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Characterization of New Cellulosic Fiber from the bark of Hardwickia Binata (Narepa)

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Abstract

This paper presents the extraction and effect of alkali on the physical, chemical, tensile and thermal characterization of Narepa fiber obtained from the bark of the tree, which is richly available near the forest areas of srisailam in Andhra Pradesh, India. To improve the properties of composites Narepa fibers were treated with sodium hydroxide. Alkali treatment was carried out using NaoH solution at 5% concentration for 2 hours. Characterization of untreated and alkali treated Narepa fiber was carried out by studying the chemical composition, surface morphology, crystallinity, tensile and thermal behavior .It was found that untreated fiber have lower cellulose content, crystallinity, tensile properties and thermal stability than alkali treated fiber. Based on the properties, it is determined that alkali treated Narepa fibers, are suitable as reinforcement in Natural fiber reinforced composites.

Keywords: Narepa Fiber, crystalinity, Morphology, Infrared spectroscopy, tensile properties.

INTRODUCTION

In recent decades, increasing global warming and environmental hazardness most of the researchers are concentrating on green materials to improve the environmental quality of the products [1]. Natural fibers are eco-friendly and bio-degradable which are extracted from the tree stem, leaves and roots are used in many automobiles, aerospace, marine and civil structures due to its low weight, specific strength and good thermal properties.[2,3] Natural fibers plays an important role in the development of bio-degradable composites to meet the current requirements[4,5]. They possess many favorable advantages over synthetic fibers in terms of low cost, low density, biodegradability, good acoustic and thermal insulation,and mechanical properties of natural fibers are incorporated as reinforcement in a wide variety of polymer matrices due to their many benefits, such as low volumetric cost, increase of heat deflection temperature, increase of stiffness of polymer composites.[6]Hence it is necessary to find new fibers which are suitable as reinforcement for the development of natural fiber composites like century fibers, Retama, Grewia tilifolia, Maize Tassel, Indian Mallow stem etc[7,8].

In the present research the newly identified Narepa Fiber obtained from the trees which are grayish brown in color, rough with deep cracks and it darkens with age from the species information [9]. The compound leaves have only two leaflets which are joined at the base. The timber obtained from the tree is the hardest and heaviest(among timbers from the trees found in India), is durable and termite resistant [10].The Scientific name of Narepa or Anjan Plant is Hardwickia Binata which belongs to the family "caesalpiniaceae" native to tropical and subtropical regions [11]. Concerning the above this investigation deals with extraction of new natural fibers from the stem of Hardwickia Binata Plant and the properties were still improved in the present study shown in view of aspect ratio ,optical micro images and sem images comparing to the previous analysis.[12]Concerning the above, this investigation deals with the extraction of new natural fibers as source from the stem of Narepa plant and analysis of the physico-chemical, mechanical and thermal properties of IMFs using X-ray diffraction method (XRD), Fourier transform infrared (FTIR) spectroscopy, Thermo gravimetric analysis (TGA) and uni-directional fiber tensile test in comparison with other natural fiber and the prime objective is to explore green composites which are environment friendly.

MATERIALS AND METHODS

Materials

Extracted Narepa fibers, analytical grade acetic acid, sodium bi-sulfite, sodium chlorite and sodium hydroxide pellets were used in the present study.

(a)

(b)

Figure 1: (a) Narepa (Hardwickia Binata) Tree (b) Stem

Fiber Extraction

Narepa plants are widely grown near thambarajapalli (forest area), and Nalamala forests near srisailam, Kurnool district in Andhra Pradesh. Previously this fiber will be using for agricultural purposes in making ropes and cots. The usual water retting process for extraction of fibers from the bark of the matured stems has been adopted. The fresh matured stems were cut from the plant and immersed in water for about 2 months and during this period, water was changed regularly. The stems were crushed by light beating; the separated single fibers were dried initially in the sun for a week. The fibers were then kept in a hot air oven for 2 hrs at 60^0c-70^0c to remove any moisture. Finally the fibers were stored in polyethylene bags and placed at 23^0c and 50% relative humidity for conditioning prior to further testing.

(a)

(b)

Figure 2: (a) Manually Extracted Fiber (b) After retting Process

Alkali Treatment

Narepa fibers were treated with 5% sodium hydroxide solution at 30^0c, maintaining a liquor ratio of 25:1 and immersed in the alkali solution for 30 minutes. The fibers were then washed with tap water repeatedly, neutralized with dilute acetic acid (Vinegar) again washed with distilled water, and dried in sun light and then in hot air oven to remove any moisture still present in the fiber.

Chemical Analysis

The chemical composition of both untreated and alkali treated Hardwickia Binata fibers was determined using the standard TAPPI (Technical Association of the Pulp and Paper Industries) and other methods for different components, namely: T 203 cm-99 (for α-cellulose) and T 222 om-06 (for lignin). The holocellulose was determined according to the method described[13]. The hemicelluloses fraction was calculated as the difference between the holocellulose and α-cellulose content. The % content of extractives like α-cellulose, hemicellulose and lignin were determined and the average values based on four samples were reported.

Fourier Transform-Infrared Spectroscopy

FT-IR spectra were used to examine the structure of Hardwickia Binata fibers that were obtained after alkali treatment. A Nicolet 560 spectrum photometer was used to obtain the spectra of each sample. The untreated and treated Hardwickia Binata fibers were grounded and the powder was compressed into plates for FT-IR analysis. The FT-IR spectra of the samples were obtained in the wavelength range of 4000–500cm^{-1}.

X-ray Analysis

The wide-angle X-ray diffractograms of both untreated and alkali treated fibers were recorded on a Rigaku Dmax 2500 diffractometer .The system has a rotating anode generator with a copper target and a wide-angle powder goniometry. The generator was operated at 40 kV and 150 mA, and the samples were scanned in the 2θ range of 5^0–70^0c

Fiber Tensile Test

The tensile test of both untreated and alkali-treated single fibers was performed using a computer controlled Instron (model-3369) machine employing a load cell of 10 kN, maintaining a gauge length of 50 mm and a crosshead speed of 5 mm/min [14]. The test was conducted at a standard laboratory atmosphere of 23^0C and 50% RH. Twenty-five each of both (untreated and alkali treated) types of fibers were tested and the average values recorded.

Table 1: Physical properties, chemical compositions, and tensile properties of Hardwickia Binata fibers

Properties	Untreated	Treated
Physical Properties		
Length(mm)	300	296
Diameter(mm)	0.26	0.18
Aspect Ratio(L/D)	1154	1644
Moisture	5.25	4.93
Chemical Composition		
Cellulose%	71.65	81.68
Hemi cellulose%	22.24	7.01
Lignin	6.09	11.28
Tensile Properties		
Max Stress(Mpa)	276	332

Table: 2. Comparison of Properties of different Natural fibers

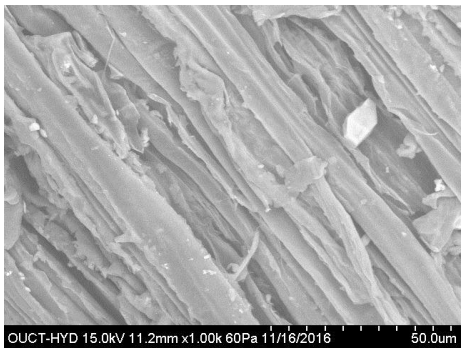
Fiber Name	Cellulose (%)	Hemi cellulose (%)	Lignin (%)	Wax (%)	Moisture (%)	Ash (%)	Density	Diameter (mm)	Ref
Jute	61-71.5	-	11.8-13	0.5	12.5-13.7	0.5-2	1.3	40-350	
Bamboo	26-43	-	1-30	-	9.16	-	0.91	240-330	
Indian Mallow	78.22	-	6.14	0.47	9.6	5.85	1.33	44.7-84.3	
Grewia Telefolia	62.8	21.2	14.9	-	2.3	-	-	-	
Dichrostachys	72.4	13.8	16.89	0.57	9.82	-	1.2	240	
Borassus	53.4	29.4	17	-	-				
Sansevieriacylindrica	79.7		3.8	0.09	3.08		0.915	230-28-	
Hardwickia Binata	81.68	7.01	11.28	-	5.25	-	1.5	260-300	*Present work*

RESULTS AND DISCUSSION

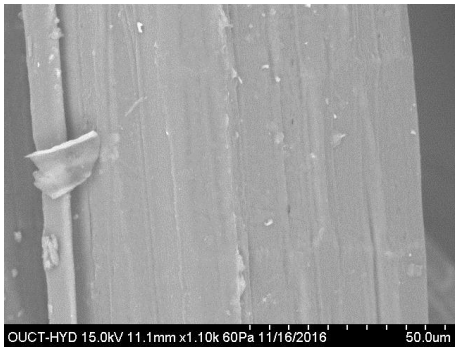
Morphology of Untreated and treated fibers

The morphology of the untreated and alkali-treated Hardwickia Binata fibers was observed using scanning electron microscope (SEM) to evaluate the fiber surface and cross-section, and the micrographs are shown in Figure3. The micrographs of the untreated fibers from figure 3(a) indicate the presence of impurities (waxes and fats) on the fiber surface and no fibrillation. The natural substances (impurities) on the fiber surface contribute to ineffective fiber-matrix bonding and poor surface wet-out. Figure 3b shows clear surface with no impurities. The cross-section of the untreated fiber Figure 3c confirms that the single fiber consists of number of hollow-type of different polygonal shape, thick microstructures. Generally, these are the micro fibrils that have hemicellulose and lignin decorating the outside. These act as the connection between the microfibrils, creating the primary structural network. A similar observation has been made in Borassus fruit fiber. The fiber morphology changes drastically by the alkalization reaction. The fibers surface becomes rough, and the fibrils are apparent in alkali-treated fiber (Figure 3d).

Untreated & Treated Hardwickia Binata Fiber



(a)



(b)

(c)

(d)

Figure 3: Scanning electron micrographs of untreated and alkali-treated Hardwickia Binata fibers: (a) Surface of untreated fiber, (b) surface of alkali treated fiber, (c) cross-section of untreated fiber and (d) cross-section of alkali-treated fiber.

FTIR Spectra Analysis

In order to confirm the chemical structures of untreated and alkali treated Hardwickia binata fibers, FTIR analysis was performed. The composition changes observed for untreated and alkali-treated Hardwickia binata fibers are shown in Figure4. The spectra show the presence of hydroxyl, carbonyl,ether groups, and absorbed water in the untreated Hardwickia binata fiber. Important changes in the FT-IR spectra are associated with the peak intensities around 1020.83, 1365.71, 1654.78, 1735.54, and 896cm^{-1}. As shown in Figure, the hemicelluloses intensity of the peak occurred [15-18] at around 1735 and 1017cm $^{-1}$ in the untreated Hardwickia binata fiber, and the spectrum is not visible in the alkali-treated fiber, which indicates that the hemicelluloses were removed completely by the alkali treatment. This is quite expected as hemicelluloses is soluble in aqueous NaOH (alkali)solution.[19] The band C=C as in tassel fiber represents the aromatic vibration at 1600cm^{-1} from methoxyl groups of lignin. This band was significantly reduced in the alkali-treated sample because of the removal of most of the hemicelluloses and lignin from the fiber through treatment. The results indicate that the intensity of the peak at around 1654cm^{-1} is attributed to the absorbed extra moisture on the surface of the fiber after alkali treatment. Finally, the result indicates that there were no considerable changes noticed in the intensity of other peaks in the FT-IR spectra. The intensity of the peaks around 3840 and 2920cm^{-1} are associated with the a-cellulose (O-H) stretching. Finally, the FT-IR results indicate that the hemicelluloses and lignin were removed from the Hardwickia Binata fibers during alkali treatment.

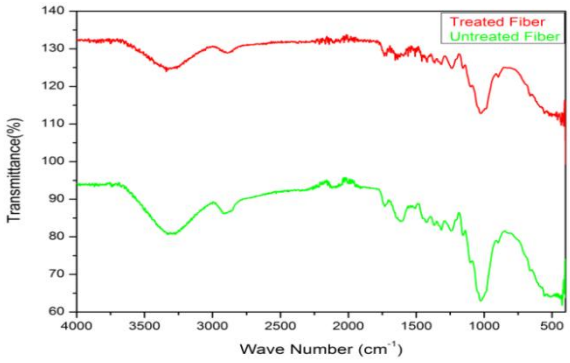


Figure: 4 FTIR Spectrum of alkali treated and untreated fiber

XRD Analysis

The X-ray diffractograms of the untreated and alkali-treated fibers are shown in Figure 5. The diffractograms shows two main reflections, corresponding to 2θ values at low-angle reflection (16.3◦), which is broad whereas the high-angle reflection (22.5◦) is sharp and intense. These reflections are attributed to the amorphous (I_{am}) and crystalline (I_{002}) components in the fiber, respectively,it is evident that the intensity of crystalline peak of the alkali-treated fibers is higher than that of the untreated fibers. The crystalline index (CI) was calculated using following equation.

$$CI = [(I_{002} - I_{am})/I_{002}] \times 100$$

The crystalline index of alkali-treated fibers is found to be higher than the untreated fibers. This may be due to the formation of new hydrogen bonds between certain of the cellulose chains because of the removal of hemicelluloses, which normally separates the cellulose chains. On the other hand, due to the removal of amorphous hemicellulose from the fibers and the rearrangement of the crystalline regions, the alkali-treated fiber exhibits a higher crystalline nature.

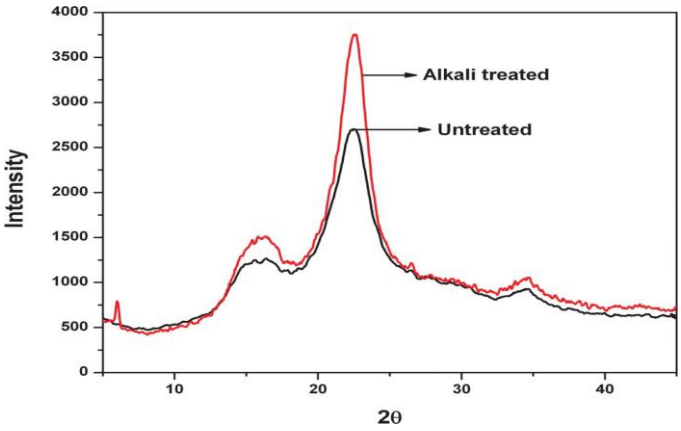


Figure: 5 XRD of Alkali treated and untreated fiber

Tensile properties of Fiber

The tensile properties (maximum stress, modulus, and elongation at break) of untreated and alkali-treated Hardwickia binata fibers are listed in Table 1. Typical stress–strain curves of untreated and alkali-treated fiber are shown in Figure 5. The untreated fiber exhibits a brittle behavior with a sudden load drop when fiber failure occurs while alkali-treated fiber appears to be nonlinear and similar to plastic deformation behavior. The tensile data (Table 1) support that for the alkali-treated fibers the maximum stress and elongation-at-break increased significantly. It can be attributed to an increase in packing density and molecular orientation due to the removal of the hemicelluloses in inter-fibrillar region. However, the Young's modulus has decreased marginally and this fact may be attributed to fiber damage caused by chemical reaction with sodium hydroxide during the treatment. A similar observation has been made in the case of single curaua fiber after alkali treatment .The tensile maximum stress and elongation at- break of alkali-treated fiber are 22.9% and 120.5% higher, respectively, while the tensile modulus is 10.4% lower than the untreated fiber.

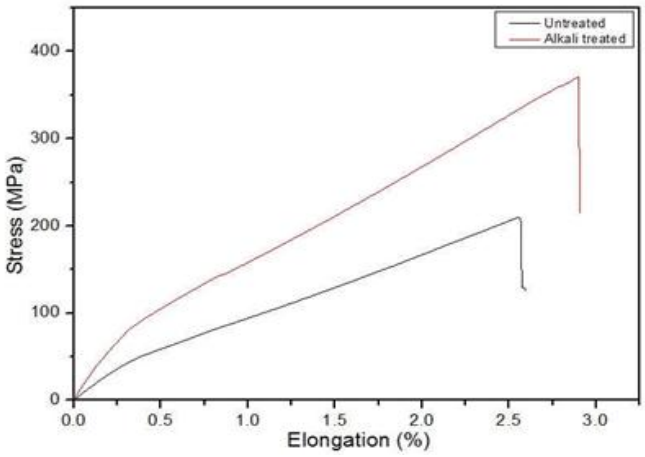


Figure: 6 Tensile property of alkali treated and untreated fiber

TGA Analysis

The thermal stability of the untreated and alkali-treated fibers was investigated by TGA, and corresponding primary thermograms are shown in Figure 6. The fiber decomposition is exhibited in two stages, indicating the presence of three different constituents. For both untreated and alkali-treated fibers, the first decomposition is found in the temperature range of 240^0C–320^0C indicating the loss of hemicellulose and some fraction of lignin. The second decomposition corresponds to the decomposition of cellulose as observed in the temperature range of 320^0C–400^0C. Lignin is the most difficult constituent to decompose; its decomposition usually extends to the whole temperature range, starting around 200^0C going up to 700^0C. The residual char content, however, has increased from 15.8% to 18.7% on alkali treatment. The temperatures at the inflection points of the curves (the maximum rate of degradation) occur at 366^0C and 376^0C for the untreated and alkali-treated fibers, respectively. This indicates the marginal difference in the thermal stability of untreated and alkali-treated fibers.

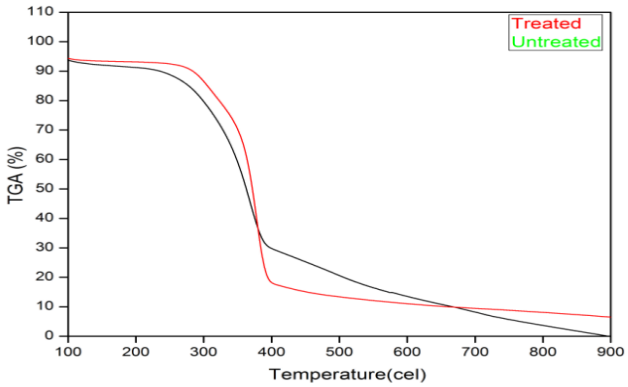


Figure: 7 TGA curve of Alkali treated and untreated fiber

CONCLUSIONS

In general, untreated natural Hardwickia fibers have relatively low physical, mechanical, and thermal properties over chemically treated fibers. The objective of this work is to further improve the physical, mechanical, and thermal properties of Nareoa fiber using an alkali treatment. This alkali treatment shows improved physical, mechanical, and thermal properties over untreated fiber. Morphology studies indicate that the diameter of the fiber decreases after alkali treatment removing hemicellulose. The elimination of amorphous hemicelluloses of the fibers to the maximum extent on alkali treatment is proved by chemical analysis and FTIR studies. The removal of hemicelluloses from fiber cells releases the internal constraint and the fibrils become more capable of rearranging themselves in a compact manner, leading to a closer packing of cellulose chains. This closer packing of the cellulose also improves the crystallinity of the fiber after alkali treatment as evidenced by XRD analysis. The tensile stress and elongation-at-break properties of the fibers are found to increase after alkali treatment. The thermal stability of the fibers improved slightly by alkali treatment. Based on improved tensile properties, renewability and environment friendly nature, Narepa fibers are considered as an effective reinforcement in bio-composites.

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