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Extraction and Characterization of Natural Fiber from the Stem of *Dombeya Buettneri* Plant for Biodegradable Polymeric Composites Application

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ABSTRACT

The global drive for a circular economy emphasizing sustainability in composite manufacturing processes has been the driving force for current ongoing research studies in natural fibers as sustainable substitutes for non-biodegradable synthetic fibers. The present study was carried out to characterize *Dombeya buettneri* fiber (DBF) extracted manually from the bark of the plant stem. Determination of physical and mechanical properties, quantitative chemical analysis, Fourier Transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), thermogravimetric analysis and scanning electron microscopy were used to characterize the extracted fiber. Fiber constituents and XRD results showed higher cellulose content (58.45%), crystallinity index (62.32%) and percent crystalline (72.63%). The fiber had a crystallite size of 2.16 nm as determined using the Debye-Scherrer's equation while FTIR analysis confirmed the presence of various functional groups of lignin, hemicellulose and cellulose on the fiber structure. The results revealed that DBF fiber had a thermal resistance that is up to 229.57°C with a maximum thermal degradation temperature of 356.27°C. Based on the results of this research that are comparable with other studies on cellulosic fiber, DBF fiber has a great potential as an alternative reinforcement for the development of polymer-based bio-composites.

摘要

全球推动循环经济，强调复合材料制造过程的可持续性，这是目前正在进行的天然纤维作为不可生物降解合成纤维的可持续替代品的研究的推动力. 本研究对人工提取的东北董贝纤维进行了表征. 采用物理力学性能测定、定量化学分析、傅立叶变换红外光谱、X射线衍射、热重分析和扫描电子显微镜对提取的纤维进行了表征. 纤维成分和XRD结果表明，纤维的纤维素含量（58.45%）、结晶度指数（62.32%）和结晶率（72.63%）较高. 使用Debye-Scherrer方程测定，纤维具有2.16 nm的晶粒尺寸，而FTIR分析证实在纤维结构上存在木质素、半纤维素和纤维素的各种官能团. 结果表明，DBF纤维的耐热性高达229.57°C，最大热降解温度为356.27°C. 基于这项研究的结果，与其他纤维素纤维研究的结果相比较，DBF纤维作为聚合物基生物复合材料的替代增强材料具有巨大的潜力.

KEYWORDS

Ecofriendly; natural fiber; sustainable; polymer composites; extraction

关键词

环保型; 天然纤维; 可持续的; 聚合物复合材料; 萃取

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Introduction

In the recent past, there has been a growing interest in the utilization of renewable and readily available materials for composites fabrication. This has been necessitated by the global drive toward circularity aimed at achieving sustainability in material production processes. Synthetic fibers majorly carbon and glass have been used to produce composite structures. Although these fibers have superior mechanical properties, they have been associated with non-biodegradability and non-renewability causing global environmental menace. To address these challenges, research is ongoing to investigate the potentials of agro-based raw materials such as natural fibers as sustainable alternatives for synthetic fibers (Khan et al. 2020a). Natural fibers are environmental-friendly, biodegradable and readily available (Bichang'a et al. 2022a, 2022b; Sanjay et al. 2019).

Natural fibers are obtained from different parts of a plant mainly the bark, stem, seed, fruit and leaves using different extraction techniques such as manual hand stripping, mechanical decortication and retting (Balogun et al. 2015, Oladele et al. 2020a; Oladele et al. 2021). Natural fiber reinforcements for composites fabrication have been considered from traditional plants such as banana, bamboo, etc (Daramola et al. 2021; Owa et al. 2021). Recently, several new fiber sources such as p*arthenium hysterophorus* stem (Vijay et al. 2019) and *acacia nilotica* L (Kumar et al. 2020) have been identified and characterized. Characterization results of these fibers have reported characteristics similar to those of traditional natural fibers like banana and bamboo (Maache et al. 2017).

This current study was carried out to evaluate the characteristics of extracted fiber from *Dombeya buettneri* plant, a naturally available plant in most African countries such as Nigeria. The bark of the stem from this plant is very fibrous and has been traditionally used to make ropes and cords to support baskets on the back as well as making nets and fishing equipment (Oladele et al. 2020b). There is scarce detailed information on the physical, mechanical and chemical properties of this fiber. Therefore, there is a need to bridge the existing knowledge gap through physical and mechanical properties measurement, Fourier Transform infrared (FTIR) spectroscopy, thermogravimetric analysis, X-ray diffraction (XRD) and scanning electron microscopy characterization. It is expected that this will lay the foundations for future research studies on the preparation of DBF fiber as a potential replacement for synthetic fibers in producing bio-composites for various applications.

Materials and methods

Extraction of *Dombeya buettneri* Fibers from the plant

Dombeya buettneri stems were cut from the plant obtained from a plantation in Akure, Ondo State, Nigeria of geographical coordinates 7.2571° N, 5.2058° E. The plant was identified in the Department of Agricultural Engineering, School of Engineering and Engineering Technology, Federal University of Technology Akure, Ondo State, Nigeria. This was followed by removal of leaves and leaf stalks from the main stem. Dombeya fibers were extracted by removing the bark from the stem, followed by manually peeling off the fibers from the bark using the hand stripping method. The extracted fibers were oven-dried at 60°C for 4 hours and then stored in airtight zip-lock polybags for further characterization as illustrated in Figure 1.

Fiber characterization

Fiber density

Fiber density measurement was conducted according to ASTM-D3800–16 standard (ASTM D3800–16 2016). The fiber sample density was calculated by dividing fiber weight in the air with computed fiber volume using Equation 1.

Figure 1. Macroscopic image of (a) *Dombeya buettneri* plant, (b) stem, (c) bark, and (d) fibers.

$$\rho_f = \frac{(m_3 - m_1)\rho_w}{(m_3 - m_1) - (m_4 - m_2)} \tag{1}$$

where ρ_f and ρ_w represent the density of fiber sample (to be determined) and density of distilled water ($\rho_w = 1\ \mathrm{gcm}^{-3}$); m_1 and m_2 are the weight of suspension wire in air and in distilled water, respectively, and m_3 and m_4 represent the weight of suspension wire and fiber sample in air and in distilled water, respectively. For repeatability and accuracy, the process was carried out five times and the average value was reported.

Fiber length

The length of the extracted *Dombeya buettneri* fiber was determined on 30 fiber samples using a meter ruler and the average length reported.

Linear density

The linear density (or fineness) of *Dombeya buettneri* fibers was determined as per ASTM D1577–07 standard (ASTM D1577–07 2012) using option A-fiber bundle weighing. From bundle weight and length, the average fiber linear density (tex) was computed using Equation (2).

$$T_d = \frac{1000W}{L\ x\ N} \tag{2}$$

where T_d represents the average fiber linear density (tex), W, L and N represent fiber bundle weight (g), length of fiber bundle (m) and number of fibers in a bundle, respectively.

Single fiber strength

The tensile strength of Dombeya buettneri fibers was determined according to ASTM D 3822–07 standard (ASTM 3822- 07 2014) on a universal testing machine (type: TH2730; model: TCTN-9110-5KN, Rycobel group, Belgium) of 5 KN load cell capacity operated at a crosshead speed of 1 mm/min. The breaking tenacity of the fibers was calculated by dividing the maximum breaking force of the fiber with the corresponding linear density (Tex) as given in Equation (3).

$$Y = \frac{F}{T_d} \tag{3}$$

where Y, F and T_d represent breaking tenacity (cN/tex), breaking force (cN) and linear density (tex), respectively.

Quantitative chemical analysis

Quantitative chemical analysis was performed to determine the percentages of each constituent present in DBF fibers following TAPPI standard methods. The samples were prepared according to TAPPI T257 cm-12 standards (TAPPI 2012). First, DBF fibers were chopped into small pieces followed by grinding using a laboratory grinder to 50–100 mesh (150–300 µm) particle sizes. The percentage of each chemical composition present was determined in triplicates and the mean values were reported. TAPPI T412 om-16 (TAPPI 2016), TAPPI T211 om-07 (TAPPI 2007a), TAPPI T204 cm-07 (TAPPI 2007b), TAPPI T222 om-06 (TAPPI 2006) and TAPPI T203 cm-99 (TAPPI 1999) standard procedures were used to determine moisture, ash, extractives, acid insoluble lignin and α-cellulose content, respectively. The percentage holocellulose content was determined following protocol established by Wise and Ratliff (Wise and Ratliff, 1947) and using Equation (4), the percentage of hemicellulose content was computed.

$$Hemicellulose\% = holocellulose\% - alpha\ cellulose\% \tag{4}$$

Fourier Transform Infrared Spectroscopy (FTIR) analysis

FTIR Spectrophotometer (FT-IR 8400S, Shimadzu Corporation, Kyoto, Japan) was used for the identification of functional groups present in DBF fiber. Powdered fibers were mixed with potassium bromide (KBr) to make the samples transparent. FTIR spectrophotometer was operated at 32 scans per minute scanning rate using 2 per cm signal-to-noise ratio resolution in 400–4000 cm^{-1} wave number region range at room temperature and relative humidity of 25 and 65%, respectively.

X-Ray diffraction (XRD) analysis

XRD analysis technique was used to study the crystallographic properties of DBF fibers using XRD diffractometer (Malvern Panalytical Aeris, Malvern, United Kingdom) with PIXcel detector and fixed slits with Fe filtered Co-Kα radiation. The XRD machine was operated at 0.02°/min scanning rate over a scattering 2θ angle range of 5° to 80° at ambient conditions. Sample crystallinity index was computed using the Segal method (Segal et al. 1959) Equation (5).

$$CrI, \% = \frac{I_{200} - I_{am}}{I_{200}} x100 \tag{5}$$

where I_{200} and I_{am} represent the peak intensity at the plane (200) and the minimum intensity at the valley between the plane (200) and (110), respectively. I_{200} represents both crystalline and amorphous elements while I_{am} represents only the amorphous elements.

Sample crystallite size was computed using Debye-Scherrer's equation (Equation 6) (Elazzouzi-Hafraoui et al. 2008) from individual XRD peaks at planes (100), (200) and (004) and the mean crystallite size (D) was reported.

$$D = \frac{k\lambda}{\beta_{1/2} cos\theta} \tag{6}$$

where k is Debye-Scherrer constant (0.91), λ is the radiation wavelength (λ = 1.79206 Angstrom = 0.179206 nm), β1/2 is the line broadening at full width at half maximum (FWHM) of the XRD peak (in rad.) and θ is the Bragg's angle of the peak (200) in degrees, half of 2θ. The full width at half maximum ($\beta_{1/2}$) in radians is given by Equation (7).

$$(\beta 1/2) = \frac{FWHM\ x\ \pi}{180} \tag{7}$$

The order of crystallite packing also referred to as percentage crystalline (P.C) or degree of crystallinity of the fibers was computed using Equation (8) (NagarajaGanesh and Rekha 2019).

$$Degree\ of\ crystallinity\ or\ P.C., \% = \frac{I_{cr}}{I_{cr} + I_{am}} x100 \tag{8}$$

where; I_{200} and I_{am} are the same as Equation (5).

Thermal analysis

Thermogravimetric analyzer, TGA (TGA4000, PerkinElmer, Massachusetts, United States), machine was used to investigate the thermal stability of DBF fibers. A powdered DBF sample weighing 10 mg was placed on an aluminum crucible placed in an oven maintained in a controlled environmental condition with 20 ml/min nitrogen gas flow rate. Using a heating temperature rate of 10°C per minute, the temperatures in an oven were increased from room temperature to 860°C. The obtained results were presented in thermograms (TG) and differential thermograms (DTG) curves.

Morphological analysis

Scanning Electron Microscopy (SEM)

Scanning electron microscope, SEM (ZEISS EVO 18, Carl Zeiss AG, Jena, Germany), with an electron beam accelerating voltage potential of 15 kV was used to examine the surface morphologies of DBF fibers at different magnifications. The conductivity of the samples was improved by coating them with a thin gold layer in a vacuum atmosphere before examination.

Energy dispersive X-ray spectroscopy (EDX)

EDX equipped with the SEM microscope was used to quantitatively identify elements such as carbon, oxygen, nitrogen, etc. present in DBF fiber.

Results and discussion

Fiber density and length

The density of natural fibers is a crucial parameter in computing mechanical performance such as the specific tensile strength of resultant bio-composites. Fiber density is influenced by compositional elements, porosity and the geometry of the fiber (Lila et al. 2020). The measured density value for *Dombeya buettneri*

fiber is 1.323 g/cm^3 which is substantially less than synthetic fibers such as carbon (1.60 g/cm^3) and E-glass (2.56 g/cm^3) (Indran and Raj 2015). The density of *Dombeya buettneri* fiber is comparable to that of sisal fibers (1.20–1.48 g/cm^3 (Bekele, Lemu, and Jiru 2022) and coconut shell (1.15–1.60 g/cm^3) (Ichim et al. 2022). The extracted *Dombeya buettneri* fiber have an average length of 55.4–70.9 cm. The fiber length was basically determined by the original length of the raw stem bark. The fiber length reported for *Dombeya buettneri* fiber in this study is lower than 98.5 cm reported for *Yucca Elephantine* leaf fiber (Azanaw, Haile, and Gideon 2019). Conversely, this fiber length is higher than 50 cm for flax (Rihouey et al. 2017); 23.1 cm for banana fiber and 22.3 cm for ramie fibers (Soraisham et al. 2022).

Tensile properties of *Dombeya buettneri* fiber

Tensile properties of textile fibers determine their ability to withstand applied tensile load before failure. The determination of tensile properties is important in assessing the suitability of textile fibers for particular applications (Azanaw, Haile, and Gideon 2019; Shaker et al. 2020). Dombeya fiber reported a maximum breaking force of 32.90 ± 6.52 N and a minimal elongation at break of 0.85 ± 0.07%. This implies that *Dombeya buettneri* fiber a low extensibility, that is, it stretches only slightly as extension increases. Similarly, *Dombeya buettneri* fiber exhibited a comparatively higher breaking tenacity of 148.53 cN/tex. The breaking tenacity of dombeya is comparable to 146.5 cN/tex reported for bamboo (Asmarea et al. 2x022). However, the reported breaking tenacity is lower as compared to other plant fibers like banana (34.86 cN/tex) and ramie (52.3 cN/tex) (Soraisham et al. 2022). Table 1 presents a comparison of tensile properties of dombeya with other cellulosic fiber.

Chemical composition of *Dombeya buettneri* fiber

Table 2 provides the quantitative chemical analysis results of DBF fibers in comparison with other cellulosic fibers. The results show lignin, cellulose and hemicellulose as the three major constituents of DBF. The cellulose content of DBF is 58.45% which is comparable with 59.15% for pigeon pea (*cajanus cajan*) pod fiber (Shyam Kumar et al. 2019) and 56.46% for *acacia nilotica L.* fiber (Kumar et al. 2020). However, the cellulose content in DBF is less than 72.4% for *dichrostachys cinerea* fiber (Baskaran et al. 2017) and higher than 55.1% for *corypha taliera* fruit fiber (Tamanna et al. 2021). The quantity of cellulose in cellulosic fibers

Table 1. Tensile properties of Dombeya and other natural fibers.

	Dombeya	Ramie	Bamboo	Castor oil fiber
Linear density (tex)	22.15 ± 0.98	0.8	5.12–95.02	4.5–13.5
Max. breaking force (N)	32.90 ± 6.52	-	1–14.46	-
Breaking elongation (%)	0.85 ± 0.07	3.85	0.26–3.43	1.2–5
Tenacity (cN/tex)	148.53	52.3	1.2–146.5	57.8–86.9
References		(a)	(b)	(c)

(a) (Soraisham et al. 2022); (b) (Asmarea et al. 2022); (c) (Belachew, Gebino, and Haile 2021).

Table 2. Quantitative chemical composition of DBF fibers and other cellulosic fibers reported in literature.

Fiber	Cellulose	Hemicellulose	Lignin	Extractives	Moisture	Ash	Reference
Dombeya buettneri fiber	58.45	11.07	14.45	4.05	8.54	3.35	PW
Corypha taliera fruit fiber	55.1	21.78	17.6	-	7.1	-	(a)
Pigeon pea (*cajanus cajan*) pod fiber	59.15	10.43	21.59	0.49	5.29	3.05	(b)
Acacia nilotica L. Fiber	56.46	14.14	8.33	-	-	4.95	(c)
Cortaderia Selloana Grass	53.7	14.43	10.32	3.1	7.6	-	(d)
Citrullus lanatus climber	53.7	12.5	10.1	3.2	14.0	2.17	(e)
Vachellia farnesiana	38.3	12.1	9.2	3.4	11.0	6.21	(f)
Dichrostachys cinerea fiber	72.4	13.08	16.89	0.57	9.87	3.97	(g)

PW – Present Work, (a) (Tamanna et al. 2021), (b) (Shyam Kumar et al. 2019), (c) (Kumar et al. 2020), (d) (Khan et al., 2020b), (e) (Khan et al., 2020a), (f) (Vijay et al. 2020), (g) (Baskaran et al. 2017).

is a critical component that enhances stiffness, stability, tensile and water absorption properties thus influencing the economic production of natural fibers for different applications.

Also, DBF contains 11.07% hemicellulose, 14.45% lignin, 4.05% extractives, 8.54% moisture and 3.35% ash content. Hemicellulose is responsible for the biodegradation, thermal degradation and water absorption properties of natural cellulosic fibers (Taj et al. 2007). The proportion of hemicellulose in DBF is comparable with 12.5% for *Citrullus lanatus* climber (Khan et al. 2020a), 12.1% for *Vachellia farnesiana* (Vijay et al. 2020) and 10.43% for pigeon pea (*Cajanus cajan*) pod fiber (Shyam Kumar et al. 2019). The rigidity, thermal stability, morphology and structure of cellulosic fibers are influenced by the lignin content of individual fibers. The percentage of lignin content in DBF fibers (14.45%) is less than 16.89% for *Dichrostachys cinerea* fiber (Baskaran et al. 2017) and 21.59% for pigeon pea (*Cajanus cajan*) pod fiber (Shyam Kumar et al. 2019).

Quantitative chemical analysis of DBF fiber reported slightly higher extractives (pectin, fat, wax, gum, etc.,) content of 4.05% compared to 3.40% for *vachellia farnesiana* (Vijay et al. 2020). The low extractive proportion increases the fiber's hygroscopic characteristics while high proportions are desirable as they prevent microorganisms' formation. The moisture percentage of DBF of 8.54% is comparable with 8.6% for *parthenium hysterophorus* stem fibers (Vijay et al. 2019).

Fourier transform infrared spectroscopy (FTIR) analysis

The FTIR spectrogram of DBF fibers observed from 4000 cm^{-1} to 400 cm^{-1} is shown in Figure 2. From FTIR spectrograms, there are clear peaks ("U" bends) at 3282, 2938, 1612, 1421, 1317, 1248, 1017, 773 and 517 cm^{-1} for DBF fibers. The first "U" shaped peak observed at 3282 cm^{-1} is due to hydroxyl O – H stretching indicating the presence of α-cellulose and water alcohol (Chandrasekar et al. 2017). Secondly, a small, but sharp peak at 2938 cm^{-1} corresponds to C – H stretching and vibration of CH_2 and CH confirming the presence of hemicellulose, cellulose and other organic compounds (Kumar and Thampi 2015). The weak peak at 2168 cm^{-1} is attributed to C – C stretching. FTIR spectrum of

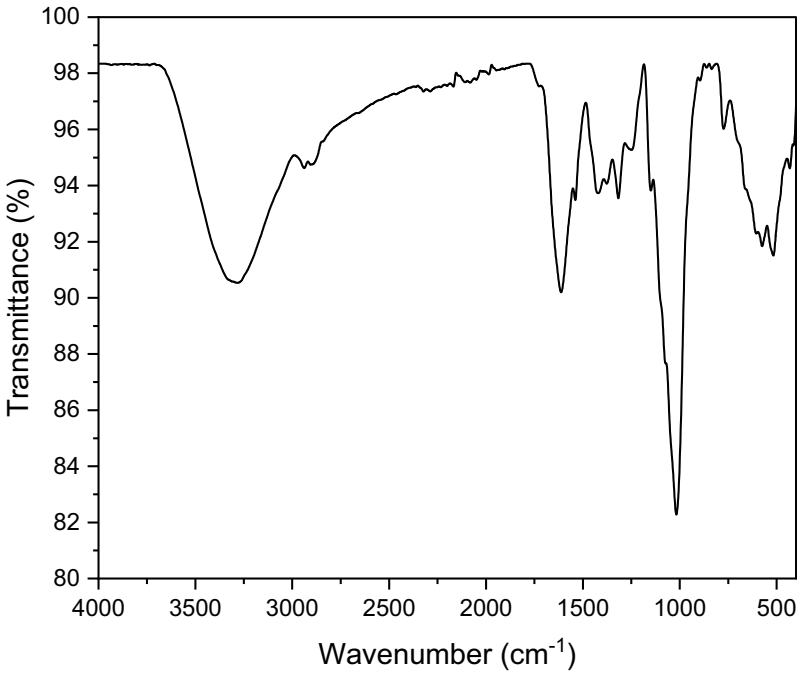

Figure 2. FTIR spectrograms of *Dombeya buettneri* fiber.

DBF has a carbonyl C=C aromatic stretching with a strong conjugated C – C bond at 1612 cm^{-1} due to lignin concentration of the fibers (Maache et al. 2017). A peak at 1421 cm^{-1} represents C – H stretching of lignin, attributed to the presence of the aromatic ring in polysaccharides (Hemmalakshmi, Priyanga, and Devaki, 2017).

From the FTIR spectrum of DBF, a C – H asymmetric and symmetric stretch vibrations of hemicellulose are found at 1317 cm^{-1}. The presence of stretch vibration of C = O and O – C–O groups of lignin is confirmed by a weak peak at 1248 cm^{-1} while the evidence of asymmetric stretching of O – C–O ester groups in cellulose is confirmed by the peak at 1017 cm^{-1} (Maache et al. 2017). Peaks at 773 cm-1, 575 cm^{-1} and 517 cm^{-1} are associated with skeletal C=C bending of cellulose (Fan, Dai, and Huan 2012). FTIR peaks, functional groups and corresponding fiber components observed in DBF are summarized in Table 3. Thus, FTIR spectrograms have been used to confirm once again the presence of lignin, hemicellulose and cellulose in *Dombeya buettneri* stem fibers.

Table 4 gives a comparative analysis indicating how the identified functional groups in DBF fibers compare with other cellulosic natural fibers reported in literature.

X-Ray diffraction (XRD) analysis

The X-Ray diffractogram of *Dombeya buettneri* stem fibers is represented in Figure 3 showing two main diffraction peaks at 2θ angles of approximately 18.12 and 25.81° corresponding to (100) and (200) lattice planes, respectively. The presence of amorphous constituents mainly hemicellulose, pectin, lignin, wax and amorphous cellulose is confirmed by the small intensity peak at 2θ = 18.12° (Manimaran et al. 2016). Whereas, crystalline constituents of DBF fiber are confirmed by the peak at 2θ = 25.81°. The two peaks are typical of natural fibers and show the presence of cellulose type I and IV (Senthamaraikannan et al. 2016).

Table 5 shows the crystallographic properties of *Dombeya buettneri* stem fibers in terms of crystallinity index, percent crystalline and average crystallite size. The percent crystalline of

Table 3. FTIR functional groups and respective fiber components in DBF fiber.

FTIR peak (cm^{-1})	Functional group	Fiber component	References
3282	O – H stretching	α-cellulose	(Chandrasekar et al. 2017)
2939	C – H stretching; vibration of CH_2 and CH	Hemicellulose and cellulose	(Kumar and Thampi 2015)
1612	C=C aromatic stretch vibration	Lignin	(Maache et al. 2017)
1421	C – H stretching	Lignin	(Hemmalakshmi, Priyanga, and Devaki, 2017)
1317	C – H symmetric and asymmetric stretching	Hemicellulose	(Maache et al. 2017)
1248	C = O and O – C–O	Lignin	(Maache et al. 2017)
1018	O – C–O asymmetric stretch vibration	Cellulose	(Maache et al. 2017)
517	β-glycosidic rings	Cellulose	(Fan, Dai, and Huan 2012)

Table 4. Comparison of DBF fiber functional groups with those other cellulosic fibers.

Band range	DBF	Sisal	Coir	UL*	CTF*	MF*	MR*	Functional group(s)
3700–3000	3282	3284	3358	3337	3417	3402	3429	O – H stretching
2900–2800	2938	-	2920	2922	2833	2921	2923	C – H stretching; vibration of CH_2 and CH
1780–1690	-	1727	1724	1738	1740	1732	1770	C=O stretching
1680–1555	1612	1597	1605	1591	1641	1636	1629	C=C aromatic stretch vibration
1508–1396	1421	1498	1460	1458	1518	1508	1506	C – H stretching
1370–1317	1317	1367	-	1370	1366	1379	1376	C – H symmetric and asymmetric stretching
1270–1190	1248	1238	1242	1237	1258	1252	1246	C = O and O – C–O
1185–1040	1018	1028	1035	1151	1026	1168	1164	O – C–O asymmetric stretch vibration
899–527	517	893	897	899	813	894	897	C=C bending
Reference	PW	(a)	(b)	(c)	(d)	(e)	(f)	

PW – present work; (a) (Benítez-Guerrero et al., 2017); (b) (Ichim et al. 2022); (c) (Njoku et al., 2020); (d) (Tamanna et al. 2021); (e) (Lila et al. 2020); (f) (Ding et al. 2022).
CTF – Corypha taliera fruit; UL – Urena lobata; MF – Munja fibers; MR-manau rattan stem fibers.

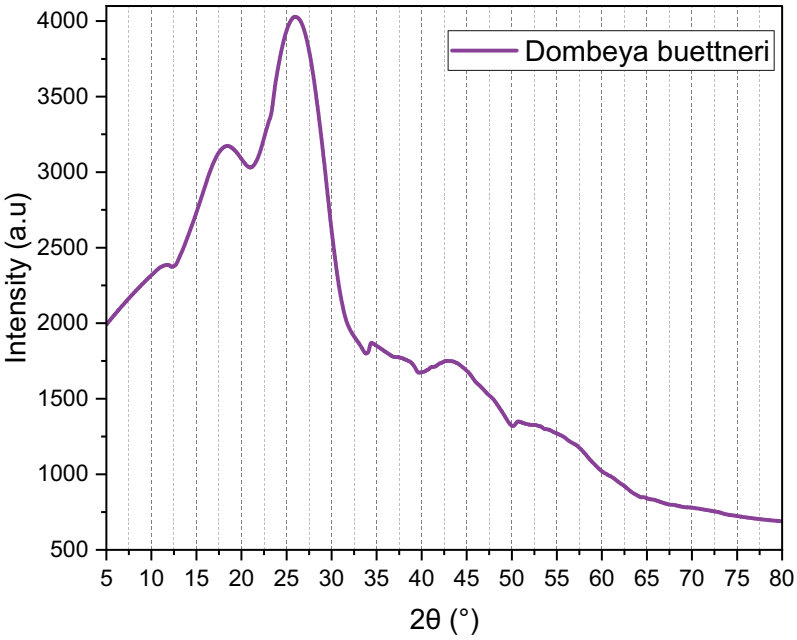

Figure 3. XRD spectrum of *Dombeya buettneri* stem fiber.

Table 5. Crystallographic properties of *Dombeya buettneri* stem fiber.

2θ (200) (°)		2θ (Amorphous) (°)		CrI, %	% Crystalline	Crystallite size, nm
Degree	Intensity (I_{200})	Degree	Intensity (I_{am})	62.32	72.63	2.16
25.81	1693.80	21.58	638.28			

Dombeya buettneri stem fibers as calculated by Equation (8) is 72.63% showing that the fiber has a good crystallite packing and strong structural arrangement for developing durable bio-composites.

The crystallinity index of *Dombeya buettneri* stem fibers as calculated by the Segal method (Equation 5) is 62.32%, which is similar to 62.5% for c*orypha taliera* fruit fiber (Tamanna et al. 2021). The crystallinity index reported in this study is higher than 40.68% for p*arthenium hysterophorus* stem fiber (Vijay et al. 2019), 33.33% for *Citrullus lanatus* climber (Khan et al. 2020a), 44.82% for *Acacia nilotica* L. fiber (Kumar et al. 2020), and 53.03% for *Coccinia indica* stem (Bhuvaneshwaran et al. 2019). Besides, the reported CrI, % is lower compared to 72.47% for aerial root banyan fibers (Ganapathy et al. 2019).

The average crystallite size computed from the XRD spectrum using the Debye-Scherrer's equation is 2.16 nm. The reported average size of the crystallite is higher than those reported for most bio-fibers such as 1.45 nm for *Corypha taliera* fruit fiber (Tamanna et al. 2021). Conversely, higher crystallite sizes of 5.81 nm for *Coccinia indica* stem fibers (Bhuvaneshwaran et al. 2019) and 18.78 nm for munja fibers (Lila et al. 2020) have been reported in literature. Better percent crystalline, crystallinity index and fairly higher crystallite size indicate the presence of higher cellulosic contents in the extracted *Dombeya buettneri* stem fiber.

Thermal analysis

The study of thermal behavior of natural fibers is crucial as the fabrication of natural fiber-reinforced thermoplastic polymer composites usually takes place at elevated temperatures. This provides information on the maximum temperatures fibers can withstand without degradation thus preserving fiber properties in the composite structure. Due to the differences in their chemical structure, the three main constituents of

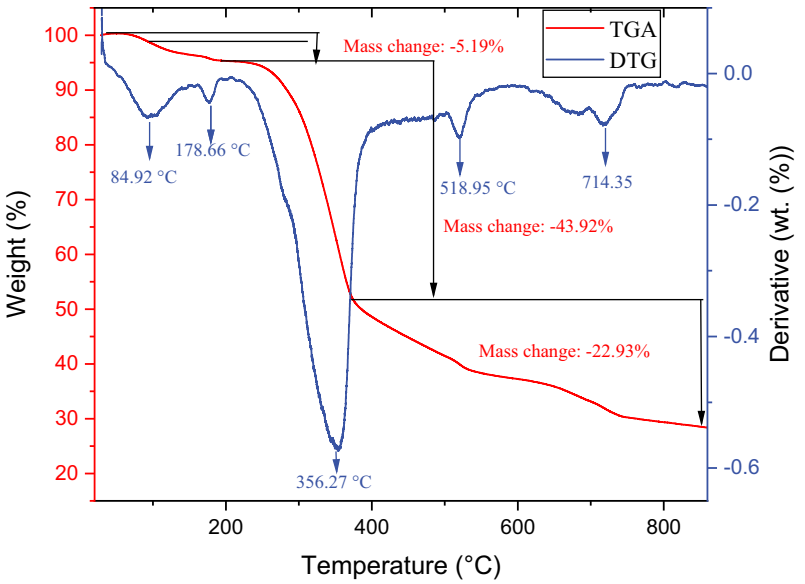

Figure 4. Thermograms (TG) and differential thermograms (DTG) curves of DBF fiber.

cellulosic fibers undergo thermal decomposition differently at different temperature ranges starting with hemicellulose (200–260°C), cellulose (240–350°C), lignin (280–500°C) and finally ash (Seki et al. 2013; Tamanna et al. 2021; Yang et al. 2007).

The effect of temperature increase with respect to weight loss and derivative weight loss of DBF fiber is presented by thermograms (TG) and differential thermograms (DTG) curves, respectively as shown in Figure 4. The thermal decomposition of DBF fiber involved four distinct phases of weight loss as indicated by DTG peaks. The first phase which occurred between 40–89°C represents moisture evaporation from the fiber surface. The second phase of degradation occurred at 165–250°C with the highest degradation obtained at 178.66°C peak representing hemicellulose decomposition. DBF fibers showed thermal resistance up to 229.57°C beyond which they underwent maximum thermal degradation.

The maximum weight loss of DBF fibers was reported between 250–375°C with a maximum decomposition rate at 356.27°C. This corresponds to the third degradation phase with an associated weight loss of 43.92%. This loss rate corresponds with the thermal degradation of α-cellulose and cellulose I (Fiore, Scalici, and Valenza 2014). Cellulose has been reported to decompose at 280–360°C for manau rattan fibers with 62.24% weight loss (Ding et al. 2022) and 267–373°C for *Cardiospermum halicababum* stem fiber with 39.36% weight loss (Vinod et al. 2019).

The last stage of thermal decomposition of DBF fiber occurred at 400–800°C with an associated weight loss of 22.93% corresponding to lignin degradation as it is the most difficult to decompose in comparison with hemicellulose and cellulose. Thus, high lignin content in cellulosic fiber imparts enhanced thermal stability characteristics. Finally, 28.3% of the initial fiber weight was retained at 860°C. This weight represents ash content with different inorganic elements such as silica which only decompose at extremely high temperatures (>1700°C) (Yang et al. 2007).

SEM analysis

The surface morphology of DBF fibers was investigated using SEM images taken at 100×, 200×, 250×, 300× and 500× magnifications. The images revealed DBF fibers as long fibers with some serrations along their length. The presence of non-cellulosic constituents mainly wax, hemicellulose and lignin in the fiber is illustrated by a white-colored substance as shown in Figure 5. These non-cellulosic constituents which act

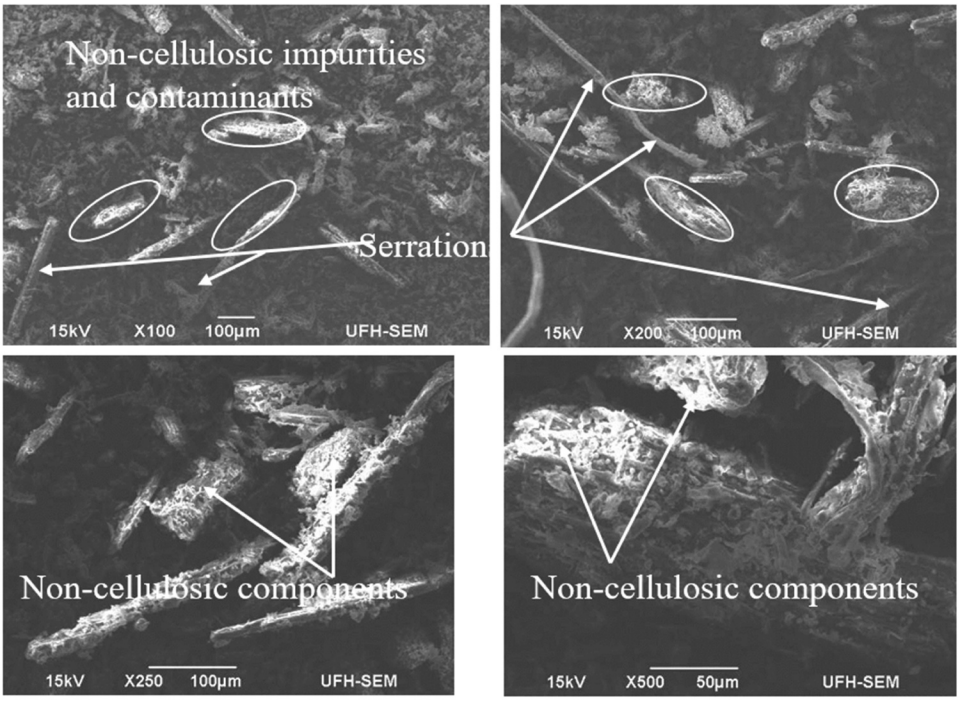

Figure 5. SEM images of Dombeya buettneri fiber at different magnifications.

as cementing substances should be removed from the fiber surface through chemical treatment such as mercerization. This enhances the surface roughness of cellulosic fibers, thus increasing interfacial adhesion between polar cellulosic fiber reinforcements and nonpolar polymeric matrices thereby increasing the mechanical properties of resultant composites.

EDX analysis

Figure 6 shows the surface characterization of DBF fiber using Energy Dispersive X-ray spectrometry. From the EDX spectrum, DBF cellulosic fiber showed high-intensity oxygen and carbon peaks which are the main components in the chemical chain structure of cellulose. The intense carbon and oxygen photoelectron peaks are found at 0.25 KeV and 0.46 KeV, respectively. The photoelectron peaks of carbon and oxygen predominantly corroborate the FTIR peaks that ratify DBF fiber as a biomaterial. Besides carbon and oxygen peaks, DBF fiber contains trace elements of sodium, potassium, magnesium, phosphorous, calcium and chlorine with different proportions as presented in Table 6. These are macronutrients plants obtain from the soil through their roots.

From elemental analysis results, DBF fibers have relatively higher oxygen content (50.66%) compared to carbon (34.13%). This is congruent with elemental analysis results of *Sansevieria roxburghiana* fiber that reported higher oxygen content (59.05%) and slightly lower carbon (40.95%) in terms of weight percentage (Krishna, Kailasanathan, and NagarajaGanesh 2020). This confirms the presence of less amount of amorphous or non-cellulosic constituents on the surface of DBF fibers. This is consistent with the chemical composition results presented in Table 2. The composition of non-cellulosic elements of DBF fiber is less (32.94%) compared to cellulose (58.45%). Further, less extractive content on the surface of cellulosic fiber is associated with slightly less carbon content compared to oxygen as illustrated by elemental analysis (Sgriccia, Hawley, and Misra 2008). The carbon/oxygen (C/O) ratio of DBF fiber is 0.67 indicating the surface of the fiber is hydrophilic. This is because a higher C/O ratio

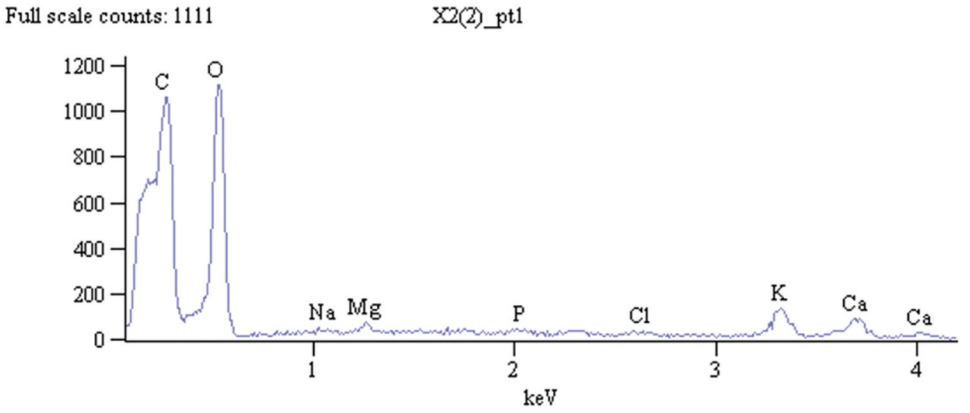

Figure 6. EDX spectrum of DBF fiber.

Table 6. Elemental analysis of DBF fiber.

Element	Weight (%)	Error (%)
C	34.13	±1.19
O	50.66	±0.82
Na	0.77	±0.12
Mg	0.92	±0.08
P	0.84	±0.11
Cl	0.79	±0.12
K	6.44	±0.42
Ca	5.46	±0.48

indicates better hydrophobicity of the surface of the fiber, a desirable characteristic in developing natural fiber-reinforced composites with enhanced properties (Ding et al. 2022).

Conclusions

In the present study, a novel cellulosic fiber from the stem of *Dombeya buettneri* plant was successfully extracted and characterized to evaluate its feasibility as bio-reinforcement in polymeric composites. From analysis results, the fiber presented high breaking force and breaking tenacity due to moderately high cellulose content with a corresponding high crystallinity index and percent crystalline confirming the presence of high crystalline cellulose as well as good crystallite packing and strong structural arrangement. Further, the FTIR analysis showed the presence of various functional groups of cellulose, lignin and hemicellulose. Thermogravimetric analysis results indicated that the extracted *Dombeya buettneri* fiber can be used as reinforcement in thermoplastic composites. SEM morphological analysis depicted a smooth fiber surface with surface impurities and contamination such as dirt. Therefore, the findings of this research suggest that DBF fiber can be used as a potential and alternative reinforcement to develop economical and environmentally-friendly polymer-based bio-composites.

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No potential conflict of interest was reported by the author(s).

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Consent

The author gives express consent to Journal of Natural Fibers to review the article and consider it for publication.

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