# Extraction and characterization of novel ligno-cellulosic fibers from *Thespesia Lampas* plant

K. Obi Reddy <sup>a</sup>\*, B. Ashok <sup>b</sup>, K. Raja Narender Reddy <sup>c</sup>, Y.E. Feng <sup>d</sup>, Jun Zhang <sup>d</sup>, A. Varada Rajulu <sup>e</sup>

<sup>a</sup> Department of Mechanical Engineering Technology, Doornfontein Campus, P.O. Box 17011, University of Johannesburg, Johannesburg-2028, South Africa

<sup>b</sup> Department of Physics, Osmania University, Hyderabad, 500007, India

° Department of Mechanical Engineering, Kakatiya Institute of Technology & Science, Warangal, India

<sup>d</sup>CAS Key Lab of Engineering Plastics, Institute of Chemistry, Chinese Academy of Sciences (CAS), Beijing, 100190, China

<sup>e</sup> Department of Polymer Science & Technology, Sri Krishnadevaraya University, Anantapur-515003, India

\*Corresponding author: K. Obi Reddy

Department of Mechanical Engineering Technology, Doornfontein Campus, P.O. Box 17011, University of Johannesburg, Johannesburg-2028, South Africa Tel.: +27 84660 6269; E-mail address: obireddyk80@gmail.com & obik@uj.ac.za

## ABSTRACT

In this work, the ligno-cellulose fibers from the plant *Thespesia Lampas* were extracted and investigated in detail. The prime objective of this work was to study the effect of alkali treatment on the chemical composition, tensile properties, morphological and structural changes and thermal degradation of *Thespesia Lampas* fibers. Chemical analysis, FTIR and <sup>13</sup>CP-NMR spectroscopic studies indicated lowering of amorphous hemicellulose content on alkali treatment. X-ray diffraction studies indicated increase in crystallinity of the fibers on alkali treatment. The tensile strength and modulus of the fibers and thermal stability increased on alkali treatment. Scanning electron micrographs revealed roughening of the surface of the fibers due to the removal of hemicellulose layer on alkali treatment. Tensile properties of *Thespesia* fibers were compared to other important natural fibers, and it indicated as an alternative suitable source for composites making.

**KEYWORDS** – *Thespesia Lampas*, Chemical composition, Morphology, Crystallinity, Thermal stability and Tensile properties

## INTRODUCTION

This century has witnessed ever-increasing demands for the utilization of polymer plastics as important raw materials. In particular polymer plastic composites are gaining more and more acceptance in various industrial sector applications such as automotives, construction, marines, aerospace and sports [1]. 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 confines which include high cost, non-biodegradable nature and high energy consumption leading to problems such as environmental pollution, skin irritation, and causing abrasion in the processing equipment [2, 3]. With the increase of environmental consciousness, the use of natural fiber-based materials has proved to be an effective solution to reduce the production of synthetic fibers are splacement of synthetic fibers, improving the environmental performance of materials and products. In the recent years, natural fibers have drawn considerable attention in various applications, e.g. automobiles, furniture, packaging and construction [6, 7].

The use of natural fibers offers several advantages such as abundance, renewability, biodegradability, low cost, low density, appropriate specific strength and non-abrading the processing equipment [5, 8]. Further, they form an important source of income for agricultural societies. However, certain drawbacks of natural fibers such as degradation during processing, poor wettability, incompatibility with some polymeric matrices and high moisture absorption make them undesirable for composite applications [9]. Another problem encountered in their use is fiber–matrix adhesion arising due to the incompatibility between the hydrophilic natural fibers and the hydrophobic polymer matrix [10]. The improvement of the fiber-matrix compatibility has generated many researchers to carry out fiber surface modification by physical (corona discharge, plasma and UV bombardment) or chemical (alkali, grafting, acrylation, permanganate, acetylation, silane and peroxide) treatment methods [11, 12, 13]. Research on a cost-effective modification of natural fibers is necessary since the main attraction of today's fiber composites market is the competitive cost of natural fibers. Alkali treatment is an inexpensive and effective method to modify the fiber surface. Numerous researchers have reported improvement in mechanical properties of natural fibers on alkalization [14, 15]. The physical and chemical treatments lead to

reduction in moisture gain, changes in the fiber surface morphology and affect the fiber mechanical properties, as well as thermal stability.

*Thespesia lampas* (synonym: Hibiscus lampas Cav.) plant belongs to Malvaceae family commonly known as 'adavi benda (Telugu)'. It is found throughout India and in Eastern tropical Africa [16]. This plant is shown in Figure 1. The plant grows to a height of 2-3 meters and the stems contain very long and soft fibers. To the best of our knowledge, none of the past studies are available on the characterization such as chemical composition, structural, tensile and thermal properties of these fibers to establish the effective utilization of the Thespesia plant. The prime objective of this study was to explore the potential of Thespesia fibers as a green composite reinforcement. The work involved extraction of fibers from the stems, alkali treatment and their structural characterization using chemical analysis, FTIR, solid-state <sup>13</sup>C CP-NMR, and other extensive studies. Studies on morphology, mechanical behavior, and X-ray diffraction were also carried out to support our objective on the application of *Thespesia* fibers.

# MATERIALS AND METHODS

## Materials

Extracted Thespesia fibers, analytical grade of acetic acid, sodium hydroxide pellets and sulfuric acid (Merck Chemicals), sodium chlorite and sodium bisulfite (S. D. Fine Chemicals) were used.

#### **Fiber extraction**

Thespesia Lampas plants are widely grown at Mahabubabad forest in Warangal district in Andhra Pradesh., India. Geographical coordinates are 17° 35' 59" N, 79° 59' 57" E. Thespesia fibers were extracted from the stems of the plant. Initially the stems were cut from the plant and immersed in water for 24 h for debarking process and then bark was alienated from the stem manually. The combined with water and mechanical retting process was adopted for extraction of fibers from alienated bark. The separated fiber strands were washed thoroughly using water and then sun dried for one week to ensure maximum moisture removal. Finally, the fibers were then kept in a hot air oven for 24 h at 105 °C to remove moisture. Concerning fiber was extracted from bark producing a fiber yield of around 60%.

#### Alkali treatment

The extracted fibers were treated with 2 % (w/v) sodium hydroxide solution for 24 h 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 h. However, both the untreated and alkali treated fibers were stored in polyethylene bags and placed at 23 °C and 50% relative humidity (RH) for conditioning prior to further testing.

#### **Determination of Chemical Composition**

The chemical composition of untreated and alkali treated Thespesia fibers was determined by using common procedures [17, 18, 19], which are described below. The fiber (untreated and alkali treated) samples were chopped and sieved through a 250 mesh size screen and used for chemical analysis. The holocellulose ( $\alpha$ -cellulose + hemicellulose) content of the fiber was determined by gradual removal of lignin, which was achieved by treating the dewaxed fiber sample (3 g of fibers) with 0.7% sodium chlorite solution (pH 4-4.2 adjusted by acetic acid-sodium acetate buffer) at 70-80 °C for 2 h. After being filtered and extensively washed with 2% sodium bisulphite and distilled water, the residue was dried at 105 °C in an oven until constant weight. The  $\alpha$ -cellulose content was determined treating the holocellulose fiber sample (3 g of fibers) with a 35 mL of 17.5% NaOH solution and then a further 40 mL of 17.5% NaOH was added slowly in 10 min. The fiber slurry was left for 35 min and 75 mL of distilled water was added and stirred the solution mixture for few minutes. After being filtered and washed with 750 mL of distilled water, steeped for 10 min in 10% acetic acid solution and thoroughly washed with distilled water until neutralization and then dried at 105 °C in an oven until constant weight. The hemicellulose content was determined from the difference between holocellulose and  $\alpha$ -cellulose contents of the fiber. Lignin content was similarly determined by solubilising all the constituents other than lignin by treatment with 72% sulphuric acid. The fiber (2 g of dried) sample was treated with 72% sulphuric acid solution at a controlled temperature of 20 °C for 48 h, followed by dilution to 3% acid concentration and refluxed for 2 h. The insoluble residue was filtered through a Gooch crucible, washed acid free with water and dried in a hot air oven at 105 °C to constant weight. The % content of  $\alpha$ -cellulose, hemicellulose and lignin were determined and the average values of five samples reported.

#### **Microscopic analysis**

Optical microscopy observations of both untreated and alkali treated Thespesia single fibers were made using a Leica DMLP polarized optical microscope with 200 X magnification. Surface morphology of both untreated and alkali treated fibers was observed by an Oxford X-Max scanning electron microscope at 10 kV. Before examination, the fiber samples were sputter coated with a thin carbon layer in a vacuum chamber.

## Fourier Transform-Infrared Spectroscopy

Fourier transform-infrared spectroscopy studies on untreated and alkali treated Thespesia fibers were carried out using a Smart iTR ATR Nicolet iS 10 FT-IR spectrophotometer. All the spectra were recorded in the 4000–500 cm<sup>-1</sup> region with 32 scans in each case, at a resolution of 4 cm<sup>-1</sup>.

# NMR (CP-MAS) spectroscopy

<sup>13</sup>C CP-MAS NMR spectra of untreated and alkali treated Thespesia fibers were run on Bruker DSX 300 MHz solid-state NMR spectrometer. The operating frequency for <sup>13</sup>C nuclei was fixed at 75.46 MHz and the samples were spun with filled 5 mm rotor at a spinning rate of 5 kHz at room temperature.

#### **X-Ray Diffraction Analysis**

Wide-angle X-ray diffractograms of untreated and alkali treated Thespesia fibers were recorded on a Rigaku Ultima IV X-ray diffractometer. The system has a rotating anode generator with a copper target and a wide-angle powder goniometer. The generator was operated at 40 kV and 30 mA, and the samples were scanned in the 20 range of  $5-50^{\circ}$ .

## **Thermogravimetric Analysis**

Thermograms of untreated and alkali treated Thespesia fibers were recorded using a thermogravimetric analyzer (PerkinElmer TGA-7). Samples of approximately 8-10 mg were placed in appropriate platinum pans and heated from 20 to 600 °C at 10 °C min<sup>-1</sup>, under dynamic flow of nitrogen (100 mL min<sup>-1</sup>).

### **Tensile Testing**

The tensile properties were determined using an Instron 3369 Universal testing machine at a crosshead speed of 5mm/min maintaining a gauge length of 100 mm. Ten 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 along with their standard deviation.

## **RESULTS AND DISCUSSIONS**

The prime work involved in the present research was to extract fibers from the Thespesia plant stems, study their chemical composition, surface morphology, structural changes, crystallinity, thermal behavior and tensile properties before and after alkali treatment of the fibers. Figure 1 b and c show representative optical microscopic (OM) images of both untreated and alkali treated single fibers which feature nearly round shape along the fiber length. Fiber diameter was evaluated through optical observations as the average of four apparent diameter measurements taken at different locations along the fibers. The average diameters of untreated and alkali treated fibers were found to be 64 and 61  $\mu$ m respectively. After alkali treatment, fiber diameter was slightly reduced due to the removal of soluble components.

The chemical composition of untreated and alkali treated Thespesia fibers was determined, and the results are summarized in Table 1. The obtained data show that the untreated fibers contained 60.63 %  $\alpha$ -cellulose, 26.64 % hemicellulose and 12.70% lignin. After alkali treatment, the fibers had  $\alpha$ -cellulose, hemicellulose and lignin as 64.12%, 20.8% and 14.98% respectively. After alkali treatment the hemicellulose content of *Thespesia* fibers reduced from 26.64 to 20.8% as hemicellulose is much more sensitive to the action of sodium hydroxide at room temperature than lignin or cellulose [15]. This indicates that after alkali treatment, the cellulose and lignin %contents increased.

A comparison of chemical composition of Thespesia fibers with some important natural fibers and agricultural residues is presented in Table 2; and these data have been obtained from the literature [12, 15, 20]. From Table 2 it is evident that banana, flax, hemp, jute, kenaf, oil palm, pineapple, ramie and sisal have higher cellulose content whereas abaca, bamboo, borassus, coir and napier

fibers have lower cellulose content when compared to Thespesia fiber. The hemicellulose content of *Thespesia* fiber is lower than that of bamboo, borassus and napier fibers but higher than all other fibers. Finally, the lignin content is lower than that of bamboo, borassus, coir, napier, oil palm, ramie and sisal fibers, it is similar to jute and pineapple fibers while higher than those of all other fibers. In conclusion, the *Thespesia* fibers had high amount of cellulose and relatively low lignin content. These fibers could be viewed as a potential source of ligno-cellulosic fibers for fiberreinforced composite materials, cellulose for the production of cellulose derivatives and production of paper pulp.

Scanning electron microscopy provides an excellent technique for examination of surface morphology of fibers. Scanning electron micrographs of untreated and alkali treated fibers are shown in Figure 2. Fig. 2 a and b show the SEM micrographs of untreated fiber from which, it is clear that the fibers contained surface impurities such as wax and fatty substances and the internal fibrils also cannot be seen (Fig 2 a). At a higher magnification in Fig. 2 b, clear smooth surfaces with lots of impurity substances and the fibrils bound together by hemicellulose can be seen. There were considerable differences in the fiber morphologies after alkali treatment compared to untreated ones. For alkali treated fibers (Figs. 2 c and d), it can be observed that the fiber had become cleaner with rough surface as the impurities were removed from the fiber surface (Fig 2c). At a higher magnification in Fig. 2(d), the fibrillation was found to occur as the binding materials were removed in the treated fibers. This roughened surface may improve the interfacial bonding when they obtained Thespesia fibers are used as absorption materials, filters and in polymeric composites.

Untreated and alkali treated Thespesia fibers were analyzed using FTIR to evaluate the various chemical constituents present in them. Figure 3 shows the FTIR spectra of the untreated and alkali treated fibers. The broad intense absorption band at 3421 cm<sup>-1</sup> arose due to O-H stretching vibrations of hydrogen bonded hydroxyl (OH) groups, present in their main components [21]. The absorption bands at 2920 and 2852 cm<sup>-1</sup> correspond to the asymmetric and symmetric stretching of methylene (–CH<sub>2</sub>–) units of main components [22]. The prominent absorption band at 1734 cm<sup>-1</sup> 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 [22]. This band intensity

considerably decreased in the alkali treated fiber due to the partial removal of the hemicellulose component. The very low intensity absorption bands in 1600-1500 cm<sup>-1</sup> region correspond to the aromatic ring C=C stretching vibrations, while the absorption band at 1458 cm<sup>-1</sup> corresponds to the C-H deformation in methyl, methylene and methoxyl groups of lignin respectively [21, 22]. The bands at 1424 and 1320 cm<sup>-1</sup> correspond to the -CH<sub>2</sub> scissoring and -OH bending vibration, while the sharp intense absorption band at 1367 cm<sup>-1</sup> corresponds to the C-H asymmetric deformation of cellulose [22]. The intensity of the band at 1235 cm<sup>-1</sup> corresponding to the -COO vibration of acetyl groups in hemicellulose was sharply weakened after alkali treatment because of the partial removal of the hemicellulose component [21]. The absorption band at 1155 cm<sup>-1</sup> corresponds to C-O antisymmetric bridge stretching of cellulose. The absorption bands at 1110 and 1030 cm<sup>-1</sup> correspond to C-O-C pyranose ring skeletal vibration of cellulose [23]. The absorption band at 899 cm<sup>-1</sup> corresponds to  $\beta$ -glucosidic linkages between the sugar units in hemicellulose and cellulose [22]. However, the intensity of the absorption bands, at 1600-1500, 1458, 1424, 1367, 1320, 1155, 1110, 1030 and 899 cm<sup>-1</sup>, slightly bands increased for the alkali treated fiber, due to the increase of lignin content. The FTIR analysis also confirmed the existence of chemical components (cellulose, hemicellulose and lignin) of Thespesia fibers and further suggests the reduction of the hemicellulose content on alkali treatment of the fibers. This is in support of the chemical analysis data of the alkali treated fibers as shown in Table 1.

The <sup>13</sup>C CP-NMR spectra of untreated and alkali treated Thespesia fibers are shown in Figure 4. The spectrum of untreated fiber shows mainly the corresponding signals for the cellulose, hemicellulose and lignin. The chemical structures of the components are shown in the same figure (Figure 4). The spectrum of untreated fibers shows a narrow peak at 21.8 ppm and a broad peak at 173.3 ppm with moderate intensity. These peaks were contributed by the methyl and carboxylic carbons of acetyl groups in hemicellulose [21, 22]. A small intense peak at 56.6 ppm was attributed to methoxyl (-OCH3) carbon. Another low intense and broad peak in the region 128.8–143.2 ppm, was mainly from aromatic ring carbons (ortho/meta) of lignin. Another inadequately resolved broad signal at 145.5–155.9 ppm was due to the aromatic rings adjacent to alkoxy/phenol functionalities of lignin [21, 24, 25]. Along with the other important peaks seen in the spectra, there is a sharp and intense signal observed in between 60-110 ppm [24, 26]. The sharp and intense peak observed at 104.60 ppm was contributed by the C1 carbons of cellulose. The sharp high

intense peaks at 72.4-75.2 ppm correspond to the C-2, C-3, and C-5 carbons of cellulose. The small intense peak at 89.4 was attributed to C-4 carbon of cellulose, while the peak at 84.4 was due to the disordered C-4 carbon of cellulose. The moderate intense peak at 65.5 ppm was due to the C-6 carbon of cellulose. Among the signals noticed for cellulose carbons, those appeared at 72.4–75.2 ppm are of highest in intensity. The chemical shift values and intensity pattern suggest that these signals were also contributed by hemicellulose fraction [22]. The observation of these characteristics peaks of the spectra was supportive of presence of cellulose, hemicellulose, and lignin in the untreated fibers.

The <sup>13</sup>C CP-MAS NMR spectrum of alkali treated fibers is also shown in Figure 4. The remarkable features of the spectrum include reduction in the intensity of carbonyl peak and deshielding of its chemical shift. The methyl and carboxylic carbons of acetyl groups in hemicellulose component observed in the raw fibers now disappeared and accordingly no signals were noticed at 21.8 and 173.3 ppm. This was also followed by no changes of lignin peaks at 56.6 ppm, 128.8–143.2 ppm and 145.5–155.9 ppm. While the other important peaks, i.e. those appearing in the region 60–110 ppm almost remained the same. The spectral features indicate that the alkali treatment affected only the hemicellulose component leaving the other two components viz.  $\alpha$ -cellulose and lignin unaffected. These observations were very much in consistent with the chemical analysis FTIR spectral data.

XRD studies of the untreated and alkali treated Thespesia fibers were carried to investigate the crystalline behavior of the fibers. The wide-angle X-ray diffractograms of untreated and alkali treated fibers are shown in Figure 5. The diffractograms of both the fibers reveal two main reflections, corresponding to  $2\theta$  values of around  $16^{\circ}$  and  $22^{\circ}$  respectively. The low angle reflection (15.7°) is broad whereas the high angle reflection (22.3°) is sharp and intense. These reflections were attributed to the amorphous (I<sub>am</sub>) and crystalline (I<sub>002</sub>) part in the fiber respectively. From the diffractograms, it is clear that the *Thespesia* fibers showed a polymorphous and the alkali treated fiber had higher crystallinity as evident by a relatively intense peak at  $2\theta = 22.6^{\circ}$ . The crystallinity index (CI) was calculated using following equation.

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

The calculated crystallinity index of untreated and alkali treated fibers were found to be 48 and 52 respectively. The crystallinity index of alkali treated fibers was found to be slightly higher than 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 [21]. This is in conformity with the results of chemical analyses, as well as FT-IR and NMR studies.

TGA is a helpful technique for the study of the decomposition of chemical components (like cellulose, hemicellulose and lignin)/thermal stability of materials. The thermal stability of the fiber is a very important parameter for the processing and usage of these materials. A number of research reports have focused on the study of thermal stability of natural fiber through TGA. The primary thermograms of untreated and alkali treated fibers are shown in Figure 6 in which both fibers exhibited a similar trend of decomposition and via in three stages of weight losses. 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 3%. The initial degradation stage occurred in the ranges of 230–360 °C and 270 – 360 °C for untreated fiber and alkali treated fiber respectively, while the weight loss in these temperature ranges was found to be about 32%. This is due to thermal depolymerization of hemicelluloses and a little fraction of lignin [27]. Another degradation stage occurs in the ranges of 360–410 °C and 360–425 °C for untreated and alkali treated fiber respectively, while the weight loss over these temperature ranges was about 70% (untreated) and 77% (alkali treated). This thermal degradation corresponds to the cleavage of glycoside bonds of cellulose structure and depolymerization of lignin [27]. This is indicated by increase of the temperatures of main degradation regions and it indicates that thermal stability of alkali treated fibers increased due to removal of hemicellulose during on alkali treatment. The % char residue at 600 °C of untreated fiber and alkali treated fiber were found to be 23.1 and 17.9% respectively. Further, these results indicate that alkali treated fibers can be used as a 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 Thespesia fiber was determined and are listed in Table 1. The stress-strain behavior of untreated and alkali treated *Thespesia* fibers (Figure 7) 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 was higher than the untreated fibers. This amend in tensile properties is attributed to the fibers tending to get 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 [15]. Hence, alkali treated fibers had greater resistance against tensile loading and exhibited higher properties compared to 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 26.5, 35.2 and 24.7% respectively. The tensile properties of Thespesia fibers were compared to other natural fibers and are presented in Table 2; these data have been obtained from the literature [12, 15, 20]. From this table, it is evident that the tensile strength of Thespesia fiber is lower than that of flax, hemp, jute, kenaf and sisal but higher than those of all other fibers. The tensile modulus of Thespesia fibers is except lower than that of hemp and pineapple and higher than those of all other fibers, while elongation at break is lower than those of all other fibers. This comparison indicates the suitability of using Thespesia fiber in the manufacture of composites.

#### CONCLUSIONS

Ligno-cellulosic fibers were successfully extracted from Thespesia lampas plant by the water retting process and then subjected to alkali treatment. The influence of alkali treatment on the chemical composition, morphology, structural, thermal and tensile properties of the fiber was studied. Scanning electron microscopy showed that the surface morphology of the fibers became rough after alkali treatment, due to the significant removal of surface impurities and hemicellulose on the fibers. Results were supported by chemical analysis, FTIR and <sup>13</sup>C CP-MAS NMR studies. The 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 on Thespesia 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 ligno-cellulosic fibers and hence these fibers show some potential as reinforcement in polymer matrix composites. This will also indirectly help local development and increase the environmental aspect of *Thespesia* plant. Further specific studies, however, should be conducted to enhance the end-products such as paper pulp or raw material for cellulose derivatives.

## ACKNOWLEDGMENTS

KOR sincerely acknowledges the Faculty of Engineering and Built Environment, University of Johannesburg, South Africa, for the award of Postdoctoral Research Fellowship. AVR thanks the Council of Scientific and Industrial Research (CSIR) of India for the award of an Emeritus Scientist Scheme [21(0842)/11/EMR-II dt: 10-05-2011].

## References

- Friedrich, K., Z. Lu, and A. M. Hager. 1995. Recent advances in polymer composites' tribology. *Wear* 190 (2): 139–144.
- 2. Cavalieri, F., and F. Padella. 2002. Development of composite materials by mechanochemical treatment of post-consumer plastic waste. *Waste Manag.* 22(8): 913–916.
- 3. Wambua, P., J. Ivens, and I. Verpoest. 2003. Natural fibers: can they replace glass in fiber reinforced plastics?. *Compos. Sci. Tech.* 63(9): 1259–1264.
- 4. Thakur, V. K., A. S. Singha, and I. K. Mehta. 2010. Renewable resource-based green polymer composites: Analysis and characterization. *Int. J. Polym. Anal. Charact.* 15(3): 137–146.
- Soroudi, A., and I. Jakubowicz. 2013. Recycling of bioplastics, their blends and biocomposites: A review. *Europe. Polym. J.* 49(10): 2839-2858.
- 6. Holbery, J., and D. Houston. 2006. Natural fiber reinforced polymer composites in automotive applications. *J. Miner. Meta. Mater. Soc.* 58(11): 80–86.
- Cheung, H.Y., M.P. Ho, K.T. Lau, F. Cardon, and D. Hui. 2009. Natural fiber-reinforced composites for bioengineering and environmental engineering applications. Compos. *Part B: Eng.* 40(7): 655–663.

- 8. Reddy, N., and Y. Yang. 2005. Biofibers from agricultural byproducts for industrial applications. *Trends in Biotech*. 23(1): 22–27.
- Athijayamani, A., M. Thiruchitrambalam, U. Natarajan, and B. Pazhanivel. 2009. Effect of moisture absorption on the mechanical properties of randomly oriented natural fibers/polyester hybrid composite. *Mater. Sci. Eng. A.* 517(1-2): 344–353.
- Singha, A. S., and A. K. Rana. 2012. Improvement of interfacial adhesion in cannabis indica/ unsaturated polyester biocomposites through esterification reaction. *Int. J. Polym. Anal. Charact.* 17(8): 590–599.
- Bozaci, E., K. Sever, M. Sarikanat, Y. Seki, A. Demir, E. Ozdogan, and I. Tavman. 2013. Effects of the atmospheric plasma treatments on surface and mechanical properties of flax fiber and adhesion between fiber-matrix for composite materials. *Compos. Part B: Eng.* 45 (1): 565-572.
- 12. John, M. J., and R. D. Anandjiwala. 2008. Recent developments in chemical modification and characterization of natural fiber-reinforced composites. *Polym. Compos.* 29(2):187-207.
- Thakur, V. K., I. Kaur, R. P. Nagarajarao, and L. Yang. 2010. Silane functionalization of Saccaharum cilliare fibers: Thermal, morphological, and physicochemical study. *J. Polym. Anal. Charact.* 15(7): 397–414.
- 14. Das, M., and D. Chakraborty. 2008. Evaluation of improvement of physical and mechanical properties of bamboo fibers due to alkali treatment. *J. Appl. Polym. Sci.* 107(1): 522–527.
- Reddy, K. O., C. U. Maheswari, M. Shukla, J. I. Song, and A. V. Rajulu. 2013. Tensile and structural characterization of alkali treated Borassus fruit fine fibers. *Compos. Part B: Eng.* 44(1): 433–438.
- Chumbhale, D.S., and C. D. Upasani. 2012. Pharmacognostic standardization stems of Thespesia lampas (Cav.) Dalz & Gibs. *Asian Pacific Journal of Tropical Biomedicine* 2(5): 357–363.
- 17. Chattopadhyay, H., and P. B. Sarkar. 1946. A new method for the estimation of cellulose. *Proceedings of the National Institute of Sciences of India*. 12(1): 23–46.
- Sarkar, P. B, A. K. Mazumdar, and K. B. Pal. 1948. The hemicelluloses of jute fibre. J. Text. Instit. Transact.39(2): 44–58.
- Macmillan, W. G., A. B. Sengupta, and A. Roy. 1952. Observations on the determination of lignin in jute. *J. Text. Instit. Transact.* 43(3): 103–107.

- Reddy, K. O., C. U. Maheswari, M. Shukla, and A. V. Rajulu. 2012. Chemical composition and structural characterization of Napier grass fibers. *Mater. Lette.* 67(1): 35–38.
- 21. Reddy, K. O., B. R. Guduri, and A. V. Rajulu. 2009. Structural characterization and tensile properties of borassus fruit fibers. *J. Appl. Polym. Sci.* 114(1): 603–611.
- Maheswari, C. U., K. O. Reddy, E. Muzenda, B. R. Guduri, and A. V. Rajulu. 2012. Extraction and characterization of cellulose microfibrils from agricultural residue-Cocos nucifera L. *Bioma. Bioener.* 46(12): 555–563.
- Pappas, C., P. A. Tarantilis, I. Daliani, T. Mavromustakos, and M. Polissiou. 2002. Comparison of classical and ultrasound-assisted isolation procedures of cellulose kenaf from (Hibiscus cannabinus L.) and eucalyptus (Eucalyptus rodustrus Sm.). *Ultrason. Sonochem.* 9(1): 19–23.
- Sievers, C., T. Marzialetti, T. J. C. Hoskins, M. B. V. Olarte, P. K. Agrawal, and C. W. Jones. 2009. Quantitative solid state NMR analysis of residues from acid hydrolysis of loblolly pine wood. *Biores. Technol.* 100(20): 4758–4765.
- Nadji, H., P. N. Diouf, A. Benaboura, Y. Bedard, B. Riedl, and T. Stevanovic. 2009. Comparative study of lignins isolated from alfa grass (Stipa tenacissima L.). *Biores. Technol.* 100(14): 3585–3592.
- 26. Kono, H., S. Yunoki, T. Shikano, M. Fujiwara, T. Erata, and M. Takai. 2002. CP/MAS 13C NMR study of cellulose and cellulose derivatives. 1. Complete assignment of the CP/MAS 13C NMR spectrum of the native cellulose. *J. Americ. Chem. Soc.* 124(25): 7506–7511.
- Kommula, V. P., K. O. Reddy, M. Shukla, T. Marwala, and A. V. Rajulu. 2013. Physicochemical, tensile, and thermal characterization of Napier grass (Native African) fiber strands. *J. Polym. Anal. Charact.* 18 (4): 303–314.



**Figure 1.** Photograph of (a) Thespesia Lampas plant, as well as optical micrographs of (b) untreated fiber and (c) alkali treated fiber



Figure 2. Surface scanning electron micrographs of Thespesia fibers (a-b) untreated fiber and (b-c) alkali treated fiber



Figure 3. FTIR spectra of untreated and alkali treated Thespesia fibers



Figure 4. <sup>13</sup>C CP-NMR spectra of untreated and alkali treated Thespesia fibers



Figure 5. X-ray diffractograms of untreated and alkali treated Thespesia fibers



Figure 6. Primary thermograms of untreated and alkali treated Thespesia fibers



Figure 7. Stress-strain curves of untreated and alkali treated Thespesia fibers

Table 1. Chemical comp	positions and tensile	properties of Thes	pesia fibers
------------------------	-----------------------	--------------------	--------------

Parameter	Untreated [SD]	Alkali treated [SD]	
Chemical composition			
α-Cellulose (%)	60.63 [0.24]	64.12 [0.75]	
Hemicellulose (%)	26.64 [0.23]	20.8 [0.91]	
Lignin (%)	12.70 [0.47]	14.98 [0.26]	
Tensile properties			
Strength (MPa)	573 [15]	780 [50]	
Modulus (GPa)	61.2 [12]	94.5 [4.7]	
Elongation at break (%)	0.79 [0.11]	1.05 [0.08]	

S.D: Standard deviation

Fiber	Cellulose	Hemicellulose	Lignin	Tensile strength	Young's modulus	Elongation	Reference
	(wt%)	(wt%)	(wt%)	(MPa)	(GPa)	at break (%)	
Abaca	56–63	20-25	7–9	400	12	3–10	12
Bamboo	26–43	30	21–31	140–230	11–17		12
Banana	63–64	19	5	500	12	5.9	12
Borassus	53.4	29.6	17	70.8	10.8	34.8	15
Coir	32–43	0.15-0.25	40–45	175	4–6	30	12
Flax	71	18.6–20.6	2.2	345-1500	27.6	2.7–3.2	12
Hemp	68	15	10	690	70	1.6	12
Jute	61–71	14–20	12–13	393–773	26.5	1.5–1.8	12
Kenaf	72	20.3	9	930	53	1.6	12
Napier	45.66	33.67	20.60	75	6.8	2.8	20
Oil palm	65		29	248	3.2	25	12
Pineapple	81	_	12.7	1.44	400–627	14.5	12
Ramie	68.6-76.2	13-16	0.6-0.7	560	24.5	2.5	12
Sisal	66–78	10-14	10–14	468–700	9.4–22	3–7	12
Thespesia	60.63	26.64	12.70	573	61.2	0.79	This work

Table 2. Comparison of chemical composition and tensile properties of Thespesia fiber with other different natural fibers