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## RESEARCH ARTICLE

# EXTRACTION AND CHARACTERIZATION OF HARDWICKIA BINATA FIBER

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**ABSTRACT**

In this work, the fibers were extracted from the plant Hardwickia Binata and investigated in detail. The effect of alkali treatment on the chemical composition, tensile properties, morphological and thermal degradation of Hardwickia Binata fibers was studied. Chemical analysis and FT-IR indicated lowering of amorphous hemicellulose content on alkali treatment. Wide-angle X-ray diffraction studies indicated increase in crystallinity of the fibers on alkali treatment. The tensile strength, modulus and thermal stability of the fiber increased on alkali treatment. Scanning electron micrographs revealed roughening of the surface of the fibers due to the removal of the hemicellulose layer on alkali treatment. Tensile properties of Hardwickia Binata fibers were compared to those of other important natural fibers, and it was indicated as an alternative suitable source for biocomposites.

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## INTRODUCTION

Synthetic fibers are widely used as reinforcement materials in polymer composites due to their high specific strength, light weight and durability. However, they have some drawbacks which include high cost, non degradable nature and high energy consumption, leading to problems such as environmental pollution, skin irritation and abrasion of processing equipment (Cavalieri and Padella, 2002; Wambua *et al*., 2003). Growing environmental concerns over the past few years have led to renascent interest in the use of natural plant fibers in most of the technical applications (Reddy and Yang, 2005). Advantages of using natural fibers in composites include low density, low cost, less skin irritation, less equipment abrasion, renewability and recyclability, as well as excellent mechanical properties such as high specific strength, specific modulus, and flexibility (Thakur *et al*., 2010; John and Thomas, 2008). Natural fibers such as bamboo, borassus, coir, flax, hemp, jute and tamarind have been investigated as reinforcements in polymer composites. However, the disadvantages of natural fibers like high moisture sensitivity, low chemical resistance, lower thermal degradation temperature, and incompatibility during the fabrication of composites (Singha *et al*., 2009). These effects have a substantial impact on the interfacial bond between the fibers and matrix. Therefore in order to improve their interfacial compatibility, physical or chemical treatment of fibers should be done (John and Anandjiwala, 2008). Knowledge of the physicochemical properties as well as mechanical behavior of natural fibers is essential in order to optimize the composites' performance. Most of the research has been carried out to study the influence of fiber treatment on chemical composition, surface morphology, crystallinity, and mechanical properties of fibers (John and Anandjiwala, 2008). The name Hardwickia Binata was named to a plant after Thomas Hardwickie by William Roxburgh (Roxburgh, William. 1819). Hardwickia is a monotypic genus of flowering plant in the subfamily Caesalpinioideae of the legumes. The only species is the Anjan, Hardwickia Binata, an Indian tree that grows some 25 to 30 m high (Hardwickia Binata information from NPGS/GRIN; Hardwickia Binata, 2013). Hardwickia Binata is a moderate-sized to large tree with drooping branches (Krishen, Pradip 2006). The bark of the tree is grayish-brown in color, rough with deep cracks and it darkens with age (Krishen, Pradip 2006). The compound leaves have only two leaflets which are joined at the base (Krishen, Pradip 2006). The tiny, white/greenish-yellow colored flowers are inconspicuous and

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are easily overlooked (Krishen, Pradip 2006). The fruits are short, flat pods about 6 cm long with a single seed attached at the end (Krishen, Pradip 2006). The timber obtained from the tree is the hardest and heaviest (among timbers from the trees found in India), is durable and termite resistant (Krishen, Pradip 2006; Saxena, 2010). The leaves are shed in April and the new leaves emerge in early May (Krishen, Pradip 2006). The flowering season is during August to September, the fruits appear after the flowering season and continue to remain till May (Krishen, Pradip 2006). The bark of the tree is used for making ropes (Saxena, 2010). The timber obtained from Hardwickia Binata is used for making agricultural equipment like cart wheels, oil mills, pestles and ploughs (Saxena, 2010; Reddy, 2007). The leaves, succulent stems and twigs serve as fodder for livestock (Singh Negi, Sharad 1996). The Hardwickia Binata bark is found to have a good sorption capacity for mercury and a modification of the bark is found to be useful for removal of most of the mercury from water under certain conditions (DESHKAR *et al*., 1990; Khare, 2008). Oleo-resin extracted from the heart wood is used in manufacture of varnishes (http://agritech.tnau.ac.in/forestry/ntfp_hardwickia_binata.
html). The leaves extracts of Hardwickia Binata showed a broad spectrum of activity against both gram-positive and gram-negative bacteria and fungi (Gunaselvi *et al*., 2010). Resin exuding from the heartwood is used for dressing the sores of elephants (Rao Sahib M Rama Rao.1914). The balsam, combined with cubebs and sandal, is used for treating sexually transmitted diseases like leucorrhoea, chronic cystitis and gonorrhea (Khare, 2008). The resin (not the Oleo-resin) derived from the tree is used as a diuretic (Khare, 2008).

Though there exists literature about the characterization of the composites prepared with Hardwickia Binata fiber and epoxy (Reddy *et al*., 2014; Kumar and Rao, 2015; Nowshoba *et al*., 2013), to the best of our knowledge, there are no past studies available on the characterization of these fibers, such as chemical composition, morphology, tensile and thermal properties to establish the effective utilization of the Hardwickia Binata fiber. The prime objective of this study was to explore the potential of Hardwickia Binata fibers as a green composite reinforcement. The work involved extraction of Hardwickia Binata fibers from its bark, alkali treatment, structural characterization using chemical analysis, FT-IR and other extensive studies. Studies on morphology, mechanical behavior and wide-angle X-ray diffraction (XRD) were also carried out to support our objective on the application of Hardwickia Binata fibers.

## MATERIALS AND METHODS

Extracted Hardwickia Binata 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

Hardwickia Binata trees are widely grown in the Nalamala forest, which is located in the Kurnool district, Andhra Pradesh, India. In this present work, barks are removed from the Hardwickia Binata tree, which is located in G Pulla Reddy Engineering College, Kurnool as shown in Figure 1(a). First, the barks were removed from the plant properly and immersed in water for around 4 months. Water and mechanical retting process was adopted for extraction of fibers from the bark. The separated fiber layers were washed thoroughly using water and then sun dried for one week to ensure maximum moisture removal as shown in Figure 1(b). Finally, the fibers were kept in a hot air oven for 24 h at 100°C to remove moisture.


**Fig.1. Photographs of (a) Hardwickia binata tree (b) Extracted Hardwickia binata fiber from its bark**

### Alkali Treatment

The extracted fibers were treated with 5% (w/v) sodium hydroxide solution for 30 minutes at room temperature, maintaining a liquor ratio of 25:1 to remove the hemicellulose

and surface impurities. Finally, the fibers were neutralized using 1% (w/v) acetic acid solution followed by water and then the fibers were dried at 100°C for 24 hours.

### Determination of Chemical Composition

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 by Wise *et al*. (Kommula *et al*., 2013). The hemicellulose 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 three samples were reported.

### Fourier Transform-Infrared Spectroscopy

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

### X-Ray Diffraction Analysis

Wide-angle X-ray diffractograms of untreated and alkali-treated Hardwickia Binata fibers were recorded on X'Pert³ Powder which is PANalytical's newest X-ray diffraction system at NIT Warangal, India. This X-ray diffraction system is based on the fully renewed X'Pert platform with its new on-board control electronics, compliance with the latest and most stringent X-ray and motion safety norms, advances in eco-friendliness and reliability. This X-ray diffraction system offers an affordable solution for high-throughput, high-quality phase identification and quantification, residual stress analysis, grazing incidence diffraction, X-ray reflectometry, small-angle X-ray scattering, pair distribution function analysis and non-ambient diffraction. The generator was operated at 45 kV and 30 mA, and the samples were scanned in the 2θ range of 5.996°–70°.

### Morphology

Morphology of the untreated and alkali-treated Hardwickia Binata fibers was examined using a scanning electron microscope. In this study, the samples were gold coated and their surface observed under an EDAX Ametek scanning electron microscope.

### Thermogravimetric Analysis

Thermograms of untreated and alkali-treated Hardwickia Binata fibers were recorded using a thermogravimetric analyzer (Perkin Elmer STA 6000). Samples of approximately 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 an Instron 3369 Universal testing machine at a crosshead speed of 5 mm/min, maintaining a gauge length of 100 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 DISCUSSION

The chemical composition of untreated and alkali-treated Hardwickia Binata fibers was determined, and the results are

**Table 1. Chemical compositions and tensile properties of Hardwickia Binata fibers**

| Parameter | Untreated Hardwickia binata fiber | Alkali treated Hardwickia binata fiber |
|---|---|---|
| **Chemical composition** | | |
| α - Cellulose (%) | 78.123 | 85.17 |
| Hemicelluloses (%) | 14.87 | 6.58 |
| Lignin (%) | 7.67 | 9.13 |
| **Tensile properties** | | |
| Strength (MPa) | 210 | 371 |
| Modulus (GPa) | 10.7 | 14.3 |
| Elongation at break (%) | 2.56 | 2.9 |

**Table 2. Comparison of Chemical Composition and Tensile properties of Hardwickia Binata 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 | (7) |
| Bamboo | 26-43 | 30 | 21-31 | 140-230 | 11-17 | - | (7) |
| Banana | 63-64 | 19 | 5 | 500 | 12 | 5.9 | (7) |
| Borassus | 53.4 | 29.6 | 17 | 70.8 | 10.8 | 34.8 | (21) |
| Coir | 32-43 | 0.15-0.25 | 40-45 | 175 | 4-6 | 30 | (7) |
| Flax | 71 | 18.6-20.6 | 2.2 | 345-1500 | 27.6 | 2.7-3.2 | (7) |
| Hemp | 68 | 15 | 10 | 690 | 70 | 1.6 | (7) |
| Jute | 61-71 | 14-20 | 12-13 | 393-773 | 26.5 | 1.5-1.8 | (7) |
| Kenaf | 72 | 20.3 | 9 | 930 | 53 | 1.6 | (7) |
| Napier | 45.66 | 33.67 | 20.60 | 75 | 6.8 | 2.8 | (22) |
| Oil Palm | 65 | - | 29 | 248 | 3.2 | 25 | (7) |
| Pineapple | 81 | - | 12.7 | 1.44 | 400-627 | 14.5 | (7) |
| Ramie | 68.6-76.2 | 13.16 | 0.6-0.7 | 560 | 24.5 | 2.5 | (7) |
| Sisal | 66-78 | 10-14 | 10-14 | 468-700 | 9.4-22 | 3-7 | (7) |
| Thespesia | 60.63 | 26.64 | 12.70 | 573 | 61.2 | 0.79 | (23) |
| Hardwickia Binata | 78.12 | 14.87 | 7.67 | 210 | 10.7 | 2.56 | This work |

summarized in Table 1. The obtained data show that the untreated fibers contained 78.123% α-cellulose, 14.87% hemicellulose and 7.67% lignin. After alkali treatment, the fibers contained 85.17% α-cellulose, 6.58% hemicellulose and 9.13% lignin. After alkali treatment the hemicellulose content of Hardwickia Binata fibers reduced from 14.87% to 6.58%, as hemicellulose is much more sensitive to the action of sodium hydroxide at room temperature than lignin or cellulose (Reddy *et al*., 2013). This indicates that after alkali treatment, the cellulose and lignin % contents increased.

A comparison of the chemical composition of Hardwickia Binata fibers with that of some important natural fibers and agricultural residues is presented in Table 2 (John and Anandjiwala, 2008; Reddy *et al*., 2013; Reddy *et al*., 2012; Reddy *et al*., 2014). From Table 2, it is evident that only pineapple has higher cellulose content than Hardwickia Binata fiber. The hemicelluloses content of Hardwickia Binata fiber is lower than that of abaca, bamboo, banana, borassus, flax, hemp, jute, kenaf, napier and thespesia fibers but higher than that of remaining other fibers. Finally, the lignin content is higher than that of banana, flax and raime fibers and lower than that of all other fibers. In conclusion, the Hardwickia Binata fibers had a high amount of cellulose and relatively low lignin content. These fibers could be viewed as a potential source of lignocellulosic fibers for fiber-reinforced composite materials and cellulose for the production of cellulose derivatives and production of paper pulp.

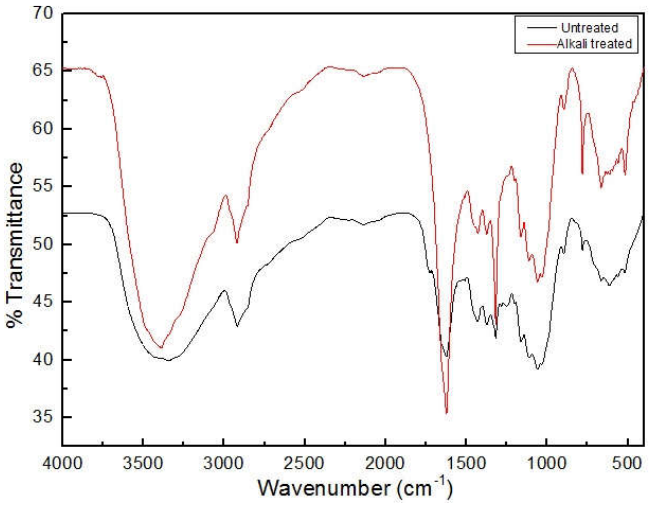


**Fig.2. FT-IR spectra of untreated and alkali-treated Hardwickia Binata fibers**

The effect of alkali treatment on the Hardwickia Binata fiber was studied using FT-IR. Figure 2 shows the comparison of IR spectra of the fibers before and after alkali treatment. From the untreated fiber spectrum, absorption bands at around 3388, 2981, and 2851 $cm^{-1}$ can be observed. These bands correspond to O–H stretching and asymmetric and symmetric stretching of methylene (–$CH_2$–) groups respectively (Reddy *et al*., 2009). The absorption band at 1623 $cm^{-1}$ corresponds to absorbed water molecules. For lignin, the absorption bands at 1618, 1435, 1375, and 1036 $cm^{-1}$ correspond to C=C stretching, –$CH_3$ asymmetric, –CH symmetric stretching, and aromatic –CH in plane deformation respectively (Sain and Panthapulakkal, 2006; Xiao *et al*., 2001). For hemicelluloses, the absorption bands at 1731 $cm^{-1}$ correspond to carbonyl (C=O) stretching (Reddy *et al*., 2014). For cellulose, the absorption bands at 1430, 1160, 1113, and 1059 $cm^{-1}$ correspond to –$CH_2$ scissoring, –OH bending, C–O antisymmetric bridge stretching, C–O–C, and C–O stretch vibrations respectively (Sun *et al*., 2005). The absorption band at 897 $cm^{-1}$ corresponds to b-glucosidic linkages between the sugar units in hemicellulose and cellulose (Sun *et al*., 2005). The spectrum of the alkali treated fiber is also nearly similar to that of the untreated fiber. However, the absorption peaks at 1731 $cm^{-1}$ disappeared, while for the absorption bands at 1618, 1435, 1375, and 1036 $cm^{-1}$ the intensity is lower after alkali treatment. Also, for cellulose bands no appreciable change is noticed. Thus, the FT-IR studies suggest a reduction of the hemicellulose content on alkali treatment of fibers. This strongly supports the chemical analysis data of the alkali-treated fibers as shown in Table 2.

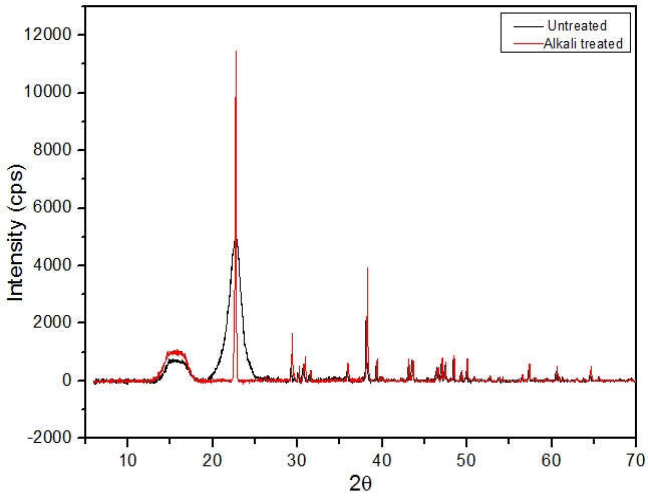


**Fig.3. X-ray diffractograms of untreated and alkali-treated Hardwickia Binata fibers**

Wide-angle X-ray diffraction studies of the untreated and alkali-treated hardwickia binata fibers were carried out to investigate the crystalline behavior of the fibers. The wide-angle X-ray diffractograms of untreated and alkali treated fibers are shown in Figure 3. The diffractograms of both types of fiber reveal two main reflections, corresponding to 2θ values of around 16° and 22° respectively. The low angle reflection (15.6°) is broad, whereas the high angle reflection (22.7°) is sharp and intense. These reflections are attributed to the amorphous ($I_{am}$) and crystalline ($I_{002}$) part in the fiber respectively. From the diffractograms, it is clear that the alkali treated fiber had higher crystallinity as evident by a relatively intense peak at 2θ = 22.7°. The crystallinity index (CI) was calculated using following equation.

$$CI = \frac{I_{002} - I_{am}}{I_{002}} \times 100$$

The calculated crystallinity indices of untreated and alkali-treated fibers were found to be 85.99 and 89.79, respectively. The crystallinity index of alkali-treated fibers was found to be slightly higher than that of the untreated fibers, due to the removal of hemicellulose. Thus, the above results demonstrate that hydrolysis took place preferentially in the amorphous region and the rearrangement of the crystalline regions in such a way that the fibers exhibited a more crystalline nature after alkali treatment (Reddy *et al*., 2009). This is in conformity with the results of chemical analysis and FT-IR.

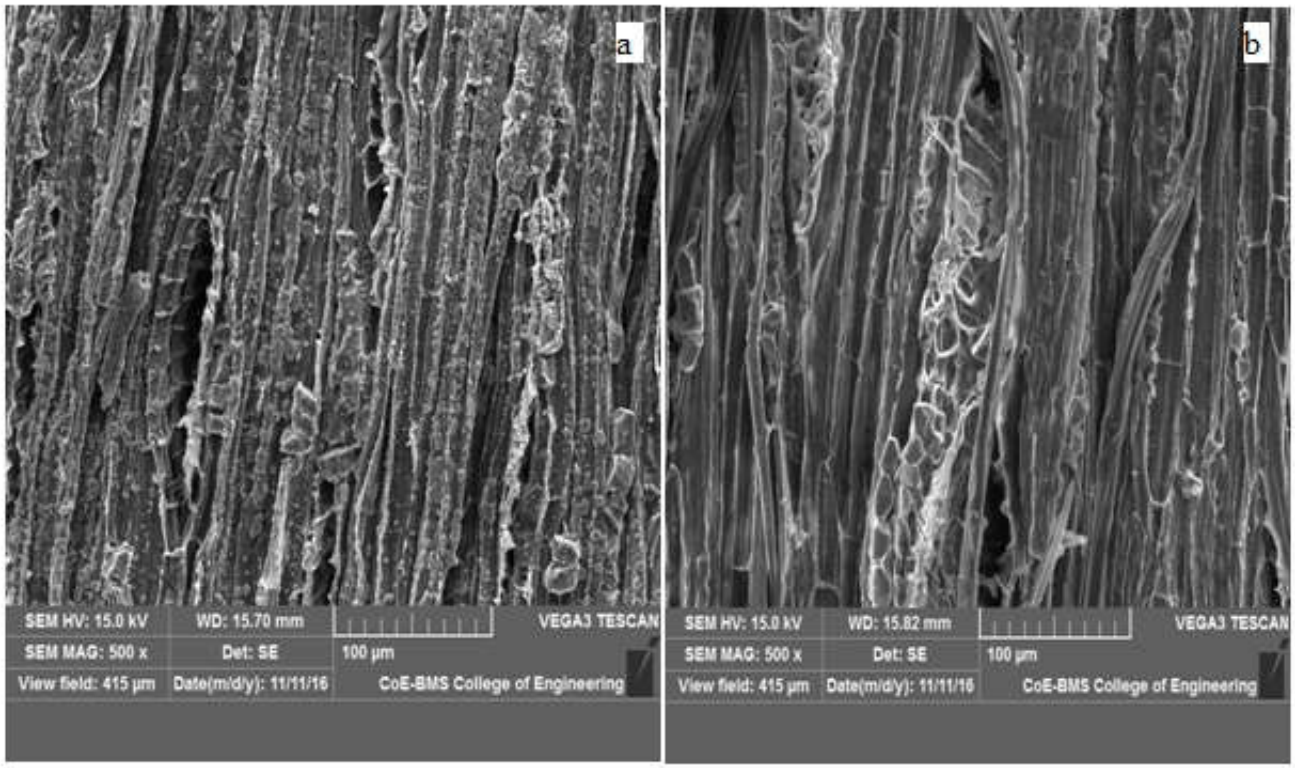


**Fig.4. Surface scanning electron micrographs of Hardwickia Binata fibers (a) untreated fiber (b) alkali-treated fiber**

Scanning electron microscopy (SEM) provides an excellent technique for examination of surface morphology of fibers. Scanning electron micrographs of untreated and alkali treated fibers are shown in figure 4. Figure 4(a) show the SEM micrograph of untreated fiber, from which it is clear that the fibers contained surface impurities such as wax and fatty substances. There were considerable differences in the fiber morphologies after alkali treatment. For alkali treated fibers as shown in figure 4(b), it can be observed that the fiber became cleaner with a rough surface, as the impurities were removed from the fiber surface. This roughened surface may improve interfacial bonding when the Hardwickia Binata fibers are used as absorption materials and filters and in polymeric composites.

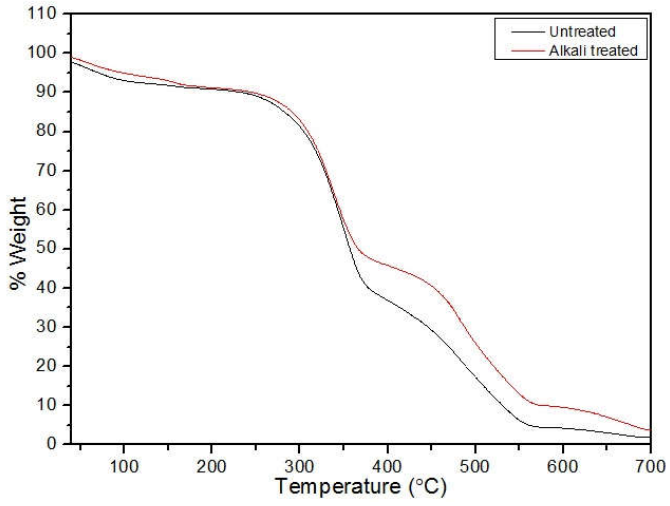


**Fig.5. Primary thermograms of untreated and alkali-treated Hardwickia binata fibers**

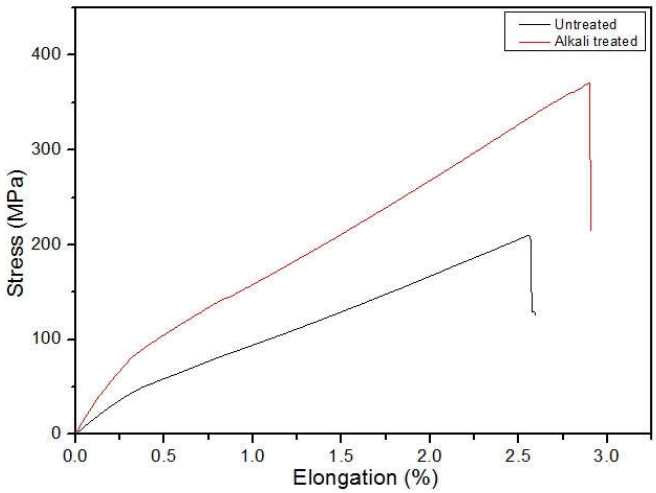


**Fig.6. Stress-strain curves of untreated and alkali-treated Hardwickia Binata fibers**

Thermogravimetric 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 100°C corresponding to loss of absorbed moisture in the fibers. The weight loss around this temperature was below 10%. The initial degradation stage occurred in the ranges of 230°–370°C and 240°–370°C for untreated fiber and alkali treated fiber, respectively, where the weight loss in these temperature ranges was found to be about 49% (untreated) and 41% (alkali treated). This is due to thermal depolymerization of hemicelluloses and a small fraction of lignin. Another

degradation stage occurs in the ranges of 370°–560°C and 370–575°C for untreated and alkali-treated fiber respectively, where the weight loss over these temperature ranges was about 37% (untreated) and 40% (alkali treated). This thermal degradation corresponds to the cleavage of glycoside bonds of the cellulose structure and depolymerization of lignin. This is indicated by increase of the temperatures of main degradation regions, indicating that thermal stability of alkali-treated fibers increased due to removal of hemicellulose during alkali treatment. The % char residues at 700°C of untreated fiber and alkali-treated fiber were found to be 1.71 and 3.50% respectively. Further, these results indicate that alkali-treated fibers can be used as reinforcement, even with thermoplastic polymers whose processing temperature is below 230°C.

The tensile properties (strength, Young's modulus, and elongation at break) of untreated and alkali-treated Hardwickia Binata fiber were determined and are listed in Table 1. From Figure 6, the stress-strain behavior of untreated and alkali-treated Hardwickia Binata fibers is fairly linear up to failure. From Table 1, it can be seen that the average tensile strength, modulus, and elongation at break of alkali treated fibers are higher than that of the untreated fibers. This change in tensile properties is attributed to the fibers tending to become closely packed owing to the removal of hemicellulose by alkali treatment and formation of new hydrogen bonds in between the chain of cellulose fibrils and stress transfer between interfibrillar regions (Reddy *et al*., 2013). Hence, alkali treated fibers had greater resistance against tensile loading and exhibited higher properties than the untreated fibers. The increase in tensile strength, modulus, and elongation at break of alkali-treated fiber over the untreated fiber was observed to be about 76.66, 33.64 and 13.28% respectively. The tensile properties of Hardwickia binata fibers were compared to those of other natural fibers and are presented in Table 2 (John and Anandjiwala, 2008; Reddy *et al*., 2013; Reddy *et al*., 2012; Reddy *et al*., 2014). From this table 2, it is evident that the tensile strength of Hardwickia Binata fiber is lower than that of abaca, banana, flax, hemp, jute, kenaf, oil palm, ramie, sisal and thespesia but higher than those of all other fibers. The tensile modulus of Hardwickia Binata fibers is significantly higher than that of napier, oil palm and sisal, while elongation at break is lower than that of abaca, banana, borassus, coir, flax, napier, oil palm, pineapple and sisal fibers. This comparison indicates the suitability of using Hardwickia Binata fiber in the fabrication of composites.

**Conclusion**

Fibers were successfully extracted from the barks of Hardwickia Binata tree by water retting process and then subjected to alkali treatment. The influence of alkali treatment on the chemical composition and morphology, thermal and tensile properties of the fiber was studied. Scanning electron microscopy showed the rough surface of the fibers after alkali treatment, due to the significant removal of surface impurities and hemicellulose from the fibers. Results were supported by chemical analysis and FT-IR studies. XRD and TGA measurements indicated an increase of crystallinity and 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 study indicated that alkali treatment of Hardwickia Binata fiber enhanced the crystallinity, thermal stability and tensile properties of the fiber. The results of the chemical composition and tensile characterization were found to be comparable to those of other common lignocellulosic fibers, and these fibers show some potential as reinforcement in polymer matrix composites. This may results in local development and increase in the environmental aspects of Hardwickia Binata plants. Further specific studies should be conducted to enhance the end products, such as paper pulp or raw material for cellulose derivatives as the cellulose content in hardwickia binata fibers is very high compared to that of other natural fibers.

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