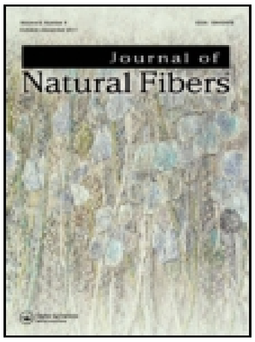

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Extraction and Characterization of Cellulose from Pretreated Ficus (Peepal Tree) Leaf Fibers

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In this work, we carried the physicochemical characterization of the cellulose extracted from the forestry residue of ficus leaves using chemical method. Detailed chemical composition of the ficus leaf fibers (both untreated and pretreated) and extracted cellulose was carried out. The chemical analysis confirmed the sequential removal of the lignin and hemicellulose components from the ficus leaves fibers. The morphology of the ficus leaf fibers and extracted cellulose was investigated using scanning electron microscopy. Structural analysis was carried out by Fourier transform infrared and nuclear magnetic resonance spectroscopy. The studies indicated that on chemical treatments the lignin and hemicellulose were removed extensively from the ficus leaf fibers. X-ray diffraction studies reveal that extracted cellulose was more crystalline than the ficus leaf fibers. The thermal stability of ficus leaf fibers and extracted cellulose was investigated by thermogravimetric analysis and the extracted cellulose had good thermal stability.

Keywords: Peepal tree leaves, chemical composition, morphology, spectroscopy, crystallinity, thermal degradation

从预处理的菩提树叶纤维提取纤维素及特征在这项工作中，我们采用化学方法对菩提树叶残余物进行提取纤维素，得到其物理化学表征。对（未处理和预处理）菩提树叶纤维进行了提取的纤维素，给出详细化学成分。化学分析证实，从菩提树的叶纤维顺序去除了木质素和半纤维素成分。利用扫描电子显微镜对菩提树叶中提取的纤维和纤维素的形态进行了研究。采用傅里叶变换红外和核磁共振波谱法进行结构分析。研究表明，化学处理菩提树叶，其的木质素和半纤维素被广泛的去除。X射线衍射分析表明，提取的纤维素比菩提树叶纤维多晶。通过热重分析，菩提树叶中提取纤维素纤维有很好的热稳定性。

关键词：菩提树叶，化学成分，形态学，光谱，结晶度，热降解

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INTRODUCTION

Cellulose is the most abundant biopolymer on the biosphere and is recognized as the major component of biomass (wood, agricultural plants, and residues), bacteria, algae, and tunicin (Angles and Dufresne 2000; Hu and Catchmark 2010; Mihranyan 2011; Reddy et al. 2014). It is the fibrillar component of biomass cell walls, and essentially a linear condensation polymer consisting of D-anhydro glucopyranose units joined together by $\beta(1\rightarrow4)$-glycosidic bonds. Cellulose continues to attract considerable attention for more than 150 years as a popular biomass resource material. It continues to remain an indispensable raw material for paper, food, textile, pharmaceutical, energy, and composite industries (El-Sherbiny et al. 2009; Shosha et al. 2002; Samir et al. 2005; Samaneh et al. 2013). The major source of cellulose is wood and its consumption is continuously increasing worldwide and even in countries where wood resources are limited. The rational and innovative utilization of agricultural residues or nonwood (plant fibers) biomass as new sources of cellulose is of potential interest worldwide.

Agricultural crops/forestry residue and plant fibers offer several environmental benefits owing to their renewable nature, abundance, low cost, low energy consumption in production, etc (Xiao et al. 2001). Cellulose is the main component of several plant fibers and agricultural crops residue such as bamboo, cotton, flax, hemp, jute, sisal, banana, and straws (rice and wheat). Generally, the natural fibers mainly consist of cellulose, hemicellulose, and lignin and then cellulose is separated from the noncellulosic substances using mechanical and chemical methods (Sun and Tomkinson 2005). Many studies were conducted on the extraction of cellulose from biomass for producing pulp, paper, and reinforced plastics (Ashori 2006; Belgacem and Gandini 2005; Cordeiro et al. 2004; Thakur et al. 2010). The present research work is aimed at the extraction of cellulose from ficus leaves by using the chemical approach. Ficus tree leaves are of forestry residues (nonwood) in the tropical region.

The ficus tree (*Ficus religiosa)* is a native of India, Sri Lanka, Nepal, China, and Vietnam. An enormous volume of ficus leaves containing cellulosic fibers are generated annually as natural waste and can form an alternative raw material for cellulose extraction. An enormous volume of the ficus leaves are generated annually as natural waste burnt in the forestry and the pollution caused by burning residues has been a serious environmental problem. The preliminary studies indicated the leaves to contain about 39% of cellulose (Reddy et al. 2010). As we consider, these residues could and should find a more rational way of exploitation, namely, as a source of cellulosic fibers. In this context, we have chosen ficus leaves for this purpose. The main aim of this work is to extract cellulose from ficus leaves and to characterize the resultant fibers and cellulose. Chemical composition of the leaf fibers (both untreated and pretreated) and extracted cellulose was determined by chemical analysis and surface morphology was investigated by scanning electron microscopy. The chemical structure of leaf fiber and extracted cellulose was ascertained by Fourier transform infrared (FTIR) and ^{13}C CP-MAS NMR spectroscopy. Crystallinity and thermal stability was determined using wide-angle X-ray diffraction and thermogravimetric analysis (TGA), respectively.

MATERIALS AND METHODS

Materials

Extracted ficus leaf fibers, benzene, sodium hydroxide pellets, sodium chlorite, acetic acid and sodium bisulphate, and ethanol (Merck Chemicals) were used.

Fiber Extraction

Ficus fibers were extracted from fallen leaves. At first, the leaves were dipped in water for three weeks and then the greenish layer at the leaf top and bottom was removed manually using a smooth brush. The separated fibers were thoroughly washed with tap water, followed by distilled water and sun dried for one week. Then the fibers were kept in a hot air oven for 24 h at 105 °C to remove the moisture. The yield of fiber from each leaf is around 35%.

Pretreatment

Ficus leaf fibers were treated with 5% sodium hydroxide (NaOH) solution at 30 °C maintaining a liquor ratio of 30:1. The fibers were kept immersed in the alkali solution for 30 min to remove the hemicellulose and other greasy materials. The fibers were then washed several times with water to remove all traces of NaOH from the fiber surface, neutralized with dilute acetic acid and finally washed again with distilled water. The washed fibers were dried at room temperature for 72 h followed by hot air oven drying at 105 °C for 24 h.

Cellulose Extraction

Pretreated fibers were chopped, sieved to 250 mesh size and dried at 105 °C for 24 h. Then the sieved fibers were dewaxed by refluxing with toluene–ethanol (2:1, v/v) for 6 h in a Soxhlet apparatus. The dewaxed fibers were then delignified with 0.7% sodium chlorite at 100 °C for 2 h in acidic solution (pH 4–4.2 adjusted by buffer solution) using a fiber/liquor ratio of 1:50. After being filtered and extensively washed with 2% sodium bisulphite, distilled water, and ethanol, the residue was dried at 105 °C in an oven until constant weight. The crude holocellulose was treated with 17.5% (w/v) sodium hydroxide solution at 20 °C for 45 min, treatment after fibers were taken out and washed with 8.3% NaOH solution. After this, the residue was washed with 10% acetic acid, tap water, and subsequently with distilled water and then 95% ethanol to neutralize the reaction. In this step, rich content of cellulose were separated by eliminating hemicelluloses from delignified fibers. Finally, the purified cellulose was dried at 105 °C in an oven until constant weight.

Chemical Analysis

The chemical composition of untreated and pretreated ficus leaf fibers and extracted cellulose was determined using the standard TAPPI (Technical Association of the Pulp and Paper Associations) methods for different components, namely: T 204 cm-07 for extractives, T 203 cm-99 for α-cellulose, T 222 om-06 for lignin, and T 211 om-07 for ash content. The holocellulose was determined according to the method described by Wise et al (1946). The hemicellulose fraction was calculated as the difference between the holocellulose and α-cellulose content. The percent contents of α-cellulose, hemicellulose, and lignin were determined, and based on five samples the average and standard deviation values reported.

Scanning Electron Microscopy

Scanning electron microscope (Oxford X-Max) was used to study the morphology of ficus leaf (both untreated and pretreated) fibers and extracted cellulose. The samples were carbon coated prior to recording the micrographs. The acceleration voltage was set at 10 kV.

Fourier Transform-Infrared Spectroscopy

Fourier transform-infrared spectroscopy studies of ficus leaf (both untreated and pretreated) fibers and extracted cellulose were carried out using a Smart iTR ATR Nicolet iS 10 FT-IR spectrophotometer. All the spectra were recorded in the 4000–500 cm^{-1} region with 32 scans in each case, at a resolution of 4 cm^{-1}.

^{13}C NMR (CP-MAS) Spectroscopy

^{13}C CP-MAS NMR spectra of ficus leaf (both untreated and pretreated) fibers and extracted cellulose was run on Bruker DSX 300 MHz solid-state NMR spectrometer (Indian Institute of Science, Bangalore, India). The operating frequency for ^{13}C nuclei was fixed at 75.46 MHz and the samples filled in 5 mm rotor were spun at a spinning rate of 5 kHz at room temperature.

X-Ray Diffraction Analysis

Wide-angle X-ray diffractograms of ficus leaf (both untreated and pretreated) fibers, and extracted cellulose 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 2θ range of 5°–50°.

Thermogravimetric Analysis

The thermal stability of ficus leaf (both untreated and pretreated) fibers and extracted cellulose was established using a thermogravimetric analyzer (Perkin Elmer TGA-7). The amount of sample for each measurement was about 10 mg. All the measurements were performed under a nitrogen atmosphere with a gas flow of 100 mL/min and heated from 50 to 600 °C at a rate of 10 °C/min.

RESULTS AND DISCUSSION

The prime work involved in the present research was an extraction of fiber from ficus tree leaves and fibers pretreatment, as well as extraction of cellulose from the pretreated fibers and their characterization. The chemical composition of the leaf fibers (both raw and pretreated) and extracted cellulose is shown in Table 1. It is evident that for ficus leaf fibers, the hemicellulose content decreased from 30.5% to 12.6% and the α-cellulose and lignin content increased from 38.1% to 46.8% and 23.4% to 36.5%, respectively, on pretreatment. However, the extractives and ash contents also decreased from 2.9% to 1.3% and 4.5% to 2.1% respectively. The aim of the fiber pretreatment with alkali was to decrease the intermolecular binding possibilities, which in turn led to an increase of solubility of noncellulosic components. The pretreatment removed part of the hemicellulose, external surface covering, and some other substance from the fiber. The α-cellulose content in extracted cellulose (from pretreated fibers) compared to that of pretreated fibers was found to increase after various chemical treatments from 46.8% to 90.6%. The hemicellulose, extractives, and ash contents decreased significantly from 12.6% to 2.5% and 1.3% to 0.6% and 2.1% to 1.1%, respectively,

TABLE 1

Chemical composition of ficus leaf (both untreated and pretreated) fibers and extracted cellulose (% on an oven-dry weight basis)

Material	*Extractives (%) [S.D]*	*Cellulose (%) [S.D]*	*Hemicellulose (%) [S.D]*	*Lignin (%) [S.D]*	*Ash (%) [S.D]*
Untreated fiber	2.9 [0.19]	38.1 [1.07]	30.5 [1.21]	23.4 [1.04]	4.5 [0.37]
Pretreated fiber	1.3 [0.15]	46.8 [1.02]	12.6 [0.94]	36.5 [0.98]	2.1 [0.38]
Extracted cellulose	0.6 [0.13]	90.6 [0.81]	2.5 [0.47]	4.7 [0.65]	1.1 [0.29]

S.D, Standard deviation.

TABLE 2
Comparative chemical composition of untreated and alkali treated ficus leaf fibers with some other plant fibers and agricultural residues

Fiber	*% Cellulose*	*% Hemicellulose*	*% Lignin*	*Reference*
Sisal	66–78	10–14	10–14	Reddy et al. (2014)
Banana	63–64	19	5	Reddy et al. (2014)
Tamarind	59	22	19	Maheswari et al. (2012a)
Bagasse	55.2	16.8	25.3	John and Anandjiwala (2008)
Bamboo	26–43	30	21–31	John and Anandjiwala (2008)
Wheat straw	33–38	26–32	17–19	Reddy and Yang (2005)
Rice straw	28–36	23–28	12–14	Reddy and Yang (2005)
Ficus	38.1	30.5	23.4	Present work

while the lignin content also decreased from 36.5% to 4.7%. This may be attributed to the effect of chemicals that were used during the cellulose extraction process. This proves that during the sodium chlorite and sodium hydroxide treatments substantial breakdown of the lignocellulosic structure took place resulting in the hydrolysis of hemicellulose fraction and depolymerization of lignin. The yield of extracted cellulose from pretreated leaf fibers was found to be 55%. A comparison of chemical composition of ficus leaf fibers with some important natural plant fibers and agricultural residues is presented in Table 2. From the table it is evident that sisal, banana, tamarind, bagasse, and bamboo have higher cellulose content, whereas wheat straw and rice straw fibers have lower cellulose content when compared to ficus leaf fiber.

The surface morphology of the ficus leaf fibers (both untreated and pretreated) and extracted cellulose is shown in Figure 1. The micrographs clearly show the difference in the surface morphology of untreated and pretreated fiber as well as extracted cellulose. In Figure 1(a) and (b) impurities were observed on the untreated fiber surface. Figure 1(c) and (d) shows partial hemicellulose and surface impurity removal after pretreatment of the fiber. As seen from Figure 1 (e) and (f), after several chemical (chlorination and alkali) treatments, most of the lignin and hemicellulose were removed from the inner part of the fiber by hydrolysis and depolymerization and then finally the fibrils were produced from original fibers by defibrillation. In ribbon shaped short fiber strands structured in irregular fragments and network form appeared with cleaned surface in the extracted cellulose.

The FTIR spectra of ficus leaf fibers (untreated and pretreated) and extracted cellulose are shown in Figure 2. All the spectra had a most representative band at 3334 cm^{-1} corresponding to the stretching of H-bonded OH groups, and at 2918 cm^{-1} corresponding to the C-H stretching vibrations (Reddy et al. 2009a). The small band at 1736 cm^{-1} corresponds to the carbonyl groups (C=O) due to the presence of acetyl ester and carbonyl aldehyde groups of hemicellulose (Reddy et al. 2009a). A decrease in the intensity of this peak for the cellulose extracted from pretreated fiber indicates the maximum removal of hemicellulose by applied chemical extraction. The band at 1629 cm^{-1} was attributed to the bending mode of the absorbed water (Sain and Panthapulakkal 2006). The bands at 1505 and 1458 cm^{-1} in the fiber represent the aromatic C=C ring stretching and C-H deformation in methyl, methylene and methoxyl groups of lignin (Maheswari et al. 2012b; Sain and Panthapulakkal 2006). The intensity of these bands decreased in the extracted cellulose, because of the most of removal of lignin by chemical treatments on cellulose extraction process. The band at 1422 cm^{-1} was attributed to the CH_2 bending and at 1367 cm^{-1} to the O–H bending of cellulose (Maheswari et al. 2012b). The absorbance band at 1315 cm^{-1} arose from the C–C and C–O skeletal vibrations of cellulose. An intense band at 1230 cm^{-1} was corresponding to the -COO vibration of acetyl groups in hemicellulose (Reddy et al. 2009a). This band slightly decreased after pretreatment as well as significantly decreased in extracted cellulose, indicating the removal of most of the hemicellulose

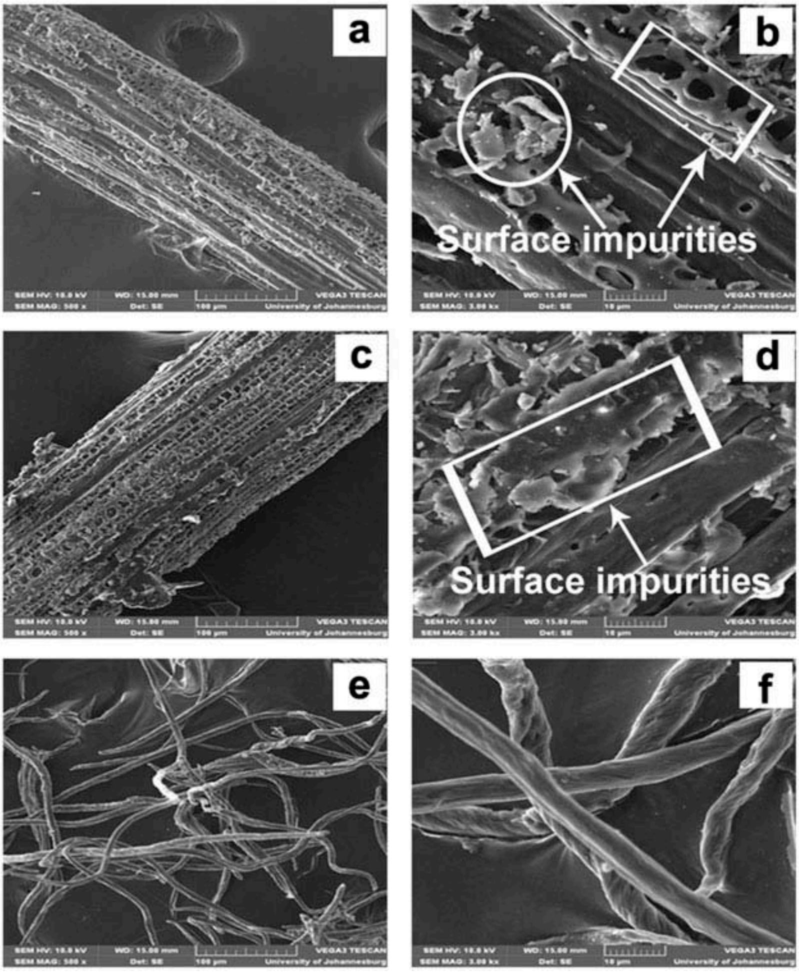

FIGURE 1 Scanning electron micrographs of ficus (a, b) untreated fiber surface, (c, d) pretreated fiber surface, and (e, f) extracted cellulose surface.

during the chemical treatments. The absorption band at 1157 cm^{-1} corresponds to the C-O antisymmetrical bridge stretching and the in-plane ring stretching. A strong absorption band at 1030 cm^{-1} arose from skeletal vibration of C-O-C pyranose ring (Pappas et al. 2002). The sharp absorption band at 896 cm^{-1} corresponds to the ring valence vibration was characteristic of β-glycosidic linkages between the sugars units (Sain and Panthapulakkal 2006). FTIR spectra agree with the removal of hemicellulose and lignin during the chemical treatments by the cellulose extraction from the pretreated leaf fibers.

To further confirm the structural features of untreated, pretreated, and extracted cellulose high-resolution ^{13}C NMR (CP-MAS) spectroscopy was used. Figure 3 shows the ^{13}C CP-MAS NMR spectra of the untreated, pretreated and extracted cellulose. The peak at 64.3 ppm for each of the three samples was assigned to C_6 corresponding to the amorphous region of cellulose. The resonance peaks for the carbons ring in the case of cellulose (C_2, C_3, and C_5) appeared in the 72.1–74.5 ppm region. However, these three peaks were found to be superimposed in the case of extracted cellulose. The narrow resonance peaks at 83.5 and 87.6 ppm were attributed to C_4 carbon in the crystalline

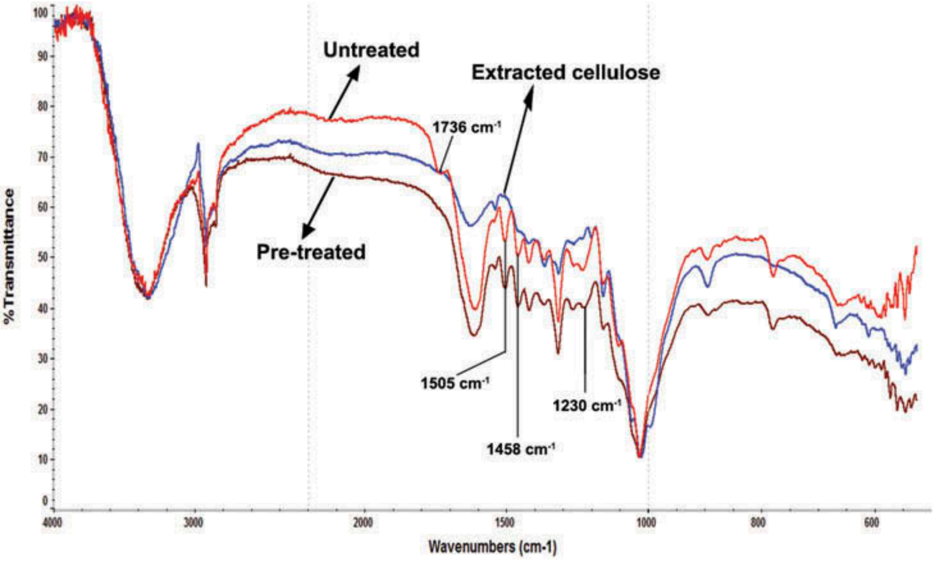

FIGURE 2 FTIR spectra of ficus untreated fiber, pretreated fiber, and extracted cellulose.

domain. Of these, the peak at 83.5 ppm was attributed to crystalline cellulose, whereas the other peak at 87.6 ppm was attributed to the crystal surface or disordered cellulose. The most downfield peak in the spectra at 104.7 ppm was associated with C_1, the anomeric carbon of nonequivalent glycosidic linkages (Pappas et al. 2002; Tang et al. 2000). However, it can be seen in untreated fiber other small peaks at 20.7 and 172.6 ppm were attributed to CH_3COO and COOH groups of hemicellulose. Further, the peaks at 12–39 ppm, 56.4 ppm, and 116–156 ppm were attributed to the methyl and alkyl carbons, methoxyl (-OCH3) and aromatic carbons of lignin (Maheswari et al. 2012b; Reddy et al. 2009a). However, in the spectrum of pretreated fibers, hemicellulose peaks were considerably decreased indicating lowering of hemicellulose content on pretreatment. While in the spectrum of extracted cellulose, the hemicellulose peaks were completely disappeared indicating most of hemicellulose removal by chemical treatments. However, in pretreated fiber the lignin peaks had no significant changes, while in extracted cellulose these peaks completely disappeared due to most of lignin removal by the chemical treatments. This indicates that hemicellulose and lignin were successfully removed as a result of the different chemical treatments by cellulose extraction process. These observations are very much in consistent with the chemical analysis and FTIR spectroscopy.

X-ray diffractograms of ficus leaf fibers (both untreated and pretreated) and extracted cellulose are shown in Figure 4. The natural fiber materials are semi crystalline in nature. The observed X-ray diffraction peaks for both materials were attributed to the crystalline scattering and the diffused background to the disordered regions. The diffractograms corresponding to ficus leaf fibers and extracted cellulose show diffraction peaks at 2θ values of 15.2° and 21.8°. Crystallinity index (CI), which is a measure of the amount of crystalline cellulose with respect to the amount of amorphous cellulose, was evaluated using the Segal empirical method.

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

Where I_{002} is the intensity of diffraction maximum of crystalline region close to 22°, while I_{am} is the intensity value for the amorphous cellulose region close to 17.3° (Li et al., 2014). The crystallinity index values of the untreated and pretreated leaf fibers, and extracted cellulose were calculated to be 50.1, 50.5, and 55.9, respectively. The extracted cellulose was found to have higher crystallinity than

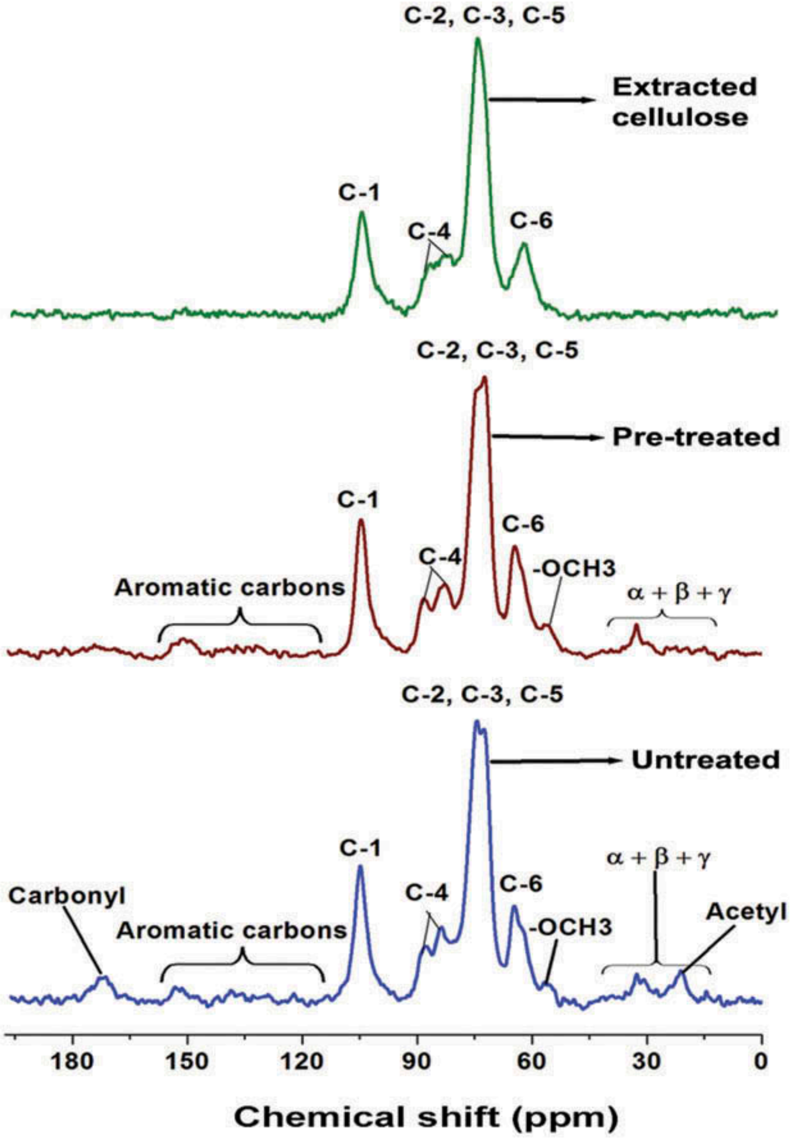

FIGURE 3 ^{13}C CP-MAS NMR spectra of ficus untreated fiber, pretreated fiber, and extracted cellulose.

untreated and pretreated fibers. This was attributed to the removal of amorphous hemicellulose and lignin and the effect of chemical treatments on cellulose during extraction. This is also supported by the chimerical analysis and FTIR spectral analysis.

Thermal stability of the ficus untreated fiber and pretreated fiber, and extracted cellulose (from pretreated fibers) was investigated by the thermogravimetric method. Figure 5 shows the primary thermograms of untreated, pretreated leaf fibers, and extracted cellulose (from pretreated fibers). The minor weight loss around 100 °C observed for all the samples corresponds to moisture loss by evaporation. The weight loss around this temperature was found to be 13% and 12% for untreated fiber and pretreated fiber respectively. The initial degradation stage occurred in the ranges of 210–310

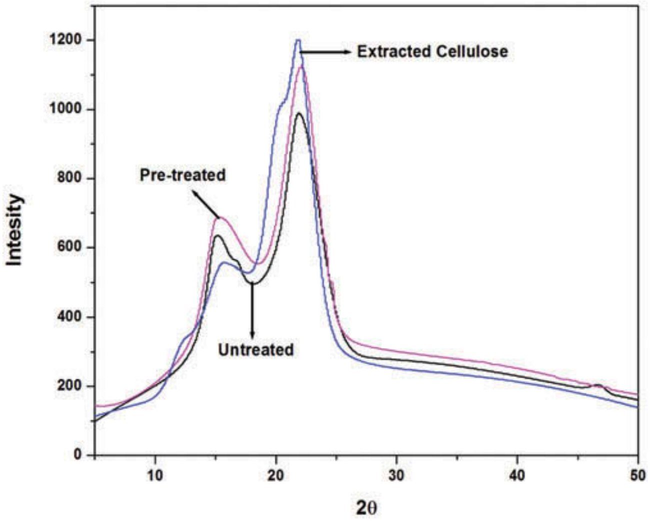

FIGURE 4 X-Ray diffractograms of ficus untreated fiber, pretreated fiber, and extracted cellulose.

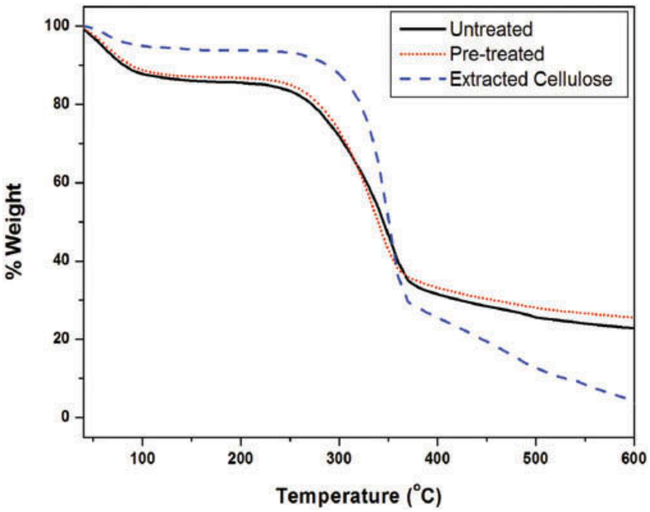

FIGURE 5 Primary thermograms of ficus untreated fiber, pretreated fiber, and extracted cellulose.

°C and 220–310 °C for untreated fiber and pretreated fiber respectively. This is due to thermal depolymerization of hemicelluloses and some fraction of lignin (Reddy et al 2009b). The weight loss in these temperature ranges was found to be about 35%. The second degradation stage occurs in the ranges of 310–370 °C and 310–365 °C for untreated and alkali-treated fiber respectively. This thermal degradation corresponds to the cleavage of glycoside bonds of cellulose structure and depolymerization of lignin (Reddy et al 2009b). The weight loss over these temperature ranges was about 66% (untreated) and 62% (pretreated). However, the extracted cellulose showed significantly different degradation behavior from untreated and pretreated fibers. The degradation of extracted cellulose microfibrils was two-step degradation. A minor weight loss around 100 °C corresponds to moisture loss by evaporation. The weight loss in the temperature was found to be about 5%. The second decomposition temperature starts at 290 °C up to 370 °C and then weight loss in this temperature range was found to be about 64%. These results clearly demonstrate that the thermal stability of the extracted cellulose was higher than that of the untreated and pretreated leaf fibers, as the extracted cellulose contain a little amount of less thermally stable substances, such as hemicellulose. The % char residue at 600 °C of untreated fiber, pretreated fiber, and extracted cellulose were found to be 22%, 25%, and 4%, respectively. The% char of extracted cellulose was found to be lower than the untreated and pretreated fibers, due to the removal of ash and this is in conformity with the results of chemical analyses.

CONCLUSION

In this work, cellulose was successfully extracted from ficus leaf fibers (pretreated) using the chemical process. The chemical composition of ficus leaf fibers (both untreated and pretreated) was established and the results indicated that the extracted cellulose (from pretreated fibers) had higher cellulose content. FTIR measurements of the extracted cellulose revealed the removal of hemicellulose and lignin in the extraction process. The cellulose extracted (from pretreated fibers) had higher crystallinity than that of untreated and pretreated fibers. The thermal stability of the extracted cellulose was found to be higher than those of untreated and pretreated fibers. The initial degradation temperature of 290 °C hints at its possible application as a natural reinforcement even in thermoplastic materials whose processing temperature is up to 300 °C. The results of this study suggest that ficus leaf fibers could prove to be an effective alternative raw material for paper pulp, biofilms, and biocomposite applications. Further, these results also demonstrate the profitable exploitation of a forestry residue for a major economic activity of cellulose production.

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