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EXTRACTION AND CHARACTERIZATION OF TERMINALIA BELLIRICA FIBER

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ABSTRACT

In the present work, fibers were extracted from the bark of terminalia bellirica tree and were investigated for their properties in detail. Fibers were treated with 5% Sodium hydroxide solution for 30 minutes and the effect of this alkali treatment on the fiber properties was studied. FT-IR studies and chemical analysis of the terminalia bellirica fibers showed the reduction of hemicellulose on alkali treatment. X Ray diffraction studies proved the increase in the crystallinity of the fibers after alkali treatment. Increase in thermal stability and tensile properties of terminalia bellirica fibers after alkali treatment was observed. Scanning electron micrographs showed the roughened surface of the fibers on alkali treatment.

Keywords: terminalia bellirica fiber, characterization, chemical composition, morphology, tensile properties, thermal stability.

INTRODUCTION

With the increase in environmental concern, the focus of researchers has been shifted more towards the eco friendly materials. There has been a high interest in the replacement of synthetic fibers with plant fibers in polymeric composites [1]. At the same time, the demand for high performance materials is rapidly growing in automobiles, aerospace and mechanical fields [2]. Natural fibers have been a promising material which can be used as reinforcement in polymer composites. Reinforcing natural fibers in a wide variety of polymer matrices will have many advantages, such as low cost, less density, biodegradability, High stiffness, etc., Therefore, natural fiber reinforced polymer composites have achieved several applications in making furniture components, inner parts of automobiles, packing pallets and many other applications [3]. The interfacial bond between fiber and polymer matrix may lead to the poor performance of the composite. Therefore proper surface treatment should be done to the fiber in order to increase the interfacial bond between them. Many methods like physical and chemical treatments are there to increase the interfacial campatability between fiber and polymer matrix. Among these treatments, alkali treatment is more economical. The knowledge about Physico chemical properties as well as mechanical behavior of natural fibers is necessary in order to get optimal performance of the fiber in composites [4].

Terminalia bellirica, a deciduous tree which belongs to Combretaceae family, is widely available in Southeast Asia [5]. It is one of the most commonly used plants in Indian traditional systems of medicine. In traditional Indian Ayurvedic medicine, the fruit of Terminalia bellirica has been extensively used as a folk medicine for the treatments of diabetes, hypertension and rheumatism [6]. Bark of this tree is blessed with some properties of tonic like cardio and diuretic. These compounds are colligated with a wide diverseness of biological activities like antibacterial, antifungal and anti-microbial [7]. The rind of terminalia bellirica fruit is used as one of the ingredients of Triphala (three fruits powder). The crude extracts of terminalia bellirica fruit is also used in Ayurveda for different treatments like treatment for cough, fever, dysentery, diarrhea and diseases related to skin [8, 9] Anti-viral activity [10, 14] including anti-HIV-1 activity, anti-bacterial, and anti-fungal activity [15, 16].

Though there exists literature about the medicinal applications of terminalia bellirica fiber, however, to the best of our knowledge, there are no previous works done on the characterization of these fibers, such as chemical composition, morphology, tensile and thermal properties to demonstrate the effective utilization of the Terminalia Bellirica fiber in the field of composites. Therefore the premiere objective of this article was to explore the potential of Terminalia Bellirica fibers as reinforcement in bio-composites.

Figure-1. Extracted Terminalia Bellirica fiber.

MATERIALS AND METHODS

Materials

Extracted Terminalia Bellirica fibers from its bark, analytical grade acetic acid, sodium hydroxide pellets, sulfuric acid, sodium chlorite and sodium bisulfite were used in this work.

Fiber Extraction

Terminalia Bellirica trees are widely grown in the Nallamala forest, which is located in the Kurnool district, Andhra Pradesh, India. In this work, the barks were cut from the Terminalia Bellirica tree and immersed in water for around 2 months. Water and mechanical retting process was adopted for extraction of fibers from its bark. The separated fiber layers were washed thoroughly using water and then dried for one week to ensure maximum moisture removal as shown in figure 1. Finally, the fibers were kept in a hot air oven for 24 hours at 100°C to remove moisture.

Determination of chemical composition

The chemical analysis was done to evaluate the different chemical compositions for both untreated and alkali treated terminalia bellirica fibers as per TAPPI (Technical Association of the Pulp and Paper Industries) standards. Both untreated and alkali treated fibers were dewaxed before undergoing these methods. The method T 203 cm-99 was used to determine α-cellulose content [17] and the method T 222 om-06 to determine lignin content [18]. The holocellulose content was determined by the procedure stated by Wise *et al* [19]. The difference bxetween α-cellulose and holocellulose content gives hemicelluloses content in the fiber.

Fourier Transform-Infrared Spectroscopy

Fourier transform-infrared spectroscopy studies on both untreated and alkali treated Terminalia Bellirica fibers were carried out using a Nicolet Smart iTR ATR and iS 10 FT-IR spectrophotometer. All spectra were recorded in the region of 4000-400 cm^{-1} with 32 scans in each case at 4 cm^{-1} resolution.

X-Ray Diffraction Analysis

Wide-angle X-ray diffractograms of untreated and alkali treated Terminalia Bellirica fibers were recorded on X'Pert³ Powder, which is a PANalytical's newest X-ray diffraction system at NIT warangal. The generator was operated at 30 mA and 45 kV. The samples were scanned in the 2 θ range of 5.996°–70°.

Morphology

Morphology of untreated and alkali treated Terminalia Bellirica fibers were examined using a scanning electron microscope. The samples were gold coated and their surface observed under an EDAX Ametek scanning electron microscope.

Thermogravimetric Analysis

The thermograms of untreated and alkali treated terminalia Bellirica fibers were recorded using a thermogravimetric analyzer Perkin Elmer STA 6000. Samples of 10 mg were placed in appropriate platinum pans and heated from 40°C to 700°C at 20°C min^{-1} under dynamic flow of nitrogen (100 mL min^{-1}).

Tensile Testing

The tensile properties were determined using Instron 3369 Universal testing machine at a crosshead speed of 5 mm/min, maintaining a gauge length of 50 mm. Four samples were tested in each case to get statistically significant data. Further, an average of properties such as tensile strength, tensile modulus, and percentage elongation at break was reported.

RESULTS AND DISCUSSIONS

Extraction of fiber from the barks of terminalia bellirica tree and study their surface morphology, crystalline nature, chemical composition, tensile properties and thermal stability before and after alkali treatment was the primary work involved in this article.

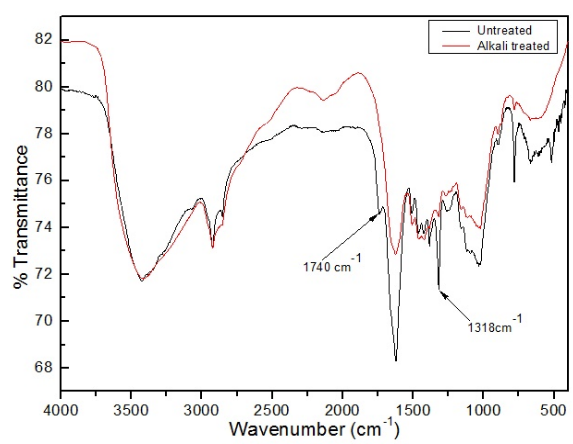

Figure-2. FT-IR spectra of untreated and alkali-treated Terminalia Bellirica fiber.

Untreated and alkali-treated terminelia bellirica fibers were analyzed using FT-IR to appraise the various chemical constituents present. Figure-2 shows the FT-IR spectra of the untreated and alkali-treated fibers. The broad intense absorption band at 3424 cm^{-1} occurred due to O-H stretching vibrations of hydrogen bonded hydroxyl (OH) groups, present in their main components [20]. The absorption bands at 2923 and 2851cm^{-1} were attributed to the asymmetric and symmetric stretching of methylene (-CH_2–) units of the main components [21]. The important absorption band at 1740 cm^{-1} in the untreated fibers was assigned to the carbonyl groups (C=O) due to the presence of acetyl ester and carbonyl aldehyde groups of hemicellulose and lignin [21]. This band intensity decreased considerably when fibers were treated with alkali solution due to the partial removal of the hemicellulose component. The absorption band at 1623

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cm^{-1} agreed to the bending mode of water absorbed water [23], while the absorption band at $1460cm^{-1}$ corresponds to C–H deformation in the methyl, methylene and methoxyl groups of lignin [20,21]. The band at 1424 cm^{-1} correspond to $-CH_2$ scissoring or bending vibration, while the absorption band at 1384 cm^{-1} corresponds to the C−H asymmetric deformation of cellulose [21]. The intensity of the band at 1318 cm^{-1}, corresponding to the C-O stretch vibration of corboxylic or alchoholic group in hemicellulose, was sharply weakened after alkali treatment because of the partial removal of the hemicellulose component [20]. The absorption band at 1156 cm^{-1} corresponds to the C−O anti symmetric bridge stretching of cellulose. The absorption bands at 1043 and 1030 cm^{-1} correspond to the C−O−C pyranose ring skeletal vibration of cellulose [22]. The absorption band at 896 cm^{-1} corresponds to β-glucosidic linkages between the sugar units in hemicellulose and cellulose [21]. FT-IR analysis also confirmed the existence of chemical components (cellulose, hemicellulose, and lignin) of terminalia bellirica fibers and further suggests the reduction of the hemicellulose content on alkali treatment of the fibers. This is also in support of the chemical analysis data of the alkali-treated fibers as shown in Table-1.

Table-1. Chemical compositions and tensile properties of Terminalia Bellirica fibers.

Parameter	Untreated Terminalia Bellirica fiber	Alkali treated Terminalia Bellirica fiber
Chemical composition		
α - Cellulose (%)	54.26	60.34
Hemicelluloses (%)	28.60	20.58
Lignin (%)	17.13	19.08
Tensile properties		
Strength (MPa)	97.45	145.99
Modulus (GPa)	10.00	7.25
Elongation at break (%)	1.30	2.32

The chemical composition of Terminalia Bellirica fibers for both untreated and alkali treated was determined and are summarized in Table-1. From Table-1, the untreated fibers contained 54.26% of α-cellulose, 28.60% of hemicellulose and 17.13% of lignin. After alkali treatment, the fibers contained 60.34% of α-cellulose, 20.58% of hemicellulose and 19.08% of lignin. Reduction of hemicelluloses from 28.60% to 20.58% after alkali treatment was observed. It is well known that the sensitivity of hemicellulose to the sodium hydroxide is more. Increase in the cellulose and lignin content after alkali treatment was observed.

A comparison is made among Terminalia Bellirica fibers and some important natural fibers in terms of their chemical compositions and tensile properties as shown in Table 2 [4, 24, 25, 26, 27]. From Table-2, it is clear that borassus, bamboo, coir and napier has lower cellulose content than Terminalia Bellirica fiber. The hemicelluloses content of Terminalia Bellirica fiber is lower than that of bamboo, coir and napier but higher than that of remaining other fibers. Finally, the lignin content is lower than that of bamboo, coir and oil palm.

Table-2. Comparison of Chemical Composition and Tensile properties of Terminalia Bellirica fiber with other natural fibers.

Fiber	Cellulose (wt. %)	Hemicellulose (wt. %)	Lignin (wt. %)	Tensile Strength (MPa)	Young's Modulus (GPa)	Elongation at break (%)	Reference
Abaca	56-63	20-25	7-9	400	12	3-10	[25]
Bamboo	26-43	30	21-31	140-230	11-17	-	[25]
Banana	63-64	19	5	500	12	5.9	[25]
Borassus	53.4	29.6	17	70.8	10.8	34.8	[26]
Coir	32-43	0.15-0.25	40-45	175	4-6	30	[25]
Flax	71	18.6-20.6	2.2	345-1500	27.6	2.7-3.2	[25]
Hardwickia Binata	78.12	14.87	7.67	210	10.7	2.56	[4]
Hemp	68	15	10	690	70	1.6	[25]
Jute	61-71	14-20	12-13	393-773	26.5	1.5-1.8	[25]
Kenaf	72	20.3	9	930	53	1.6	[25]
Napier	45.66	33.67	20.60	75	6.8	2.8	[27]
Oil Palm	65	-	29	248	3.2	25	[25]
Pineapple	81	-	12.7	1.44	400-627	14.5	[25]
Ramie	68.6-76.2	13.16	0.6-0.7	560	24.5	2.5	[25]
Sisal	66-78	10-14	10-14	468-700	9.4-22	3-7	[25]
Thespesia	60.63	26.64	12.70	573	61.2	0.79	[24]
Terminalia Bellirica	54.26	28.60	17.13	97.45	10.0	1.30	This work

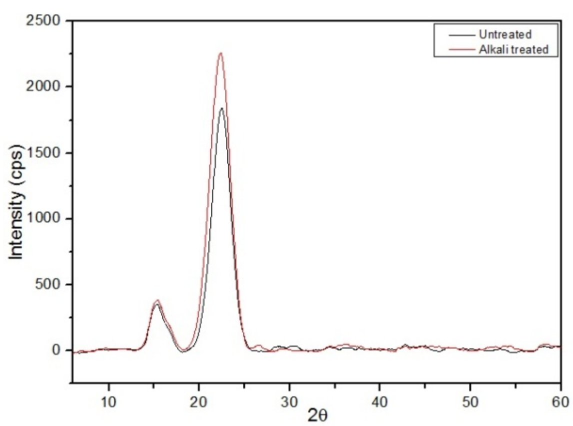

Figure-3. X-ray diffractograms of untreated and alkali treated Terminalia Bellirica fibers.

In order to examine the crystalline nature of the fiber, X ray studies was carried out for both untreated and alkali-treated Terminalia Bellirica fibers. From Figure-3, both treated and untreated fibers showed two main reflections, corresponding to 2 θ values at around 16° and 22° respectively. The eflection at 16.65° is somewhat broad when compared to the reflection at 22.88° (sharp with high intensity). These sharp and broad reflections are ascribed to the crystalline (I_{002}) and amorphous (I_{am}) part in the terminalia bellirica fiber respectively. From this Figure-3, it is evident that the alkali-treated fiber has higher crystallinity when compared to untreated fibers. Further from the following equation, the crystallinity index (CI) was calculated.

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

Crystallinity index for untreated fibers was calculated and its value is 80.78. The same for alkali treated fibers is found to be 83.08. due to the partial removal of hemicelluloses after alkali treatment, the rearrangement of crystalline portions were taken place in fibers, so that the crystallinity index of the fibers was increased after alkali treatment. Which means that the preferential amount of hydrolysis took place. This is in support with results obtained from chemical analyses as well as FT-IR.

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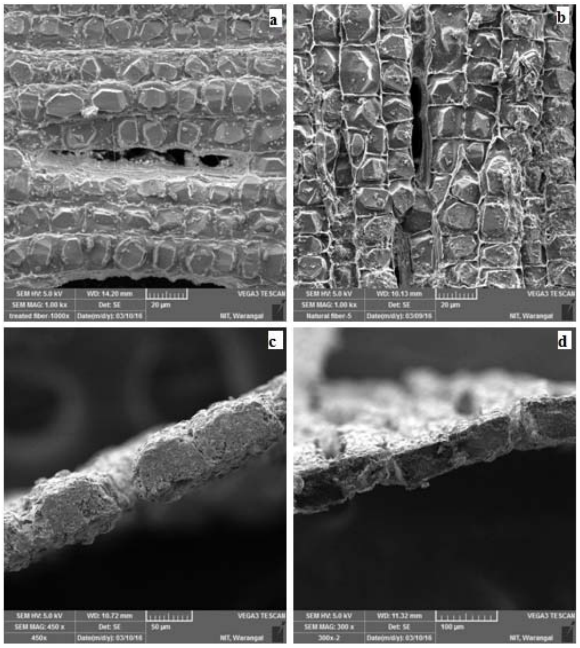

Figure-4. (a), (b) SEM images of Untreated and alkali treated fiber surfaces respectively. (c), (d) SEM images of Untreated and alkali treated fiber cross sections respectively.

SEM images of both untreated and alkali-treated fibers are shown in Figure-4. From Figures 4(a) and 4(c), it is clear that the untreated fibers contained impurities (wax, fatty substances, etc.). From Figures 4(b) and 4(d), roughened surface of the fiber can be observed after alkali treatment as the impurities were washed off with the effect of alkali treatment. Also the slight reduction in diameter of the fiber after alkali treatment was observed due to the removal of impurities as shown in figure 4(d). Thus roughened surface may amend the interfacial bonding between the fibers and matrix when the fibers are reinforced in a polymer matrix.

The stress-strain behavior of untreated and alkali treated Terminalia Bellirica fibers which are linear up to failure as shown in Figure-5. The tensile properties for both untreated as well as alkali-treated Terminalia Bellirica fiber were determined and are listed in Table-1. From Table-1, increase in the tensile strength and elongation at break of alkali-treated fibers was observed. The reason for the increased tensile strength is due to the tending of fibers to pack closely due to the gaps formed in removal of hemicelluloses after alkali treatment so that the new hydrogen bonds can be formed in between the cellulose chains which are responsible for the transfer of stress from fibril to fibril. Therefore alkali treated fibers always exhibits better tensile properties than untreated fibers. The tensile properties of other natural fibers and are listed in Table 2 [4, 24, 25, 26, 27]. From Table-2, it is evident that the tensile strength of Terminalia Bellirica fiber is higher than that of borassus, pineapple and napier and lower than that of remaining all other fibers. The tensile modulus of Terminalia Bellirica fibers is significantly higher than that of coir, napier and oil palm and lower than those of remaining all other fibers. While the elongation at break is higher than that of thespesia. Thus this comparison reflects the compatability of using Terminalia Bellirica fiber as reinforcement in bio composites.

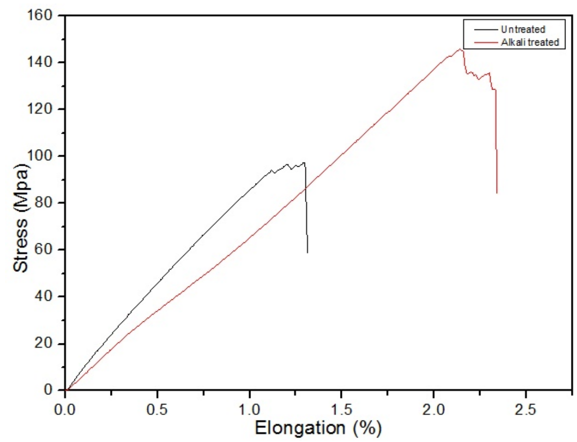

Figure-5. Stress-strain curves of untreated and alkali-treated Terminalia Bellirica fibers.

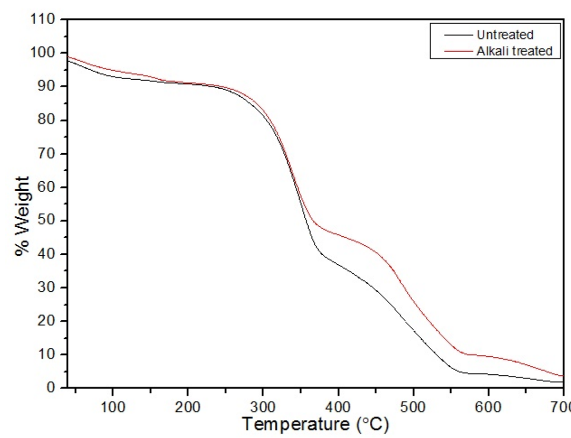

Figure-6. Primary thermograms of untreated and alkali-treated Terminalia Bellirica fibers.

The TGA was conducted to access the thermal stability of the treated and untreated terminalia bellirica fibers as shown in the Figure-6. The fiber decomposition occurred in multi stages where in first stage which correspond to moisture/lesser volatile elimination. The next stage often known as medium temperature degradation stage can be observed where hemicelluloses andsome part of lignin was degraded [28]. The last stage of the process where high temperature range takes part has associated with degradation of α cellulose and lignin. Here it can be noted that almost all cellulose and lignin decomposition occurred Thermo gravimetric analysis (TGA) is helpful to study the thermal stability of materials and also about decomposition of chemical components like cellulose, hemicellulose and lignin. The primary thermograms of untreated and alkali-treated fibers are shown in Figure 5, in which both fibers exhibit a similar trend of decomposition in three stages of weight loss. Both untreated and alkali-treated fibers thermograms show a weight loss at around were pyrolyzed as it .happens almost in most of the natural fibers [29, 30, 31]. The points of inflection for these curves occurred at 332.32 °C and 360.89 °C for treated and untreated fibers respectively. Through these thermograms, different parameters like % moisture content, char content inflection point, IDT, FDT, 25% and 50% degradations were found and are mentioned in Table-3. From these indications it is significant that thermal stability parameters of treated fibers were high than untreated fibers. Further, these results indicate that alkali-treated fibers can be used as reinforcement, even with thermoplastic polymers whose processing temperature is below 260°C.

Table-3. Thermal degradation parameters of Terminalia bellirica fibers.

Parameter	Untreated	Treated
% moisture content	5.2	4
% char content	3.33	1.94
IDT (^oC)	218	260
25% degradation temperature (^oC)	304	325
50% degradation temperature (^oC)	350	369
FDT (^oC)	690	700

CONCLUSIONS

Extracted fibers from the stems of Terminalia Bellirica tree by Mechanical water retting process were subjected to 5% alkali treatment. The influence of alkali treatment on the chemical composition, morphology, thermal and tensile properties of the fibers was studied. Scanning electron micrographs showed the roughened surface of the fibers after alkali treatment which is due to the significant removal of hemicellulose as well as surface impurities. These obtained results have been supported by FT-IR studies and chemical analysis. X Ray difractograms indicated an increase in crystallinity on alkali treatment and TGA studies indicated thermal stability of the fibers on alkali treatment. The tensile properties of alkali-treated fibers were found to be superior to those of untreated fibers. This article indicated that alkali treatment of Terminalia Bellirica fiber improved the crystallinity, thermal stability and tensile properties of the fiber. The results like chemical composition and tensile characterization were compared to those of other commonly used lignocellulosic fibers, and these terminalia bellirica fibers show some potential only after alkali treatment as reinforcement in polymer matrix composites. This may results in local development and increase in the environmental aspects, as terminalia bellirica plants have got many medicinal applications.

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