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Investigating the characterization of fiber extracted from *Wrightia tinctoria* (Roxb.) R. BR

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Abstract
Natural fibers are one of the good alternative sources for replacing synthetic fiber and reinforcing polymer matrices because of their eco-friendly nature. The present study was undertaken to investigate the fibers extract from *Wrightia tinctoria* (Roxb.) R. Br. (Apocynaceae). seedpod and to analyze their mechanical behavior of the fiber. Physical and mechanical characterizations were carried out on *Wrightia tinctoria* (Roxb.) R. Br fiber (*WTF*): fibre strength, elongation, fiber length, fineness, diameter and chemical composition. The microstructure and functional elements of fiber were studied using Field Emission Scanning Electron Microscopy and FTIR spectroscopy for better understands their behavior. Thermal analysis was studied using Differential Scanning Calorimetry (DSC). The crystallinity indexes (CrI) and crystallite size (CS) of *WTF* were studied using X-ray diffraction (XRD) spectroscopy. The results showed a strong influence of extraction parameters on the characteristics of fibers. The XRD showed the highest degree of crystallinity and the lower moisture content and water absorption. The *WTF* has no hemicelluloses, as verified by FTIR. Thus the above characteristics confirm that this fibre has wide scope in the field of textiles application.

Keywords: Wrightia tinctoria R. Br. FESEM, FTIR, XRD spectroscopy, Crystallinity and Textile application etc.

1. Introduction
Wrightia tinctoria (Roxb.) R.Br. (Sans: Asita-Kutaja) belonging to family *Apocynaceae* is a small deciduous tree with pale grey, smooth bark, distributed in tropical Africa and Asia. It is considered to be therapeutically very effective jaundice plant in Indian indigenous system of medicine [1]. *W. tinctoria* commonly called as veppalaii (in Tamil). These species have been important in the traditional healing. However, the former one is widely recognized medicinal plant [2]. In recent years, multiple drug resistance in human pathogenic microorganisms has been developed due to indiscriminate use of commercial antimicrobial drugs commonly used in the treatment of such diseases [3]. The whole plant or its specific parts (bark, leaf, seed and root) are known to have medicinal properties and have a long history ofuse by indigenous communities in India [4].
Wrightia tinctoria is a small to medium-size deciduous tree. The plant grows up to 18 m tall and to 20 cm DBH (Diameter at Breast Height) with green marks on the stem and producing milky-white resin. The bark is smooth, somewhat corky and pale grey. Leaves large up to 10 cm long by 5 cm wide, simple, opposite, decussate and glabrous (sometimes puberulous beneath). Young leaves are bluish with reddish nerves. Flowers white, fragrant, 1-5 cm long, arranged in lax dichasial cymes (5 cm long). Fruit a green follicle, 0.5 cm in diameter by up to 50 cm long, pendulouspairs and coherent only at the tip. Seed linear, pointed at the ends, 1.2-1.8 mm long, light yellowish-grey, crowned with a tuft of white silky hairs. W. tinctoria coppices well and also produces root suckers. It's slow to moderate growing. The saplings start to flower and fruit when they are 5-8 years old. The planted seedling should be protected from livestock browsing The tree is sensitive to frost and is damaged by drought. It shows tolerance to high uranium levels in soils in India. The pods contain floss, which is used for stuffing cushions. The cream-coloured latex has a rubber content varying from 2 to 28% that can be exploited commercially [5]. Leaves, flowers, fruits and roots constitute the source of an indigo-yielding glucoside, which produces a blue dye or an indigo dye [6].
The crushed fresh leaves when filled in the cavity of delayed tooth relieve tooth ache. A decoction of the leaves and bark is taken as a stomachic and in the treatment of abdominal pain.

The bark and seeds are effective against psoriasis, bilious infections, leprosy, asthma, various skin diseases and nonspecific termatitis[7]. Concerning the above, this investigation deals with the extraction of new natural fibers as *Wrightia tinctoria* fibers (*WTF*) from the seedpod and analysis of the physical and mechanical properties.

2. Materials and Methods

2.1 Material and extraction of *WTF*

The plant material *WTF* was collected from in and around Mettupalayam, Coimbatore District Tamil Nadu. The matured seed pod samples were shade dried at room temperature (28±2°C) for 3 days and the fibres were extracted from the seedpod by hand stripping method. The collected fibers were then bundled in plastic bag and stored in normal and air tight condition for 5 days for the experiments. The plant specimen was identified and confirmed by Botanical Survey of India, Coimbatore, Tamil Nadu, India.

a) Fiber in seed pod **b)** Hand stripped conditioned fiber

Fig 1: *Wrightia tinctoria* Fiber

2.2 Characterization of *WTF*

2.2.1 Chemical composition of *WTF*

The main purpose of this analysis was to identify the different components present in *WTF*. The chemical compositions such as cellulose, hemicellulose, and lignin content of the *WTF* were determined at SITRA (South India Textile Research Association, Coimbatore, India) laboratory using the standard test procedures. Using Mettler Toledo XSZ05 balance method, we evaluated the density of *WTF*. The wax content of the *WTF* was assessed using Conrad procedure and the ash content was calculated as per the ASTM E1755-61 standard. Presence of moisture in the *WTF* was determined by the conventional weight loss method[8].

2.2.2 Length and Diameter of *WTF*

The fibre length analysis using advanced instrument was not possible due to the brittle nature of the fibre. Hence the fibre length was measured manually using a calibrated steel scale[9]. The Fiber diameters were measured from FESEM photography by drawing straight lines along the diagonals of an image and measuring fibers that crossed the lines using Carl Zeiss, Germany instrument [10].

2.2.3 Tensile properties of *WTF*

Physical properties of fibers can be classified into moisture absorption, mechanical, electrical, and thermal properties. The most important mechanical properties of fibers are their tensile properties [11]. The mechanical properties of the natural fibers mostly depend on the percentage of cellulose content. The high cellulose content and small micro fibril angle contribute in improving the tensile strength and stiffness of the fiber the mechanical properties of the *WTF* were determined by performing the single-fiber tensile test [12]. The tensile properties of cotton and milkweed fibres were determined as per ASTM D 3822-01 standard in an Vibrodyn 500 tester tester.

2.2.4 Thermal analysis of *WTF*

The physical properties measured by common thermal analysis techniques such as heat, mass, dimension, strain, and stress. Test environmental conditions are typically an inert gas such as nitrogen, an oxidizing gas such as air, humidified nitrogen, and stress or strain. Differential scanning calorimetry (DSC) is the most commonly used thermal analysis techniques [13]. DSC allows a rapid detection and measurement of the physical and chemical changes that a polymer undergoes when subjected to heating [14]. The Thermal stability of *WTF* assessed using DSC (NETZSCH STA 449F3) the temperature range starts from 30°C/20.0(K/min)/1000°C.

2.2.5. FTIR spectroscopy

FTIR analysis was performed to study the structural and chemical information of the fibre materials [9]. Fourier transform-infrared spectra of *WTF* were recorded using Thermo Fisher Scientific, Nicolet 6700 spectra in a KBr matrix with a scan rate of 32 scans min^{-1} and a resolution of 2 cm^{-1} in the wavenumber region 4000–400 cm^{-1}. *WTF* samples were chopped into small particles using scissors, and ground to a fine powder using a mortar and pestle. This powder was mixed with KBr and pelletized by pressure to record the FT-IR spectra under standard conditions [15].

2.2.6 X-ray Diffraction spectroscopy

The crystallinity index (CrI) and crystallite size of *WTF* were studied using X-ray diffraction (XRD) spectroscopy with monochromatic CuKα radiation of 0.154 nm wavelength at a current of 30 mA with an accelerating voltage of 40 kV. The analysis was carried out in the 2θ ranges from 10° to 80° at a scanning speed of 5° per min [12]. According to Segal's method, the index of crystallinity and crystallite size (CS) of *WTF* is calculated from the heights of the amorphous (Iam) and the total intensity (I) [16].

$$CI = \frac{(Ic - Iam)}{Ic} \quad X\ 100$$

where I_C represents maximum intensity of the peak (2 0 0) which corresponds to the crystalline fraction and Iam represents minimum intensity of the peak (1 1 0) which corresponds to the amorphous fraction. The crystallite size (CS) of the IMFs was calculated for the crystallographic plane (2 0 0) using Scherer's formula (2):

$$CS = \frac{K\lambda}{\beta COS\theta}$$

where λ is the Scherer constant (0.94), k is the X-ray wavelength (0.154 nm), ß is the peak's full-width at half-maximum and h is the Bragg angle [17].

2.2.7 Morphological Study of *WTF*

The morphology of the *WTF* was characterized using Field Emission Scanning Electron Microscopy (FESEM) Carl Zeiss tester. The *WTF* were dried at room temperature and fibers were gold coated before analysis[18]. The FESEM studies were conducted by scanning the samples with a high-energy electron beam at an accelerating voltage of 0.2-30 kV in a vacuum chamber and then image captured in different Magnification.

2.3 Results and discussion

The *WTF* testing reports were studied and the results are reported below.

2.3.1. Chemical composition of *WTF*

The detailed comparison of properties of seed fibre like cotton, milkweed, and kapok with *Wrightia tinctoria* are shown in table 1.

Table 1: Comparison of Chemical composition of Cotton, Milkweed, Kapok and *WTF* fibers

S. No	Chemical Composition	Cotton fiber	Milkweed fiber	Kapok fiber	*Wrightia tinctoria* fiber
1	Cellulose content wt%	80-90%	55%	35-50%	75.23%
2	Lignin content wt %	-	18%	15-22%	13.77%
3	Wax content wt %	0.5-1%	-	2 3%	0.55%
4	Ash content (on dry basis) wt %	1-1%	-	-	2.40%
5	Moisture content wt %	8.5 %	10.5-10.9%	10-10.73%	7.77%
6	Density g/cc	1.54	1.4	1.5	1.264

Above table 1 shows that *WTF* contained the higher percentage of cellulose content (75.23%) and lower percentage of lignin content (13.77 wt%) compared to other seed fiber. Normally high level of cellulose content and lower microfibrillar angle provide better strength properties and it is hydrophilic in nature due to high cellulose content [19].The higher content of lignin made the fiber tougher and stiffer compared to other fiber. *WTF* had the lowest lignin content which suggested the this material can undergo bleaching more easily and with the utilization of lower amount of chemical compared to milkweed and kapok fiber[20].The ash content 2.40wt% is higher compared to other fiber increase in ash content decrease the fire resistance characterstitic. The average moisture content of *WTF* material determined by weighing of sample before and after drying for 24 h at 105°C was found 7.77 wt%. The fibre density is considering the air inside the hollowness of fibres. The linear density of *WTF* calculated using Vibrodyn and gravimetric method was found to be around 1.26g/cc respectively which is low compared to the other seed fibers.

2.3.2 Length and Diameter of *WTF*

The staple lengths of *WTF* were calculated manually the average mean value is 36.1mm and the standard deviation and co-efficient of variation is 34.5and 1.03% respectively. The short fiber percentage was high and there is wider variation in fiber length. The fibre diameter observed on Field Emission Scanning Electron Microscopy. The dimension was found to be in the range of 100µm with a mean value of *WTF* is 51.08 µm.

2.3.3 Tensile properties of *WTF*

The tensile properties of single *WTF* were determined using an Vibrodyn 500 tester it measure strength and elongation of individual fibers based on the constant rate of elongation. The sample tested methods BISFA 2004, Chapter 6 & ASTM D 3822/D3822M-14 at relative humidity 65%+/-2% and temperature of 21°C+/-1 °C. The tensile properties of *WTF* single fibre results were given in the below table 2.

Table 2: Tensile properties of *WTF* is compared with cottyon and milkweed fiber

S. No	Tensile properties	Cotton fiber	Milkweed fiber	*Wrightia tinctoria* fiber
1	Mean Denier	1.25	1.05	1.36
2	CV% of denier	12.7	18.6	16.14
3	Tenacity g/Denier	4.1	3.73	2.41
4	CV% of Tenacity	34.1	37.6	46.11
5	Elongation %	8.1	3.05	1.95
6	CV% of Elongation	23.2	33.7	26.28
7	Initial Modulus (gf/den)	101.8	210.89	197.23
8	CV% Initial Modulus	42.33	33.7	52.23

From the Table 2, it is noticed that the *WTF* have lower dinear, tenacity and elongation values compared to the cotton fiber. The lower tenacity and elongation values means the fiber is finer then the other fiber.Such lower elongation values could result in fibre breakage during opening in blow room and carding processes. The initial modulus (young modulus) is the slope of this linear line and represents the stiffness of a fiber [21]. The maximum tensile force was found to be 7.36N, the initial modulus of *WTF* is 197.36 (52.23%) significantly higher than the cotton fibres 101.8 (42.33%) which could be due to higher cellulose content and study results confirmed that *WTF* is one of the best alternatives for synthetic fibers.

2.3.4 Thermal analysis of *WTF*

Thermal stability studies were carried out for *WTF* as most of the natural fibres are low in thermal stability. The degradation temperature of cellulosic fibres depends on their molecular weight, polymer morphology and crystallinity [22].

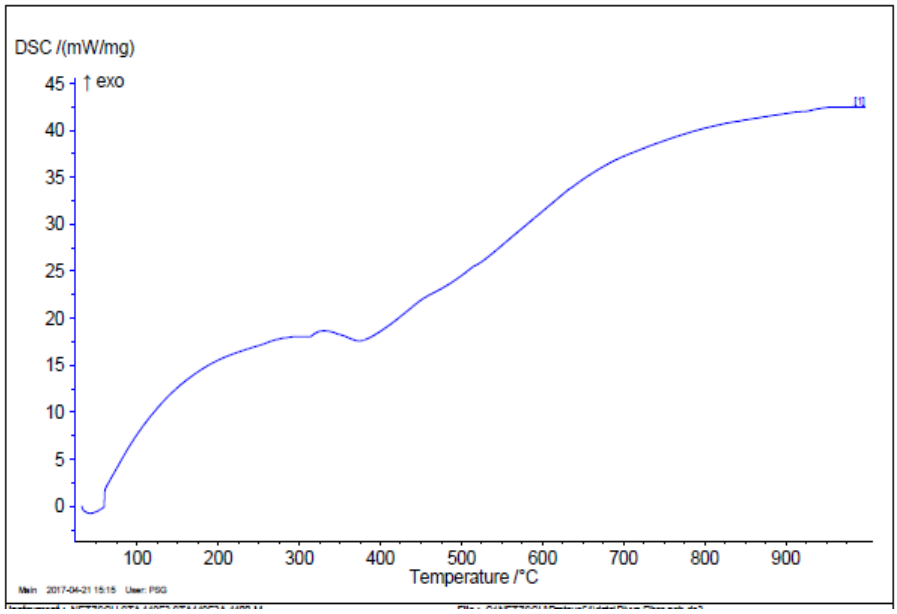

Fig 2: Thermal analysis of *WTF* fiber

Above Figure 1 (a) DSC showed board endotherm peak 971°C. that extended upto 996°C.This is attributed to the moisture present in the fiber calculated from this endotherm area was roughly around 50%. The Small endotherm observed around 350°C in this fiber is more likely attributed to the melting and volatilization of the fatty and wax materials present in the fiber.

2.3.5 FTIR spectroscopy

FTIR analysis is performed to study the organic and inorganic components present in the materials [23]. The IR spectra of *WTF* were shown in below Figure 3.

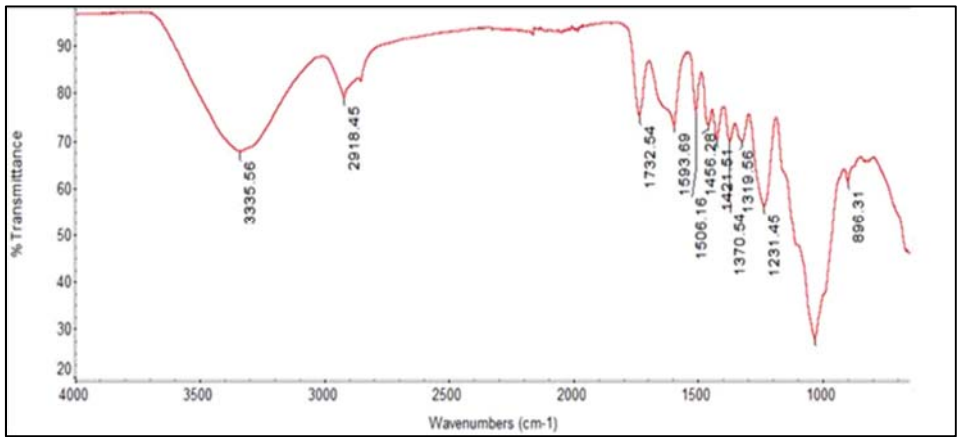

Fig 3: FTIR Analysis

The above figure 3 which illustrates the FTIR spectrum of the *WTF*, totally 11 well defined peaks were observed, broad peak at 3335.56 cm^{-1} and 2918.45, 1732.54, 1593.69, 1506.16, 1456.28,1421.51, 1370.54,1319.56, 1231.45 and 896.31 cm^{-1}. The broad peak observed at 3335.56 cm^{-1} corresponds to the stretching vibration mode of intra- and intermolecular hydroxyl (–OH) bond of cellulose[12] and the peak 2918.45 cm^{-1} corresponding to the asymmetric and the symmetric stretching of methylene ($–CH_2–$) groups in long alkyl chains. These peaks prove the presence of waxes in *WTF* [24]. The vibration located at 1,732.54 cm^{-1} is attributed to C=O stretching vibration and could originate from esters or amide [25]. the C-C stretching vibrations of the title compound are observed at 1593.69 cm^{-1} in the FTIR spectrum [26]. The peak 1506.16 cm^{-1} indicates C=C aromatic symmetrical stretching of *WTF* [27], and 1456.28 cm^{-1} showing the presence of aromatic group [C=C] [28]. The band at the 1421.51cm^{-1} represented the CO [29]. The peak 1319.56 cm^{-1} proved the presence of alcohols, carboxylic acids, esters, ethers and aliphatic compounds [30]. The peak seen at 1370.54 cm^{-1} indicates the in-plane bending vibrations of the CH_2 and CH groups of cellulose [12]. The CO group stretching in hemicelluloses was observed at 1231.45 cm^{-1} [14]. The crystalline structure and the band at 896.31 cm^{-1} is attributed to CH deformation in the amorphous region of the cellulose [28].

2.3.6 X-ray Diffraction spectroscopy of *WTF*

XRD studies were done to determine the crystallinity index which measures the orientation of cellulose crystals in a fibre axis and the patterns are shown in the below figure 4.

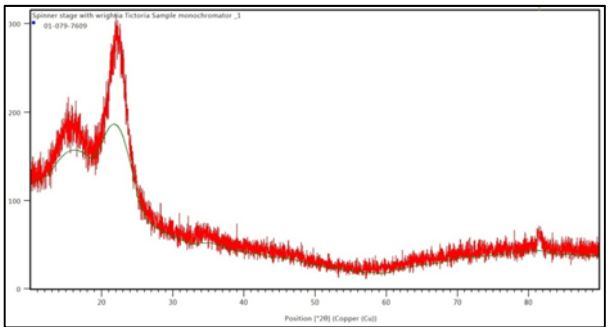

Fig 4: X-ray diffraction (XRD) spectroscopy

The X-ray diffraction (XRD) is a technique of structural analysis based on the diffraction of X-rays on a sample. It allows determining the micro and poly crystalline phases of materials. The X-ray diffractogram of *WTF* sample is shown in figure 4. This diagram represents two main peaks. The first peak at 2θ = 21.9° corresponds to the crystallographic plan [0 0 2] of cellulose I, and the second one, observed around 2 θ = 15.9°, corresponds to the amorphous fraction. The calculated crystalline index (CrI) is 62.22 %. and Crystalline size is 2.55nm. The *WTF* have a lower crystalline index and lower Crystalline size compared to other fibers. The lower crystalline fraction due to the lower amount of cellulose compared with the other lingo cellulosic fibres: 75% for sisal, 83% for cotton, 63.5% for jute, 72.4% for ramie, 71% for flax and 63.5-82.2% for hemp [31].

2.3.7 FESEM Analysis of *WTF*

The surface morphological Study of raw *WTF* was done by using FESEM analysis and the SEM image, as shown in figure 5.

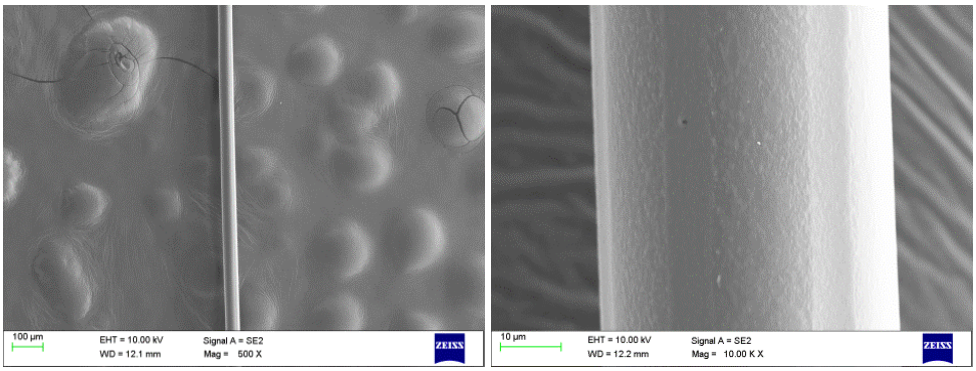

Fig 5: FESEM *(Wrightia tinctoria* fiber) at magnification of 100µm and 10 µm

Above figure 5 It is obvious that the *WTF* are typically cylindrical with an outer diameter of 51.08 µm. According to the SEM observation, the thin lines with tiny holes on the entire surface of the fiber. The small ridge on the entire fiber surface shows the presence of lignin, celluloses, and trace amount of impurities that cover the cellulose [12]. It can be seen traces of waxy materials and some damage in the fiber surface due to manual extraction process [32].

Conclusion

The, complete characterization of natural *WTF* were studied. The obtained results showed in composition test the higher cellulosic content of *WTF* which shows better strength properties and it is hydrophilic and low lignin contant which means the fiber is soft compared with other seed fiber. Physical properties show this fibre has good tensile strength and low elongation compared with other seed fiber. Length and diameter of the fiber indicates that this fibre can be quit difficult to made 100% of *WTF* yarn. Thermal stability DSC measurement reveals that *WTF* has unique property specifically capable of with standing high temperature The result of XRD shows that semi – crystalline nature of the fibre. The degree of crystallinity was found out as crystalline index (CrI) is 62.22 %. and Crystalline size is 2.55nm. *WTF* showed the major crystallinity degree because it does not have hemicellulose as it was seen by FTIR analysis. By considering the specific properties of *WTF* has good thermal insulation and low density light weight it can be utilizes for varies technical application towards the field of technical textile product development. Due to the limited number of studies reported, there are various aspects of *Wrightia tinctoria* fiber research and development that remain open for further work.

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