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Research Paper

ISOLATION AND CHARACTERIZATION OF JUTE NANO FIBRES

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The Present work was to investigate the possibilities of breaking down the sub-micron fibrillar structure to fabricate submicron of Jute nanofibres by high energy ball milling. The particle size distributions of the jute fibres were obtained to determine the variations occurring in the particle size and size span when ball milling was applied. Nano fibres of jute fibre were characterized by using XRD analysis at different time periods at 10hrs, 20hrs, 40hrs, 60hrs and 80hrs of milling by varying ball diameters in steps and speeds of the range from 100 to 200 rpm. The fibre size in diameter is decreased by the longest milling time investigated. The faster milling speed led to smaller particles, with wider size distribution while the size was hardly effected. The utilization of smaller ball diameter resulted in slightly smaller particles only after longer milling time, with narrow size distributions. Fourier Transformation Infra red spectroscopy (FTIR) analysis showed that the lignin and hemicelluloses was removed for NoaH 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.

Keywords: Jute fibre, Cellulose, Chemical treatment, Ball milling, Characterization, Micro and nanofibres

INTRODUCTION

Natural fibres have been subject of Investigation for their use as reinforcement in Polymer matrices. Lignocelluloses fibres like Jute, Sisal, Flax, Hemp and Wood have been used as reinforcements in polymer matrices. Jute fibres are sustainable and biodegradable with many advantages of low density, low cost and high specific properties.

Nanotechnology has rapidly become an interdisciplinary field and one exciting research area is the isolation of nano cellulose from bio resources using top-down technique (Bhatnagar, A Sain M, 2005; and Mehdi Jonoobi *et al*., 2009). Nano particles take advantage of their dramatically increased surface area to volume ratio (Bei wang *et al*., 2007; and Kani Akkay *et al*., 2004). The polymer composites can be reinforced by nano particles resulting in novel materials which can be used as light weight replacements for metals. Such nano technologically improved materials enable a weight

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reduction accompanied by an increase in stability and an enhanced functionally (M N Belgacem and A Gandini, 2008; and E Sinha and S K Rout, 2009). 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.

So far the research has been done to obtain nano cellulose by acid hydrolysis technique. This study was focused on the development of a new isolation technique to extract cellulose nano fibres from jute by mechanical process using high energy ball milling (Ayse Alemdar and Mohini Sain, 1999; P A Zielinski *et al*., 1995; and Eichhorn S J, *et al*., 2010). The morphology and crystallinity of the nano particles of jute fibres were characterized by Scanning electron microscope, (SEM) and X-ray diffraction analysis (XRD), Fourier transformation infrared analysis (FTIR). This research aims to prepare the nanofibres of jute and characterize them in order to evaluate their suitability as reinforcement for biocomposite applications which influence of nanofibre morphologies on the mechanical properties.

EXPERIMENTAL WORK

Jute fibres were collected from Nellimarla jute Mills Limited, Vizianagram, Andhra Pradesh, India. The fibres are washed with distilled water and dried in an oven at 80°c under vacuum to obtain constant weight. The jute fibers were cut into 2 cm length and weighed before a Chemical treatment was applied. The weighed jute fibres were soaked into 4% of sodium hydroxide solution at 80 °c using a Remi Water bath shaker for 5 hours.

The jute fibres were Chopped to become fine enough 10 mm snippets by using Pulveriser, 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 milled jute powder was driven out by rotary generated air flow and subsequently collected using a cyclone. A Planetary ball milling with a 100ml Tungsten carbide vessel container was charged with Tungsten carbide balls of 20 mm diameter as grinding media. The collected jute powder (g) to ball weight (g) ratio was maintained at 1: 40 for 20 hours at a speed of 100 rpm. To avoid temperature increase during milling, the milling period of 60 minutes was alternated with a 2 minutes rest time. In the present study wet ball milling as a potential means to decrease particle size of jute fibre with minimum loss of jute fibres was investigated. The diameter of the ball and milling speed were varied in the investigation. As a result of high energy ball milling the size of the jute particles were observed to decrease. Further particle size reduction with 10 mm diameter Tungsten carbide balls were used at a speed of 120 rpm for 20 hours. Final particle size reduction with finer media 5mm and 3mm diameter of Zirconium balls were used at a speed of 150 and 200 rpm respectively for 20 hours each. The particle size distribution of the original jute and the ground samples obtained to monitor the variations occurring in the size of the particles.

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, 4% Naoh treated jute fibres, and jutenano fibre samples were pattelized with KBr powder with 42 scans and the resolution of $8cm^{-1}$.

X-Ray diffractometer (Phillips made X Pert Pro Diffractometer model) analyzed the nano fibres of jute at a scanning rate 4°/min with Cu, K?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. The nano jute fibre particles exhibited spherically as well as elliptical shape.

RESULTS AND DISCUSSION

The jute fibres morphology was changed from the micro to the nano scale during the process. It is clearly observed that the chemical treatments are reducing the fibre size and the surface roughness compared to the untreated fibre. The fibre morphology is affected by cryo crushing. The milling process imparted sufficient energy to break the bundles into the single fibre. The finer particles of fibres are defibrillated and the size is reduced from micro to nanometer level. The milling speed 100 rpm to 200 rpm and the ball diameter 20mm to 3mm were varied in the experiments carried out for different period of time as shown in Table No:1

Table 1: Shows the Different Milling Speeds with Different Ball Diameters

Ball diameter mm	Milling speed Rpm	Duration period in Hrs.	Size reduction
20	100	20	820 μn
10	120	20	500 μn
5	150	20	100 μn
3	200	20	33 nm

The reduction in the particle size was generally higher for the faster milling speed with the smaller diameter balls after 20 hours of milling. As the ball diameter is decreased and speed of the ball milling increased, so the finer particles are expected to be obtained. Dispersions and aggregation are regarded to be the two main process taking place during the milling of the fibres. At the first stage of milling, dispersion may be expected to be dominant; indicating a decrease in particle size, while at the later periods of milling the particle size is further reduced by increasing the speed and reducing the ball diameter.

FT-IR Analysis

FT-IR spectrascopy is used to measure the change of surface composition of the fibre chemical of untreated, treated and nano jute particles were shown in Table No. 2.

Table 2: Chemical Structure of Cellulose Jute Fibre

Bond-Type	Raw jute (untreated)	4% Naoh treated at 80°c	Jute nanofibres
-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 peaks of interest in the study have been identified in the FT-IR analysis of raw jute, 4wy.% NaoH 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. 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 .The peaks 1735 cm seen in untreated fibres disappears upon alkali treatment. This is due to removal of the

carboxylic group by alkali treatment. The Transmittance % 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 peak 1647 cm^{-1} (C=C stretching) is reduced to (1635-1647) cm in alkali treated jute fibres. This may be due to the removal of un sa- turation present in the traces of jutefibres.. The absorbed peak at 1373cm^{-1} shows diminishing intensity as the subjected higher concentration of caustic soda. The dis- appearance of the peak at 1249cm^{-1} after alkalization indicates the complete removal of hemicelluloses which indicates that hemi- celluloses are easily removed by alkalization. The C-OH bending peak is observed at 516-601 cm^{-1} .

Figure 1: FT-IR Analysis of Raw Jute

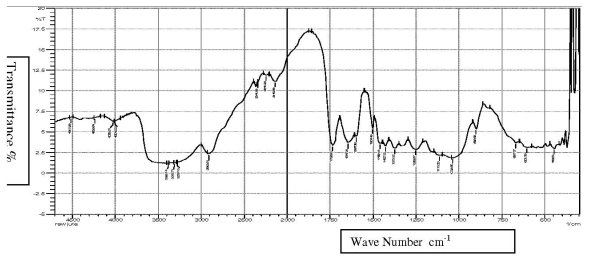
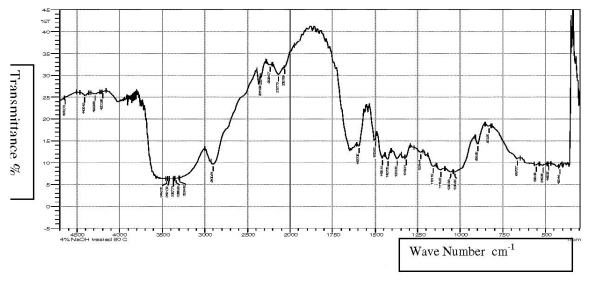
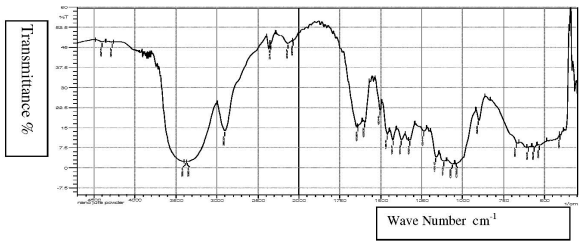


Figure 2: FT-IR Analysis of 4% Noah treated at 80°c of Jute



The FTIR analysis of i) raw jute, ii) 4% NaoH treated jute at 80°c and iii) Nanojute particles were shown in the Figures 1, 2 and 3 respectively as below.

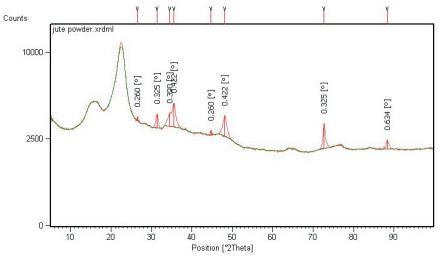
Figure 3: FT-IR Analysis of Jute Nanofibres



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, Kα radiation at 45 kv and 40mA.

Figure 4: XRD



The size 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

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

Where

k = Shape factor (0.9 spherical particles)

β= 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.

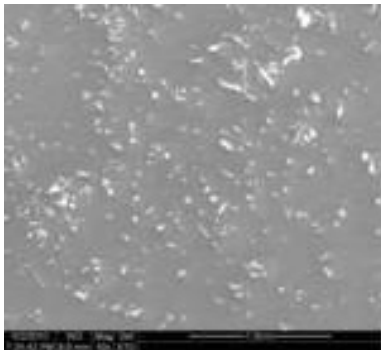
Scanning Electronic Microscope

The Scanning Electronic microscope (SEM) images of jute fibres and micro fibrils were taken with JEOL model Scanning Electronic microscope. SEM analysis observed the effect of chemical treatment of jute fibres in the following figures at different milling hours of 20 hrs, 40 hrs and 80 hrs of Ball milling.

Figure 5: SEM Images of Jute Fibres After 20 hrs Milling

Figure 6: SEM Images of Jute Fibres After 40 hrs Milling

Figure 7: SEM Images of Jute Nanofibres After 80 hrs Milling



SEM observation was carried out to verify the microstructures characteristics. The SEM images at different milling hours demonstrates that the obtained cellulose jute fibrils are micro scale to Nano scale with diameter range from 30 nm to 100 nm and a length of several microns. The crystalline structure of the jute fibres did not collapse completely even when long milling times were employed. The Nano jute finer particles exhibited spherically as well as elliptical shape.

CONCLUSION

In this work the nanofibres of jute were prepared by using High energy planetary ball milling. There are some inherent advantages in processing. Nanofibres by high energy ball milling techniques such as excellent versatility, scalability and cost effectiveness. Chemical treatment of the jute fibres shows that the increase in cellulose content and a decrease in lignin and hemicelluloses contents as compared with raw jute fibres. Rotary milling was not very effective in producing fine particles, the particles obtained in rotary milling was remained in fibre segiment after many passes. Ball milling could produce fine particles when milling parameter such as powder to media ratio, media size. The faster milling speed led to

smaller particle size while the crystalline was hardly effected, when smaller ball diameter was utilized. The experimental results shows that the size of produced jute nanofibre were within the range of 20nm to 52 nm at an average of 33 nm from the XRD analysis. FTIR is used to detect functional groups and understand the structure of the nanofibres. The morphologies of jute fibres were analyzed by Scanning Electronic microscope. The spherical and elliptical shape of the Nano jute fibres were in corporate as reinforcing filler in a biocomposite matrix.

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