Synthesis of Bio – Degradable Banana Nanofibers

P. SURYA NAGENDRA
Dept. of Marine Engineering
Andhra University
Visakhapatnam, India

V.V.S. PRASAD
Dept. of Marine Engineering
Andhra University
Visakhapatnam, India

K. RAMJI
Department of Mechanical Engineering
Andhra University College of Engineering
Visakhapatnam, India

GANGADHARA PUSTY
Dept. of Mechanical & Manufacturing Engineering
University of New South Wales
Australia

Abstract - The present work includes the development of composites in nano based, reinforced with natural fibers. The fibers to be studied come from mechanical extraction from banana. The process, in general, consists of mechanical extraction. Further, the fibers are cut, classified in the adequate mesh and later on undergoes a chemical pulping process. The pulp is hydrolized and filtered, in order to produce nanofibers. A great deal of attention has been paid recently to cellulose nanofibrillar structures as components in nanocomposites. Present paper is aimed for production of Nano Banana fibers using high energy ball milling method. Banana stem powder was soaked in sodium hypochlorite solution for 24 h at room temperature then ball milled at variable milling time to produce banana stem powder dispersions with variable particle size. The effect of ball milling time on the particle size and morphology of the banana stem powder particles was examined. Results showed the mean particle size of the banana stem powder reduced progressively with milling time. The size of chemically treated fibers is reduced down to Nano crystalline level by high energy ball milling. Wet ball milling was carried out for the production of Banana Nano fibers. Wet milling with toluene as medium was carried out for different milling hours viz. 20, 40, 60 and 80 hours. The nanofibers are characterized by X-ray diffraction to measures the crystallinity.

Keywords - Biodegradability, Lignocellulosic fibers, Speed duration, Cellulose Extraction, High Energy Ball Milling

I. INTRODUCTION

There is a tremendous interest for using natural fiber nanoparticles to applied in the area of composites in material research. Synthesis and characterization of nanostructured materials have received a great attention because their superior mechanical properties [1]. Nano material is defined as the materials with the microstructure having least on dimension in nanometer range. It has appeal of miniaturization, imparts enhanced, mechanical, electronic, magnetic, optical and chemical properties to a level that cannot be achieved by conventional materials. Currently, natural fiber reinforced polymer composites technology is focused on creating low cost, high performance, and lightweight materials. There have been intensive research and product development of composite materials reinforced with natural fibers. Composites made of natural fibers are increasing attention for a variety of applications. Natural fibers such as coir, jute, banana, sisal etc. are widely available in India, Burma, Srilanka, Malaysia, Bangladesh and some of the African countries but are not optimally utilized. Over the last thirty years composite materials, plastics and ceramics have been the dominant emerging materials. The volume and number of applications of composite materials have grown steadily, penetrating and conquering new markets relentlessly [2]. Modern composite materials constitute a significant proportion of the engineered materials market ranging from everyday products to sophisticated niche applications. While composites have already proven their worth as weight-saving materials, the current challenge is to make them cost effective. The efforts to produce economically attractive composite components have resulted in several innovative manufacturing techniques currently being used in the Composites industry. It is obvious, especially for composites, that the improvement in manufacturing technology alone is not enough to overcome the cost hurdle. It is essential that there be an integrated effort in design, material, process, tooling, quality assurance, manufacturing, and even program management for composites to become competitive with metals.

Thus the shift of composite applications from aircraft to other commercial uses has become prominent in recent years. Increasingly enabled by the introduction of newer polymer resin matrix materials and high performance reinforcement fibers of glass, carbon and aramid, the penetration of these advanced materials has witnessed a steady expansion in uses and volume [3]. The increased volume has resulted in an expected reduction in costs. High performance FRP can now be found in such diverse applications as composite armoring designed to resist explosive impacts, fuel cylinders for natural gas vehicles, windmill blades, industrial drive shafts, support beams of highway bridges and even paper making rollers[4].

The present paper makes an attempt to generate the nano crystalline size banana fibers from micro sized banana fibers by using a high energy ball milling. The
milling media, milling balls and time of milling is taken as the process parameters. This nano structures banana fiber can be used as reinforcing fillers in making polymer matrix nanocomposites [5-9]. The expected gains these materials will be introduced in industrial sectors that require high performance composites, with the additional advantages of being renewable, recyclable and with great social importance, mainly in the case of sisal, jute, banana fibers.

II. NATURAL BANANAFIBER MATERIALS

A. Raw Materials

Lignocellulosic natural fibres are excellent raw materials for production of wide range of composites for different applications; Banana fibers are produced from the cellulose fibers, Banana Nano Fiber procured from M/s. Sri Laxmi Group of textiles, Mangalagire, Guntur, Andhra Pradesh, Fig:1 show the received banana fibers. The chopped banana fiber cut into small pieces as shown Fig.2. The interest in using natural fiber such as different plant fiber as reinforcement in polymers increased during last year’s. The fiber is a reinforcing material separated by an interface from the matrix. Natural fibers have a structural hierarchy in addition to that of the composite. The Physical Properties of banana fiber (wt.%) and mechanical properties are given Table.1 and Table.2 respectively [10].

| TABLE1: PHYSICAL PROPERTIES OF BANANA FIBER(WT.%) |
|------------------|------------|-----------|----------|------------|
| Lignin           | Hemicellulose | Moisture  | Pectin   | cellulose  |
| 5%               | 19%         | 10-11%    | 11%      | 63-64%     |

| TABLE 2: MECHANICAL PROPERTIES OF THE AS RECEIVED FINE BANANA FIBERS |
|-----------------|-------------|------------|
| Tensile strength, MPa | Young’s modulus, GPa | Density is Kg/m³ |
| 650-750         | 5.62        | 1350       |

B. Chemical Treatment

The fibers are cleaned normally in clean running water and dried. A glass beaker is taken and initially 8% NaOH is added in 80% of distilled water is added and a solution is made. After adequate drying of the fibers in normal shading for 2 to 3 hours, the fibers are taken and soaked in the prepared NaOH solution. Soaking is carried out for different time intervals depending upon the strength of fiber required. The fibers are soaked in the solution for three hours. After the fibers are taken out and washed in running water, these are dried for another 2 hours. The fibers are then taken for the next fabrication process namely the procasting process. Chemical treatment with NaOH removes moisture content from the fibers thereby increasing its strength. Also, chemical treatment enhances the flexural rigidity of the fibers. Last, this treatment clears all the impurities that are adjoining the fiber material and also stabilizes the molecular orientation. Loss of weight of banana fibers is observed after treatment. This loss is due to loss of lignin present in the banana fibers. As the percentage of NaOH is increasing the weight loss of banana fibers is increasing linearly up to 8% NaOH and later it is in decreasing for higher concentration of NaOH, as shown Fig.4 The addition of NaOH of banana fibers in hot condition the following reactions are takes place between lignin in banana and NaOH. Thus 8% NaOH is the optimum concentration of NaOH in removing lignin content present in the fibers.

Chemical analysis tests are conducted to find out the cellulose content in the fibers. As received banana fibers are cut into an average size of 1cm and washed with distilled water. The cut pieces of fibers are shown in Fig: 2. About 25 grams of the fiber taken in a dish containing 400cc of 2% NaOH solution [11]. This solution along with banana fibers is boiled for 6 hrs in circulating water bath in a chemical treatment unit at 100°C temperature. Periodically small quantity of water is added to the solution while boiling to avoid the vigorous boiling, evaporation and to keep the concentration of the solution constant. After 6hours boiling the contents in the solution are filtered in a filter paper and these boiled fibers are washed with fresh water and also dried in an oven at 30°C per one hour. The NaOH chemical treated banana fiber,
washed and dried fibers are remove lignin completely. After wash the fibers again dried to remove wetting completely. [12] The percentage of lignin removed is measured as the difference in the weight loss of fibers before and after chemical analysis are repeated for 6%, 8% & 10% NaOH solution.

\[
\% \text{ of Lignin removed} = \frac{W_i - W_f}{W_i} \times 100
\]

![Graph showing percentage of lignin removed with concentration of NaOH](image)

**III. PREPARATION STEPS INVOLVED FOR CELLULOSE NANO FIBERS**

The schematic diagram of the preparation of cellulose fibers by different stages of treatments were illustrated below in Fig. 5.

**IV. SYNTHESIS OF BANANA NANO FIBER BY HIGH ENERGY BALL MILLING**

The reduction in particle size of banana fibers to nano level is carried out by using high energy ball mill (Model: Restech, PM 100, Germany). The dried and chemically treated fibers are taken for milling Fig. 6. Shows the different milling hours. XRD analysis also supports the evidence for the reduction in size and the crystallinity of the fiber. These processed fibers can be used a better reinforcement in polymeric matrices. Mechanical properties of developed nanofiber reinforced Polypropylene composites were investigated. Fig. 7. shows the milling variation in XRD. The treated and untreated banana fibers were done to investigate the crystalline behavior and size of the fibers. From the XRD graphs, it is clear that the treated banana fibers show a crystalline nature [13]. The fibers show increasing orientation along a particular axis as the fibers are treated under different processing conditions.

**TABLE 3: EXPERIMENTAL DETAILS OF MILLING PROCESS**

<table>
<thead>
<tr>
<th>Time (hrs)</th>
<th>Image</th>
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<tbody>
<tr>
<td>20 hrs</td>
<td><img src="image" alt="Image" /></td>
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<td><img src="image" alt="Image" /></td>
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<td>80 hrs</td>
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**V. X-RAY DIFFRACTION STUDIES**

XRD analysis was studied using the room temperature powder X-ray diffraction (Model: PW 1830 diffractometer, Phillips, Netherland) with filtered 0.154 nm Cu Kα radiation. Samples are scanned in a continuous mode from 10°-80° with a scanning rate of 20 per minute except for fiber, for which scanning range is 50-450°. From XRD analysis crystallite size and degree of crystallinity is calculated [14]. XRD plots as shown in Fig. 7., in the Banana nano fibers after ball milling have been monitored with the help of wide angle X-ray diffraction studies. The average crystallite size is determined from the full width at half maximum (FWHM) of the X-Ray diffraction peak using Scherrer’s equation.

\[
D = \frac{K\lambda}{B \cos \theta}
\]

Where D is the particle diameter, \(\lambda\) is the X-Ray wave length, B is the FWHM of the diffraction peak, \(\theta\) is the diffraction angel and K is the Scherrer’s constant of the order of unity for usual crystals. The magnified view of the major peaks of 80 hrs ball milling is given; it is observed that the peaks of banana fibers are matched with the XRD pattern of cellulose. Fig.8 shows the variations in crystallite size of Banana fibers with milling time.
It is observed that from Table 4, the crystallite size of the banana fibers is reduced with increasing milling time; the size reduction goes into saturation after 80 hours of milling. 80 hours is the optimum milling time as shown in Table 4.

**Table 4: Crystallite Size of different Milling hours**

<table>
<thead>
<tr>
<th>Milling hours</th>
<th>Crystallite Size (nm)</th>
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<tbody>
<tr>
<td>20</td>
<td>120</td>
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<tr>
<td>40</td>
<td>84</td>
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<tr>
<td>60</td>
<td>72</td>
</tr>
<tr>
<td>80</td>
<td>45</td>
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**Fig. 7 X-ray diffraction patterns**

**Fig. 8 variations in crystallite size of Banana fibers with milling time**

**VI. CONCLUSIONS**

The lignin present in the Banana fibers is removed by treating with 8% NaOH. The phase present in the ball milled banana fibers is identified as cellulose by XRD analysis. The size of banana fibers is brought to nano level using high energy ball milling and it is concluded that 80 hours is the optimum milling time.

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**REFERENCES**


