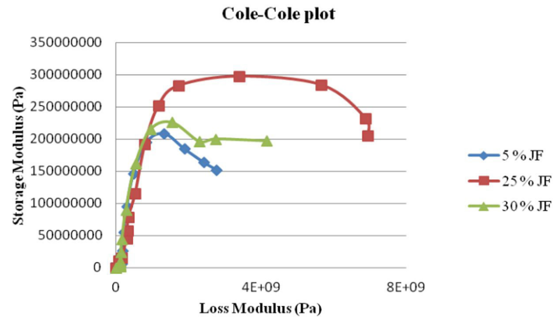

Figure 16. Cole-Cole plot of the Jute fibre reinforced polyester/clay nanocomposites at 10Hz frequency.

Table 2. Values of Tg and Peak height from tan δ at 10Hz frequency.

Sample	Tg (Tan δ) (°C)	Peak height	Peak width at half height
5wt% JF NC	109	0.34	4.86
10wt% JF NC	110	0.33	4.92
15wt% JF NC	112	0.31	5.03
20wt% JF NC	114	0.25	5.07
25wt% JF NC	115	0.23	5.13
30wt% JF NC	113	0.28	6.22

Table 3. Experimental properties of Tan δ value of chemically treated jute reinforced polymer composites with 5wt% montmorillonite clay as filler.

5wt% NC + 15wt% JF	Dynamic Mechanical Analysis Tg (Tan δ) (°C)
Untreated	112.0
NaOH	112.3
$KMnO_4$	112.5
Both NaOH & $KMnO_4$	113.0

4. Conclusions

The following observations were made during this study of montmorillonite nanoclay as a filler agent in unsaturated polyester resin along with chemically treated jute fibre as reinforcement.

- The inclusion of montmorillonite nanoclay with jute fibre reinforced polyester composite enhances the mechanical properties in all the cases of investigation;
- The maximum increase in the inter laminar shear strength is found as 28.03 MPa with the inclusion of 5wt% clay and 25wt % jute fibre which is 54.44% increase when compared to 5wt% clay and 5wt% jute fiber. This shows the existence of enhanced bond characteristics, better interfacial areas and distinctive phase morphology of the fibre reinforced polyester clay nanocomposites;

- The surface treatment of fibre by alkali and $KMnO_4$ shows better mechanical and thermal property which aids in improved interphase adhesion between reinforcements and matrix. Hence, the mechanical properties namely tensile, flexural and impact are improved by 16.70%, 21%, and 13.87% respectively when compared to untreated fibre;
- From the dynamical mechanical analysis, it is found that the addition of nanoclay increases the storage modulus due to the imparting effect of filler reinforcements which are better rigid than the polymer matrix[38]. The increase in the glass transition temperature was found from 109 to 115 °C, however it reduces the damping factor. It may be due to the requirement of high thermal energy to induce uniform motion of molecules.

Based on the experimental analysis, it was found that the jute fibre reinforced clay filled polyester composite can be used as a substitute material for the medium and light weight applications like automotive industries, biomedical equipments, food packaging industries and other consumer applications with enhanced mechanical, thermal and damping properties.

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