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Mechanical Properties of Jute Nanofibres Reinforced Composites

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Abstract- Cellulose nanofibres were extracted from natural fibre Jute by a chemical and mechanical technique to examine their potential for use as reinforcement fibres in biocomposite applications. The Present work was to investigate the possibilities of breaking down the sub-micron fibrillar structure to fabricate submicron and nano particles by high energy ball milling. Nano fibres of jute were characterized by X Ray Diffraction and its structural morphology has studied by Scanning electron microscope. FT-IR spectroscopy analysis showed that the lignin and hemicelluloses was removed for NaoH treated jute fibres. The XRD analysis that the particle size distribution reduced from micro to 20-50 nm. SEM observations revealed that the nano particles of jute fibre were exhibited spherical and elliptical shape. Nanofibre composites were prepared with different weight percentages (1 wt. % to 5 wt. %) via hand lay-up technique. The mechanical properties of nanofibre reinforcement has improved when compared with the virgin composite. The maximum improvements were observed in 3 wt. % Jute nanofibre composites.

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Abstract- Cellulose nanofibres were extracted from natural fibre Jute by a chemical and mechanical technique to examine their potential for use as reinforcement fibres in biocomposite applications. The Present work was to investigate the possibilities of breaking down the sub-micron fibrillar structure to fabricate submicron and nano particles by high energy ball milling. Nano fibres of jute were characterized by X Ray Diffraction and its structural morphology has studied by Scanning electron microscope. FT-IR spectroscopy analysis showed that the lignin and hemicelluloses was removed for NaoH treated jute fibres. The XRD analysis that the particle size distribution reduced from micro to 20-50 nm. SEM observations revealed that the nano particles of jute fibre were exhibited spherical and elliptical shape. Nanofibre composites were prepared with different weight percentages (1 wt. % to 5 wt. %) via hand lay-up technique. The mechanical properties of nanofibre reinforcement has improved when compared with the virgin composite. The maximum improvements were observed in 3 wt. % Jute nanofibre composites.

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I. Introduction

Nature continues to be generous with mankind by providing all kinds of resources in abundance for his living and existence. In this technological era the products produced essentially depend on the new varieties of materials with special characteristics. In this regard the metal composites, plastic and fibre reinforced polymer composites are playing key role in all the fields of engineering and technology. Jute fibres are sustainable and biodegradable with many advantages of low density, low cost and high specific properties. The interest in natural fibre reinforced polymer composite has grown rapidly due to high performance in mechanical properties, significant processing advantages. Jute fibres have been applied to reinforcement to eco-composites and bio- composites. Natural fibres reinforced polymer composites form an interesting class of materials, which seem to have good potential as a substitute for the fibre reinforced materials, i.e. automotive and structural and non structural applications.

Nanotechnology has rapidly become an interdisciplinary field and one exciting research area is the isolation of nano cellulose fibre from bio resources using Top-down technique [2]. Nano fibres take advantage of their dramatically increased surface area to volume ratio [3 and 4]. The polymer composites can be reinforced by nano finres resulting in novel materials which can be used as light weight replacements for metals. Such nano technologically improved materials enable a weight reduction accompanied by an increase in stability and an enhanced functionally [5 and 6]. The direction of using nano fibres from natural fibres as reinforcing materials in plastics may bring changes in manufacturing scenario. Since these jute fibres are biodegradable and eco-friendly.

This research was focused on the development of a new isolation technique to extract cellulose nanofibres from jute by mechanical process using high energy ball milling [10, 11, and 12]. The morphology and crystallinity of the nanofibres of jute fibres were characterized by Scanning electron microscope, (SEM) and X-ray diffraction analysis (XRD), Fourier transformation infrared analysis (FTIR) The nanofibre reinforcement for biocomposite applications which influence of nanofibre morphologies on the mechanical properties.

II. Experimental Work

a) Materials

Jute fibres were collected from Nellimarla jute Mills Limited, Vizianagram, Andhra Pradesh, India. The matrix used in the present study was an Diglycidyl ether of bisphenol-A, a di-functional epoxy resin (LY 556), Tri– ethylene tetra amine (TETA) araldite hardener (HY951) and Woven roving glass fibre mat (WRM) of 410 gsm was taken from M/s ECMAS Pvt. Ltd, Visakhapatnam.

b) Synthesis of Nanofibres

i. Chemical treatment

The weighed jute fibres were cut into 5cm in length and are soaked into 4% NaoH solution at 80°c maintaining a liquor ratio of 15:1. The jute fibres were kept immersed in the alkaline solution for 5 hours by using a Remix Water bath shaker. The fibres were then washed with water to remove any NaoH sticking to the fibre surface and finally washed again with distilled water. The fibres are then dried at room temperature for 24 hours followed by oven drying at 100°c for 5 hours.

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ii. *Mechanical Milling*

The milling process is taken in two steps, the primary process is rotary milling and second process is high energy planetary ball milling by Retsch model PM100. The Jute fibre snippets were passed through a rotary mill fitted with a 0.08mm sieve which operates at a speed of 3000 rpm. The collected jute powder was wet milled in a high energy ball milling to synthesize nanofibres.

c) Characterization of Nano fibres

i. *Fourier –Transform infrared (FT-IR) analysis*

Fourier –transform infrared (FT-IR) spectra of jute fibre samples was obtained from thermo Nicolet Avatar 380 FT-IR, in which samples of untreated jute fibres , 5% Noah treated jute fibres, and nano jute fibre samples were pattelized with KBr powder with 42 scans and the resolution of 8cm^{-1}.

ii. *X-Ray Diffraction*

X-Ray diffractometer (Phillips made X Pert Pro Diffractometer model) analyzed the nano particles of jute at a scanning rate 4^0/min with Cu, K_α radiation at 45 kv and 40 mA. The size of the jute fibres were determined by using Scherrer formulae.

iii. *Scanning electron microscope*

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. The nano jute fibre particles exhibited spherically as well as elliptical shape.

d) Preparation of Jute nanofibre reinforced 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. %) were 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 testing the mechanical properties as per ASTM standards.

i. *Studies on Mechanical Properties*

Sufficient tensile strength, impact strength, hardness and damping are required for engineering materials. Especially this is an important mechanical test for polymer based nanocomposites. The mechanical properties of plastic as well as composite materials, tensile properties are probably the most frequently considered. These properties are an important indicator of the material's behavior under loading in tension. Tensile strength were carried out using a Universal testing machine (Instron corp.,) to evaluate the tensile strength of nanocomposites as per ASTM D-638 Type-I.

The impact properties of the polymeric materials depend mainly on the toughness of the material. Toughness can be described as the ability of the polymer to absorb applied energy. The molecular flexibility has a great significance in determining the relative brittleness of the material. Impact energy is a measure of toughness and the impact resistance is the ability of a material to resist breaking under a shock–loading In the present investigation notched izod impact strength of the specimens of dimensions 63.5x12.7x3mm was evaluated using an Impactometer (Ceast, Italy) as per ASTM-D-256 with a notch depth of 2.54 mm and notch angle of 45°.

The resistance of a material to an indentation deformation is hardness. The higher the hardness, the better the material resistance to the indenting deformation will be. For thermosetting materials, the hardness with the shore A reference is used for low modulus materials. The test enables the determination of the penetration depth in the sample under a given load and using a fixed time lap the indentation device is not supposed to be deformable. In the present work the Jute nanofibre reinforced polymer composite specimens are tested as per ASTM D 2240 by Barcoll hardness tester.

Damping is also a significant factor for the fatigue life and impact resistance of structures. Damping varies with different environmental effects such as frequency, amplitude of stress, temperature and static load.

III. Results and Discussion

a) Surface Modification

The most important factor in obtaining good fibre reinforcement in the composite is the strength of adhesion between the matrix polymer and the fibre. The extent of adhesion depends upon the structure and polarity of the materials. Owing to the presence of hydroxyl and other polar groups in various constituents of jute, the moisture regain is high which leads to poor wettability with the matrix and weak interfacial bonding between the fibres and the more hydrophobic matrices. Therefore in order to develop composites with improved mechanical properties, It is necessary to impart hydrophobocity to the fibres by suitable chemical treatment.

i. *FT-IR Analysis*

FT-IR spectra is used to measure the change of surface composition of the fibre chemical of untreated, treated and Jute nanofibres were shown in Table No.1. The absorbance peaks of interest in the study have been identified in the fig 1, 2 and 3 of raw jute, 4% Noah

treated at 80°c and nanojute particles. Alkaline treatment reduced hydrogen bonding due to removal of the hydroxyl groups by reacting with sodium hydroxide. The result in the increase of the– OH concentration evident from the increased intensity of the peak between1000 and 1500 cm^{-1} compared to the untreated fibre.

Table 1 : Chemical Composition of Jute Fibers

Bond-Type	Raw jute (untreated)	4%NAOH treated at 80°c	Nano jute fibre particles
-OH stretching	3383.14	3448.72	3556.14
C-H Vibration	2912.51	2900.94	2900.94
C=O stretching	1735.93	Nil	Nil
C=C stretching	1647.21	1635.	1635.64
C-H bending	1373.32	1319	1319
C-H bending	1249.87	1234.44	1234.44
-OH	601.79	516.92	524.64

The absorbance between this ranges are indicative of the hemicelluloses. The hydroxyl groups are also involved in hydrogen bonding with the carboxyl groups, perhaps of the fatty acids, available on the fibre surface of jute fibre. This is indicated by the reduction of the peaks between (3383-3556) cm^{-1}. The peaks 1735 cm^{-1}seen in untreated fibres disappears upon alkali treatment. This is due to removal of the carboxylic group by alkali treatment. The reduction in the peak intensity found in alkali treated jute fibres indicates the particular reaction of the C=O bonds of hemicelluloses, which shows that hemicelluloses of jute is removed by alkalization. The intensity of peak1647 cm^{-1} (C=C stretching) is reduced to (1635-1647) cm^{-1} in alkali treated jute fibres. This may be due to the removal of un saturation present in the traces of jutefibres. The absorbed peak at 1373cm^{-1} shows diminishing intensity as the subjected higher concentration of caustic soda. The disappearance of the peak at 1249cm^{-1} after alkalization indicates the complete removal of hemicelluloses which indicates that hemicelluloses are easily removed by alkalization. The C-OH bending peak is observed at 516-601 cm^{-1}. From this analysis it is observed that several reactions take place during alkalization.

b) X-Ray Diffraction

X-Ray diffractometer (Phillips made X Pert Pro Diffractometer model) analysed the Nano particles of jute at a scanning rate 4°/min with Cu, K_α radiation at 45 kv and 40mA.

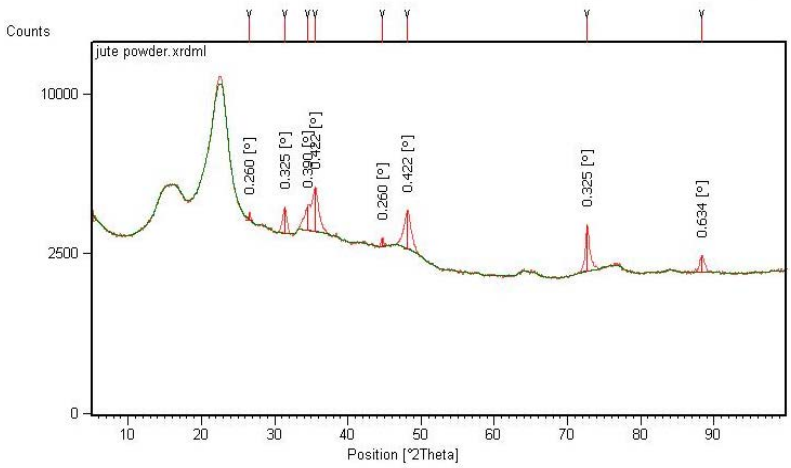

Figure 1 : XRD analysis of Nano jute particles

The sizes of the jute fibres were obtained 22nm to 52 nm with an average size of 33nm after 80 hours of milling. The crystalline size was calculated by using Scherrer formulae.

Scherrer formulae: Crystalline size (d) $= \frac{k\lambda}{\beta cos\theta}$

Where k = Shape factor (0.9 spherical particles)

λ = Wave length of copper K_α =1.540598

θ = Centre of the peak in degrees.

The crystallinity values obtained from the X-ray diffraction shows that the crystallinity values may be observed to decrease with increased milling time. The peak intensities of jute powder samples were observed to be reduced. It is concluded that the average crystalline size of the jute powder finer particles were at 20 nm to 50 nm. The sample Nano jute powder particles are scanned from 20 to 90 degrees of 2 Theta.

c) Scanning Electronic Microscope

The Scanning Electronic microscope (SEM) images of jute fibres and micro fibrils were taken with

JEOL model Scanning Electronic microscope. Before placing the samples into machines, the sample are coated with gold palladium using a sputtering machine to prevent charging. All images are taken at 1000 magnification. SEM analysis observed the effect of chemical treatment of jute fibres in the following figures at different milling hours of 20 hrs, 40 hrs, 60 hrs and 80 hrs of Ball milling.

Figure 2 : SEM images of Jute fibres after 20 hrs milling

Figure 3 : SEM images of Jute fibres after 40 hrs milling

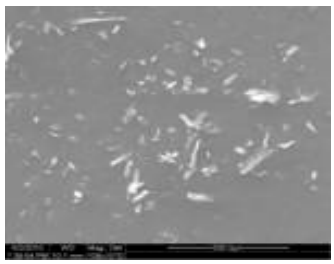

Figure 4 : SEM images of Jute fibres after 60 hrs milling

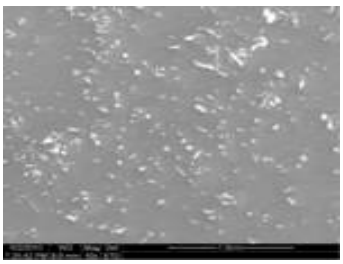

Figure 5 : SEM images of Jute nanofibres after 80hrs milling

d) Mechanical Properties of nanofibre composites

In order to study the reinforcement effects of different surface modified nanofibre with polymer resin matrix. The tensile strength, impact strength, hardeness and damping were determined.

i. *Tensile test*

Tensile test specimens were prepared as per ASTM D-638 and results are taken an average of the five tested samples. The mechanical properties are found to be increased with the reinforcement of jute nanofibres of (1wt. % to 5wt. %) composites and reached maximum of 96% improvement for 3wt. % nanocomposites. The nanoscale fibre reinforcement most strongly influences the matrix-dominated mechanical properties, while the in –plane ones are typically dominated by the fibre reinforcement.

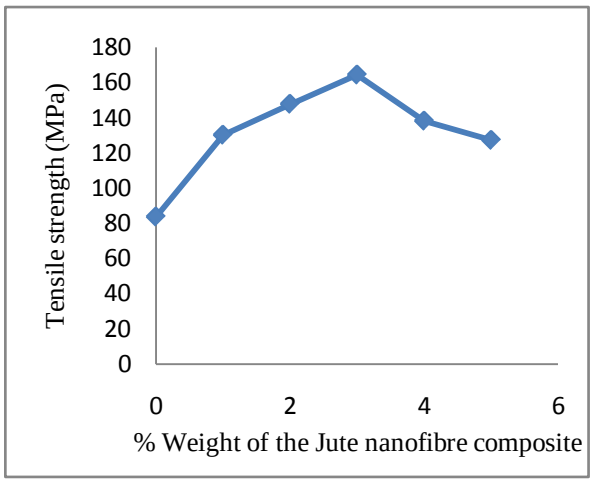

Figure 6 : Tensile properties of base composite and JNFC

ii. *Impact Test*

The impact test specimens are prepared as per ASTM D 256 and results are found to be increased for the reinforcement of nano jute particles. The maximum improvement is 38.5% for 3wt% nanojute composite. The result of the Izod test is reported as energy lost per unit cross-sectional area at the notch in J/mm^2. The variation of impact strength with (1wt% to 5wt %) JNF composites had been graphically represented in the results.

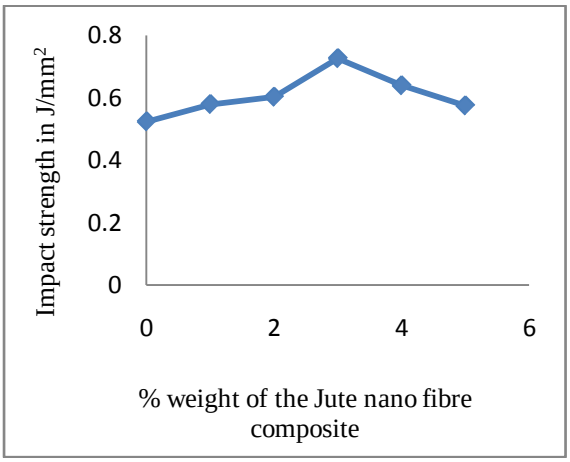

Figure 7 : Impact properties of base composite and JNFC

iii. *Hardness*

Hardness tests were prepared using Barcoll Hardness tested machine and testing specimens are prepared as per ASTM D-2583. The specimen was placed under the indenter of the Barcoll hardness tester and a uniform pressure was applied to the specimen until the dial indication reaches maximum. The depth of penetration was converted into absolute Barcoll numbers. Average of 5 specimens were reported for each nano composite. These tests were carried out at for different (1wt% to 5wt. %) JNF composites.

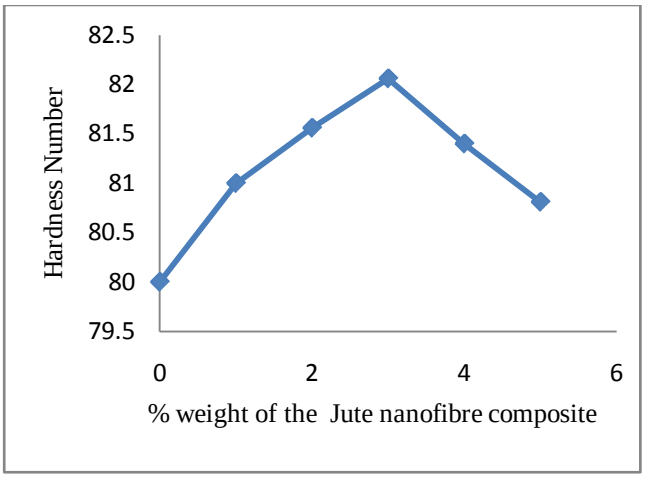

Figure 8 : Hardness properties of base composite and JNFC

The Barcoll hardness of the nano composites increases from to 80 to 82.5 when the nanofibre is reinforced in epoxy composites. The maximum improvement in hardness is obtained by 2.57% in 3 wt.% in Jute nanofibre composites. This is because of the strong interfacial bonding strength between the fibre and matrix which greatly increases the hardness of nanofibre reinforced composites. Due to an increase in the Jute nanofibres content the composite becomes stiffer and harder, and thus there is an increase in hardness with increasing fibre content.

iv. *Damping Ratio*

The damping behavior of nano jute fibre reinforced polymer composite is investigated experimentally with different weight percentage of composites at different modes. The effect of the natural frequency and the damping ratio are analyzed because polymer matrix composites have temperature dependent mechanical properties. It is observed that

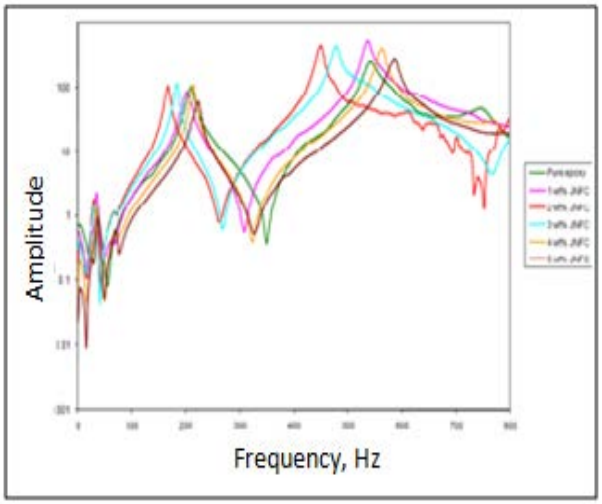

Figure 9 : Damping property of Base Composite and JNFC

Figure 7 shows the improvement in the natural frequency as noticed for both modes of nanofibre composites (1 wt.% to 5 wt.% JNF) when compared with basecomposite. The increased natural frequency is due to the good dispersion of Jute nanofibre reinforcement in the matrix which results in improved stiffness. It may be noted that the addition of Jute nanofibre has a considerable effect on the damping behaviour of nanocomposites. This may be due to the composites possessing high stiffness on account of the high modulus of nanofibres and its uniform distribution. The nanofibres are uniformly distributed, which results in a good bonding between the reinforcement and the matrix. This provides a large interfacial area between the matrix and nanofibres. This increases the modulus value as well as the energy dissipating interface.

IV. Conclusions

The development of nanocomposites based from nanofibre material is a rather new but rapidly evolving research area. Natural fibres are abundant in nature, biodegradable and relatively cheap, besides being promishing nano-scale rinforcement materials for Polymers. In this work the nanocomposites were fabricated using jute nano fibre reinforcement in epoxy matrix. The mechanical properties of the jute nanofibre composites were studied through different experimental approaches.

The chemical structure of jute fibre after treatments showed an increase in cellulose content and a decrease in lignin and hemicellulose as compared with the original jute fibres.Jute fibres have been synthesized to produce nanofibres and used as reinforcement in the epoxy resin system to fabricate nanocomposites, which have imparted improved mechanical tensile, impact, hardness, and damping properties compared to the base composite.

The nano fibre composite with 1 wt.%, to 5 wt.% JNF were reinforced in epoxy resin to prepare nanocomposites. The tensile strength of nanocomposites were increased from 50% to 96% with an increasing jute nanofibre 1 wt.% to 5 wt.% content. The tensile strength,impact and hardness improvement was noticed for the 3 wt% JNF composites. The increase in strength can be attributed to strong interaction between the polymer and Jute nanofibres. The damping properties are improved by improving damping ratio for fibre reinforced composites by reinforcing nanofibres of jute upto 3 wt.%, thus leading to a higher performance in damping properties than those with jute nanofibre loading levels. The reinforment of jute nanofibre results in good damping.

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