

Research Article

## Characterization of New Cellulose Fiber Extracted from *Pithecellobium dulce* Tree

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## Abstract

Lightweight materials are continuously required for various parts of automobile and aerospace applications. In this connection, natural fibers are widely used to develop polymer composites due to their being biodegradable and lightweight. The demand for natural fiber for developing the lightweight polymer composite is needed for new fibers. The present work extracts and characterizes the bio-fiber from the *Pithecellobium dulce* (PDs) plant. The *Pithecellobium dulce* fiber (PDF) has a cellulose content of 63.45 wt.%, hemicellulose content of 14.56 wt.%, lignin content of 8.45 wt.%, wax content of 0.37 wt.%, moisture content of 11.71 wt.%, and ash content of 4.85 wt.%. The physical density and crystallinity index of PDF was 1097 kg/m<sup>3</sup> and 9.7 %. The tensile strength and Young's modulus properties were identified as 317–1608 MPa and 8.41–69.61 GPa. The thermal stability of PDF showed at higher temperatures of 339.1°C. This revealed higher cellulose content leading to the higher bonding of cellulose structure. The properties of PDFs can be used to make green polymer composites.

Keywords: Pithecellobium dulce, Chemical analysis, Thermal stability, Mechanical properties, Natural fiber

## 1 Introduction

Natural fibers are widely used in automobiles and aerospace interior parts and many researchers are working on synthetic and natural fibers to pass on the requirement and applications. Synthetic fibers, such as glass, aramid, nylon, and carbon fiber have been used for many years [1]. Considering the weight reduction and biodegradable materials, many researchers are focussing on developing various natural fiber composites by identifying new natural fibers. Researchers have identified various natural fibers, such as *Sansevieria ehrenbergii* fibers [2], *Acacia leucophloea* bark fiber [3], Urtica dioica [4], Dichrostachys cinerea [5], Sansevieria roxburghiana Schult [6], Sida rhombifolia [7], sisal [8], Artisdita hystrix leaf [9], Sida cordifolia [10], Furcraea foetida [11], Ficus racemosa [12], Acacia arabica [13], Hibiscus vitifolius [14], Cyperus pangorei [15], Elephant grass [16], Passiflora foetida stem [17], Perotis indica [18], Shwetark Stem [19], Borassus Fruit [20], Carica papaya bark [21], Prosopis juliflora bark [22], and Acacia planifrons [23]. Natural fibers have less production cost and environmental benefits, which are gaining popularity in composites. Natural fibers are essential in the field of composites because they are environmentally friendly, easy to dispose of, and take less energy to process and extract fiber from plants. Natural fibers can easily replace synthetic fibers due to remarkable qualities, such as low cost, eco-friendliness, and lower density [24], [25].

The chemical and manual extraction processes are less expensive than mechanical retting processes because chemical treatment removes the cellulose and ligneous materials. The fundamental goal is to use natural fibers and fillers as a new reinforcement in lightweight composite structures. Mechanical properties of composites are improved by varying the weight content of fiber and altering the number of layers [26], [27].

Natural plants generate different properties of natural fiber based on the various forms, but on the other hand, their properties are based on specific locations and natural conditions. Research into the differentiation of unique natural fibers has provided additional information on potential applications. Future demand for unique natural fibers is expected to rise as new goods. The global demand for natural fiber requirement is increasing day by day. The main aim is to fulfill the above demand by finding new natural fibers from various natural plants [28], [29].

One such new plant is identified that the *Pithecellobium dulce* (PD) tree is a type of spiny tree, native to the Pacific Coast and surrounding highlands of Mexico, Central America, and Northern South America. It is cultivated throughout India's plains and in the Andaman Islands. The fiber is identified from the skin of the PD plant. The physical, tensile, chemical and thermal properties of PD's bark fiber have yet to be determined and before reinforcing fiber in a polymer matrix the above characterizations must be required. This study is to use single-fiber tensile testing to explore the physical, mechanical, thermal and chemical properties of PD fibers isolated manually from plant stalks.

### 2 Materials and Methods

#### 2.1 Pithecellobium dulce plant and its fiber extraction

PD is known as Manila tamarind, Madras thorn, or camachile. It is a flowering plant of the Fabaceae family [Figures 1(a) and (b)]. PD skins were manually separated from its plant [Figures 1(c) and (d)]. The



**Figure 1**: (a) and (b) *Pithecellobium dulce* (PD) bark tree, (a) and (d) Peeling of PD tree skins, (c) PD skins (e) Peeled PD fibers, and (f) and (g) Skin tipped in NaOH solution and dry PD fibers.

fibers [Figure 1(e)] were extracted by manual process. The PDs were properly cleaned with tap water before getting cleaned with distilled water [Figure 1(e)]. The fibers [Figures 1(f) and (g)] were washed in 2% NaOH solution and dried for a week at ambient temperature. Table 1 shows the various extraction method for natural fibers.

 Table 1: Natural fibers extraction methods

Fiber's Name	<b>Extraction Method</b>	Ref.	
Acacia leucophloea Bark	Water with microbial degradation	[3]	
<i>Urtica dioica</i> L	Dew/microbial retting	[4]	
Wax in cotton fiber	Alcohol extraction method	[30]	
Flax	Enzymatic method	[31]	
Sisal	Mechanical decortification	[8]	
Furcraea foetida	Water retting	[11]	
Ficus racemosa	Water retting	[12]	
Hibiscus vitifolius	Water/microbial retting	[14]	
Cyperus pangorei	Water	[15]	



## 2.2 Chemical analysis of PDFs

Chemical properties of PDFs such as hemicellulose, wax, pectin, cellulose, ash, and moisture content were calculated by standard test methods [11]. PDF density is obtained using the Mettler Toledo xsz05 testing method. The moisture content of the PDF was examined using the electrical humidity analysis, and the ash component was quantified according to ASTM standard E1755-01 (Sartorious, model MA45). The wax mixture is calculated in the Conrad method. Pectin is analyzed in the form of ammonium oxalate KOH (Potassium hydroxide).

#### 2.3 Lignin

The biomass solvent extraction process was used to measure the amount of lignin. The sample was kept at atmospheric temperature for one day before being boiled at 100 °C using a hot plate. The solution become filtered and the strong residue was washed in the deionized water. During the washing, the detected sulfate ion was mixed in 10% of the Barium Chloride solution. This sample was dried in a hot air oven at 110 °C until constant weight was obtained. The residue weight was measured to record the lignin content.

## 2.4 Hemicellulose

The biomass solvent extraction was used to determine the amount of hemicellulose. The 150 mL of NaOH (0.5 mol/L) was added to 1 g of *Pithecellobium dulce* (B). The solution was heated at 80 °C using a hot plate for 3.5 h. The deionized water was used to wash and detected Na<sup>+</sup>. The sample was dried in a hot air oven at 110 °C. Finally, the weight of the residual was measured to find the hemicellulose content.

#### 2.5 Moisture

Moisture content in fiber affects a substance's physical qualities, such as weight, density and conductivity. Weight reduction after drying is a common indicator to find moisture content. To perform the moisture test 5 g of samples were used. Three moisture dishes were first rinsed in water and properly dried. The weight of each dish was determined using a weight balance. Next step, 5 g of samples were put into moisture dishes, followed by these three samples were placed in a moisture oven for three hours until they reached a consistent weight. After that, the dishes were placed in a desiccator, and each sample weight was measured as soon as they were cool. The average moisture content of each sample was determined by calculating weight losses in each sample. Finally, the moisture content is calculated by Equation (1) [2]–[5].

Moisture (%by weight) = 
$$\frac{W_1 - W_2}{W_1 - W} \times 100$$
 (1)

Where,

W is the Weight of dish (g)  $W_1$  is Weight of dish + Weight before dying (g)  $W_2$  is Weight of dish + Weight after dying (g)

#### 2.6 Ash

The inorganic residue (minerals) left following full oxidation of organic matter is referred to as ash content. Dry ashing entails heating the sample to 500-600 °C in a muffle furnace. To perform the ash test, it needs a 5 g of sample. The procedure for the test is to place samples in a cool muffle furnace and burn for 1 hour at 550 °C. The muffle furnace is turned off and allowed it to cool to at least 250 °C by gently opening the door to avoid losing any fluffy ash. The safety tongs is used to transfer the crucibles to a desiccator, cover the crucibles, and close the desiccator to cool to room temperature before weighing. Finally, the ash content is calculated by Equation (2) [2], [5], [30].

$$Total ash content = \frac{(W_2 - W_1) \times 100 \times 100}{(W_1 - W) \times (100 - M)}$$
(2)

 $W_1$  is Crucible weight + sample (g))

 $W_2$  is Crucible weight + ash content (g)

*W* is Empty crucible weight (g)

M is Moisture content in % of the sample.

## 2.7 Pectin

Pectic was found between the primay and secondary walls of the natural fiber, which was the derivatives of carbohydrate and methylated polymer of galacturonic acid. 50 g of the blended sample is mixed with 400 mL of 0.005 N HCl, and sample is boiled for 2 h at 80 and

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90 °C in 400 mL of water. Water loss was compensated by adding sufficient water to cool and fiber sample was transferred to a volumetric flask of 500 mL, and do the filter process. 100 mL aliquot of sample was filled into a conical flask and subsequently add 250 mL of distilled water, and then followed to neutralize the acid with 1N NaOH using phenolphthalein as indicator, followed to add another 10 mL of 1N NaOH and allow standing overnight. Hereafter, 50 mL of 1N acetic acid and 25 mL of 1 N calcium chloride were added to the solution and then allow to stand for 1 h and boil for 1–2 min. After boiling, a solution was filtered through a previously weighed filter paper. The precipitate was washed in free of chloride with hot water. The precipitate was dried and in a desiccator and weighed again. Finally, the ash content is calculated by Equation (3) [2], [5], [30].

$$Pectin (\%) = \frac{Weight of precipiate}{100 ml of filtration taken for estimation \times weight of sample}$$
(3)

## 2.8 Cellulose

Cellulose is the important element in plants. It is composed of glucose molecules joined together within the form of repeating units of cellobiose with several cross-linkages. Initially, the selected sample is taken in a tube with addition of 10 mL of H<sub>2</sub>SO<sub>4</sub> (Sulfuric acid) and allowed to stand for one hour. After that 0.5 g of vortex mixer are add to the sample and reagent with 3.0 mL acetic/nitric, followed to place the tubes in a waterbath at 100 °C for 30 min. The tube with mixture was cooled and centrifuged at 1200 rpm for 15-20 min. The residue contain the discard tube was filled with distilled water and add 10 mL of H<sub>2</sub>SO<sub>4</sub>, and then allow to stand for one hour. Then, it was diluted 1.0 mL of the solution to 100 mL with distilled water. After that, 1 mL of this diluted solution with a blank of distilled water were mixed with 10 mL of anthrone reagent was added to the tubes, which was mixed properly and kept in the boiling water tub for 10 min. Then, it was cooled and measured the concentration of cellulose.

#### 2.9 Physical analysis of PDFs

A single long fiber was splitted into short lengths. A microscope was used to project the fiber surface onto

a screen. A ruler was used to measure it in millimeters. The linear density was calculated using the weights and lengths of the objects investigated. To determine the average diameter, 100 fibers were investigated, and 10 fibers were tested to get the average linear density. Equation (4) was used to compute the volume density of natural fibers using the average linear density and average diameter of fibers [2].

$$\rho_f = \frac{M}{\frac{\pi d^2}{4}l} \tag{4}$$

where M is mass of natural fiber, d is average diameter of the fiber, and l is length of natural fiber.

# 2.10 Fourier Transform-Infrared Spectroscopy (FTIR)

FTIR spectroscopy was plotted using the Perkin Elmer Spectrum spectrometer. The scanning rate of 30 scans per minute had been used to identify the spectrum. At room temperature and relative humidity of 65%, the total wavenumber area varied from 500 to 4000 cm<sup>-1</sup>. The fiber bundle was cut into 2–3 mm pieces and processed into powder form using a mortar. The collected powder was combined with another KBr matrix and compressed into a pellet. The spectra were recorded using the pellet.

#### 2.11 Analysis of x-ray diffraction

An X-ray diffractometer (Model: Dmax 2500) had been used to record the SEFs' broad-angle XRD spectra. X-ray tube derived monochromatic Cu K $\alpha$ radiation. Using a high-intensity spectrum, the crystallinity index was calculated by a reflection medium at a scan speed of 4 °C/min. All samples were tested for 2 h at a 5–50° angle [2].

$$CS_{\frac{1}{4}} = \frac{H_{002} - H_{am}}{H_{002}}$$
(5)

Where,  $H_{21.72}$  and  $H_{15.81}$  are the higher peaks at  $2\theta = 42.96^{\circ}$  and  $38.79^{\circ}$ , respectively. *CS* is calculated using the diffraction pattern of the cellulose's, 200 lattice planes. Using Scherrer's formula, the *CS* was computed using Equation (5)



$$CS_{\frac{1}{4}} = \frac{K\lambda}{B\cos\theta} \tag{6}$$

Where, *K* is Scherrer's constant (0.89),  $\beta$  is the complete width of the peak at half maximum in radian,  $\lambda$  (*delta*) is the radiation wavelength (0.15406), and  $\theta$  (*theta*) is Bragg angle in radian [Equation (6)].

## 2.12 Field Emission Scanning Electron Microscopy (FESEM)

The surface structure of PD fiber was examined by using high-resolution FESEM (Model: Thermo scientific Apreo S). The scanning specification was a resolution of 1.5 nm and 30 voltage acceleration, a working distance of 48 mm and 5X magnification.

## 2.13 Thermogravimetric Analysis (TGA) and Derivative Thermogravimetry (DTG)

The TGA deals with the amount of fiber material eradicated in percentage against the temperature (when heated), while DTG exhibits the rate at which these fiber materials are eradicated in the ratio of the percentage of the fiber to the time. The Jupiter simultaneous was used to analyze the thermal behavior of PDFs (Model: STA 449F3, Make: NETZSCH, Place: Germany). DTA is a temperature difference between the obtainable sample and the pre-known values reference sample (dT = T obtainable-T known). From the above inference, it can clearly stated that the DTG plot is the derivative plot of TGA [2].

$$In\left[In\left(\frac{1}{y}\right)\right] = -\left(\frac{E_a}{R}\right)\left[\left(\frac{1}{T}\right) + k\right]$$
(7)

Where, R = 8.32 J/mol K, which is the universal gas constant, *T* is maintained temperature (Kelvin), *K* is constant, *y* is the proportion of initial molecules that have not yet decomposed  $(w_t/w_o)$ , where  $w_t$  denotes the sample weight at any time *t* and  $w_o$  denote the fiber sample's beginning weight.

#### 2.14 Differential thermal analysis

Differential thermal analysis (DTA) is a way of figuring out and quantitatively studying the chemical composition of materials by way of gazing at the thermal behavior of a pattern as it is heated. The powder form of fibers was heated from room temperature to 1000 °C in nitrogen gas atmospheric at a heat rate of 10 °C/min. During heating, the nitrogen gas was supplied at a rate of 20 mL/min.

#### 2.15 Single fiber test

A single-fiber tensile test was carried out with different sizes of fibers in order to prove the maximum strength of the testing fiber. The INSTRON universal testing machine was used to conduct the tensile test with a load cell of 5 kN. Equation (8) is used to calculate the total cross-head displacement ( $\delta t$ ) during fiber testing [2].

$$\frac{\delta t}{F} = \left[\frac{1}{EA}\right]L + C \tag{8}$$

where F is applied load (Newton), L is gage length (mm), E is Young's modulus, A is cross-sectional area of fiber, and c is machine compliance. Twenty numbers of fibers were tested for calculating the tensile properties.

#### **3** Results and Discussion

#### 3.1 FESEM analysis of PDFs

A morphological study of fiber surface using FESEM showed the presence of many small cracks near half the length of the fiber surface [Figure 2(a)]. Due to the manipulation of individual fiber components in the fiber masses, the outside of the fiber showed roughly [Figure 2(b)]. By adopting a connecting technique, the greater stiffness of the fiber might make it simpler to attach to the fiber matrix. The whitish material in the fiber zone [Figure 2(c)] indicates the availability of amorphous cellulose, hemicellulose, and lignin [8]. The existence of sticky polysaccharides or contaminants between the filaments is also shown by the FESEM picture (Figure 2). When the fiber is chemically treated, the contaminants are filtered out, and a robust fiber structure is generated, improving fiber-matrix adhesion and The diameter of PDFs is ranging from 20.5-86 µm.



Figure 2: SEM of the PDFs.

## 2.1 PDFs chemical analysis

Table 2 lists the chemical characteristics of PDFs and numerous other plant fibers. Natural fiber's functional qualities are mostly determined by its cellular value and lignin concentration. Natural fibers with more cellulose content have high tensile strength and modulus, also high lignin content has a longer lifespan. In comparison to coir of 37 wt.%, Kenaf of 53.14 wt.%, Palm oil of 65 wt.%, Jute of 72 wt.%, and hemp fibers of 74 wt.%, PDFs have a comparatively high quantity of cellulose (74.89 wt.%). The PDFs contain 12.80 wt.% lignin, which is higher than flax fiber (3 wt.%), hemp fiber (4 wt.%), *Sida rhombifolia* fiber (7.48 wt.%) [7], and kenaf fiber (8.18 wt.%).

Table 2: Comparison of chemical compositions of raw PDFs with various natural fibers

Fiber's Name	Cellulose (%)	Hemicellulose (%)	Lignin (%)	Wax (%)	Moisture Content (%)	Ash (%)	Ref.
Pithecellobium dulce	63.45	14.56	8.45	0.37	11.71	84.85	-
Acacia leucophloea	68.09	13.6	17.73	0.55	8.83	0.08	[3]
Sida rhombifolia	75.09	15.43	7.48	0.49	12.02	4.07	[7]
Artisdita hystrix	59.54	11.35	8.42	-	-	-	[9]
Furcraea Foetida	68.35	11.46	12.32	0.24	5.43	6.53	[11]
Acacia arabica	68.1	9.36	16.86	0.49	-	-	[13]
Hibiscus vitifolius	75.09	13.34	10.42	0.17	11.31	0.94	[15]
Cyperus pangorei	68.5	-	17.88	0.17	9.19	-	[15]
Perotis indica	68.4	15.7	8.35	0.32	9.54	4.32	[18]
Acacia planifrons	73.1	9.41	12.04	0.57	8.21	4.06	[24]



## 3.3 FTIR analysis

The FTIR determines the main active groups contained in PDFs throughout a wavelength range of  $500-4000 \text{ cm}^{-1}$  (Figure 3). The OH expansions of free hydroxyl groups and cellulose hydrogen are shown by the lowest exposure at 3880 cm<sup>-1</sup> and 3414 cm<sup>-1</sup>, with a wide waveform peak at 3697 cm<sup>-1</sup> [19]. A narrow band of 2985 cm<sup>-1</sup> shows the growing C-H vibration. The 2387 cm<sup>-1</sup> band is mostly caused by lignin ion vibrations [2]. The peak at 2005 cm<sup>-1</sup> is attributed to the presence of higher crystalline cellulose, which displays CH<sub>2</sub> vibration [2]. A significant height of 1658 cm<sup>-1</sup> shows lignin (guaicycl) cellulose corresponds to the respiratory guaiacyl ring with a substantial C-O stretch. A prominent peak at 1072 cm<sup>-1</sup> shows C-O-C extraction cellulose absorption. A maximum value of 908 cm<sup>-1</sup> is associated with cellulose structural connections. OH group bending outside the plane may be calculated up to 561  $\text{cm}^{-1}$  [2].

## 3.4 XRD Analysis

The PDF XRD pattern is shown in Figure 4. The diffractogram structure revealed two well-defined peaks with angles of  $2\theta = 17.45^{\circ}$ , suggesting the presence of monoclinic Cellulose-I crystals [1]. The crystalline cellulose has supported high stability and strength in natural fibers, and the crystallinity index (CI) is used to quantify its existence [14]. When utilizing the Segal equation provided in the scale (4), the PDF CI value was found to be 27.20%. This number is relatively less compared to that of fibers such as Borassus (38.4%) [20], Acacia



Figure 4: XRD spectrum of PDFs.

*leucophloea* (42%), and *Furcraea foetida* (42.7%) [11]. The Scherrer formula calculated the crystallite PDF size, which came out at 1.38 nm. Table 3 shows the CS and CI values of PDFs and other natural fibers.

Fiber's Name	Crystalline Index (%)	Crystal Size (nm)	Tensile Strength (MPa)	Tensile modulus (GPa)	Elongation (%)	Density (kg/m³)	Ref.
Pithecellobium dulce	9.70	1.38	513-1226	6.9–9.6	0.8-1.37	1097	-
Dichrostachys cinerea	57.82	-	$873\pm14$	-	-	1240	[5]
Sida rhombifolia	56.6	2.75	$673\pm14$	-	-	1320	[7]
Sida cordifolia	56.92	18	$703.95\pm23.73$	$42.84\pm2.1$	$2.89\pm0.24$	1330	[10]
Furcraea foetida	52.6	28.36	$623.52\pm45$	$6.52\pm1.9$	$10.32\pm1.6$	778	[11]
Cyperus pangorei	41	-	$196\pm 56$	$11.6\pm2.6$	1.69	1102	[15]
Passiflora foetida	67.36	4.23	248–942	11 - 48	1.38-4.67	1328	[17]
Perotis indica	48.3	15	317-1608	8.41 - 69.61	1.38-4.24	785	[18]

Table 3: Physico-mechanical properties of PDFs and other natural fibers

#### 3.5 TG AND DTG analysis

The thermal behavior of PDFs was evaluated using the DTG and TG curves (Figure 5). The initial thermal breakdown of the fiber occurred at 68.57 °C due to moisture loss and evaporation [1], [14]. The second phase of PDF thermal deterioration was discovered to occur between 100 and 150 °C, with a maximum temperature of 138.05 °C. A quick increase to 334.73 °C caused a considerable weight loss of around 59.76% due to a reduction in cellulose temperature. Acacia leucophloea fiber [3], Sida rhombifolia fiber [7], Shwetark fiber [19], and Cyperus pangorei fiber show a similar trend at 346.8 °C, 328.2 °C, 350 °C, and 331.1 °C. The molecular structure of lignin makes it more thermally stable compared to other fiber components [2]. At 548°C, the fiber residual mass percent is 23.35 percent. The activation potential associated with the thermal degradation of PDFs at temperatures ranging from 80 °C to 360 °C is calculated using the Broido equation. The overall PDF regeneration power was 55.295 kJ/mol based on the structure ln(1/y) compared to T-1 in Figure 6.

## 3.6 DTA analysis

The derivative analysis of the fiber is investigated, and the DTA curve is given in Figure 7. The comparison for DTA with temperature in (°C) shows the complex peak arises at 69.3 °C with an onset of 68.4 °C. The area is shown as 7.085 J/g.

#### 4 Mechanical Properties of PDFs

Stress versus strain curves for ten PDF samples are shown in Figure 8. The stress structure determines the strength of PDFs subconsciously. The fiber's ultimate resistance to the solid load and value was determined to be 513–1226 MPa, whereas the strength of various natural fibers, such as palm, petiole bark, kenaf bast, bananas and bamboo was found to be 377, 185.52, 427–519, 600, and 503 MPa, respectively [14]. The tensile modulus revealed by PDF was derived from the slope of the stress structure, which was found to be 6.9–9.6 GPA, and the extension to PDF failure was predicted to be 0.8–1.37%. As a result, when compared to the majority of plant fibers in Table 3, PDFs were discovered to have significantly powerful



Figure 5: TG and DTG curves of PDFs.



**Figure 7**: Derivative Thermogravimetry analysis of PDFs.



Figure 8: The stress vs. strain curves of PDFs.

solvents. The high cellulose content in PDFs is responsible for their great potency in mechanical properties. Another important aspect affecting natural fiber mechanical properties is the angle at which cellulose microfibrils are lodged in the lignin, commonly known as the microfibrillar angle [17]. Natural fibers with a loss microfibrillar angle are known for their strength and durability, whereas those with a high microfibrillar angle are known for their ductility [22]. The microfibrillar angle of the computer-generated PDF with Equation (7) is 11.28°. In addition to mechanical properties, plant fiber density is critical in reducing weight and the rate at which it is utilized in biocomposites. It was observed that PDF has a density of 1097 kg/m<sup>3</sup>.

## 5 Conclusions

Physicomechanical, chemical, morphological, and thermal studies were used to determine the appropriateness of cellulosic fibers derived from Pithecellobium dulce for use in combination. The capacity of PDF to reinforce the polymer matrices was validated by character separation investigations and intensive testing. The inclusion of a high cellulose concentration (63.45%), a good moisture level (11.71%), and a low wax content (0.37%) improves PDF strength and promotes improved fiber-matrix adhesion. PDF has a microfibrillar angle of 11.28°, and natural fibers with a microfibrillar angle of less than 10° have greater ductility with increasing stress. PDF has a high crystallinity index (9.70%) and a tiny crystallite size (1.38 nm) compared to other natural fibers, making it excellent for the manufacturing of polymer composites. TGA found thermal stability (up to 360 °C) and kinetic regeneration capacity (55.295 kJ/mol) in PDF, which are important properties for compounding. The PDF will be treated with various chemicals for improving the mechanical strength and making good bonding with the polymer matrix. This study found that PDFs may be utilized to make a perishable polymer composite that can be used to make automotive parts, wall panels, construction partition boards, and sports equipment.

## **Author Contributions**

T.P.S.: conceptualization, investigation, reviewing and editing; P.N.: investigation, methodology, writing an original draft; L.R.K. & G.R.K.: research design, data analysis; G.V.S. & S.S.K.: conceptualization, data curation, writing—reviewing and editing, All authors have read and agreed to the published version of the manuscript.

## **Conflicts of Interest**

The authors declare no conflict of interest.

## References

- T. P. Sathishkumar, P. Navaneethakrishnan, S. Shankar, R. Rajasekar, and N. Rajini, "Characterization of natural fiber and composites–A review," *Journal* of *Reinforced Plastics and Composites*, vol. 32, no. 19, 2013, doi: 10.1080/15685543.2013.816652.
- [2] T. P. Sathishkumar, P. Navaneethakrishnan, S. Shankar, and R. Rajasekar, "Characterisation of new cellulose *Sansevieria ehrenbergii* fibers for polymer composites," *Composite Interfaces*, vol. 20, no. 8, pp. 575–593, 2013, doi:10.1080/ 15685543.2013.816652.
- [3] V. P. Arthanarieswaran, A. Kumaravel, and S. S. Saravanakumar, "Characterisation of new natural cellulosic fiber from *Acacia leucophloea* Bark," *International Journal of Polymer Analysis and Characterization*, vol. 20, pp. 367–376, 2015, doi: 10.1080/1023666X.2015.1018737.
- [4] L. Bacci, S. D. Lonardo, L. Albanese, G. Mastromei, and B. Perito, "Effect of different extraction methods on fiber quality of nettle (*Urtica dioica* L.)," *Textile Research Journal*,

vol. 81, no. 8, pp. 827–837, 2010, doi: 10.1177/0040517510391678.

- [5] P.G. Baskaran, P. Senthamarainkannan, K. Marimuthu, and S. S. Saravanakumar, "Characterisation of new natural cellulosic fiber from the bark of *Dichrostachys cinerea*," *Journal of Natural Fibers*, 15, no. 1, pp. 62–68, 2017, doi: 10.1080/ 15440478.2017.1304314.
- [6] M. G. Krishna, C. Kailasanathan, and B. Nagarajaganesh, "Physico-chemical and morphological characterisation of cellulose fibers extracted from *Sansevieria roxburghiana Schult*. & *Schult*. F leaves," *Journal of Natural Fibers*, vol. 19, no.9, pp. 3300–3316, 2020, doi: 10.1080/ 15440478.2020.1843102.
- [7] R. Gopinath, K. Ganesan, S. S. Saravanakumar, and R. Poopathi, "Characterisation of new cellulosic fiber from the stem of *Sida rhombifolia*," *International Journal of Polymer Analysis and Characterization*, vol. 21, no. 2, pp. 123–129, 2021, doi: 10.1080/1023666X.2016.1117712.
- [8] J. Naveen, M. Jawaid, P. Amuthakkannan, and M. Chandrasekar, "Mechanical and physical properties of sisal and hybrid sisal fiberreinforced polymer composites," *Woodhead Publishing Series in Composites Science and Engineering*, vol. 1, pp. 427–440, 2019, doi: 10.1016/B978-0-08-102292-4.00021-7.
- [9] M. K. Pandiarajan, P. Senthamaraikannan, and S. S. Saravanakumar, "Physicochemical properties of new cellulosic Artisdita hystrix leaf fiber," *International Journal of Polymer Analysis and Characterization*, vol. 21, pp. 663–668, 2006, doi: 10.1080/1023666X.2016.1194636.
- [10] P. Manimaran, M. Prithiviraj, S. S. Saravanakumar, V. P. Arthanarieswaran, and P. Senthamaraikannan, "Physicochemical, tensile, and thermal characterisation of new natural cellulosic fibers from the stems of *Sida cordifolia*," *Journal of Natural Fibers*, vol. 15, no. 6, pp. 860–869, 2017, doi: 10.1080/15440478.2017.1376301.
- [11] P. Manimaran, P. Senthamaraikannan, S. M. Rangappa, M. K. K. Marichelvam, and M. Jawaid, "Study on the characterisation of *Furcraea foetida* new natural fiber as composite reinforcement for lightweight applications," *Carbohydrate Polymers*, vol. 181, pp. 650–658, 2018, doi: 10.1016/j.carbpol.2017.11.099.

- [12] P. Manimaran, S. P. Saravanan, M. Prithiviraj, "Investigation of physico chemical properties and characterization of new natural cellulosic fibers from the bark of *Ficus racemosa*," *Journal* of Natural Fibers, vol. 18, no. 2, pp. 274–284, 2021, doi: 10.1080/15440478.2019.1621233.
- [13] P. Manimaran, S. S. Saravanakumar, K. Mithun, and P. Senthamaraikannan, "Physicochemical properties of new cellulosic fibers from the bark of *Acacia arabica*," *International Journal* of *Polymer Analysis and Characterization*, vol. 21, pp. 548–553, 2016, doi: 10.1080/1023666X. 2016.1177699.
- [14] N. M. Pannirselvam, R. Gopinath, T. P. Sathishkumar, "Physico-mechanical, chemical composition and thermal properties of cellulose fiber from *Hibiscus vitifolius* plant stalk for polymer composites," *Journal of Natural Fibers*, vol. 19, no. 3, pp. 1–16, 2021, doi: 10.1080/15440478.2021.1941484.
- [15] M. Ramesh, M. T. Selvan, L. Rajeshkumar, C. Deepa, and A. Ahmad, "Influence of Vachellia nilotica subsp. indica tree trunk bark nano-powder on properties of milkweed plant fiber reinforced epoxy composites," Journal of Natural Fibers, vol. 19, pp. 13776–13789, 2022, doi: 10.1080/15440478.2022.2106341.
- [16] K. M. M. Rao, A. V. R. Prasad, M. N. V. R. Babu, K. M. Rao, A. V. S. S. K. S. Gupta, "Tensile properties of elephant grass fiber reinforced polyester composite," *Journal of Material Science*, vol. 42, no. 9, pp. 3266–3272, 2007, doi:10.1007/s10853-006-0657-8.
- [17] T. Natarajan, A. Kumaravel, and R. Palanivelu, "Extraction and characterisation of natural cellulosic fiber from *Passiflora foetida* stem," *International Journal of Polymer Analysis and Characterization*, vol. 21, no. 6, pp. 478–485, 2016, doi:10.1080/1023666X.2016.1168636.
- [18] V. Bhuvaneswari, M. Priyadharshini, C. Deepa, D. Balaji, L. Rajeshkumar, and M. Ramesh, "Deep learning for material synthesis and manufacturing systems: A review," *Materials Today: Proceedings*, vol. 46, pp. 3263–3269, 2021, doi: 10.1016/j.matpr.2020.11.351
- [19] K. Raja, B. Prabu, P. Ganeshan, V. S. Chandra Sekar, B. N. Ganesh, "Characterisation studies of natural cellulosic fibers extracted from



shwetark stem," *Journal of Natural Fibers*, vol.18, no.11, pp. 1934–1945, 2021, doi: 10.1080/15440478.2019.1710650.

- [20] N. Kumar, A. Singh, K. Debnath, and N. Kumar, "Water absorption and mechanical behaviour of Borassus fruit fibre-reinforced composites," *Emerging Materials Research*, vol. 9, no. 1, pp.10–17, 2019.
- [21] A. S. Kumaar, A. Senthilkumar, T. Sornakumar, S. S. Saravanakumar, and V. P. Arthanariesewaran, "Physicochemical properties of new cellulosic fiber extracted from *Carica papaya* bark," *Journal of Natural Fibers*, vol. 16, no. 2, pp. 175– 184, 2019.
- [22] S. S. Kumar and V. M. Raja, "Processing and determination of mechanical properties of *Prosopis juliflora* bark, banana and coconut fiber reinforced hybrid bio composites for an engineering field," *Composites Science and Technology*, vol. 208, Art. no. 108695, 2021.
- [23] P. Senthamaraikannan, S. S. Saravanakumar, S. M. Rangappa, M. Jawaid, and S. Siengchin, "Physico-chemical and thermal properties of untreated and treated *Acacia planifrons* bark fibers for composite reinforcement," *Materials Letters*, vol. 240, pp. 221–224, 2019.
- [24] L. Rajeshkumar, M. Ramesh, V. Bhuvaneswari, D. Balaji, and C. Deepa, "Synthesis and thermomechanical properties of bioplastics and biocomposites: A systematic review," *Journal* of Materials Chemistry B, 2023, doi: 10.1039/ D2TB02221D.
- [25] U. V. Akhil, N. Radhika, B. Saleh, S. A. Krishna, N. Noble, and L. Rajeshkumar, "A comprehensive review on plant-based natural fiber reinforced polymer composites: Fabrication, properties, and applications," *Polymer Composites*, 2023, doi: 10.1002/pc.27274.

- [26] V. Bhuvaneswari, L. Rajeshkumar, T. P. Sathishkumar, G. Rajeshkumar, S. M. Rangappa, and S. Siengchin, "Effect of fiber orientation on physical and mechanical properties of Typha angustifolia natural fiber reinforced composites," *Applied Science and Engineering Progress*, vol. 16, no. 3, 2023, doi: 10.14416/j. asep.2022.11.004.
- [27] M. Ramesh, L. N. Rajeshkumar, N. Srinivasan, D. V. Kumar, and D. Balaji, "Influence of filler material on properties of fiber-reinforced polymer composites: A review," *e-Polymers*, vol. 22, no. 1, pp. 898–916, 2022.
- [28] T. P. Sathishkumar, J. de-Prado-Gil, R. Martínez-García, L. Rajeshkumar, G. Rajeshkumar, S. M. Rangappa, S. Siengchin, A. M. Alosaimi, and M. A. Hussein, "Redeemable environmental damage by recycling of industrial discarded and virgin glass fiber mats in hybrid composites—An exploratory investigation," *Polymer Composites*, vol. 44, no.1, pp. 318–329, 2023.
- [29] M. Ramesh, R. Janani, C. Deepa, and L. Rajeshkumar, "Nanotechnology-enabled biosensors: A review of fundamentals, design principles, materials, and applications," *Biosensors*, vol. 13, no. 1, Art. no. 40, 2022, doi: 10.3390/bios1301004.
- [30] C. M. Conrad, "Determination of wax in cotton fiber, a new alcohol extraction method," *Industrial Engineering Chemistry Analytical Edition*, vol. 16, no. 12, pp. 745–748, 1994, doi: 10.1021/i560136a007.
- [31] J. A. Foulk, W. Y. Chao, D. E. Akin, R. B. Dodd, and P. A. Layton, "Enzyme-retted flax fiber and recycled polyethylene composites," *Journal of Polymers and Environment*, vol. 12, no. 3, pp. 165–171, 2004, doi: 10.1023/B:JO OE.0000038548.73494.59.