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Synthesis and Characterization of Sisal Fibers

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***Abstract*— Natural fibers are very widely being used in various applications like construction, materials, automotive etc. Out of the available natural fibres sisal is one such fibre which is presently being worked on. All the research presently going on with natural fibres deals with nano scale study. This requires a lot of chemical treatment involved and long hours of ball milling for converting the material to a nano scale. Though the advantages with nano are high the process of converting the material to nano scale is a bit time taking and cumbersome. The present work highlights a method adopted for chemical treatment of raw sisal fibers which minimizes the ball milling time required.**

***Index Terms*—Natural fibers, nano scale, ball milligng, harvest.**

I. INTRODUCTION

Natural fibres play a key role in the emerging green economy based on energy efficiency, the use of renewable feed stocks in polymer products, industrial processes reduce carbon emissions and recyclable materials that minimize waste.

Natural fibres are a renewable resource, *par excellence* – they have been renewed by nature and human ingenuity for millennia. They are also carbon neutral: they absorb the same amount of carbon dioxide they produce. During processing, they generate mainly organic wastes and leave residues that can be used to generate electricity or make ecological housing material. And, at the end of their life cycle, they are 100% biodegradable. FAO et.al,(2009)

The environmental benefits of natural fibre products accrue well beyond the production phase. For example, fibres such as hemp, flax and sisal are being used increasingly as reinforcing in place of glass fibres in thermoplastic panels in automobiles. Since the fibres are lighter in weight, they reduce fuel consumption and with it carbon dioxide emissions and air pollution.

But where natural fibres really excel is in the disposal stage of their life cycle. Since they absorb water, natural fibres decay through the action of fungi and bacteria. Natural fibre products can be composted to improve soil structure, or incinerated with no emission of pollutants and release of no more carbon than the fibres absorbed during their lifetimes.

Sisal fibre is obtained from *Agave sisalana*, a native of Mexico. The hardy plant grows well in a variety of hot climates, including dry areas unsuitable for other crops. After harvest, its leaves are cut and crushed in order to separate the pulp from the fibres. Lustrous and creamy white, **sisal** fibre measures up to 1 m in length, with a diameter of 200 to 400 microns. It is a coarse, hard fibre unsuitable for textiles or fabrics. But it is strong, durable and stretchable, does not absorb moisture easily, resists saltwater deterioration, and has a fine surface texture that accepts a wide range of dyes Today, sisal can be found in speciality paper, filters, geotextiles, mattresses, carpets and wall coverings.It is used as reinforcement in plastic composite materials, particularly in automotive components, but also in furniture. Another promising use is as a substitute for asbestos in brake pads. By-products from sisal extraction can be used for making bio-gas, pharmaceutical ingredients and building material.

In the automotive industry the use of cellulosic fibres as renewable raw material in fibre reinforced composite materials has received much attention as "green" development and is showing much promise Jan E.G et al (2011). Since its introduction a decade ago the use of natural fibres in automotives has shown increasing trends. Among those, flax and hemp non-woven find a growing outlet in compression moulded trim panels and dashboards. The natural fibre reinforced injection moulded composite parts show less demands on the fibre processing and handling (as compared to textile fibre production). The biggest advantage over glass fibre reinforced composites is the weight reduction resulting in lower fuel consumption in the use phase. Other advantages are the lower wear of equipment in the production phase and easier end-of-life recycling by incineration

II. LITERATURE SURVEY

K.Joseph et.*al*.,(1999) Natural fibres are prospective reinforcing materials and their use until now has been more traditional than technical. They have long served many useful purposes but the application of the material technology for the utilization of natural fibres as reinforcement in polymer matrix took place in comparatively recent years. Economic and other related factors in many developing countries where natural fibres are abundant, demand that scientists and engineers apply appropriate technology to utilize these natural fibres as effectively and economically as possible to produce good quality fibre reinforced polymer composites for housing and other needs. Among the various natural fibres, sisal is of particular interest in that its composites have high impact strength besides having moderate tensile and flexural properties compared to other lignocellulosic fibres**.** Sisal fibres have good potential as reinforcements in polymer (thermoplastics, thermosets and rubbers) composites. Due to the low density and high specific properties of sisal fibres, composites based on these fibres may have very good implications in the automotive and transportation industry.Tara Sen et.*al*.,(2011) Natural fibres such as sisal, bamboo, coir and jute are renewable, non-abrasive to Process equipment, and can be incinerated at the end of their life cycle for energy recovery as they possess a good deal of calorific

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value. V. K. Baheti et.*al*.,(2012) has prepared nano cellulose from jute fibres by mercerisation with 18% NaOH at room temperature for two hours, 1M H2SO4 acid treatment at 80°c for 1 hour and 4% NaOH treatment at 80°c for one hour. After chemical treatment, pulverization of chemically treated jute fibres was carried out using a high-energy planetary ball mill in a sintered corundum container with zirconia balls of 10 mm diameter for initial 10 min of dry milling and 3 mm diameter for further 3 hours of wet milling in deionised water. The ball mill was loaded with ball to material ratio (BMR) of 10:1. The rotation speed of the planet carrier was 850 rpm. It was observed that as wet milling time increased, the bimodality in distribution reduced giving narrower size distribution results. The results show that particle size is around 500 nm. Juan I. Moran et.*al*.,(2007) The fibers were washed with distilled water several times and dried in an oven at 80 C for 24 h. Then they were chopped to an approximate length of 5–10 mm. Finally a de-waxing step was carried out: boiling in a mixture toluene/ethanol (2:1 volume/volume) in a soxhlet for 6 h. The de-waxed fibers were then filtered, washed with ethanol for 30 min and dried. Subsequently, two different procedures were used for cellulose extraction. The first is (I) Pre-treatment with 0.1 M NaOH in 50% volume of ethanol at 45 C for 3 h under continuous agitation; (II) Treatment with hydrogen peroxide at pH = 11.5 (buffer solution) and 45 C: (a) 0.5% H2O2, (b) 1.0% H2O2, (c) 2.0% H2O2 and (d) 3.0% H2O2 for 3 h each one under continuous agitation ; (III) Treatment with 10% w/v NaOH—1% w/v Na2B4O710 H2O at 28C for 15 h, under continuous agitation; (IV) Treatment with HNO3, 70% + HAc, 80% (1/10 v/v) at 120 C for 15 min (V) Washing with 95% ethanol; washing with water and washing again with 95% ethanol; (VI) drying at 60 C in oven until constant weight. Second procedure is (I) Treatment with 0.7 w/v.% sodium chlorite NaClO2: holocellulose (a-cellulose + hemicellulose) production by the gradual removal of lignin; at pH 4 (buffer solution) boiling for 2 h using a fiber to liquor ratio of 1:50 and treatment with sodium bisulphate solution 5% w/v.; (II) treatment of holocellulose with 17.5 w/v.% NaOH solution; (III) filtering, washing with distilled water and drying at 60 C in a vacuum oven until constant weight. After cellulose was prepared, acid hydrolysis was carried out in order to produce cellulose nanofibers the diameter of the cellulose fibers was in the range of the nanometer with an average size of 30.9 ± 12.5 nm K.P. Kumar et.*al*.,(2008) The sisal nanofibres are prepared in two steps. In the first step the alkali treated sisal fibres are chopped into small lengths of about 10 mm and fed to D.P.Pulveriser, which runs at a speed of 6000 rpm. The fibres are collected in the form of whiskers from the D.P.Pulveriser. Pulverizing is followed by ball milling for about 140 hours at 60 r.p.m with heavy weight aluminum balls in M.S.vessel to realize them in the form of nanofibres. The obtained nanofibres are characterized by Transmission electron microscope (TEM). and are observed that the particles are having spherical shape and they are approximately 12 nm in diameter. From the literature presented it is obvious that natural fibres are being effectively used in various areas like automotive, construction, composites, green technology etc. The researchers have used various methods for synthesizing and characterizing the fibres to a nanoscale. Observing all the methods presented, an attempt has been made to optimize the effort and time required to synthesize the sisal fibre to a nanoscale.

III. EXPERIMENTAL PROCEDURE

A. Chemical Treatment

The obtained Sisal fibers are dried and chopped into a size of 10 to 20 mm by a mechanical chopper. The fibers are then chemically treated with 8% NaOH Solution for about 72 Hours and then washed with distilled water. Then the fibres are air dried for 24 to 48 hours to ensure that surface water is completely dried and afterwards for 10 hours at 80^{o} C in an Hot Air oven. After the fibers are dried, they are treated with 1M H_2SO_4 solution for 30 minutes under continuous strirring at 80^{o}C in a hot water bath. After the treatment the fibers are washed thoroughly with 50% Distilled water and then completely dried in a Hot air oven at 70^{o}C for 20 to 30 minutes. After the fibers are dried they are subjected to Ball Milling process.


Raw Sisal Fiber


Air Drying


Drying in Oven


Treatment with H_2SO_4


Hot water Bath


Treated Sisal Fiber

B. Ball Milling

Ball Milling is a process of converting the size of a material into a nanoscale by mechanically bombarding the material by balls in a mill chamber rotating at high speed about its axis. In this particular work the the Ball mill used is from "Insmart Systems". The Ball mill has 4 chambers two of Stainless Steel and two of Tungsten carbide material. In this work the Steel chambers are used and a ball to weight ratio of 8:1 is maintained and milling is done at a speed of 300rpm.The sample is milled for 36 Hours and the sample is sent for XRD Analysis .


Sample in Ball Mill


After 36 Hours


Sample for XRD


XRD Analysis

C. X-Ray Crystallography

X-ray crystallography is a tool used for identifying the atomic and molecular structure of a crystal, in which the crystalline atoms cause a beam of incident X-rays to diffract into many specific directions. By measuring the angles and intensities of these diffracted beams, a crystallographer can produce a three-dimensional picture of the density of electrons within the crystal. From this

electron density, the mean positions of the atoms in the crystal can be determined, as well as their chemical bonds, their disorder and various other information.

D. Scherrer Equation

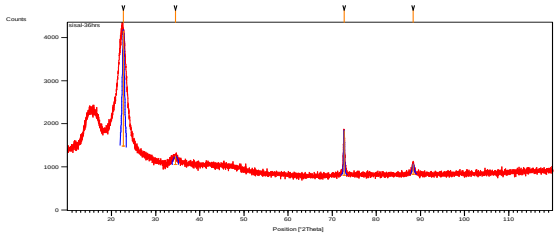
The Scherrer equation ,in X-ray diffraction and crystallography, is a formula that relates the size of sub-micrometre particles, or crystallites, in a solid to the broadening of a peak in a diffraction pattern. Paul Scherrer.et al,(1918) Patterson et.al,(1939). It is used in the determination of size of particles of crystals in the form of powder.The Scherrer equation can be written as:

$$\tau = \frac{K\lambda}{\beta Cos\theta}$$

where:

- τ is the mean size of the ordered (crystalline) domains, which may be smaller or equal to the grain size;
- K is a dimensionless shape factor, with a value close to unity. The shape factor has a typical value of about 0.9, but varies with the actual shape of the crystallite;
- λ is the X-ray wavelength;
- β is the line broadening at half the maximum intensity (FWHM), after subtracting the instrumental line broadening, in radians. This quantity is also sometimes denoted as $\Delta(2\theta)$;
- θ is the Bragg angle.

The results of the XRD analysis done and the values obtained are shown in the figure and the table below.



Pos. [°2Th.]	Height [cts]	FWHM [°2Th.]	d-spacing [Å]	Rel. Int. [%]
22.6700	2670.34	0.5712	3.91920	100.00
34.4673	199.77	1.1424	2.60000	7.48
72.6816	1034.59	0.2448	1.29989	38.74
88.3548	237.51	0.4896	1.10535	8.89

From the above table the relevant values are obtained and after the calculation it is found that the average size of the particle is 14nm.

IV. CONCLUSION

In the above study, various approaches for characterizing sisal fibres have been studied . It is observed that all the methods require chemical treatment. If the fibres are subjected only to surface treatment long milling hours of the range 80 to 120 hours are required for obtaining nano size. Subjecting the fibres to a series of acid treatments reduces the milling hours but it is a time taking process. So by combining both the methods an optimal way for chemical treatment has been identified and adopted. The method adopted in this particular work showed a considerable reduction in the milling time from 100 hours to 36 hours for obtaining sisal nano fibre.

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