Pertanika J. Sci. & Technol. 31 (S1): 125 - 138 (2023)

SCIENCE & TECHNOLOGY

Journal homepage: http://www.pertanika.upm.edu.my/

Extraction and Characterization of Novel Ligno-cellulosic Fiber from *Wrightia tinctoria* and *Cebia pentandra* Plant for Textile and Polymer Composite Applications

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ABSTRACT

Natural fibers derived from cellulose and ligno-celluloses materials have many advantages, such as being renewable, low density, inexhaustible, and cheap rather than synthetic fibers. Researchers and scientists are searching for a new fiber source that can be processed environmentally sustainable. The aim is to produce an organic and Eco-friendly product. The present investigation aims to extract and characterize ligno-cellulosic fiber from the seedpod of *Wrightia tinctoria* (WT) and *Cebia pentandra* (CP) plants. The extraction of WT fibers (WTFs) and CP fibers (CPFs) was carried out using the hand-stripping method. The structural and functional Characterization of WTFs and CPFs were determined using Scanning Electron Microscope (SEM), Fourier Transform Infrared (FT-IR) spectroscopy, Chemical analysis, X-ray diffraction studies (XRD), and the thermal behavior of fibers determined by using Thermo Gravimetric Analysis (TGA). The results indicated that WTFs composed of 75% cellulose, 14% lignin, and 0.55% wax content were, as the CPFs were composed of 38% cellulose, 15% lignin, and wax content of 2.34%. The SEM micrograph confirms that both fibers were hollow structures with thin cell walls and luminous because of the wax content presence on the surface of the fiber. The crystallinity percentage of WTFs and CPFs was calculated from XRD studies and is

ARTICLE INFO

Article history:
Received: 06 March 2023
Accepted: 24 July 2023
Published: 27 October 2023

DOI: https://doi.org/10.47836/pjst.31.S1.07

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ISSN: 0128-7680
e-ISSN: 2231-8526

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valued at 62% and 52%. Thermo gravimetric analysis revealed that WTFs and CPFs were thermally stable up to 460°C and 350°C. The above characterization results confirm that WTFs and CPFs have a wide scope in textile and polymer composite applications.

Keywords: *Cebia pentandra*, lignocelluloses, SEM, TGA, *wrightia tinctoria*, XRD

INTRODUCTION

India has many natural fibers with approaching use in various industrialized products based on existing technology (Ramasamy et al., 2022). Natural fibers derived from plant materials are more eco-friendly, low-cost, renewable, and bio-compatible than petroleum-based synthetic fibers. Cellulose fiber has ample applications in the textile industry (Durai et al., 2022). During past decades, the usage of natural products has gained more attraction globally. The cellulose fiber materials show interest among the researchers to search for the best materials for each application (Mohan et al., 2022). The natural, conventional lignocellulosic fibers derived from jute, hemp, sisal, and ramie have been widely studied and reported. On the other hand, a few nonconversational lignocellulose fibers remain unutilized and are going as natural disposal (Divya, Jenish, et al., 2022). Two different natural fibers were collected from *Wrightia tinctoria* R.Br. and *Ceiba pentandra* only because of local seed pod-based fibers. It is available in large quantities to analyze and select a suitable product for futuristic applications.

Wrightia tinctoria R.Br. is a small deciduous tree that grows in a wide range of soil types. All parts of the plant have numerous meditational properties. In the Tamil Nadu state of India, the tree is called a "Jaundice curative tree" (Desore & Narula, 2018). The blackish-green fruits with white dots resembling two or more fruits were united at the tip (Ramalakshmi et al., 2012). The fibers were collected from matured dried fruit-like white hairs attached to the seed at the chalaza end. Numerous investigations have been developed to find a new product to replace the existing one (Subramanian et al., 2005).

Ceiba pentandra (CPFs) is one of the less given consideration agricultural products (Macedo et al., 2020). The fibers are grown in arid environments and distributed throughout warmer parts of India. The CPFs is a hollow lumen fiber with a hydrophobic character due to its waxy cutin; it can be used as a sorbent in oil. Fiber characteristics will vary depending on the soil and climatic conditions. Much research was carried out on CPFs in foreign countries, but in Indian fiber, only limited works were documented (Rangappa et al., 2022). Concerning the above, this recent report deals with the extraction of two lignocellulose natural fibers, *Wrightia tinctoria* (WT) and *Cebia pentandra* (CP) fiber, from the seedpod of plant and analyzes important properties like morphology, crystallinity, chemical composition, FT-IR spectroscopy and thermal properties concerning plantation environment.

MATERIALS AND METHODS

Extraction of Fibers

WTFs and CPF are collected from healthy and matured seedpods were obtained from the plants located at Therampalayam village, Mettupalayam Taluk (Latitude and longitude coordinates are: 11.289087, 76.940971), Coimbatore district of Tamil Nadu, India. The fibers were removed from the pods, seed, and cover. The entangled fibers were separated by hand and dried at room temperature at (28 ± 2°C) to remove moisture (Figure 1) (Raja et al., 2021). The collected fiber was authenticated from the Botanical Survey of India and the Institute of Forest Genetics and Tree Breeding, Coimbatore, Tamil Nadu, India.

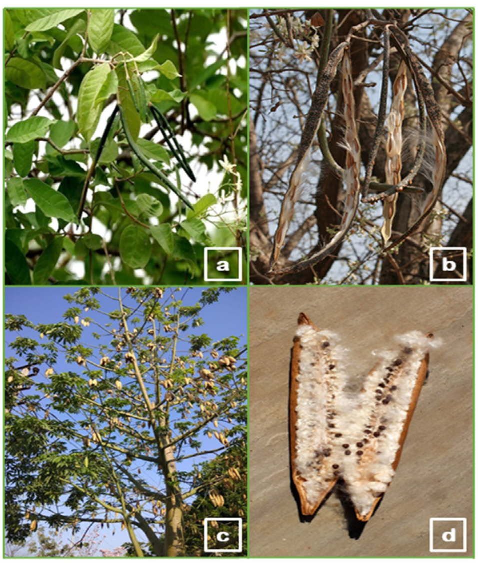

Figure 1. (a & b) *Wrightia tinctoria* tree and its seed pod fiber (b & C) *Cebia pentandra* tree and its seed pod fiber

Chemical Analysis

A typical testing procedure studied the chemical composition of cellulose, lignin, wax, ash, and moisture contents of CPFs and WTFs. The density of the fibers was assessed using the Mettler Toledo XSZ05 balance method. The wax content of CPFs and WTFs was estimated using the Conrad procedure, and the ash content was calculated by the ASTM E1755-01 method. The moisture content of fibers was assessed by the conversion weight loss method (Conrad, 1944; Divya, Suyambulingam, et al., 2022; Selvan et al., 2022).

Fourier Transform Infrared Analysis (FT-IR)

FT-IR spectrometer analysis was carried out to determine the presence of organic and inorganic components in the fibers using the SHIMADZU spectrometer. The infra-red passed on the surface of the sample, which may be solid, liquid, or gaseous form; the material absorbs the light, and the absorbency of the energy was measured at various wavelengths, which produces the output of an Infrared spectrum(Sanjay et al., 2019; Senthamaraikannan et al., 2019). The WTFs and CPFs were powdered, approximately 2 mg of powder mixed with 200mg potassium bromide (KBr), and compressed to 1 mm thick disks. The spectrum recorded in the frequency range from 4000-500cm^{-1} with a resolution of 2 cm^{-1}.

X-ray Diffraction Analysis (XRD)

The Super molecular structure of the WTFs and CPFs was investigated by X-ray diffraction analysis (XRD) using BRUKER ECO d8 Advance analyzer and PANALYTICAL system Netharland (Powder XRD) using Cukα radiation (λ = 1.5406 Å), operating 40 kV and 30 mA. Both fiber crystallinity index was calculated using the peak height method and Segal empirical equation (1) (Segal et al., 1959):

$$\mathrm{Crl} = \frac{I_{max} - I_{min}}{I_{max}} \; X \; 100 \tag{1}$$

Where I_{max} is a Crystalline Fraction, and I_{min} is a Diffracted intensity. Scherer's relation was admirable in establishing the crystallite size of the optimally surface-modified fiber, which is given in Equation 2. In this relation, 'K' represents the constant (0.89), 'λ' indicates the intensity of radiation, 'β' symbolizes the full width at half-maximum (FWHM), and 'θ' signifies the Bragg's angle (Saravanakumar et al., 2013; Pillai et al., 2022).

$$CS = \frac{K\lambda}{\beta \cos\theta} \tag{2}$$

Thermo Gravimetric Analysis (TGA)

Thermal gravimetric analysis is used to determine the thermal stability of the materials. Usually, cellulosic fibers decompose easily at low temperatures depending upon the fiber characteristic's morphology, crystallinity, and molecular weight (Khan et al., 2021a; Vinod et al., 2021). The thermograph of both fibers was recorded using the NETZSCH STA 449F3 A-1100M instrument. TG-DSC experiment was examined with an Alumina crucible with a lid in a programmed temperature range of 30 to 1000°C under a nitrogen gas atmosphere at 10°C/min.

Scanning Electron Microscopy Analysis (SEM)

The Surface morphology of WTFs and CPFs are examined using a Scanning Electron Microscope (SEM) Carl Zeiss tester. The SEM analysis is a powerful magnification tool to characterize the sample shape, size, and surface structure. A focused electron beam with low energy is radiated on the sample's surface, producing the sample's images by scanning (Bharath et al., 2016; Witayakran et al., 2017). The dried fibers were sputtered with gold before analysis to make the fiber conductivity, and then observed the image at an accelerating voltage of 30 kV.

RESULTS AND DISCUSSION

Chemical Analysis

The main components of the natural fibers are cellulose, lignin, wax, and Ash content. The chemical component in the plant fiber will differ from place to place due to the plant's maturity, relative humidity, temperature, and soil conditions (Karthik & Murugan, 2013). The evaluated WTFs and CPFs had WTFs composed of (75 wt%) cellulose, (14 wt%) lignin, (0.55 wt%) of wax content and (2.40%) of ash content were as the CPFs Cellulose (38 wt%), lignin (15 wt%), wax content (2.34 wt%) and Ash (2%) (Rantheesh et al., 2023; Sundaram et al., 2021). The Density of the CPFs and WTFs was 1.5 g/cc and 1.2 g/cc, slightly higher than other natural fibers. The analyzed Chemical composition of WTFs and CPFs are compared and are listed in Table 1. *Wrightia tinctoria* (WT) and *Cebia pentandra* (CP).

Table 1

Chemical composition of Wrightia tinctoria *(WT) and* Cebia pentandra *(CP) fibers*

S. No	Chemical Composition	WT (Wt.%)	CP (Wt.%)
1	Cellulose	75	42
2	Lignin content	15	20
3	Wax	2	23
4	Moisture	8	10
5	Ash content	2	1

Fourier Transform Infrared analysis (FT-IR)

FT-IR measure the main functional group present in the WTFs and CPFs by absorbance of infrared light at various wavelength. The IR spectrum of both samples is illustrated in Figures 2a and b. There were a total of eight well-defined peaks absorbed, and some similar absorption bands were observed for both fibers. WTFs broad absorption band is at 3335,2918,1732, 1506, 1421,1370,1231, and 896 cm^{-1}. CPFs show peaks at 3350,2887,1739, 1506,1375,1246,1055 and 896 cm^{-1}. The elongated absorption peaks at 3350 and 3335cm^{-1} represent cellulose's stretching vibration mode (–OH) bond in both fibers (Gandhi et al., 2022). The WTFs band peak of 2918 cm^{-1} in the spectrum indicates the asymmetric and symmetric stretching of methylene (–CH2–) groups in long alkyl chains (Ilangovan et al., 2018; Jagadeesan et al., 2023). The stretching vibrations (NH_3) of free amino acids were found at a wavelength of 2887cm^{-1} in CPFs. The carbonyl group (C=O) peak in 1739 and 1732 cm^{-1} shows the presence of acetyl groups C=O, degree of acetylation (Babu et al., 2020; Indran et al., 2018; Rajeshkumar et al., 2021). The medium absorption peak of CPFs and WTFs at 1375 and 1370 cm^{-1} indicates bond stretching CH bending. The bands in the region 1506 cm^{-1} are for C=C aromatic symmetrical stretching(Vijay et al., 2019). The CPFs' broad absorption peak at 1055 cm^{-1} is associated with the carbohydrate structure's stretching vibrations (C-C) (Babu et al., 2020). These peaks prove the presence of waxes. The Absorption at 1421.51cm^{-1} represents the symmetrical CH_2 bending vibration of crystalline cellulose (Indran et al., 2016; Jagadeesan et al., 2022). The peak 1231 cm^{-1} provides the presence of COH bending at C_6. The peak at 896 indicates the anomeric region of carbohydrates in both fibers(Sunesh et al., 2022).

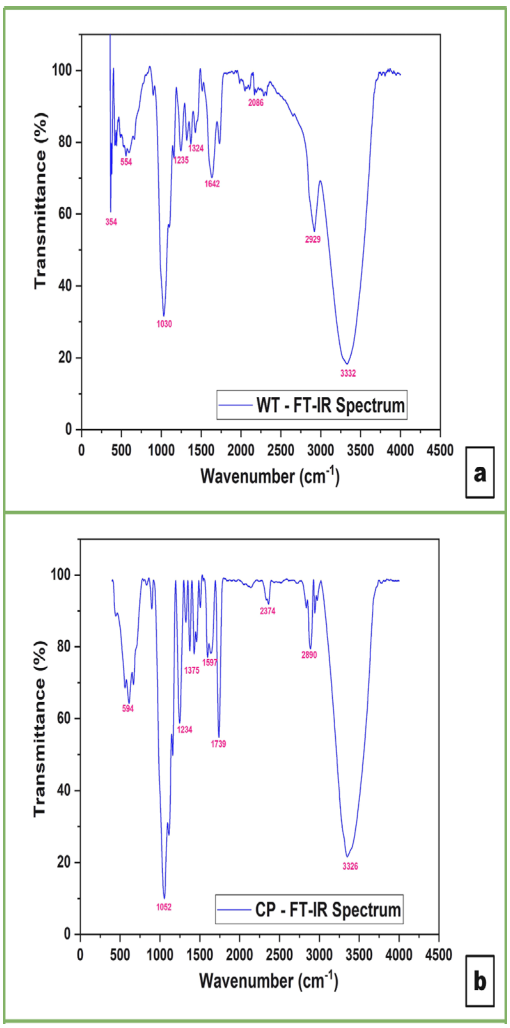

Figure 2. FT-IR analysis of (a) *Wrightia tinctoria* (WT) and (b) *Cebia pentandra* (CP) fibers

X-ray Diffraction Analysis (XRD)

The X-ray diffractogram of WTFs and CPFs has been recorded and shown in Figure 3. From the graph by XRD analysis of both fibers, crystalline size and the percentage of the crystallinity index were calculated. The crystalline size of the fiber reflects the diffusion of particles into the complex structure of the fiber (Gandhi et al., 2022). The particle size and crystallinity index will coincide, reflecting the fiber's moisture absorbency capacity and chemical reactivity (Divya, Jenish, et al., 2022). CPFs and WTFs major intensity peak at $2\theta = 23°$ and $2\theta = 21.9°$ and corresponds to the crystallographic part of cellulose, where the second peak at $2\theta = 18°$ and $2\theta = 15.9°$ corresponding to the amorphous part of cellulose (Moshi, Ravindran, Bharathi, Indran et al., 2020; Moshi, Ravindran, Bharathi, Padma, et al., 2020; Rajan et al., 2022; Surendran et al., 2022). The crystallinity Index CrI of both the fibers calculated using equation (1) is 57% for CPFs and 62.22% for WTFs, and the crystalline size of CPFs was 2.12 nm and WTFs fiber was 2.55 nm. The crystalline size of both fibers was lower than other lignocellulosic fibers: Luffa cylindrical fiber 4.52 nm, cotton 4.7 nm, and curaua fiber 3.4 nm, respectively (Azwa et al., 2013).

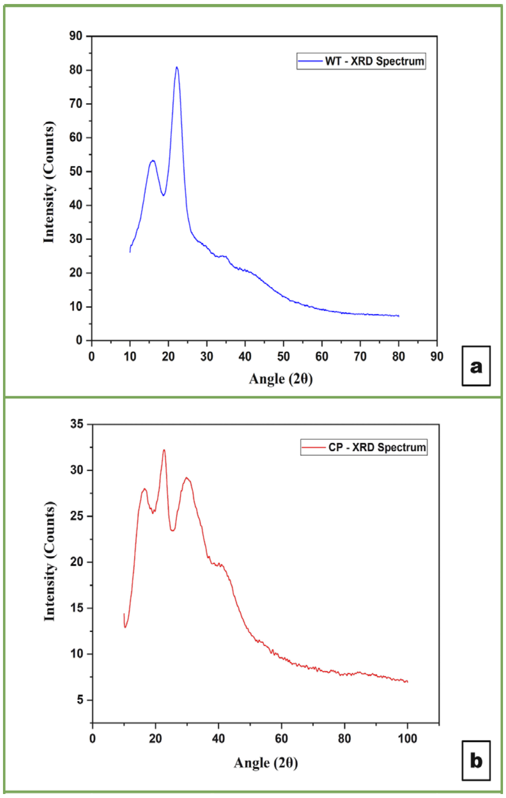

Figure 3. X-ray diffraction spectroscopy of (a) *Wrightia tinctoria* (WT) and (b) *Cebia pentandra* CP fibers

Thermogravimetric Analysis (TGA)

The thermal analysis was carried out on WTFs and CPFs to analyze the thermal stability and evaluate the sample's mass loss at respective times and temperatures. The thermal stability of the materials depends upon the characteristics, crystallinity, and molecular weight; most of the natural fibers are low in thermal stability (Ilangovan et al., 2020). The thermal decomposition and weight loss are observed in three main stages: first-stage water evaporation, second-stage devolatilization of organic matters like cellulose, hemicelluloses, and lignin content and final-stage decomposition and charcoal formation (Khan et al., 2021b).

From the TGA result (Figures 4a and 5b), WTFs and CPFs indicate a similar fiber degradation process. In the first stage, minor mass loss is below 100°C for both fibers due to moisture evaporation (Divya et al., 2021; Divya, Jenish, et al., 2022; Iyyadurai et al., 2023; Raja et al., 2022). The second mass loss starts at below 230–325°C for CPFs and 275–330°C for WTFs` which can be attributed to the degradation of lignin and cellulose. The third stage CPFs show pyrolysis between 360–900°C

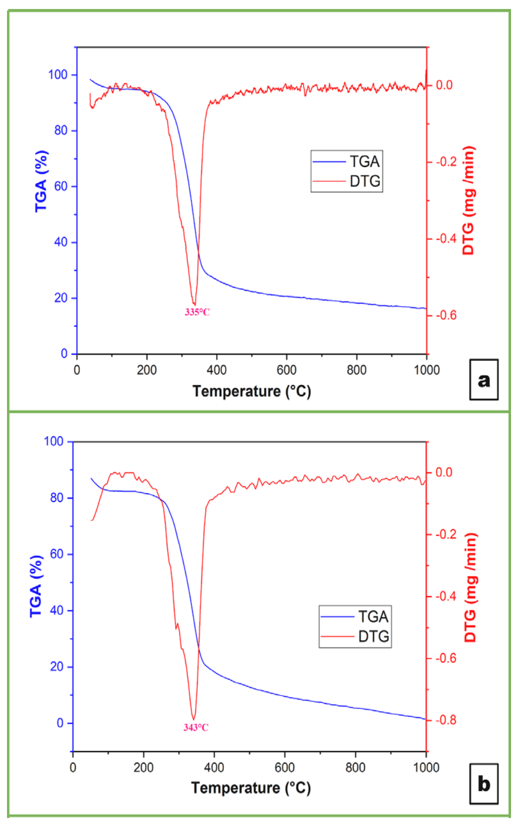

Figure 4. Thermal analysis images of (a) *Wrightia tinctoria* (WT) and (b) *Cebia pentandra* (CP) fibers

corresponding to the charcoal formation residual mass 16.34% at 998.3°C. Above 400°C of WTFs shows a slow decomposition of lignin, cellulose, and charcoal formation, and the charcoal yield at 997.8°C and the residual is 1.42% (Loganathan et al., 2020; Madhu et al., 2019).

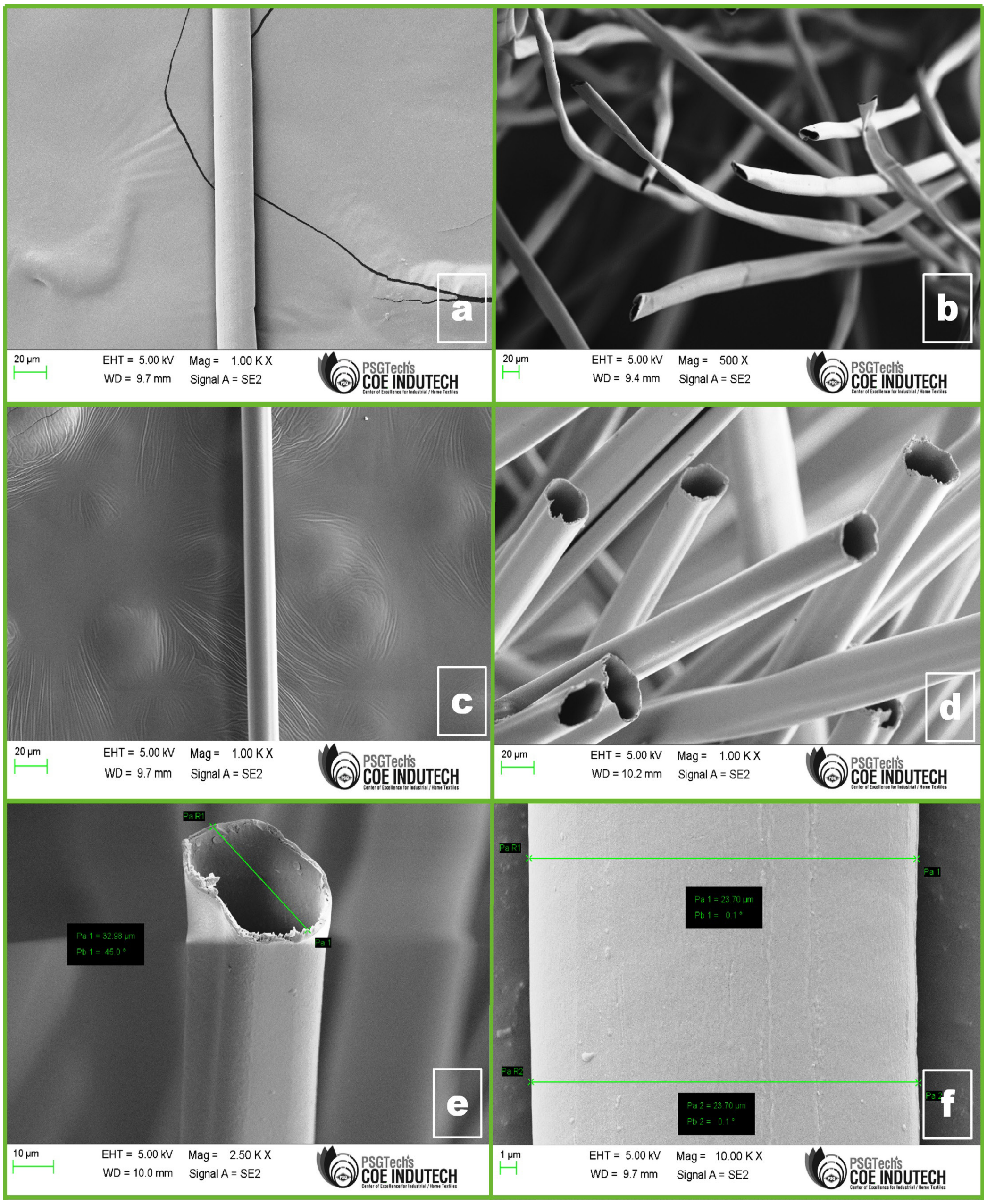

Figure 5. SEM images of (a, c) Longitudinal morphology of *Wrightia tinctoria* (WT) and *Cebia pentandra* (CP), (b, d) Cross-sectional morphology of WT and CP, and (e, f) Diameter of WT and CP

Scanning Electron Microscopy Analysis (SEM)

The Surface morphology of the WTFs and CPFs was examined in a cross-sectional and long-sectional direction using a Scanning Electron Microscope (SEM) at different magnifications (Figure 5a–d). Figure 4a and b, cross-section and long sectional view of CPFs at the magnification of 500x (20 µm), shows a thin fiber wall covered with a large layer of wax, making the fiber hydrophobic. The oval and hollow structure shows that the fiber retains warmth and is lightweight. The fiber contains large luminous which leads to a lower density of fiber (Fatma & Jahan, 2018; Manimekalai et al., 2021; Moshi, Ravindran, Bharathi, Indran, et al., 2020; Moshi, Ravindran, Bharathi, Padma, et al., 2020; Sumesh et al., 2021). The average diameter of fiber was measured and computed as 27.30 µm from Figures 5b, 4c, and 4d, which show the cross-section and long-sectional view of WTFs at the magnification of 1 kx (20 µm) and 250 kx (10 µm), showing thin cell wall, cylindrical structure with a thin line and small microfibers on the entire surface on the fiber which means the higher porosity and good air permeability in nature—the diameter of the fibers calculated as 22.54 µm which clearly shows the WTFs lighter then CPFs. The staple length of both fibers was manually calculated; the mean average value of the CPFs was 37.42 mm, and the WTFs were 35 mm. Both fibers have analogous characteristics because of their similar morphology (Sari et al., 2021).

CONCLUSION

This investigation analyzes the complete characterization of WTFs and CPFs to select futuristic applications using surface morphology, FT-IR, Chemical analysis, crystallinity, and thermal analysis techniques. SEM micrograph of both the fibers shows a smooth surface and hollow structure with a large lumen, which leads to a hydrophobic nature. The diameter and length of the fibers were calculated as 27.30 µm and 37.42 mm for CPF and 22.54 µm and 35 mm. which indicates that both fibers have less staple length when compared with other natural fibers, which means it is quite difficult to construct 100% fabric. The cellulose content of CPFs and WTFs is 38 and 75%, with WTFs showing high cellulose content with low density, contributing largely to the end product's feature. FT-IR studies indicate the presence of functional groups in the fibers. CPFs and WTFs showed similar absorption bands and confirmed the fiber's hydrophobicity. The crystallinity index (CrI) of CPFs and WTFs was found to be 57% and 62.2%, and crystalline size was 2.12 nm and 2.15 nm, which confirms the presence of crystalline cellulose, affecting the fiber's moisture absorbency. The thermal analysis indicates that fibers were thermally stable up to 330°C for CPFs and 360°C for WTFs. Since considering the deserved properties of CPFs and WTFs, like good thermal resistance and lightweight, they can be utilized for technical textile products. Future recommends giving mild alkali treatment to the fiber, which can also be utilized in the apparel industry.

ACKNOWLEDGEMENTS

The first author acknowledges the Natural Composites Research Group Lab, Department of Materials and Production Engineering, The Sirindhorn International Thai-German Graduate School of Engineering, King Mongkut's University of Technology North Bangkok, Bangkok-10800, Thailand, for supporting the completion of this research work.

This research was funded by the National Science, Research and Innovation Fund (NSRF) and King Mongkut's University of Technology North Bangkok, Thailand with Contract no. KMUTNB-FF-66-01.

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