Cellulose
https://doi.org/10.1007/s10570-024-06211-1

ORIGINAL RESEARCH

Characterization of Ligno-cellulosic fibers from Wild turmeric (*Curcuma aromatica*) petiole for potential textile applications

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Received: 22 March 2024 / Accepted: 2 October 2024

Abstract The rhizomes of the wild turmeric plant are harvested while the aerial part is discarded as waste. The current study aims to extract fibers (WTPF) from the petiole portion of the wild turmeric plant by manual (mechanical) retting using metal comb. The fiber extraction is carried out at two stages: the first extraction yielded long and strong fibers (WTPF-1) ranging in length between 20 and 40 cm and the second extraction from the residual biomass yielded comparatively shorter fibers (WTPF-2) ranging in length between 1 and 5 cm. The extracted fibers are evaluated for physical, chemical, thermal, structural and morphological properties. The diameter of both the fibers is observed to be 20 µm and the tensile strength is 2.17 Mpa. The fibers exhibited significant difference in their properties, say the hemi-cellulose content, elongation at break, density, young's modulus while the cellulose, lignin, wax, pectin, ash and moisture content did not vary much. Based on the properties, the WTPF fiber has potential application in technical textiles field as it could be made into non-wovens and composites.

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Keywords Agro waste fiber · Wild turmeric fiber · Circular economy · Natural fiber · Non-woven · Composites

Introduction

Increasing environmental awareness in the society opens a new horizon for the search of eco-friendly materials. Hence, natural fibers are preferred by researchers (Khan et al. 2019). Plants are good sources of natural fibers, especially the ligno-cellulosic fibers. Natural fibers could be obtained from various plant parts such as the root, stem, bark, leaf, petiole, fruit, seed etc (Vijay et al. 2018). Generally, the factors that influence the properties of plant fibers are the geographic location, the plant part from where the fibers are obtained (Kumar et al. 2017). Fundamentally, the fibers extracted from plant parts possess cellulose and lignin in varied proportions and hence they are called ligno-cellulosic fibers (Ahmed et al. 2021). Several researchers have already proved that synthetic fibers could be effectively replaced by natural alternatives such as jute, hemp, sisal, cotton etc (Sarikanat et al. 2013). Research using agro waste has increased rapidly due to increase in demand for sustainable materials. Fibers from agricultural waste, including: maize tassel (Maepa et al. 2015) banana core stem fiber (Karuppuchamy et al. 2024) cassava bagasse (Pasquini et al. 2010) banana rachis (Zuluaga et al. 2009), corn stalks (Reddy and Yang 2005),

Published online: 17 October 2024

coconut husk fibers (Rosa et al. 2010), wheat straw and soy hulls (Alemdar and Sain 2008) have been used by researchers for various applications.

There are few multipurpose agricultural crops that can provide many finished products or produce. The main purpose of growing most important crops, including sugarcane, wheat, corn, and rice, is for their produce. Unfortunately, the quantity of produce that these crops yield looks small in comparison to the waste or residues that they produce. For example, there is far less corn harvested than the residue or byproducts of leaves, husks, stalks, and cobs. These remaining substances are typically burned or buried. This indicates that the crop was not grown with optimal utilization of the energy and natural resources available. Many efforts are being made to use the agricultural resources because they are abundantly available, affordable, and sustainable residue for industry (Ilangovan et al. 2018). Agricultural biomass is a good source of eco-friendly materials with several useful applications in people's daily lives. A low-density textile fiber extracted from the core part of the banana pseudostem is extremely soft and feels like cotton. Because they are nontoxic, biodegradable, environmentally friendly, and produce less pollution and waste, natural fibers are attracting a lot of attention from researchers (Karuppuchamy et al. 2024).One such source of natural fibers is the stalk or petiole of the wild turmeric plant. Many types of turmeric plants are available: *Curcuma longa* (common turmeric), *Curcuma aromatica* (wild turmeric), *Curcuma caesia* (black turmeric), *Curcuma amada* (mango ginger), *Curcuma angustifolia* (Indian arrowroot) (Jyotirmayee and Mahalik 2022).Wild turmeric is a medicinally important species belonging to the Zingiberaceae family (Promod et al. 2018) known in Ayurveda as Vanaharidra. It is widely used as coloring as well as flavoring agent. It is also used in many traditional medicines in many countries of Southeast Asia (Umar et al. 2020).

Curcuma aromatica is a well-known medicinal herb with powerful antibacterial properties. In Chinese medicine, it is said to help both prevention and treatment of cancer. *Curcuma aromatica* is commonly used to eliminate cell build-up like cancer. It contains aromatic essential oils that reduce inflammation, platelet aggregation and the removal of excess lipids from the circulatory system (Rachana et al. 2014). The present study investigates natural fibers from *Curcuma aromatica* (wild turmeric) plants collected in Tamil Nadu, India. The wild turmeric plant and its parts are shown in Fig. 1. The stalk grows to approximately 20–30 centimeters tall and is topped with enlarged pink-tipped colored bracts. Fully mature plants can grow high up to 40 cm. Fiber is extracted from the petioles of *Curcuma*

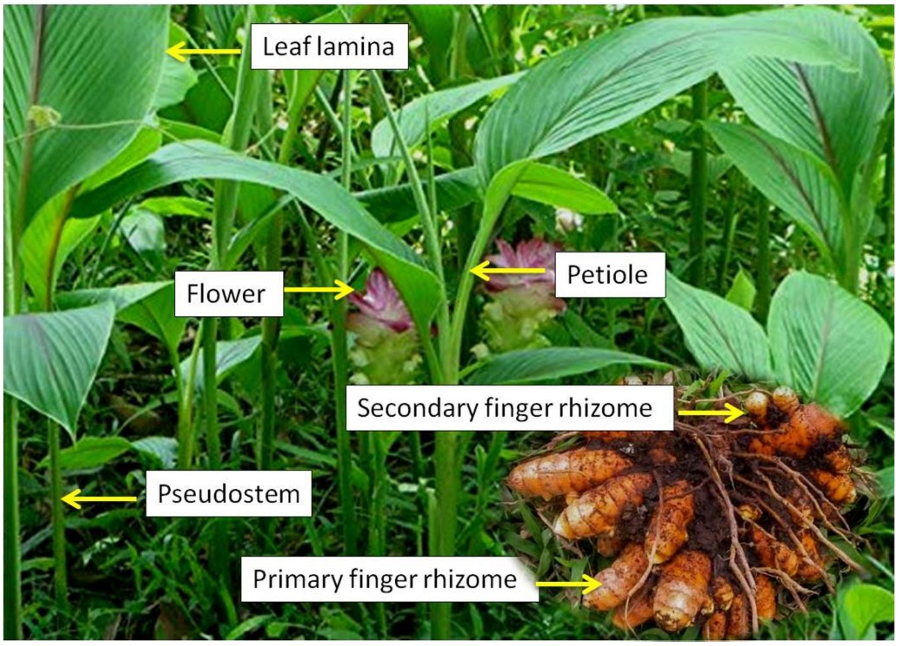

Fig. 1 Parts of wild turmeric plant

aromatica plants. The fiber is evaluated for physical, chemical, morphological, structural (FTIR and XRD), and thermal (DSC and TGA) properties.

Materials and methods

The rhizomes of the *Curcuma aromatica* (wild turmeric) plants are harvested for use, while the aerial plant parts are discarded as waste, resulting in a huge biomass. The wild turmeric biomass is collected from an organic farm [Thirumoorthy Organic Farm] at Sathyamangalam, Tamilnadu, India. The geological coordinates are 11.50445, 77.24456.

Extraction of WTPF

Natural fibers are generally extracted from the plant sources by various retting methods such as mechanical, chemical or water retting (Kommula et al. 2013). In the present study, the fibers present in the petioles of wild turmeric plant are extracted by manual retting using metal comb. Then the fibers are dried in the shade so that excess moisture content is removed from the fibers. The long fibers acquired in the first extraction are referred to as WTPF- 1 and the re-extracted, comparatively short fibers are called WTPF-2. Fig. 2 shows the method of fiber extraction from the petiole of *Curcuma aromatica*.

Physical and mechanical properties of WTPF

Using a calibrated metal ruler the length of the WTPF-1 and WTPF-2 has been measured. To prevent elongation during measurement, the fiber is properly straightened at right tension on an even surface. The findings are given in centimeter. A scanning electron microscope (FESEM) is used to measure the fiber diameter. By analyzing a fiber's different regions, the average value can be determined. Standardized, in house test method (SITRA/CP/01-2015) has been used to determine the density of the fibers.

The fibre fineness (Denier) of the WTPF-1 and WTPF-2 is tested according to SITRA/CP/01-2015 (Cut & Weigh Method). Single fibre strength and elongation of WTPF-1 and WTPF-2 are tested using Zwick/Roell tensile strength tester machine. The machine's working principle is constant rate of traverse (CRT) where the preconditioned fiber sample is held between the two jaws. The tensile strength test was performed for 20 single fiber samples. The test settings are: uniform gauge length of 50 mm, Preload: 0.5 N, Young's modulus speed: 1 mm/min, test speed 30 mm/min. The parameters such as mean breaking strength Fmax (MPa), Tenacity (g/den) and Youngs modulus Emod (GPa) are measured and recorded.

Fig. 2 WTPF extraction process **a** wild turmeric plant **b** Petiole **c** fiber extraction **d** WTPF-1 fiber **e** WTPF-2 fiber

Chemical composition

Basically, ligno-cellulosic fibers are composed of cellulose, hemicellulose, lignin, pectin, wax, ash, and moisture. The chemical composition of WTPF is found by following methods: Cellulose Content (SITRA/TC/FCC/01), Lignin Content (SITRA/TC/FCC/02), Wax Content (SITRA/TC/GT/09); Ash Content (on dry weight basis) (IS 199), Moisture Content (IS 199), Pectin (SITRA/TC/FCC/04), Hemi Cellulose (SITRA/TC/FCC/05).

Morphological analysis

The morphology and the cross section of WTPF are observed under the field emission scanning electron microscopy (FESEM) by the instrument FEI Quanta 250 Field Emission Gun SEM. High resolution images obtained can be used to find the structure of the fiber and arrangement of fibrils. The fiber specimen is coated with nano scale conductive material as a protection against high energy electron beam.

Structural properties

FTIR provides the absorption or emission spectrum of a material in the form of solid, liquid or gas. This provides the chemical nature of the material under analysis. The fiber samples are cut into small pieces and made into the pellet using KBr disk sample method. Then it is analyzed in the wavelength ranging from 500 cm^{-1} to 4000 cm^{-1} using Shimadzu IR Prestige 21. As is the case with most natural fibers, the WTPF has both crystalline and amorphous regions in its structure. The measurement of crystallinity index of the fiber can be calculated from the diffracted peaks obtained by the XRD analysis. Bruker Eco D8 is used for the current analysis with following parameters: X-ray tube generating monochromatic diffracted Cu–Kα radiation at wavelength of λ=1.54 Å, the scan range maintained between 5º ≤ 10º≤ 90º (2θ) and scan speed of 5º/min. The accelerating voltage is maintained at 40 kV along with 30 mA electric current.

The crystallinity Index (CI) is calculated using equation (Segal et al. 1959; Park et al. 2010)

$$CI = 100 \times \left(I_{200} - Iam\right)/I_{200} \quad (1)$$

where I_{200} represents the intensity of the 200 peak, and Iam represents the amorphous content, measured at the minimum intensity between the 110 and 200 peaks, at about 18.6°. The crystallite size (CS) was calculated using the Scherrer (1918) equation, equation 2

$$CS = \frac{K\lambda}{\beta \cos\theta} \quad (2)$$

where Scherrer's shape factor K=0.89, β represents the peak full width in radians at half maximum height, and λ is the wavelength of the radiation. θ is half of the 2θ angle.

Thermal properties

Thermogravimetric analysis and Differential scanning calorimetry (TGA and DSC) are used for analysis of thermal properties of WTPF. The analyzer (Netzsch STA model 449F3) has been employed for the current study of TGA and DSC. The powdered fiber sample is kept in an alumina pan and then heated over a temperature ranging from 30°C to 550°C under nitrogen atmosphere.

Results and discussion

The basic properties of any material reveal its characteristics which in turn gives an idea about the suitability of the material for a specific end use. Testing and evaluation help in assessing the characteristics of the WTPF.

Physical and mechanical properties of WTPF

The physical properties such as density and diameter of WTPF are summarized in Table 1. The fiber length and diameter are measured: the long fibers measured in the range of 20-40 cm and short fibers 1-5 cm while the diameter is found to be 20μm (FESEM analysis). The density of the WTPF-1 and WTPF-2 fibers was found to be 1.31 g/cm^3 and 1.45 g/cm^3 respectively. Tenacity has been found to be 1.39 g/den and 1.56 g/den respectively for WTPF-1and WTPF-2.

Tensile strength is appropriate measurement of mechanical properties of any material. The factors that influence the tensile properties of natural fiber

Table 1 Physical and thermal properties of WTPF-1 and 2 compared with other natural fibers

Fiber	Physical properties		Tensile properties			References
	Diameter (μm)	Density (g/cm^3)	Tensile strength (MPa)	Youngs Modulus (GPa)	Elongation at break (%)	
WTPF 1	20	1.31	2.17	0.157	1.0	Current study
WTPF 2	20	1.45	2.17	0.094	1.6	Current study
Sunnhemp	20	1.22	–	–	–	Srikavi and Mekala (2023)
Red banana peduncle	150–250	0.99	440 ± 13.4	12.41 ± 13.4	1.57 ± 0.04	Manimaran et al. (2019)
Prosopis juliflora	20	0.58	570 ± 8.4	5.87 ± 1.7	1.55 ± 0.12	Madhu et al. (2020)
Saharan aloe vera	80.61	1.32	805.5	42.29	2.39	Senthamaraikannan and Kathiresan (2018)
Furcraea foetida	12.8	0.778	623.52 ± 45	6.52 ± 1.9	10.32 ± 1.6	Senthamaraikannan and Kathiresan (2018)
Musa Cavendish (Pseudostem)	–	–	700–800	27–32	2.5–3.7	Manimaran et al. (2019)
Pithecellobium dulce	~144.3 ± 46.63	0.865 ± 0.026	600	0.007	–	Manimaran et al. (2020)
Mendong Grass (*Fimbristylis globulosa*)	33.8 ± 5.6	0.89	452 ± 47	17.4 ± 3.9	–	Suryanto et al. (2014)
Acacia leucophloea Bark	–	1.38	317–1608	8.41 69.61	–	Prithiviraj et al. (2016)
Coconut Tree Primary Flower Leaf Stalk Fiber	179.08	1.11	2008–2653	1.52–2.75	1.25 – 1.12	Thirumurugan et al. (2021)
Cyperus pangorei	133.1 ± 17	1.102	196 ± 56	11.6 ± 2.6	1,69	Mayandi et al. (2016)
Acacia leucophloea	30	0.13	317 – 1608	8.41 – 69.61	1.38 – 4.24	Arthanarieswaran et al. (2015)
Phoenix sp. fiber. (Untreated)	570	1.25	348 ± 26.71	7.62 ± 1.18	–	Rajeshkumar (2020)
Coccinia grandis.L	27.33 ± 0.3789	0.12 ± 0.22	273 ± 27.74	10.17 ± 1.261	2.703 ± 0.2736	Senthamaraikannan and Kathiresan (2018)
Epipremnum aureum stem	105–150	0.65	317 – 810	8.41 – 69.61	–	Maheshwaran et al. (2018)
Dichrostachys cinera	100	0.12	873 ± 14	–	–	Baskaran et al. (2018)

Not reported

are chemical composition (higher the cellulose percentage, higher the tensile strength) the degree of crystallinity (higher the crystallinity, higher the tensile strength) of the polymeric material. The softening process reduces the lignin content which also leads to increase in the tensile strength and elongation of the fibers (Zhang et al. 2013). The tensile strength of fibers are found to be 2.17 MPa for both WTPF-1 and 2, with elongation at break of WTPF-1 1.0% and WTPF- 2 1.6%. An average Young's modulus value of WTPF-1 is 0.157 and WTPF-2 is 0.094 GPa due to deviation in fiber diameter. The tensile properties

Table 2 The chemical composition of WTPF -1 and 2 compared with other natural fibers

Fiber	Cellulose	Hemi cellulose	Lignin	Wax	Ash	Moisture content (%)	Pectin	References
WTPF 1	73.71	36.30	7.80	0.36	3.23	11.92	2.80	Current study
WTPF 2	70.95	24.07	7.12	0.40	4.56	13.70	2.47	Current study
Sunnhemp	66.31	18.60	14.90	0.93	4.16	10.55	4.75	Srikavi and Mekala (2023)
Red banana peduncle	72.90	11.01	15.99	0.32	2.79	9.36	–	Manimaran et al. (2019)
Prosopis juliflora	60.42	15.21	15.11	0.58	5.2	8.48	–	Madhu et al. (2020)
Saharan aloe vera	67.4	8.2	13.7	0.24	–	5.8	–	Senthamaraikannan and Kathiresan (2018)
Furcraea foetida	68.35	11.46	12.32	0.24	6.35	5.43	–	Senthamaraikannan and Kathiresan (2018)
Musa Cavendish (Pseudostem)	60–65	6–8	5–10	–	1.2	10–12	–	Manimaran et al. (2019)
Pithecellobium dulce	75.15 ± 0.26	10.23 ± 3.45	12.14 ± 1.56	0.22 ± 0.08	2.13 ± 0.56	6.24 ± 1.26	–	Manimaran et al. (2020)
Mendong Grass (*Fimbristylis globulosa*)	72.14	20.2	3.44	–	–	4.2–5.2	–	Suryanto et al. (2014)
Acacia leucophloea Bark	68.09	13.6	17.73	0.55	0.08	8.83	–	Prithiviraj et al. (2016)
Coconut Tree Primary Flower Leaf Stalk	71.7	6.03	11.57	0.12	3.31	9.03	–	Thirumurugan et al. (2021)
Cyperus pangorei	68.5	–	17.88	0.17	–	9.19	–	Mayandi et al. (2016)
Acacia leucophloea	68.09	13.60	17.73	0.55	0.08	8.83	–	Arthanarieswaran et al. (2015)
Phoenix sp. fiber. (Untreated)	61.13	12.56	19.91	0.32	7.69	10.41	–	Rajeshkumar (2020)
Coccinia grandis.L	62.35	13.42	15.61	0.79	4.388	5.6	–	Senthamaraikannan and Kathiresan (2018)
Epipremnum aureum (golden pothos)	66.34	13.42	14.01	0.37	4.61	7.41	–	Maheshwaran et al. (2018)
Dichrostachys cinerea	72.4	13.08	16.89	0.57	3.97	9.82	–	Baskaran et al. (2018)
Caribbean royal palm tree	61.67	14.52	9.15	0.21	12.12	2.48	-	Gurupranes et al. (2022)

Not reported

of WTPF are compared with other natural fibers as shown in Table 1.

Chemical composition

The chemical composition of a natural fiber greatly influences its physical, mechanical and thermal characteristics. Natural ligno-cellulosic fibers are generally composed of cellulose, hemicellulose, lignin, pectin, wax, ash and some moisture. Variations in these proportions are observed due to factors such as the fiber source, the method of extraction and the age of the fiber (Razali et al. 2015). The chemical composition of WTPF-1and 2 is compared with other natural fibers in Table 2. The assessed cellulose content of WTPF-1 is 73.71% and WTPF-2 is 70.9% which are higher than some other natural fibers. The hemicellulose content is observed to be 36.30% and 24.07% for WTPF-1 and WTPF-2. The lignin content is 7.80%

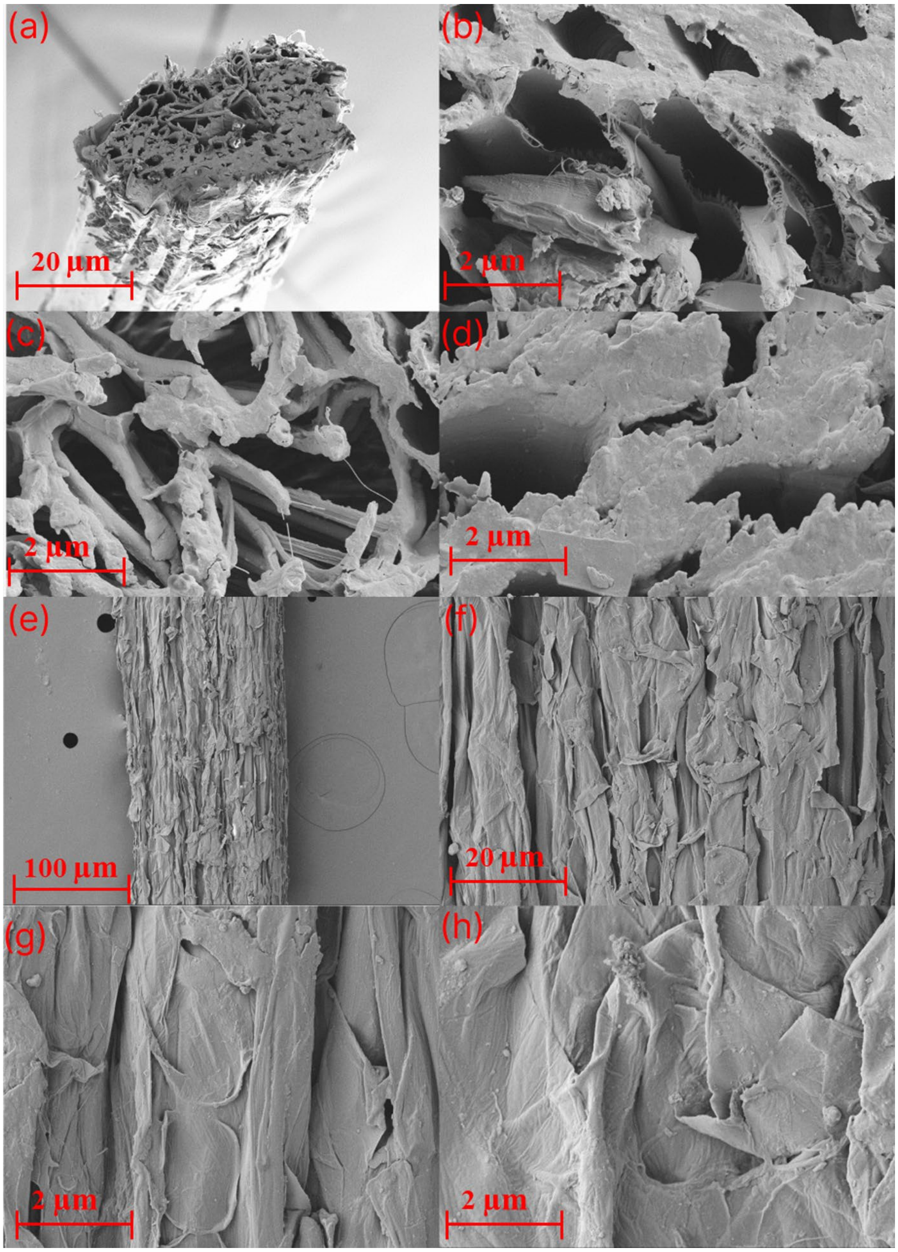

Fig. 3 Field emission scanning electron micrograph images of WTPF 1 – Cross sectional view **a** 1000X **b** 6000X **c** 10000X **d** 15000X and longitudinal view **e** 250X **f** 1200X **g** 5000X **h** 10000X

and 7.12%. The presence of lignin will also influence the fiber structure, properties and morphology of the fiber (Prithivirajan et al. 2019). The wax content is 0.36% and 0.40%. Fiber with lesser wax content is preferred for composites because it reduces the bonding characteristics of the fiber (Srikavi and Mekala 2023). The moisture content (11.92% and 13.30%), ash content (3.23%and 4.56%) and pectin content (2.80% and 2.47%) present in the WTPFs. This analysis reveals that the WTPFs will be very much useful in making lightweight composites and nonwoven.

FESEM analysis

The longitudinal and cross-sectional view could be analyzed through the field emission scanning electron microscope. The lumen that surrounds the cell wall (Fig. 3 a, b) depicts the cross-section of a WTPF-1 fibre magnified by 1000X. Multiple voids between the fibrils, some of which are wider in size, are observed in the fibers. To increase the surface area, the fibrils can be arranged firmly (Fig. 3c). Each fiber's cross section contains pores in the core (Fig. 3d). At a magnification of 250X, (Fig. 3e) depicts the surface view of a single fiber with a width of around 300 µm. Further magnifications at 1200X, 5000X, and 10000X (Fig. 3f) show the fibre surface to be scaly with fine lines and vacuoles. WTPF has more irregular surface which may give more binding if used in composites. Also, it enhances the cohesion between fibers when nonwovens are made with these fibers (Fig. 3g, h). The parallel arrangement of many fibrils that is held together as a single fibre by lignin polymer is obvious in the FESEM images.

The morphology of the WTPF-2 fibre as shown in Fig. 4 is found to be similar to that of WTPF-1. One particular fibre taken for cross-sectional study has a large void at the centre and small voids all over the fibre. Similar results have been reported by kenaf bast fiber (Amel et al. 2013). They found that all of the cross-sectional shapes of the individual fibers provided were polygonal to round. In general, bundle shape, single fiber shape, and observed lumen diameter differs depending on the extraction method. The larger lumen size contributes to higher water absorption and more moisture content of the fiber.

FTIR analysis

Fig. 5 shows the chemical groups of the extracted fibers WTPF-1 and WTPF-2. The O-H stretching of cellulose is represented by the peak that appeared at 3320 cm^{-1}, and the C-H stretching of cellulose is represented by the peak that showed up at 2919 cm^{-1}. O-H stretching in carboxylic acid and C=O stretching of the alpha-keto carboxylic acid in the lignin or ester group of hemicellulose, respectively, can be observed by peaks at 2850 cm^{-1} and 1628 cm^{-1}. The typical peak at 1023 cm^{-1} is attributed to lignin's symmetric C–OH stretching. The band at 1318 cm^{-1} is attributed to polysaccharide-derived t-butyl stretching. The C-H stretching vibration of CH in the cellulose component may be responsible for the peak identified at 2922 cm^{-1}. The fiber's cellulose exhibited CH2 stretching, which ultimately peaked at 1424 cm^{-1}.

XRD analysis

The crystalline and amorphous phases of materials may be distinguished using XRD. The XRD patterns for WTPF-1 and WTPF-2 are shown in Fig. 6. One peak is at $2\theta = 15.5°$ which is an overlapped peak arising from the (1-10) and (110) planes. These peaks have similar d-spacings and overlap because the crystallites are small and therefore broad. French and Santiago Cintron (2013) show how the 1-10 and 110 peaks overlap more and more as the crystallite size gets smaller and smaller. The peak at 22.0° has Miller indices of 200. These patterns are typical of samples with the cellulose I_{am} structure. Crystallinity index values for WTPF-1 and WTPF-2 are around 86.3% and 88%, respectively. Crystallite sizes are estimated as 3.29 and 2.7 nm for WTPF-1 and WTPF-2, respectively, comparable to flax fiber (2.8 nm) but decreased in the fiber's capacity to absorb water and its chemical reactivity of the fiber (Manimaran et al. 2016).

Thermogravimetric analysis

The thermal properties of the WTPF are investigated by using a TGA analysis under a nitrogen atmosphere. The Fig. 7a, b represents the TGA analysis of extracted fibers WTPF-1 and WTPF-2. The initial weight loss of sample WTPF-1 and WTPF-2 is due to the deoxylation of water at 100ºC. The fiber reduction in lignin and hemicellulose content caused

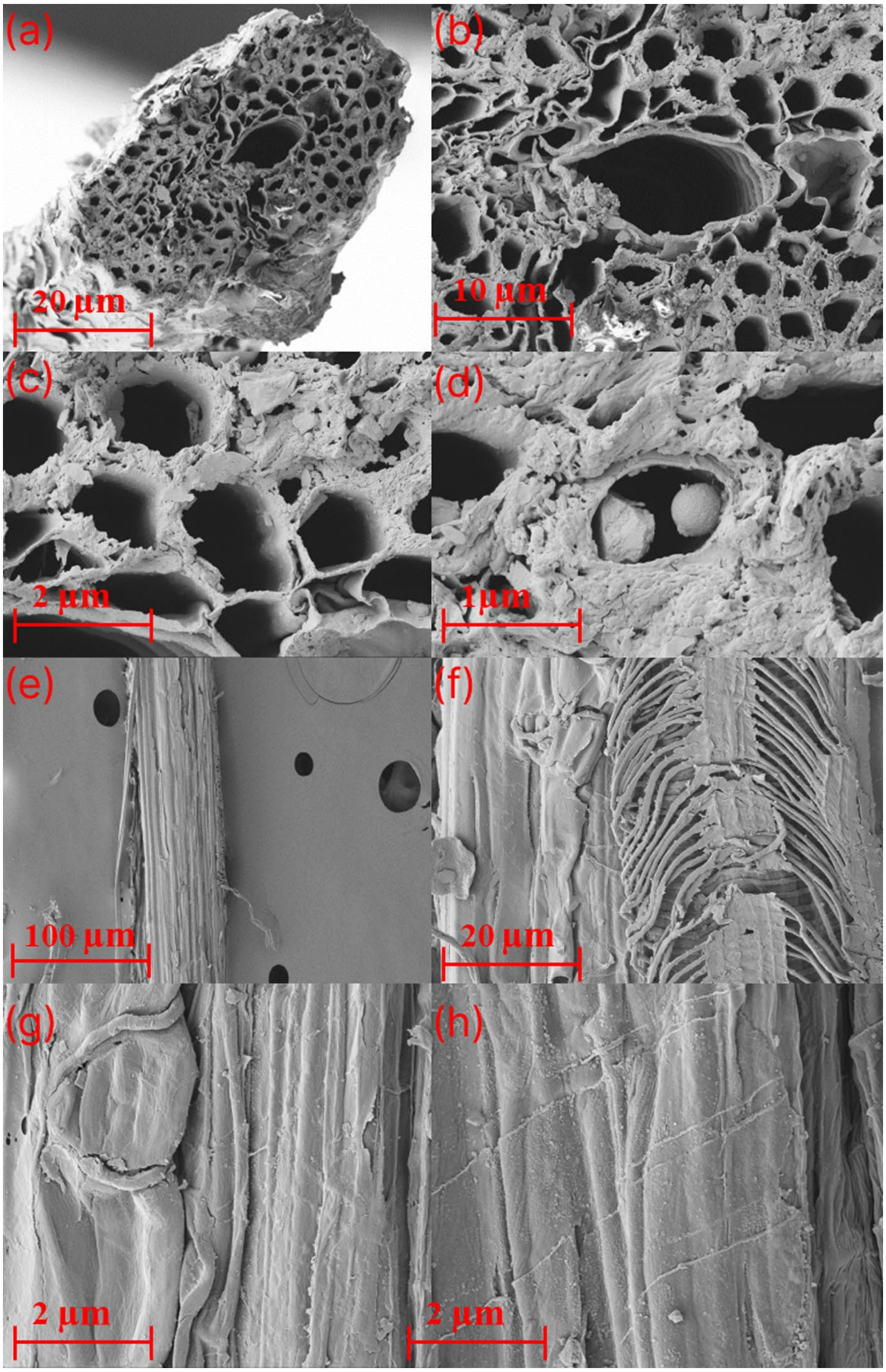

Fig. 4 Field emission scanning electron micrograph images of WTPF 2 Cross sectional view **a** 1000X **b** 2400X **c** 6000X **d** 15000X and longitudinal view – **e** 250X **f** 1200X **g** 2400X **h** 10000X

the stability of the WTPF-1 and WTPF-2 fibers to drop by approximately 56% and 74%, respectively, at 350ºC. WTPF-1 decomposes completely when the

temperature is raised to 500ºC. But in contrast to it, WTPF-2 has high thermal stability. Even at 500ºC, WTPF-2 shows superior stability than WTPF-1, with degradation reaching around 70%.

By comparing the thermal performance of the two fiber samples at a constant heat flow rate, the WTPF-1 and WTPF-2 are shown to have decreased

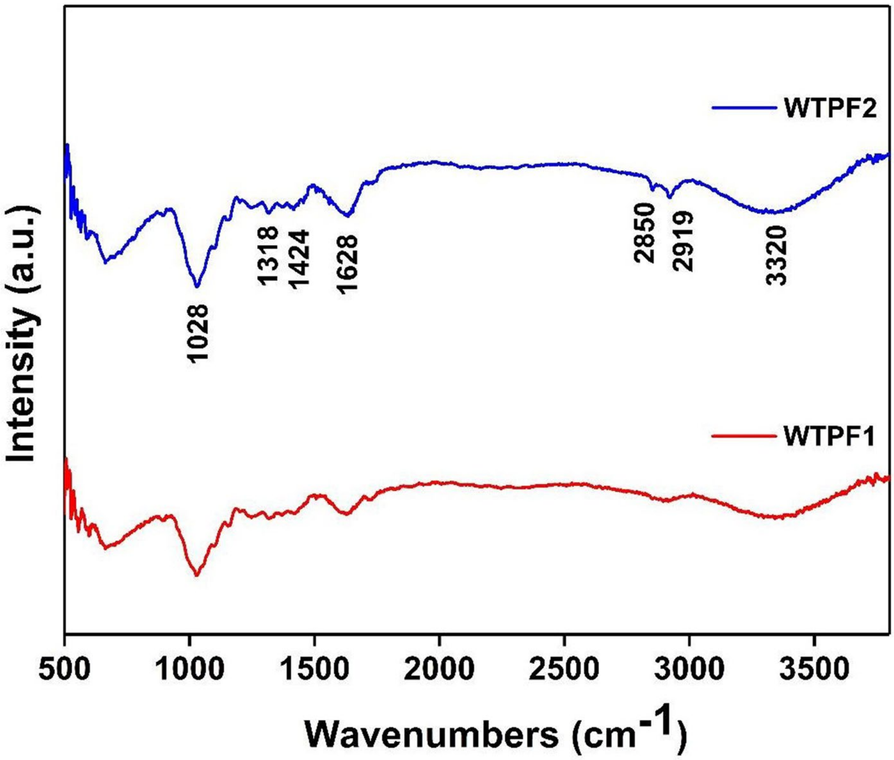

Fig. 5 FTIR spectrum of WTPF-1 and WTPF-2

crystallinity, as measured by the DSC technique. The WTPF-1 curve, in comparison with WTPF-2, was stable and at precisely 320ºC a clear high exothermic peak was observed for both WTPF-1 and WTPF-2. This peak is caused by a thermal transition that involved the breakdown and deterioration of the structural components of the fibers. The breakdown of chemical bonds in the proto-lignin in the fiber is the reason for the larger endothermic peak found for WTPF-1 at the temperature range of 520ºC. The WTPF-2 fiber has better stability than WTPF-1, with degradation reaching around 70%, whereas the TGA plot indicates that the WTPF-1 has entirely decomposed at 500ºC.

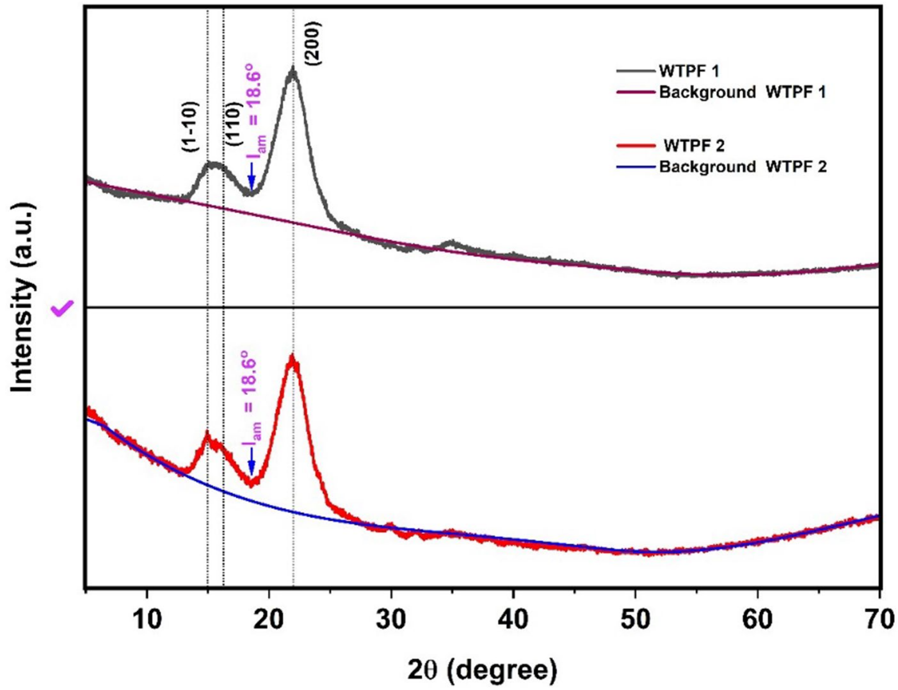

Fig. 6 X-ray diffractogram of the WTPF 1 and WTPF 2

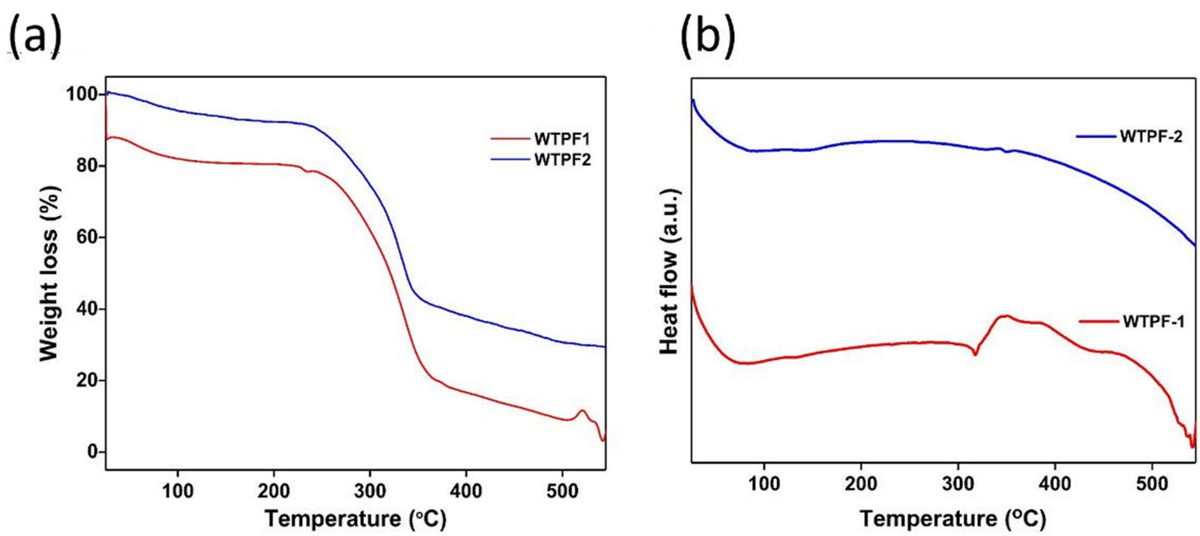

Fig. 7 **a** TGA curve **b** DSC thermogram of WTPF-1 and WTPF-2

Conclusion

The wild turmeric petiole fiber obtained from the agricultural bio-mass residue ensures circular economical approach. The higher cellulose content and better crystallinity of the fiber attributes to potential technical textile applications such as non-wovens and composites. It could be used in agro-textiles, geo- textiles, pack-textiles, acoustic textiles, hygiene textiles and as thermal insulations. The fiber is expected to be compatible with cotton and other ligno-cellulosic fibers. The authors are developing non-wovens by blending the WTPF with other natural fibers.

Acknowledgements The authors like to acknowledge the organic farmer Mr. Thirumoorthy for providing the wild turmeric plant waste for the study.

Author contribution Ramya K > > Experiment, Analysis, Original draft writing. Amutha Karuppuchamy > > Conceptualization, Supervision, Editing.

Funding The work receives no external funding from any agency.

Data availability The datasets generated during and/or analyzed during the current study are available from the corresponding author on reasonable request.

Declarations

Conflict of interests The authors have not disclosed any competing interests.

Ethical approval There are no human subjects in this article.

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