Extraction and Characterization of Natural Cellulosic Fiber from Coccinia Indica Stem for Polymer Composites

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Because of its low weight and environmentally benign characteristics, the primary emphasis of this work is the potential use of Coccinia Indica (CI) fiber as reinforcement in structural polymer composite materials. The dried stems were combed with a metal teeth brush to remove the fibers. A thorough analysis was conducted on the morphological structures, chemical compositions, and thermo-physico-mechanical properties of CI fiber. Chemical examination confirmed that CI fiber had a larger proportion of cellulose and a lower percentage of sparse lignin, hemicellulose, ash, and wax. Through FTIR analysis, the functional groups and their corresponding peaks were shown. According to XRD tests, the CI fiber has a crystallite size (L) of 5.81 nm and a crystallinity index (CrI) of 53.03%. The fiber has a multicellular structure and a cylindrical form, according to SEM analysis. It was also established that the CI fiber's cell wall contains parenchyma and chlorenchyma tissues, which improve the composite's mechanical qualities and adhesion. The main temperature range for CI fiber breakdown, according to TGA research, is 204.16°C to 376.3°C. The cellulose-based CI fiber has a tensile strength that is on par with other conventional fibers and suitable for use as a reinforcement material in composite construction.

KEYWORDS
Coccinia Indica fiber; technical properties; morphology; reinforcement; conventional fibers; medicinal plant

Introduction

For the creation of composites, researchers are currently mostly concentrating on biodegradable materials, or natural fiber, which has many benefits including being safer, more affordable, recyclable, and free of health hazards. Because of its benefits, including low cost, low density, acceptable specific strength, and high thermal characteristics, cellulose-based fibers were employed as reinforcing elements in bio-composites instead of traditional reinforcement materials (Thakur, Thakur, and Gupta 2014; Harish et al. 2009). The mechanical qualities of a plant are determined by its maturity and the extraction procedure used (Mohanty, Misra, and Drzal 2001). Therefore, scientists focus on natural fibers that have the potential to be reinforced using either a thermo-set or thermo-plastic matrix. Examples of these fibers include coir (Dicker et al. 2014), sisal (Li, Mai, and Ye 2000; Oksman et al. 2002), flax (Goutianos et al. 2006), jute (Ahmed and Vijayarangan 2007; Mohanty and Misra 1995; Sever et al. 2012), and hemp (Madsen et al. 2007; Shahzad 2011). Because natural fibers have limited ranges, identifying them has become difficult for researchers working in the bio-composite material sector. For example, from the past five years, cellulose-based fibers such as cissus quadrangularis root fiber (CQRF) (Indran, Raj, and Sreenivasan 2014), manicaria saccifera (Porras, Maranon, and Ashcroft 2015), arundo donax (Fiore, Scalici, and Valenza 2014), ferula communis (Seki et al. 2013), sansevieria ehrenbergii (Sathishkumar et al. 2013), polyalthia cerasoides (Jayaramudu et al. 2009) and calotropis gigantea (Ganeshan et al. 2018) have been recommended as remarkable alternatives to reinforce novel composites. Urtica dioica natural fiber was investigated and reported that it can be used to fabricate eco-friendly natural fiber-reinforced composite (Bodros and Baley 2008). Composites are extensively used in the automobile industries (Thakur, Thakur, and

Gupta 2014). It was discovered that a manufactured home phone stand made of natural banana fiber and epoxy resin was environmentally beneficial. This technology will also be used to create affordable home appliances (Sapuan and Maleque 2005). Recent work suggests that natural fiber composites are important, and lightweight composites have been made using low-density natural fibers. In this study, the Coccinia Indica (CI) fiber's thermo-physico-mechanical characteristics and morphological characterizations are examined in an effort to present them as a novel natural reinforcement for composites.

Materials and experiments

Extraction of fiber

The matured CI plants were collected from Kanuvukarai Village, Coimbatore District, Tamil Nadu State, India, which is a medicinal plant under the family of Cucurbitaceae (Ajithabai et al. 2011; Aliet al. 2008; Sivasankari, Anandharaj, and Gunasekaran 2014). Figure 1 shows the CI stems and extracted fibers after the microbial degradation process. In this process, fibers were separated fromthe CI plant stem and it was dipped into the water for 3 weeks to allow for microbial degradation. Then, the soaked stems were cleaned with freshwater and dried in the open atmosphere for a week.

Figure 1. Fiber extraction process (a) sliced CI stems immersed in water (b) CI stems in water after 3 weeks (c) extracted fibers.

Finally, the fibers were extracted from the dried stems by combing process with the metal teethbrush.

Chemical–physical property analysis

To compute the maximum tensile strength of the single fiber, tensile tests were carried out on the extracted fiber prepared as per ASTM D3822 M14 standard. INSTRON 5500R tensile testing machine with the aid of 1 kN load cell was employed in this study. During testing, 50 mm gauge length specimen was subjected to a crosshead speed of 5 mm/min, and the entire test was performed under room temperature. The chemical analysis was done to determine the content of cellulose, lignin, wax, moisture, and density as per the standards (Boopathi, Sampath, and Mylsamy 2012).

Fourier transform infrared (FTIR) analysis

To figure out the functional groups in CI fiber, Fourier Transform Infrared (FTIR) analysis was carried out using Perkin Elmer RXI FTIR tester with the scanning rate of 30 scans per minute ata resolution of 5 cm^{-1} in the wavenumber region of 500–4000 cm^{-1}. The fine powder was prepared using ball miller from the chopped samples and then mixed with KBr. Then, the powder was pelletized by applying pressure to prepare the specimen for FTIR spectra under standard conditions.

X-ray diffraction (XRD) analysis

The crystallinity index (CrI) of CI fiber was calculated using powder X-Ray Diffraction (XRD) method with the help of X'PERT PRO diffractometer. The parameters used in XRD

analysis are monochromatic Cu-Kα radiation with the wavelength of 0.154 nm, current of 30 mA, copper anode and the signal absorption ranges between 10°C and 80°C.

Thermogravimetric-difference thermogravimetric (TG-DTG) analysis

Thermogravimetric (TG) and Difference Thermogravimetric (DTG) analysis was performed on the CI fiber samples using Jupiter Simultaneous Thermal Analyzer (Model STA, NETZSCH). Nitrogen inert gas was used to avoid the oxidation of samples during testing. The sample was kept on the ceramic crucible to establish proper contact between the thermocouple and CI fiber samples. The heating rate of 10°C/min over a range of 30–600°C was utilized in this experiment.

Scanning electron microscopy (SEM) analysis

Scanning Electron Microscopy (SEM) analysis was carried out to reveal the surface morphology of the CI fiber. SEM analysis was carried out using the CARL ZEISS model V18. Gold sputtering was done on the surface of samples before SEM analysis to avoid electrical charge accumulation.

Results and discussions

Chemical–physical property analysis

The chemical analysis was performed on the CI fiber and the results are tabulated in Table 1. Generally, the presence of cellulose and lignin content offers good tensile property and rigidity to the fiber, respectively. In this study, the tested CI fiber contains high cellulose and lignin contents with low wax and ash contents. Especially, the cellulose percentage makes an impact on mechanical properties of the fiber (Indran, Raj, and Sreenivasan 2014). Mechanical properties were determined from randomly selected 20 samples of a given bundle of fibers, and the average tensile strength

Table 1. Chemical-physical properties of the CI fiber in comparison with other cellulose-based natural fibers.

Fiber	Cellulose (%)	Hemi-cellulose (%)	Lignin (%)	Wax (%)	Moisture (%)	Density (kg/m^3)	Elongation (%)	Tensile strength (MPa)	Ref.
CI	64.56	14.09	12.55	0.25	7.27	1370	4.46	645	Present work
CQRF	77.17	11.02	10.45	0.14	7.30	1510	3.57–8.37	1857–5330	[16]
Arundo Donax	43.20	20.50	17.20	-	-	1168	3.24	248	[18]
Ferula Communis	53.30	8.50	1.40	-	-	-	4.20	476	[19]
Sansevieria Ehrenbergii	80.00	11.25	7.80	0.45	10.55	887	2.80–21.70	50–585	[20]
Polyalthia Cerasoides	64.50	22.70	12.60	-	-	-	2.50	44	[21]

values are also tabulated in Table 1. Chemical and physical properties of the CI fiber were compared with other natural fibers, and it clearly depicts that tensile strength of the cellulose-based CI fiber is enough for its usage as reinforcing material in composite manufacturing.

FTIR analysis

The FTIR analysis to determine the functional groups present in the CIF was conducted on the prepared samples and the results are presented in Figure 2 and Table 2.

XRD analysis

The XRD result depicts in Figure 3 inferred that the couple of well-defined peaks could be observed. The peak at $2\theta = 22.37°$ (200) clearly indicates the cellulose content and the peak at

2θ = 15.66° (100) shows the typical non-cellulose materials which could be lignin, wax, etc. Commonly, CrI was used

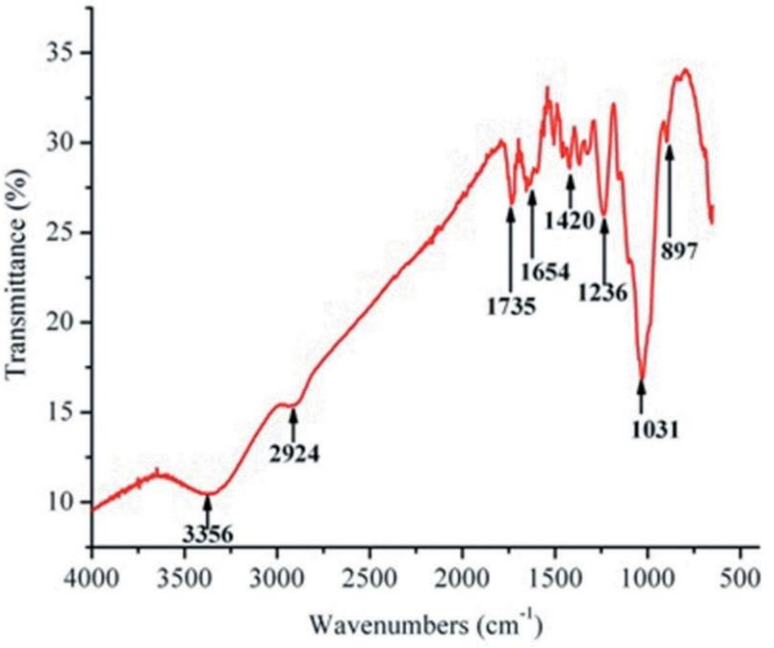

Figure 2. FTIR spectrum of CI fiber.

Table 2. Functional groups and their corresponding peaks (cm^{-1}) of CI fiber.

Bond/stretching	CI fiber peak (cm^{-1})	Assigned functional groups	Ref.
O-H	3356	Presence of cellulose	[16]
C-H	2924	Vibration of cellulose	[29]
C = O	1735	Hemicellulose	[17]
C = C	1654	Lignin, calcium pectates and water	[30]
C-O	1236	Cellulose and hemicellulose	[31]
C-O-C	1031	Modes of hydroxyl and cellulose	[32]
C-OH	897	Bending in cellulose	[33]

Figure 3. XRD spectrum of CI fiber.

to measure the crystalline of cellulose with respect to the amorphous materials. The following expression was employed to compute the CrI (Segal et al. 1959) of CI fiber.

$$CrI = \frac{(I_{002} - I_{AM})}{I_{002}} \times 100 \quad (1)$$

CrI denotes the relative degree of the crystallinity, I_{002} refers the maximum intensity, and I_{AM}represents the minimum intensity. CrI of the CI fiber was found to be 53.03%. The crystallite size (L)of CI fiber was determined by Scherer's equations (Park et al. 2010) and it was found to be 5.81 nm.

$$L = \frac{k\lambda}{\beta \cos\theta} \quad (2)$$

where k is Scherer's constant (0.94), β is the peak's FWHM (field width at half maximum) and λ is the wavelength of the radiation.

TG-DTG analysis

Thermal characteristics of the CI fiber were studied using TG and DTG curves obtained from the thermogravimetry analysis as shown in Figure 4. It is inferred from the figure that the degradation of CI fiber occurred in different stages. The first stage (up to 69.16°C) shows the loss of moisture anda mass change was found to be 9.1% as predicted (Moran et al. 2008). Similarly, mass change of 19.26% was found at next peak shown from 69.16°C to 204.16°C, where pyrolysis of lignin is initiated (French 2014). The third stage (between 204.16°C and 376.3°C) is the degradation of cellulose and part of lignin with the mass change of 51.02% (Silverio et al. 2013), similar peaks were observed at 321°C, 309.2°C, and 308.2°C for bamboo, kenaf, and hemp fibers, respectively (Yaoet al. 2008). The reason for major mass change in the third stage is due to the degradation of cellulose which is higher in the CI fiber. In this stage, the chemical analysis shows the higher amount of cellulose in the temperature above 376.3°C is due to breaking of aromatic structure present in the lignin, dehydration and methane gas release were taking place simultaneously. In the final stage, the temperature ranges above 376.3°C, the degradation of part of cellulose and lignin occurs and themass change was observed to be 7.52%.

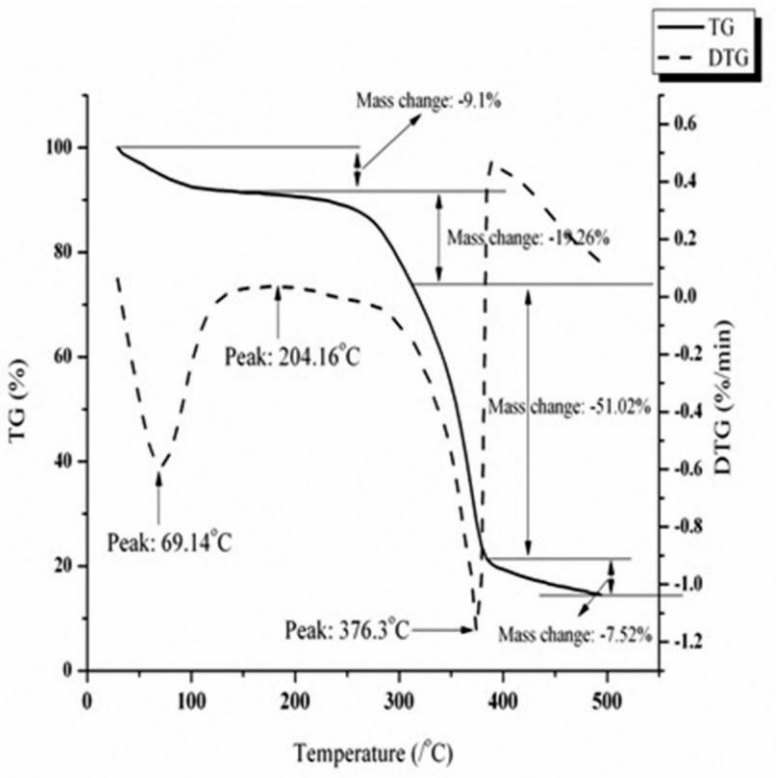

Figure 4. TG/DTG curves of CI fiber.

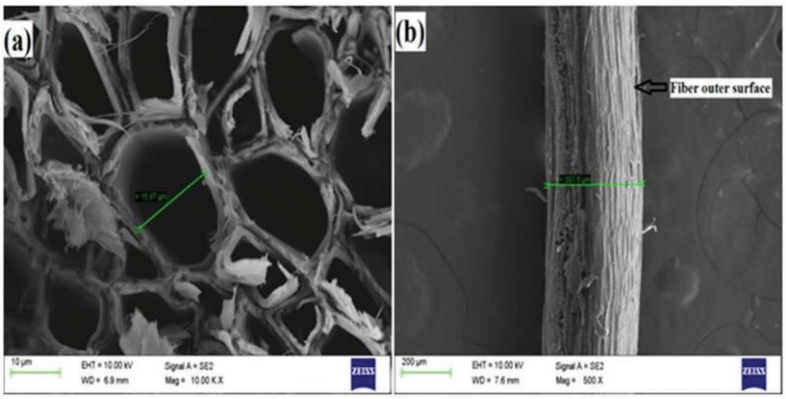

Figure 5. SEM images of single CI fiber at a magnification of (a) 10 µm and (b) 200 µm.

SEM analysis

It is inferred from Figure 5(a) that the CI fiber possesses a multi-cellular structure with a diameter of 15 µm approximately. Moreover, from Figure 5(b), it is observed that the CI fiber is soft in nature and looks like a cylindrical shape with a diameter of 390 µm approximately. The cylindrical morphology in the natural fiber increases the specific area and thus favors the chemical processes. Similarly, it is observed from Figure 5(b) that the functional groups of parenchyma and chlorench- yma tissues were present in the cell wall of the CI fiber plant stem which offers better adhesion and mechanical properties to the composite.

Conclusion

Investigation on the thermo-physico-mechanical properties, chemical compositions and morpholo- gical structures of CI fiber was presented to evaluate the possibility of using them as reinforcement in polymer composite materials. The chemical analysis and spectral analysis results showed that the higher percentage of cellulose content (64.56 wt.%) was present in the CI fiber and the rest includes lignin, hemicellulose, wax, and moisture. The mechanical properties with a crystalline index of 53.03% and a crystallite size of 5.81 nm are comparable with the other conventional fibers. The thermal analysis results indicate that the CI fiber is stable up to 204.16°C, which is higher than the polymerization process temperature. The density of CI fiber is significantly lower (1.37 g/cm^3) in comparison with synthetic fibers such as E-glass fiber (2.5 g/cm^3). Therefore, CI fiber could be used as a reinforcement material in structural polymer composites because of being lightweight and eco-friendly.

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