

## Characterization of Natural Cellulosic Fiber Isolated from Malaysian *Cymbopogon citratus* Leaves

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A novel natural fiber derived from the *Cymbopogon citratus* plant was investigated for the first time. The characterization of the *C. citratus* fibers was conducted, and the chemical composition and physical, thermal, mechanical, crystallinity, and morphological characteristics were studied. The chemical composition analysis of *Cymbopogon citratus* fiber revealed that the suggested fiber was rich in cellulose contents (37.6%). The tensile test of *C. citratus* fiber demonstrated the fiber's average tensile strength of  $43.81 \pm 15.27$  MPa and modulus of elasticity of  $1.046 \pm 0.33$  GPa. Further analysis with X-ray diffraction (XRD) confirmed that the crystallinity index of *Cymbopogon citratus* fiber was 35.2%, and the crystalline size was estimated as 4.28 nm. The *Cymbopogon citratus* fiber's thermal stability was investigated via thermogravimetric analysis (TGA) and observed to be thermally stable (230 °C). A morphological investigation was employed on the fiber via a scanning electron microscope (SEM). The morphological study result exhibited that the fiber had a perforated and rough surface with the lumen in the center. Thus, the findings revealed that the *Cymbopogon citratus* fiber was a promising potential reinforcement for thermoplastic green composite applications.

**Keywords:** *Cymbopogon citratus*; Natural fiber; Mechanical properties; Crystallinity index; Thermogravimetric analysis; Scanning electron microscopy

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### INTRODUCTION

Global warming, extreme emissions, and natural resource scarcity are among the significant risks that will be faced by the future generations. Finding a bio-based material originating from a natural renewable source as a substitute for synthetic materials is one way to minimize the depletion of the earth's raw materials and the impact on the environment. In recent decades, natural fibers are attractive due to their nature, e.g., biodegradable, abundant availability, low cost, non-toxic, low density, eco-friendly, recyclable, and having a high strength-to-weight ratio (Ilyas *et al.* 2018b; Jumaidin *et al.* 2020; Kamaruddin *et al.* 2020). The massive discrepancy between the existing demand and the cultivation rate of conventional fibers has prompted the emergence of new natural fibers that are chemically comprised of cellulose, hemicellulose, lignin, wax, ash, and other

soluble substances (Senthamaraikannan and Kathiresan 2018). Cellulose is classified as a linear polysaccharide polymer composed of glucose monosaccharide unit groups. In general, the high content of cellulose in natural fibers gives the fibers more strength and stability (Ishak *et al.* 2010). When characterizing a new natural fiber, its crystallinity index value is normally computed to determine its degree of crystalline or amorphous nature. The natural fibers' mechanical, physical, and chemical properties vary from each other because of variations in the chemical composition, maturity, and the plant's part of the derived fiber, location of the plant cultivation, soil micronutrients level, and environmental conditions (Saravanakumar *et al.* 2013). From the same work, the natural fibers' importance was discovered from a comprehensive literature review. A previous study found that the percentages of the fiber cellulose content and the crystallinity index directly affected the effectiveness (Ilyas *et al.* 2018). Using natural fibers as reinforcements in composite materials development is effective, as confirmed by Hyness *et al.* (2018). They also reported that the percentage of cellulose and the value of the crystallinity index influence the fiber's effectiveness. However, the crystalline cellulose and amorphous fractions of natural fibers (hemicellulose, lignin, and wax) vary relying on the place and conditions of the plant growing. The polymer composites' characteristics depend on a few factors: resin nature, the fiber alignment, as well as the strong attachment between the fiber and the matrix (Chakravarthy *et al.* 2020).

*Cymbopogon citratus* is an aromatic member of the Poaceae family. It is versatile due to the massive available products that can be produced from this plant (Ranitha *et al.* 2014). Throughout Malaysia, the *Cymbopogon citratus* plant can be widely cultivated in home gardens in the rural areas of Johor, Kedah, Kelantan, Perak, Pahang, Selangor, and Negeri Sembilan (Wifek *et al.* 2016). The plant can be generally found throughout the country because this species naturally grows in various areas. The fresh consumption of *Cymbopogon citratus* is increasing every year. The *Cymbopogon* species from China have been reported to have potential as a reinforcement in composite materials (Bekele *et al.* 2017). In China, there are various species of *Cymbopogon* such as *Cymbopogon goeringii*, *Cymbopogon tortilis*, and *Cymbopogon mekongensis* (Huang *et al.* 2013). Unlike in China, *Cymbopogon citratus* is the main species in Malaysia. Usually, the *Cymbopogon citratus* leaves are disposed of as waste materials. To exclusively consume the waste in manufacturing useful products, the current study focuses on the fiber originated from *Cymbopogon citratus* leaves of Malaysia. Generally, there are many varieties of *Cymbopogon*, e.g., *Cymbopogon flexuosus*, *Cymbopogon nardus*, *Cymbopogon winterianus* (Jowitt), *etc.* (De Oliveira *et al.* 2011; Biosci *et al.* 2015).

In general appearance, the *Cymbopogon citratus* leaves resemble those of other grasses that grow in large tufts. Each leaf is composed of two distinct components: the split sheath and the blade, which are connected at the blade joint. Based on the previous study, the anatomy of the leaf epidermis is similar to that of grasses in general, with an alternating band pattern related to the tissues beneath it. The epidermis is composed of three distinct cell types: (1) long cells, (2) suberized short cells, and (3) silica cells. In addition, there are the bulliform cells of the upper epidermis. The long cells are rectangular in shape, with occasionally pointed or concave ends. Surface preparations stained with chlorozinciodide revealed that the walls of the long cells are bright blue in color and that the middle lamella is undulating and strongly silicified, resulting in a white wavy line across the surface. The long cells vary in sizes, measuring in the intercostal region of the blade up to 63  $\mu\text{m}$  long, 17  $\mu\text{m}$  wide, and 14  $\mu\text{m}$  high, respectively. Meanwhile, the short cells usually occur in pairs with the long cells. The cells typically are rectangular in shape, especially when lying over

the veins. However, they can occasionally be broadly circular or even triangular. The silica cells have a greater degree of uniformity. They are typically rectangular and parallel to the leaf axis; however, they can be oval, rounded, or even broader than long. They typically appear somewhat constricted in the middle when viewed from the surface. Silica cells do not exist in the blade's lower epidermis (Balbaa and Johnson 1955; Berteau *et al.* 2003; Eltahir and Abu Reish 2010; Shah *et al.* 2012; Yeşil and Akalin 2015).

A recent study was conducted on natural fibers, including thermal, mechanical, morphological properties, as well as chemical composition (Ishak *et al.* 2012; Yusriah *et al.* 2014). Yusriah *et al.* (2014) discussed the impact of betel nut husk (BNH) maturation (raw, ripe, and matured) on its characteristics of mechanical, physical, thermal, and morphological properties. The same work discovered that the fiber of the ripe type has the most excellent tensile strength. The sugar palm fibers' tensile and thermal properties were characterized, and the greatest tensile strength is obtained from green fiber due to optimum chemical composition, which includes a high cellulose content, as well as hemicellulose and lignin (Ishak *et al.* 2012).

Even though there are studies reporting on the characterization of lignocellulosic fibers as reinforcement in composite materials, none has been found on the characterization of natural cellulosic fiber obtained from Malaysian *Cymbopogon citratus* leaves. Therefore, the objective of this work was to understand the chemical, physical, mechanical, and thermal properties of the Malaysian *Cymbopogon citratus* fibers in comparison to other natural fibers known. The findings obtained through this study will be beneficial to understand the potential of this natural fiber for various biodegradable materials applications. Though, to this date, there have been few investigations on the utilization and the application of *Cymbopogon citratus* fiber and its composites. In the present work, the properties of Malaysian *Cymbopogon citratus* fiber were characterized *via* a series of testing for their properties that include density, chemical composition, mechanical properties, thermal stability, morphology, and functional groups. The findings were presented and compared with other natural fibers.

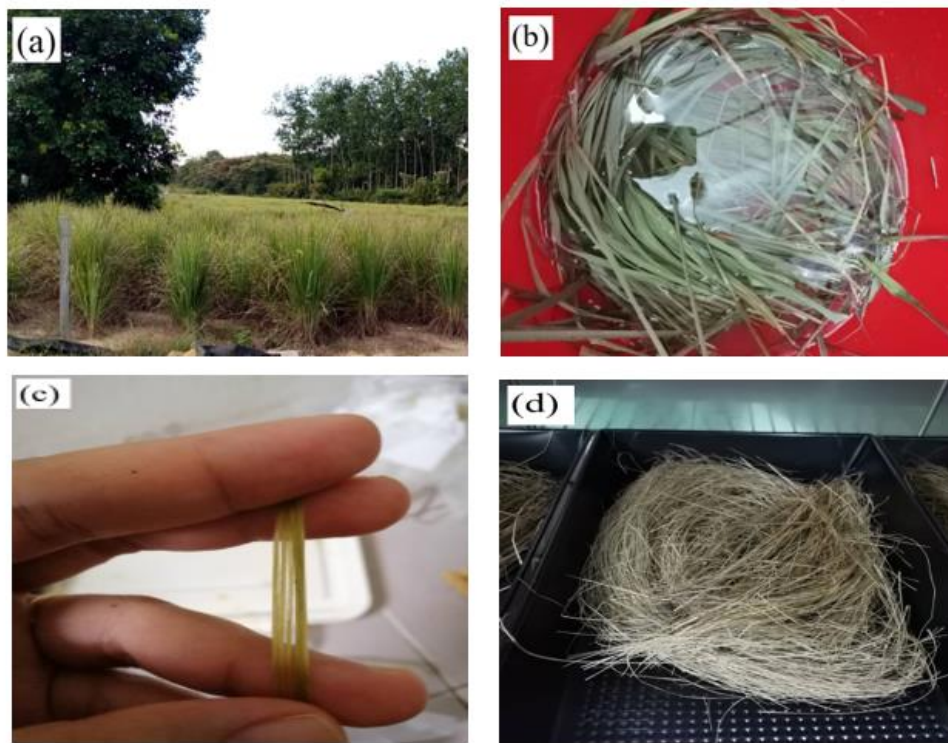
## EXPERIMENTAL

### Materials

The *Cymbopogon citratus* plant leaves used in this study were collected from trees located in Beranang, Selangor (Malaysia). The leaf lamina is linear and green in color.

### Preparation of Fibers

Unwanted particles were eliminated from the surface of the collected *Cymbopogon citratus* by washing with fresh water. Next, the cleaned leaves were retted by immersing them in normal fresh water for the microbial degradation process for 14 d in a water tank. The materials in the tank were periodically monitored to ensure that fibers were loosened to be extracted. The fibers were manually removed from the leaves after the retting process. To remove the dust particles, if any, the collected fiber was washed using distilled water and later sun-dried for 48 h before oven-dried for another 24 h at 85 °C to further eliminate the residual moisture (Huzaiyah *et al.* 2017). Approximately 1 kg of fresh *Cymbopogon citratus* leaves were required to yield about 30 g of fibers. The flow processes of water retting are displayed in Fig. 1 (a) through (d).



**Fig. 1.** (a) *Cymbopogon citratus* plant, (b) water retting of *Cymbopogon citratus* fibers, (c) extraction of *Cymbopogon citratus* fibers, and (d) *Cymbopogon citratus* fibers

### Chemical Composition

The *Cymbopogon citratus* fiber's chemical composition was evaluated via neutral detergent fiber (NDF), acid detergent fiber (ADF), acid detergent lignin (ADL), as well as ash content analysis (Razali *et al.* 2015). Using Eqs. 1 and 2, the cellulose and hemicellulose percentages can be determined respectively:

$$\text{Cellulose} = \text{ADF} - \text{ADL} \quad (1)$$

$$\text{Hemicellulose} = \text{NDF} - \text{ADF} \quad (2)$$

### Physical Properties

#### Diameter

*Cymbopogon citratus* fibers diameter was evaluated under an optical microscope, Zeiss (Axiovert 200; Carl Zeiss Light Microscopy, Gottigen, Germany) that measured 15 individual fiber samples. The average diameter was randomly measured at three different positions of each image and the average value was determined (Razali *et al.* 2015).

#### Density

*Cymbopogon citratus* fiber density was measured using an AccuPyc 1340 TEC pycnometer (Micromeritics Instrument Corporation, Norcross, GA, USA). This pycnometer was employed to measure the volume and density in accordance with ASTM D792 (2008) of powdered fiber. This experiment utilized helium gas at a constant temperature. Three replicates of analysis were considered for this sample, and an average value was used for data analysis (Ilyas *et al.* 2017).

### Moisture content

Moisture content evaluation was performed for five samples that were prepared and heated in the oven for a period of 24 h at a temperature of 105 °C. Moisture content calculation requires the initial weight prior to heating ( $M_i$ , g) as well as final weight after heating ( $M_f$ , g) (Ilyas *et al.* 2017). Next, the *Cymbopogon citratus* fiber's moisture content was calculated using Eq. 3:

$$\text{Moisture content (\%)} = \frac{M_f - M_i}{M_i} \times 100 \quad (3)$$

### Water absorption

Five *Cymbopogon citratus* fiber specimens with a length range of 40 mm to 50 mm were prepared, and each sample's average water absorption percentage was computed using Eq. 4. The specimens were weighed at room temperature before ( $M_i$ ) and after ( $M_f$ ) soaking in fresh water for 24 h at room temperature (Huzaifah *et al.* 2017), as shown in Eq. 4:

$$\text{Water absorption (\%)} = \frac{M_f - M_i}{M_i} \times 100 \quad (4)$$

### Tensile Properties

The ASTM D3379 (1998) guideline was adopted for the evaluation of the tensile properties of *Cymbopogon citratus* fiber by using Instron universal testing machine (5556, Norwood, MA, USA) with 5 kN load cell capacity. Experimentation was performed with 25 mm gauge length and 1 mm/min crosshead speed. The *Cymbopogon citratus* fiber was carefully chosen under an optical microscope prior to the testing to guarantee that there was no damage to the fibers (Razali *et al.* 2015). Each fiber was fastened on the sample holder as displayed in Fig. 2, and the *Cymbopogon citratus* fibers with 15 replicates were used for the measurements of tensile characteristics.

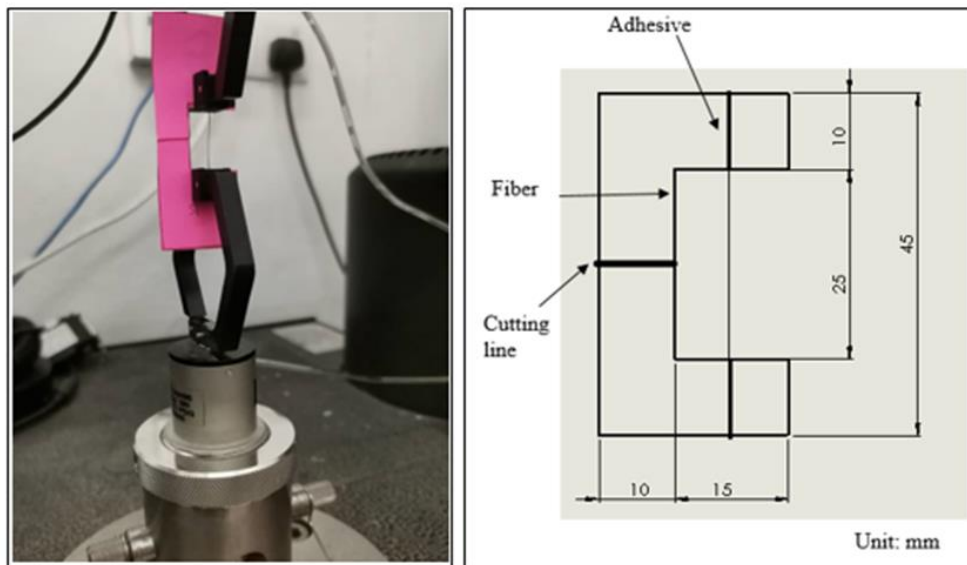


Fig. 2. Sample preparation for tensile test

## Thermal Characterization

### *Thermogravimetric analysis (TGA)*

For the study of thermal stability of the *Cymbopogon citratus* fiber, a Thermogravimetric analyzer from TA Instruments (Mettler-Toledo AG, Schwerzenbach, Switzerland) was deployed to investigate the strength of the fibers for high-temperature applications with the thermal analysis Q series. The needed amount of *Cymbopogon citratus* fibers was weighed and put inside an alumina crucible and placed in a heating chamber. The study was carried out in a nitrogen environment from 25 °C to 900 °C at a 10 °C/min constant heating rate. Before the thermal study, the samples were pre-conditioned for at least 2 d at 53% (Jumaidin *et al.* 2017).

## Scanning Electron Microscopy (SEM)

The cross-section and morphology of the *Cymbopogon citratus* fibers were conducted using a scanning electron microscope (SEM) instrument model Hitachi S-3400N (Kyota, Japan). The acceleration voltage used was 15 kV, and the surface of the *Cymbopogon citratus* fibers was pre-coated using a thin gold layer. The sample was 50 mm long and the results were acquired by enlarging at different magnifications (Arul Marcel Moshi *et al.* 2020).

## Fourier Transform Infrared Spectroscopy

Fourier transform infrared (FTIR) investigation of all samples was performed to determine the functional groups' presence in the *Cymbopogon citratus* fiber. This analysis was performed on an IR spectrometer (Nicolet 6700 AEM, Thermo Nicolet Corporation, Madison WI, USA) experimental setup. The samples for this examination were powdered and mixed with potassium bromide. An approximate 2 mg of powdered sample was combined with potassium bromide (KBr) and later pressed into a 1-mm-thick disc. The samples' FTIR spectra were recorded in the range of 4000 to 400  $\text{cm}^{-1}$  (Jumaidin *et al.* 2020).

## X-ray Diffraction Analysis (XRD)

The X-ray diffraction approach was chosen to evaluate the crystallinity index and crystalline size of the *Cymbopogon citratus* fiber using a Rigaku D/max 2500 X-ray powder diffractometer (Rigaku, Tokyo, Japan) with Cu radiation run at 40 kV and 30 mA with 0.15406 nm light wavelength. The scanning rate of 2 °min<sup>-1</sup> in the range of diffraction angle 10° to 40° at room temperature was used to scan the samples. The crystallinity index (CI) of *Cymbopogon citratus* fiber was computed using subsequent Segal expression Eq. 5 (Ilyas *et al.* 2018a),

$$CI = \frac{I_{002} - I_{am}}{I_{002}} \times 100\% \quad (5)$$

where  $I_{002}$  and  $I_{am}$  represent peak intensities of the crystalline and amorphous fractions, respectively. The crystallite size (CS) was determined using Scherrer's formula shown in Eq. 6,

$$CS = \frac{k\lambda}{\beta \cos\theta} \times 100\% \quad (6)$$

where  $k = 0.89$  (Scherrer's constant),  $\lambda = 0.1541$  nm is the radiation wavelength,  $\beta$  is the peak's full-width at half-maximum in radians, and  $\theta$  is the corresponding Bragg angle.

## RESULTS AND DISCUSSION

### Chemical Composition

Natural fiber contains three main constituents, *e.g.*, celluloses, hemicellulose, and lignin. Cellulose is known to be the key framework portion of the fiber structure. Hemicellulose and lignin compounds cover cellulose domains and give fiber strength, rigidity, and structural stability (Kabir *et al.* 2012). The natural fibers' chemical composition analysis is vital in determining the fibers' mechanical strength that is influenced by the quantity of numerous chemical components present on the natural fibers' surface (Moshi *et al.* 2020). The comparison of the results from the analysis of the chemical composition of the *Cymbopogon citratus* fiber with different natural fibers is presented in Table 1. A total of 37.6% cellulose was found on the fibers' surface, of the *Cymbopogon citratus* fibers, which was relatively higher than other natural fibers, *e.g.*, bamboo (26 to 43%) (Kulandaivel *et al.* 2020), *Mikania micrantha* Kunth (21%) (Ganapathy *et al.* 2019), piassava (28.6%) (Indran and Raj 2015), coconut tree leaf sheath (27%) (Indran and Raj 2015), and less than *Carica papaya* (58.71%) (Arul Marcel Moshi *et al.* 2020), Nendran banana (73.2%) (Manimaran *et al.* 2020), Saharan aloe vera (67.4%) (Balaji and Nagarajan 2017; Manimaran *et al.* 2020), and sugar palm (44.53%) (Huzaifah *et al.* 2017).

**Table 1.** Chemical Composition of *Cymbopogon citratus* Fiber with Other Natural Fibers

Fibre	Cellulose (%)	Hemicellulose (%)	Lignin (%)	Ash (%)	Reference
<i>Cymbopogon citratus</i> leaves	37.56	29.29	11.14	4.28	Current study
Piassava leaves	28.6	25.8	45	-	(Indran and Raj 2015)
Coconut Tree Leaf Sheath	27	14	27.7	-	(Indran and Raj 2015)
Bamboo pulp	26 to 43	-	21 to 31	-	(Kulandaivel <i>et al.</i> 2020)
<i>Mikania micrantha</i> Kunth leaves	21.31	14.61	20.67	9.11	(Ganapathy <i>et al.</i> 2019)
Sugar Palm trunk	44.53	10.01	41.97	0.955	(Huzaifah <i>et al.</i> 2017)
Jute bark	45 to 71.5	13.6 to 21	13-26	0.5 to 2	(Li <i>et al.</i> 2007)
Hemp stem	55 to 77	14 to 22.4	3.7-13	0.8	(Sathishkumar <i>et al.</i> 2013b)
Saharan Aloe Vera leaves	67.4	8.2	13.7	-	(Balaji and Nagarajan 2017; Manimaran <i>et al.</i> 2020)
<i>Carica Papaya</i> bark	58.71	11.8	14.26	4.7	(Arul Marcel Moshi <i>et al.</i> 2020)
Nendran Banana peduncle	73.2	10.85	15.32	2.59	(Manimaran <i>et al.</i> 2020)

Researchers in this field have stated that with a higher cellulose content, the mechanical properties comprising elasticity modulus and tensile strength are improved.

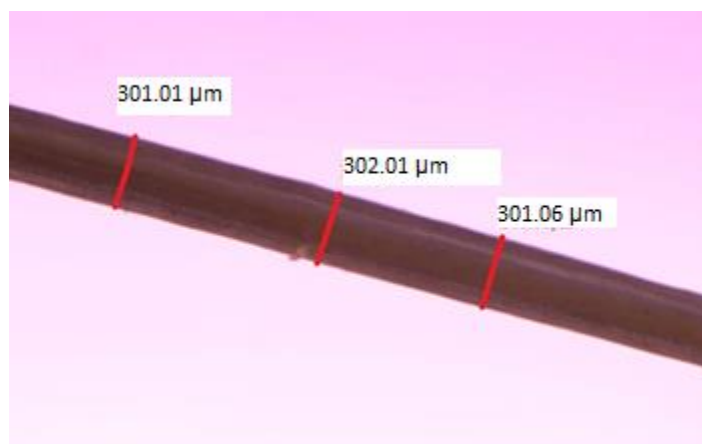


However, the higher content of hemicellulose in natural fiber has an adverse effect on fiber strength (Ilaiya Perumal and Sarala 2020). In this study, a hemicellulose content of 29.3% was found on the surface of *Cymbopogon citratus* fiber. The lignin content of *Cymbopogon citratus* fiber was 11.1%; it acts as a protective guard against bacterial attacks (Balaji and Nagarajan 2017). The lower amount of ash (4.28%) for *Cymbopogon citratus* fiber can explain its mechanical properties, such that the *Cymbopogon citratus* fiber has the essential properties for use as a potential reinforcement material.

## Physical Properties

### Density and diameter

Table 2 shows the measurements obtained for the diameter of *Cymbopogon citratus* fiber. The diameter measurement is important because it is essential in determining the fiber's mechanical properties, especially tensile strength. Measurement of the *Cymbopogon citratus* fiber's diameter was conducted using an optical microscope, as displayed in Fig. 3. The fiber's average diameter was calculated as  $326.67 \pm 45.77 \mu\text{m}$ , which was comparable with that red banana peduncle (150 to 250  $\mu\text{m}$ ) (Chakravarthy *et al.* 2020), *Agave sisalana* fiber (50 to 300  $\mu\text{m}$ ) (Moshi *et al.* 2020), and banana fiber (80 to 250  $\mu\text{m}$ ) (Moshi *et al.* 2020). The source of the fiber, plant condition, plant maturity, and extraction process are the key factors affecting the physical properties (Reddy and Yang 2005).



**Fig. 3.** Optical microscopy image of the *Cymbopogon citratus* fiber

Density is an essential factor for natural fibers mass determination and influences the mass of the natural fibers composite materials that will be produced. The density of *Cymbopogon citratus* fiber was measured using the pycnometer setup (Micromeritics Instrument Corporation, Norcross, GA, USA) and recorded as  $0.251 \pm 0.002 \text{ g/cm}^3$ , which was comparatively lesser than some other natural fibers, *e.g.*, Bambusoideae ( $0.600 \text{ g/cm}^3$ ) (Arul Marcel Moshi *et al.* 2020), *Prosopis juliflora* ( $0.58 \text{ g/cm}^3$ ) (Saravanakumar *et al.* 2013), and *Furcraea foetida* ( $0.778 \text{ g/cm}^3$ ) (Manimaran *et al.* 2018). The comparisons of diameter and density of *Cymbopogon citratus* fiber with different natural fibers are presented in Table 2. The density of *Cymbopogon citratus* fiber revealed that it will be a great candidate for producing lightweight composite products.



**Table 2.** Diameter and Density of *Cymbopogon citratus* Fiber and Other Natural Fibers

Fibre	Diameter ( $\mu\text{m}$ )	Density ( $\text{g}/\text{cm}^3$ )	References
<i>Cymbopogon citratus</i>	$326.67 \pm 45.77$	$0.251 \pm 0.002$	Current study
<i>Bambusoideae</i>	14	0.600	(Arul Marcel Moshi <i>et al.</i> 2020)
<i>Prosopis juliflora</i>	20	0.580	(Saravanakumar <i>et al.</i> 2013)
<i>Furcraea foetida</i>	12.8	0.778	(Manimaran <i>et al.</i> 2018)
Red Banana Peduncle	150 to 250	0.99	(Chakravarthy <i>et al.</i> 2020)
<i>Agave sisalana</i>	50 to 300	1.500	(Moshi <i>et al.</i> 2020)
Banana	80 to 250	1.350	(Moshi <i>et al.</i> 2020)

### Moisture content

One of the primary factors that must be deliberated throughout the evaluation of a novel material as a possible reinforcing agent in polymer composites is moisture content. Low moisture content fibers will have a good bond between a composite material's fiber and polymer matrix (Jayaramudu *et al.* 2010). The hydrophilic behavior of natural fibers has noticeable effects on their mechanical performance, dimensional stability, and porosity of natural fiber in a composite material, resulting from high moisture absorption in wet conditions (Razali *et al.* 2015). Hence, low moisture containing fibers are preferable for the application. As shown in Table 3, *Cymbopogon citratus* fiber recorded the moisture content of  $5.20 \pm 2.28$ , which was lower compared to other natural fibers (sugar palm, roselle, banana, flax, jute, sisal, ramie, and hemp) with moisture contents of approximately 5.5 to 22% (Akil *et al.* 2011; Razali *et al.* 2015; Huzaifah *et al.* 2017). Therefore, increasing the cellulose content percentage in the fibers will result in an increased moisture absorption rate. This phenomenon is associated with the cellulose chemical structure itself that consists primarily of water-accessible hydroxyl groups. Moisture and hemicellulose contents of natural fibers are key factors of their moisture absorption level, thermal, and biological degradation. Therefore, natural fiber's low moisture content is the most preferable criterion for polymer composites. Moreover, high moisture content fibers cause some negative effects on the composites, *e.g.*, degradation, cracking, mechanical properties loss, and inviting decay fungi (Sapuan *et al.* 2013).

### Water absorption

Natural fibers possess numerous benefits as reinforcing materials and have relatively similar characteristics to synthetic fibers. The water absorption of *Cymbopogon citratus* fibers was computed based on the samples' weights before and after their immersion in water for 24 h. The main common disadvantage of using natural fiber is its high hydroxyl cellulose content, making it prone to water absorption and negatively affecting the composites' mechanical properties. This contributes to the hydrophilicity of the fibers resulting from rising water absorption with increasing cellulose composition because of the increasing free hydroxyl groups present in the fiber (Sahari *et al.* 2011). The water absorption is dependent on the fiber's temperature, content, orientation, permeability, surface area exposed, void content, as well as hydrophilicity of each component (Akil *et al.* 2011). All lignocellulosic materials exhibit water absorption behavior due to the water molecules that are attracted to hydrophilic groups in the fiber and interact with the hydroxyl group (-OH) of cellulose molecules, forming hydrogen bonds. The results displayed in Table 3 indicate the value of water absorption for *Cymbopogon citratus* fiber compared to other natural fibers. From the results, the water absorption

percentages of *Cymbopogon citratus* fiber were lower than roselle fiber but higher than sugar palm fiber. The size of the lumen in the *Cymbopogon citratus* fiber has a major impact on its water uptake behavior. As more lumens exist in the unit cell of *Cymbopogon citratus*, this creates simple passages of water molecules entry into the cell unit and spread deeper through the fiber's cell wall, thereby increasing the water absorption of the *Cymbopogon citratus* fiber.

**Table 3.** Physical Properties of *Cymbopogon citratus* Fiber with Other Natural Fibers

Fiber	Water Absorption (%)	Moisture Content (%)	References
<i>Cymbopogon citratus</i>	224.03 ± 23.34	5.20 ± 2.28	Current study
Roselle	320	5.8	(Razali <i>et al.</i> 2015)
Sugar Palm	161.96 ± 34.04	6.45 ± 1.07	(Huzaifah <i>et al.</i> 2017)

### Tensile Properties

Table 4 compares the tensile characteristics of *Cymbopogon citratus* fiber samples to those of different renowned natural fibers. The average single fiber's tensile strength, elasticity modulus, and elongation rate were  $43.81 \pm 15.27$  MPa,  $1.046 \pm 0.33$  GPa, and  $0.84 \pm 0.28\%$ , respectively. The tensile strength of *Cymbopogon citratus* is dependent on plant age, source, method of fiber extraction, as well as fiber microstructure, where cracks initiated from the bigger flaw will lead to fiber failure (Bezazi *et al.* 2014). Hence, it is vital to test a minimum of 15 replications of each fiber sample to finalize the tensile properties. The high cellulose content and index of crystallinity are the main factors contributing towards achieving greater tensile strength in natural fibers (Saravana Kumar *et al.* 2019). In the comparison with different natural fibers, although the strength of *Cymbopogon citratus* fiber was lower than Nendran banana (65.5 MPa) (Manimaran *et al.* 2020) and *Sansevieria ehrenbergii* (50 to 585 MPa) (Sathishkumar *et al.* 2013a), the tensile strength of *Cymbopogon citratus* showed a moderate value with coconut tree leaf sheath (46.4 MPa) (Kulandaivel *et al.* 2020) and oil palm fruit (49 MPa) (Arul Marcel Moshi *et al.* 2020). The *Cymbopogon citratus* fiber also revealed higher strength than the *Pennisetum purpureum* (13.2 MPa) (Arul Marcel Moshi *et al.* 2020) and aerial roots of the banyan tree (19.4 MPa) (Ganapathy *et al.* 2019). Natural fibers possess similar morphology; however, factors, *e.g.*, the lumens' internal area, lumen number, fiber cells' number and size, secondary cell walls' thickness, and actual cross-section of the cells, differ from each other. The cross-sectional areas of each fiber are interrelated with its tensile behavior because they are not perfectly circular and exhibit some variations. Furthermore, the results of tensile tests can be influenced not only by morphology but also by chemical composition. The most important factor in determining the fiber's strength is the cellulose content. For example, the Nendran banana and piassava had approximately 73.2% and 28.6% cellulose content, respectively (Indran and Raj 2015; Manimaran *et al.* 2020). Therefore, the Nendran banana might experience greater strength that was not only because of its morphological characteristics but also its higher cellulose content. From the tensile test results, there is a possibility for the *Cymbopogon citratus* fiber to be applied as reinforcement for polymer composites.

**Table 4.** Tensile Properties of *Cymbopogon citratus* Fiber with Other Natural Fibers

Fiber	Tensile Strength (MPa)	Tensile Modulus (GPa)	Elongation at Break (%)	Reference
<i>Cymbopogon citratus</i>	43.81 ± 15.27	1.046 ± 0.33	0.84 ± 0.28	Current study
<i>Pennisetum purpureum</i>	13.15	88.4	0.99	(Arul Marcel Moshi <i>et al.</i> 2020)
Aerial Roots of Banyan Tree	19.37	1.8	1.8	(Ganapathy <i>et al.</i> 2019)
Coconut Tree Leaf Sheath	46.4	2.3	2.84	(Kulandaivel <i>et al.</i> 2020)
Oil Palm Fruit	49	2.763	7	(Arul Marcel Moshi <i>et al.</i> 2020)
Nendran Banana	65.51	49.5	2.17	(Manimaran <i>et al.</i> 2020)
<i>Sansevieria ehrenbergii</i>	50 to 585	1.5 to 7.97	2.8 to 21.7	(Sathishkumar <i>et al.</i> 2013a)

### Thermogravimetric Analysis

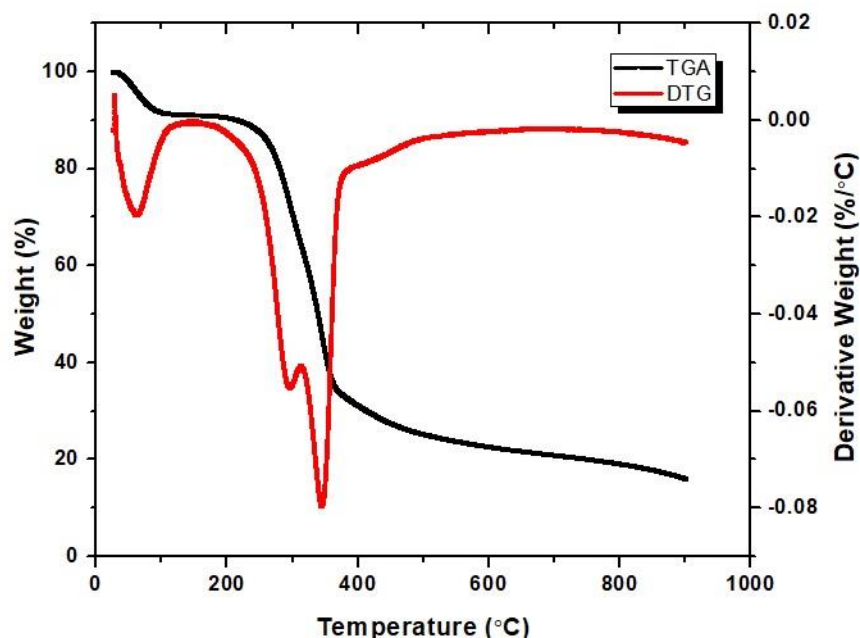
Natural fibers have limited thermal stability at high temperatures, which is one of the drawbacks of their application as a reinforcing agent in structural composite materials. The TGA studied the *Cymbopogon citratus* fiber's thermal behavior and determined the suitability of the *Cymbopogon citratus* for high-temperature structural applications. From the TGA curves (Fig. 4), there were three major degradation phases. The first degradation corresponded to the vaporizations of highly volatile components and moisture from the fiber. The earliest devolatilization took place at a temperature of 30 °C to approximately 110 °C due to the vaporization of water or moisture as reported by Ilyas *et al.* (2017). Moisture evaporation from the *Cymbopogon citratus* fiber occurred between 80 and 95 °C and the weight loss of the fibers was 14.2%, as shown in Table 5.

The second degradation peaks were found at 280 and 340 °C, which indicated the elimination of hemicellulose from the fiber's surface and the mass loss of around 44.73%. The typical hemicellulose decomposition occurs at 220 °C and becomes complete at 315 °C (Yang *et al.* 2007). The result attained from this work was parallel with the reported hemicellulose degradation data, where the hemicellulose degradation took place from 280 °C and was completed at approximately 300 °C, as displayed in Fig. 4. The first component to degrade during thermal analysis is hemicellulose because it comprises heterogeneous polysaccharides, *e.g.*, galactose, xylose, glucose, and mannose that exist in the amorphous state, and hence are not difficult to devolatilize at low temperatures (Yang *et al.* 2007).

The next thermal decomposition was cellulose as demonstrated by the clear U-shaped peak found at 338 °C from the derivative thermogravimetry (DTG) graph displayed in Fig. 4 occurred from 280 to 340 °C. The last component decomposed is lignin degradation from the fiber's surface. Lignin and other non-cellulosic chemical components degraded as confirmed by the final clearly observable thermal degradation peak near 490 °C. The leftover material after complete lignin degradation is called the residual mass. For *Cymbopogon citratus* fiber, the residual mass was in the range of 15 to 20%. Overall, the thermal stability of the *Cymbopogon citratus* fiber was concluded to be similar to other well-known bast fibers, such as kenaf, roselle, sugar palm, and jute, as shown in Table 6.

**Table 5.** Thermal Degradation Analysis of *Cymbopogon Citratus* Fiber

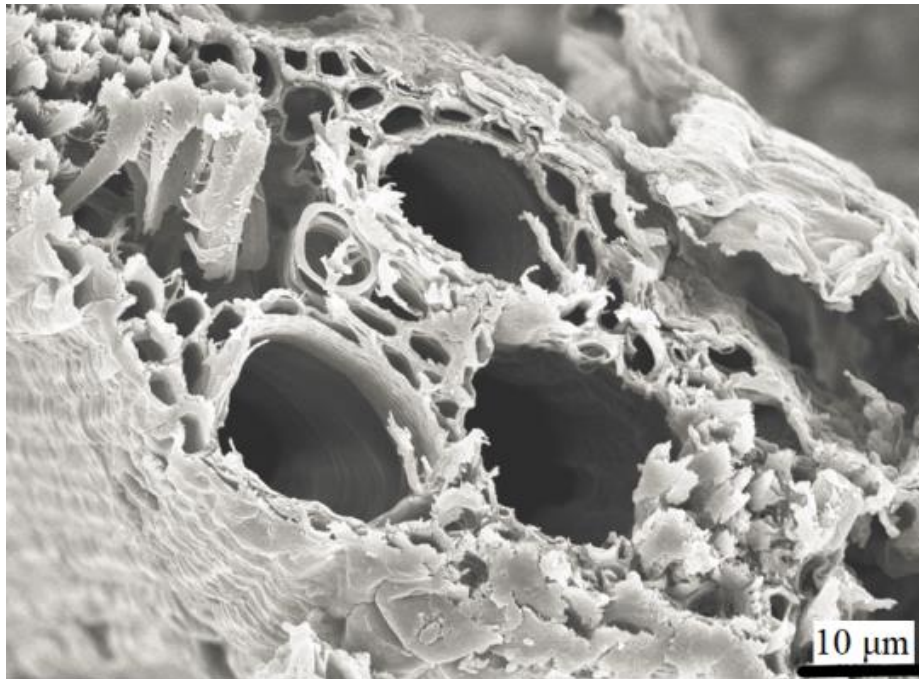
Sample	Weight Loss (%) From 30 to 100 °C	1 <sup>st</sup> Thermal Degradation			2 <sup>nd</sup> Thermal Degradation			Char Residue (wt%)
		$T_1$ (°C)	Weight Loss (%)	$T_{peak}$ (°C)	$T_2$ (°C)	Weight Loss (%)	$T_{peak}$ (°C)	
<i>Cymbopogon citratus</i> fiber	9.72	140 to 280	14.18	245.03	280 to 340	44.73	338.62	14.29

**Fig. 4.** TG and DTG curves for *Cymbopogon citratus* fiber**Table 6.** Temperatures of Degradation for Selected Natural Fibers

Natural Fiber	Temperature of Initial Decomposition (°C)	Maximum Decomposition Temperature (°C)	References
<i>Cymbopogon citratus</i>	230	338	Current study
Sugar Palm	228	312	(Huzaifah <i>et al.</i> 2017)
Roselle	210	366.08	(Razali <i>et al.</i> 2015)
Kenaf	219	284	(De Rosa <i>et al.</i> 2009)
Jute	205	283	(De Rosa <i>et al.</i> 2009)
Hemp	250	390	(De Rosa <i>et al.</i> 2009)

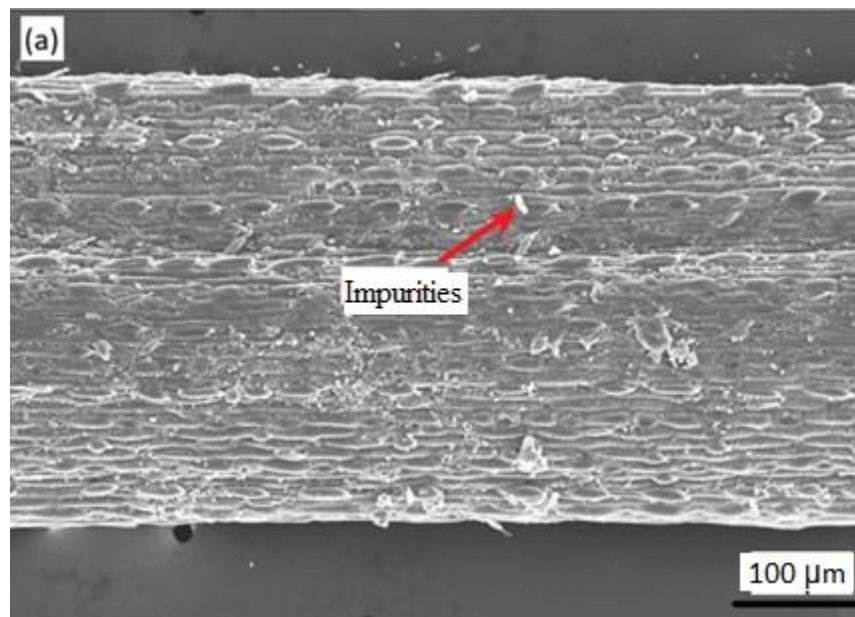
### Morphological Properties

Natural fibers' morphology is among the important factors influencing the mechanical and physical characteristics of the fibers and indicates their suitability as reinforcing material for composites. The *Cymbopogon citratus* fiber's cross-section was examined under SEM, and results are shown in Fig. 5. The water absorption greatly depends on lumen size as the larger lumen and porous structures make it much easier for the fiber to absorb more water and promote good penetration when used as polymer composite's reinforcement.

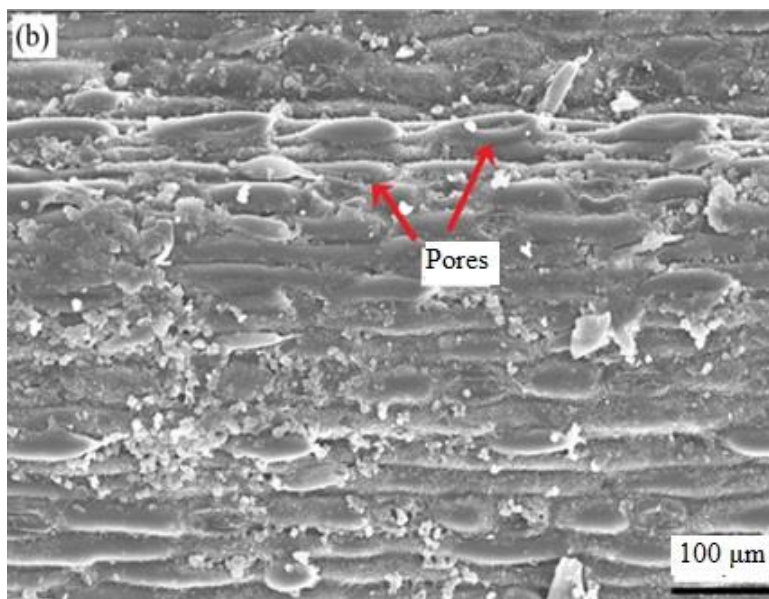


**Fig. 5.** SEM images of *Cymbopogon citratus* fiber in cross-sectional view

The *Cymbopogon citratus* fiber's surface morphology was examined at different magnifications and the surface fiber's images in the longitudinal view are displayed in Fig. 6 (a) and (b).



**Fig. 6(a).** SEM images of the surface structure of *Cymbopogon citratus* fiber (a) 200X magnification and (b) 500X magnification



**Fig. 6(b).** SEM images of the surface structure of *Cymbopogon citratus* fiber (a) 200X magnification and (b) 500X magnification

The SEM images of the surface showed irregular rough surface structure and the presence of pores of the fiber. It is predicted to promote better bondability with the polymer as well as enhancing mechanical interlinking between fiber and polymer matrix in the composite (Maheshwaran *et al.* 2018; Chakravarthy *et al.* 2020). Hence, the *Cymbopogon citratus* fiber was shown to be an excellent candidate for reinforcement in polymer composites. Each unit cell of fiber comprises cellulose surrounded by lignin and hemicellulose. At higher magnification, the hemicellulose was observed on the fiber's surface indicated by the image of a white layer that might be simply eradicated with the fiber's alkali treatment. The treatment also resulted in the improvement of the mechanical characteristics of the *Cymbopogon citratus* fiber by facilitating the elimination of hemicellulose as well as other undesired contaminants from the surface of the *Cymbopogon citratus* fiber (Moshi *et al.* 2020).

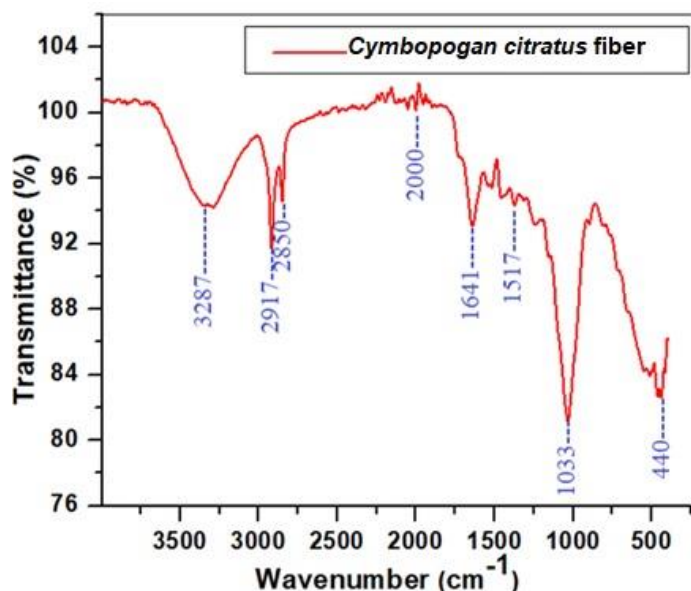
### FTIR Analysis

The FTIR spectrum obtained from *Cymbopogon citratus* fiber is shown in Fig. 7, in which noticeable FTIR peak positions, the composition of the *Cymbopogon citratus* fiber, and chemical functional groups were revealed. From Fig. 7, a peak was detected at  $3287\text{ cm}^{-1}$ , indicating the presence of the O-H chemical bonds in carboxylic acid group cellulose constituents (Ilaiya Perumal and Sarala 2020; Vijay *et al.* 2020). The next two consecutive peaks nearby for *Cymbopogon citratus* fiber were noticed at  $2917\text{ cm}^{-1}$  and  $2850\text{ cm}^{-1}$  corresponded to the C-H stretching vibration of CH and CH<sub>2</sub>, which indicated the presence of cellulose and hemicellulose (Ilyas *et al.* 2017; Kulandaivel *et al.* 2020). A band peak at  $2000\text{ cm}^{-1}$  was associated with CH<sub>2</sub> symmetrical stretching of wax. A small observable peak at  $1641\text{ cm}^{-1}$  was ascribed to the hemicellulose components (Gurukarthik Babu *et al.* 2019). The peak at  $1517\text{ cm}^{-1}$  indicated the existence of a small number of moisture particles, and the peak at  $1033\text{ cm}^{-1}$  was ascribed to lignin in the form of C-OH molecules (Manimaran *et al.* 2020). Table 7 provides FTIR peak locations and distributions of stretching in the *Cymbopogon citratus* fiber.



**Table 7.** FTIR Peak Positions and Chemical Stretching Allocations in the *Cymbopogon citratus* Fiber

Wavenumber (cm <sup>-1</sup> )	Allocations
3287	OH stretching of $\alpha$ -cellulose
2917 and 2850	CH stretching of $\alpha$ -cellulose
2000	C=C stretching of wax
1641	C=O stretching of hemicellulose
1033	C-OH vibration of lignin

**Fig. 7.** FTIR spectrum analyses for *Cymbopogon citratus* fiber

### X-ray Diffraction Analysis

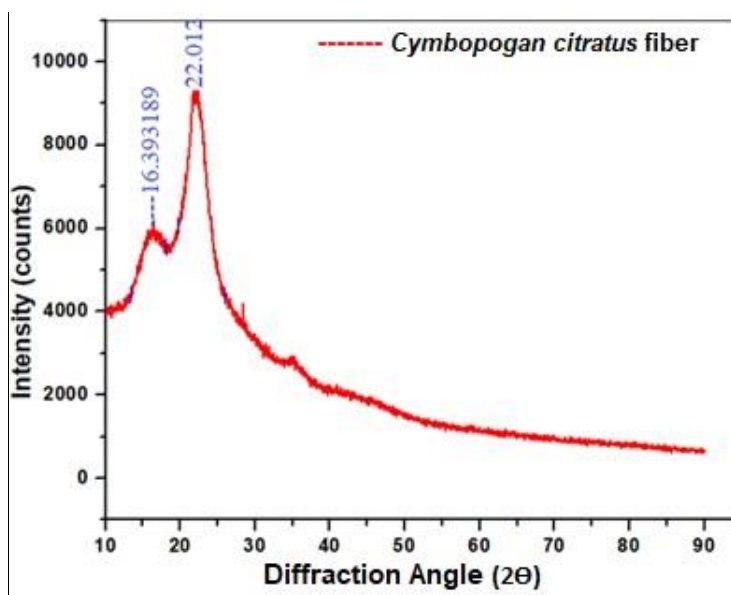
Figure 8 depicts the X-ray diffractogram of the *Cymbopogon citratus* fiber. Two clearly observed peaks were found in the XRD spectra of *Cymbopogon citratus* fiber at around  $2\theta = 16.39^\circ$  and  $22.01^\circ$ . The first peak was noted at a near  $16.39^\circ$  diffraction angle, which indicated the existence of amorphous components in *Cymbopogon citratus* fiber. The next peak showed the crystalline constituents in the *Cymbopogon citratus* fiber. The high crystallinity index (CI value) of the fiber showed excellent mechanical properties and molecular arrangement (Indran *et al.* 2014). The degree of crystallinity in the fiber structure can be recognized by observing the diffraction peak's sharpness, where the sharper the peak of diffraction, the higher the fiber's degree of crystallinity (Alemdar and Sain 2008). The CI value of the *Cymbopogon citratus* fiber was computed in accordance with Segal empirical method, as shown in Eq. 5, which was 35.2% and the crystalline size was estimated as 4.28 nm. The CI value of the *Cymbopogon citratus* fiber was similar to other natural fibers as displayed in Table 8, which was similar to oil palm fruit (34.1%) (Jebadurai *et al.* 2019), *Calotropis gigantea* fruit bunch (36%) (Narayanasamy *et al.* 2020), and *Tridax procumbens* (34.5%) (Chakravarthy *et al.* 2020). The value also was lower than that of Saharan aloe vera cactus (52.6%) (Balaji and Nagarajan 2017) and tilifolia (41.7%) (Narayanasamy *et al.* 2020), jute (65.8%), sisal (71%), flax (80%), and hemp (88%) (Kulandaivel *et al.* 2020). Thus, the crystallinity value depends on the cellulose content and the types of plants. There are relationships between fiber stiffness, crystallinity degree region, as well as cellulose content. Increasing the crystallinity region will increase the



fibers' stiffness. Higher fiber crystallinity is interconnected with the fibers' cellulose content. From the X-ray diffraction analysis, there is a possibility for the application of *Cymbopogon citratus* fiber as a reinforcement for polymer composites.

**Table 8.** Crystallinity of *Cymbopogon citratus* Fiber and Other Natural Fibers

Fiber	Crystallinity Index (%)	References
<i>Cymbopogon citratus</i>	35.2	Current study
Oil Palm Fruit	34.1	(Jebadurai <i>et al.</i> 2019)
<i>Calotropis gigantea</i> Fruit Bunch	36.0	(Narayanasamy <i>et al.</i> 2020)
<i>Tridax procumbens</i>	34.46	(Chakravarthy <i>et al.</i> 2020)
<i>Tilofilia</i>	41.7	(Narayanasamy <i>et al.</i> 2020)
Saharan Aloe Vera Cactus	52.6	(Balaji and Nagarajan 2017)
Jute	65.8	(Kulandaivel <i>et al.</i> 2020)
Sisal	71	(Kulandaivel <i>et al.</i> 2020)
Flax	80	(Kulandaivel <i>et al.</i> 2020)
Hemp	88	(Kulandaivel <i>et al.</i> 2020)



**Fig. 8.** X-ray diffraction pattern of *Cymbopogon citratus* fiber

## CONCLUSIONS

This study emphasizes the fibers extracted from the leaves of the *Cymbopogon citratus* plant in order to evaluate the possibility of using them as potential biodegradable materials and adds value to the existing knowledge on *Cymbopogon citratus*.

1. The higher lignin content of the *Cymbopogon citratus* can offer relatively higher rigidity in comparison to the existing other natural fiber.
2. The average tensile strength of *Cymbopogon citratus* from single fiber testing was  $43.81 \pm 15.27$  MPa and the tensile modulus of *Cymbopogon citratus* fiber was

found to be  $1.046 \pm 0.33$  GPa, making it an ideal alternative reinforcement material to the conventional fibers.

3. The density of the *Cymbopogon citratus* fiber was found to be lower in comparison with many other reported natural fibers and few synthetic fibers, which may enable lightweight applications.
4. Thermogravimetric analysis indicated that the *Cymbopogon citratus* fiber was thermally stable up to 230 °C, which was estimated within the temperature range of 200 to 350 °C. Thus, the material is applicable for high-temperature applications.
5. Furthermore, this study will be extended to identify the suitability of the *Cymbopogon citratus* fiber with biodegradable matrix derived from natural sources in order to analyze the mechanical and thermal properties of this new kind of composite, the fiber/matrix adhesion, and to evaluate the necessity of a chemical pre-treatment of the fibers. In conclusion, the findings showed that *Cymbopogon citratus* has potential as biodegradable materials, especially for reinforcement in bio-based composites.

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