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A new study on characterization of Pithecellobium dulce fiber as composite reinforcement for light-weight applications

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**A New Study on Characterization of Pithecellobium Dulce  
Fiber as Composite Reinforcement for Light  
weight Applications**

Journal:	<i>Journal of Natural Fibers</i>
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**Reviewer(s)' Comments to Author:****Reviewer: 1****Comments to the Author**

WJNF-2018-0082, "A New Study on Characterization of Pithecellobium Dulce Fiber a Composite Reinforcement for Light weight Applications" The manuscript presents the characterization of a new bio-fiber, Pithecellobium Dulce, aimed to be used as reinforcement of polymeric matrices in light weight composites. The topic is adequate for the Journal but the paper, in the present form requires modifications before it can be published, as detailed below.

**General comments:**

The English of the manuscript should be carefully revised. There are grammar problems (i.e. noun + verb conjugation, use of comparative adjectives, etc.). Some examples (but notice that they are not the only ones) of sentences that requires adequacy are indicated below:

**Page 2, lines 11-13:** "As a bio-fiber, Pithecellobium Dulce is abundantly available in the all over the world and has higher cellulose content (75.15 wt.%) and low density (865 kg/m<sup>3</sup>)." Higher cellulose content than what?? The use of comparative adjectives should be revised. Moreover, as the properties of the natural fibers present great variability the experimental values obtained should consider it. In this sense, at list the mean value  $\pm$  standard deviation of the properties should be reported although a statistical study should be better.

**Incorporated in the revised version.**

**Page 2, lines 18-20:** "The Chemical functional group and Crystallinity index (48.4%) of the PDFs were obtained...". Revise the use of the capital letters. Moreover, just one chemical functional group is going to be assessed? If not, "group" should be replaced by "groups".

**Incorporated in the revised version.**

**Page 2, lines 40-45:** "In recent days the increasing awareness in utilizing natural fiber as an alternate reinforcement for synthetic fiber in polymer matrix composites especially due to the environmental impact of synthetic fibers...". The main verb in the sentence is missed.

**Incorporated in the revised version.**

**Page 3, lines 5-11:** "Due to the above advantages natural fiber reinforced composites used in many fields such as automotive, electrical industrial and construction (Arpitha et al. 2017)." The main verb is missed.

**Incorporated in the revised version.**

**Page 3, lines 28-31:**

"The performance of natural fiber composites are primarily..." Revise number agreement between noun and verb.

**Incorporated in the revised version.**

### Specific comments

**Page 3, lines 17-24:** "A number of bio-fibers, such as *Sansevieriacylindrica*, *Sansevieriaehrenbergii*, *Cyperuspangorei*, *Acacia leucophloea* and sisal have previously been proven to replace the synthetic fiber in several applications". There are much more common natural fibers that are being used thoroughly as filler/reinforcement for polymeric matrices (i.e. abaca, curaua, henequen, pineapple, sisal, banana, cotton, flax, hemp, jute, ramie, coir, oil palm, etc.), why list those few and practically unknown ones?

**Page 4, line 45:** "...and single tensile test". Do you want to say "single fiber" tensile test?

**Page 5, equation 1:** if I replace the corresponding masses and density in the equation I obtain something with units = Kg fibers / (Kg toluene \* m<sup>3</sup> toluene), which is not consistent with the units expected for the density of a natural fiber. Thus, calculations should be revised.

Sorry for the inconvenience. Due to the typographical error. There is a mistake (negative sign missing) in equation (1).

Please refer the corrected one.

$$\rho_{PDFs} = \left( \frac{m_2 - m_1}{(m_3 - m_1) - (m_4 - m_2)} \right) \rho_T \quad (1)$$

**Page 7, lines 44-47:** "From the existing literature, the cross-sectional area of the fiber was assumed as circular cross-section." This "existing literature", or at least part of it, should be cited.

**Page 8, lines 5-6:** "The fiber diameter is approximately 60–230 μm.". These boundary values are too different, as compared with the range of fiber diameters of the natural fibers reported in table 1. Thus, enough fibers should be measured to perform a statistical analysis.

Diameter of 25 samples has been measured and reported the mean and standard deviation values of diameter in table 1 and Weibull analysis carried out (Figure 2 b).

**Page 8, lines 12-13:** In the same line, the density of different fibers should be measured, to get information about the limit values that can be expected.

Incorporated in the revised version.

**Page 8, lines 19-52:** The previous comment also applies to the chemical characterization of the fibers. Moreover, the chemical composition of more common natural fibers should be added to Table 2 for comparison.

Incorporated in the revised version.

**Page 9, X-Ray Diffraction (XRD) Analysis:** the crystallinity index (CI) should be calculated from several samples and presented as mean value ± standard deviation, at least. The CI of common natural fibers should be added to the manuscript for comparison.

Incorporated in the revised version.

1  
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5 **Page 10, lines 10-15:** "A large crystal size of the fiber means condensed surface area as a result  
6 lower water and chemical absorption of fibers." This sentence should be further explained and  
7 related with the crystal size measured for the PDF fiber.

8 **Incorporated in the revised version.**

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11 **Page 10,** Thermogravimetric analysis: the average moisture content of the fibers should be  
12 calculated, it is an important parameter when dealing with natural fibers. Obviously replicate  
13 samples should be tested. A list of thermoplastics resins with working temperature below 170°C  
14 should be added, give an idea of the potential matrices for these fibers.

15 **We can use thermosets as matrices for these fibers.**

16  
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18  
19 **Page 10-11,** Surface Morphological Analysis: re-phrase this section, it is quite repetitive.

20 **We have written this surface morphological analysis based on literature and validate this with**  
21 **other references.**

22  
23  
24 **Page 11,** Tensile behavior: why studying the dependence of the tensile properties with the  
25 "gauge length"? Is not obvious that the properties will vary with it? Is this procedure indicated in  
26 the ASTM D 3822-07 standard? On the other hand and taking into account the enormous  
27 variation in the fiber diameter, a study about the influence of the fiber diameter on the tensile  
28 response should be much more interesting and useful.

29 **We have done single fiber tensile testing as per ASTM D-3822-07. Thank you for your**  
30 **suggestion, In future, we will do study about the influence of the fiber diameter on the tensile.**

31  
32  
33  
34  
35 **We have incorporated all the comments and suggestions given by the you in the revised**  
36 **manuscript. Thank you for your valuable comments and corrections. I request you to please**  
37 **kindly accept our paper for publication in this Journal.**

#### 38 39 **Reviewer: 2**

40 **Comments to the Author**

41 what about economic potential of *Pithecellobium* (PD) Dulce fibres- please, describe clearly in  
42 the text.

43 **On page 3,** materials and methods, please describe in which region of the world and  
44 geographical latitude is PD growing.

45 ***Pithecellobium Dulce* tree is grows in Mexico, America, India, Bangladesh and Philippines.**

46  
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50 What is currently system of exploring /use this fiber PD?

51 **It is a new study, PD fiber is not currently used in any system.**

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54 In fibre extraction from the tree on fig. 1 /c there is nothing about conditions of retting and where  
55 are retting tank on the picture? Describe it in the text and complete.

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3 Incorporated in the revised version.  
4  
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6 **From page 4** up to page 6 authors presented different test methods; please explain in the text  
7 task of these tests (why did you do it?).  
8

9 Explained in the revised manuscript.  
10

11 **On page 7**, second line from the top, please, explain why there are so big differences in diameter  
12 of these fibres from: 60 -230  $\mu\text{m}$ .  
13

14 Now we have tested 25 similar samples and results is incorporated in the revised manuscript.  
15

16 **Line 4, p. 7** please, add the word: some to be: Table 1 shows the diameter of some other natural  
17 fibers.  
18

19 Incorporated in the revised version.  
20

21 **Page 7, line 5**, please, explain shortly in this text why PDF can be used as reinforcement material  
22 in natural fiber composites for light –weight applications.  
23

24 Because of chemical analysis and XRD results exposed that the PDFs has a high amount of  
25 cellulose content ( $75.15 \pm 0.26$  wt. %) with better crystallinity index ( $49.2 \pm 2.45\%$ ). The FTIR  
26 analysis confirms the cellulose, hemicellulose, lignin and other functional group present in the  
27 PDFs. The thermogravimetric analysis shows that the PDFs are withstand upto  $170^\circ\text{C}$ . The SEM  
28 analysis encounter that PDF has rough surface. The tensile strength of the PDFs is around 600  
29 MPa and Young's modulus is about 7 MPa. From these results concluded that PDF may use as a  
30 better reinforcement for green composites in light weight applications.  
31  
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33 **Page 10 and 11, conclusion:** from this manuscript and from conclusion it is not clear -why the  
34 PDF fibre can be applied in automotive application, please, explain it in the text. If authors can't  
35 prove it- it is not suggested to write about these matters.  
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37 Incorporated in the revised version  
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## A New Study on Characterization of *Pithecellobium Dulce* Fiber as Composite Reinforcement for Light weight Applications

### Abstract:

A bio-fiber, *Pithecellobium Dulce* is abundantly available in all over the world and it has a higher cellulose content ( $75.15 \pm 0.26$  wt.%) and low density ( $865 \pm 26$  kg/m<sup>3</sup>). To acquire fundamental knowledge about *Pithecellobium Dulce* Fibers (PDFs), its physico-Chemical, crystalline, tensile and morphological properties were examined and compared with other plant fibers. The chemical functional groups and crystallinity index ( $49.2 \pm 2.45\%$ ) of the PDFs were obtained via FTIR analysis and XRD respectively. The TGA results of PDFs exhibit thermal stability up to 170°C. The surface morphology of PDF was analyzed by scanning electron microscopy (SEM). The attained results conclude that PDFs are appropriate fibres for acting as reinforcement in manufacturing of green composite product.

**Keywords:** *Pithecellobium Dulce* Fibers, Crystallinity index, Chemical functional groups, Tensile strength, Surface morphology.

### Introduction

In recent days there is an increasing awareness in utilizing natural fiber as an alternate reinforcement for synthetic fiber in polymer matrix composites, especially due to the environmental impact of synthetic fibers (Sanjay et al 2018a; Sanjay et al 2018b). The other disadvantages are high cost, higher power spending during production of fiber, creating skin annoyance to processing labors and higher abrasion of processing equipment. However the natural fibers can overcome this troubles by its renewable origin, low density, high specific strength, high toughness, biodegradability, non-corrosiveness, reduced wear, low cost, easy

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3 availability, good thermal and insulating properties along with less power consumption during  
4 processing (Jawaid et al. 2011; Rajesh Jesudoss Hyness et al. 2017). Due to the above  
5  
6 advantages natural fiber reinforced composites are used in many fields such as automotive,  
7  
8 electrical industrial and construction (Arpitha et al. 2017). The properties of natural fiber  
9  
10 composites depend on the type of resin, fibers, and interfacial bonding between fiber and resin  
11  
12 (Sanjay et al 2018a; Senthamaraikannan et al. 2018). So, it is essential to detect new bio-fibers  
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14 that allow simple and economical extraction processes without deteriorating fiber properties. A  
15  
16 number of bio-fibers, such as banana, jute, coir, oil palm, pineapple, abaca, flax, ramie,  
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18 *Sansevieria cylindrica*, *Sansevieria ehrenbergii*, *Cyperus pangorei*, *Acacia leucophloea* and sisal  
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20 have previously been proven to replace the synthetic fiber in several applications (Madhu et al.  
21  
22 2017; Madhu et al. 2018; Pothan et al. 2006; Biagiotti et al. 2004; Sreenivasan et al. 2011;  
23  
24 Sathishkumar et al. 2016; Mayandi et al. 2016). However, the recognition of novel bio-fibers  
25  
26 and fabrication of composites by these fibers is necessary in the field of materials research,  
27  
28 particularly for eco-friendly inventions. The performance of natural fiber composites is primarily  
29  
30 subjected to the physical, thermal, structural and tensile properties of the fiber. Before using a  
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32 fiber reinforcement in polymer matrix composites, investigations in the characterization of fiber  
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34 are significant (Manimaran et al. 2018a; Madhu et al. 2017). In this study, we investigate the  
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36 physico-chemical, structural, thermal and tensile characterization of *Pithecellobium Dulce* fiber  
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38 by chemical analysis, X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FT-IR),  
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40 Thermo gravimetric analysis (TGA), Scanning electron microscopy (SEM) and single fiber  
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42 tensile test.  
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## MATERIALS AND METHODS

### Materials

*Pithecellobium Dulce* (PD) is an average-sized evergreen tree. PD is a rapidly growing tree that can attain a height of 10 m in 5-6 years in satisfactory environmental conditions. *Pithecellobium Dulce* tree is grows in Mexico, America, India, Bangladesh and Philippines. PD has a small, dark stem (30-100 cm in diameter) that bears low uneven branches. PD is a multiuse plant. Its fruits are having sweetish acidic paste and they can be consumed in raw form or converted into a juice. Oil can be obtained from the seeds of PD and can be utilized for making soaps.

### Fiber Extraction from the tree

The matured barks of PD were collected from M. Kalluppatti village located near Elumalai in Perayur taluk, Madurai district, Tamilnadu, India. The barks of PD were detached from the stem of the plant as presented in Figure 1(b). The inner bark was slit out using a knife. The inner bark were entirely submerged into a tank occupied with water for 15 days at ambient temperature for biological degradation as shown in Figure 1(c) (retting process). After retting is over, PD barks were pullout mechanically by a metallic comb with the teeth. Then the PDFs was thoroughly cleaned with running water and it was dried in the room temperature to dehydrate for a week, before preparing a specimen for analysis (Senthamaraikannan et al. 2016) (Figure 1(d)).

### Physical analysis of PDFs

The cross-section and density of the PDF is presented in this section. The cross-section of the fiber was analyzed by SEM image of a primary and secondary wall of the fiber. The average

cross-section diameters of fiber were measured by an image processing software (Image J, NIST) using the SEM images (Rajkishore et al. 2013). Pycnometer was used to estimate the density of the PDFs (Mettler Toledo xsz05 balances). The density of PDFs ( $\rho$ ) was calculated by using following equation (Murali Mohan Rao et al. 2007; Sathishkumar et al. 2013) ;

$$\rho_{PDFs} = \left( \frac{m_2 - m_1}{(m_3 - m_1) - (m_4 - m_2)} \right) \rho_T \quad (1)$$

where  $m_1$  is the mass of the empty pycnometer (kg),  $m_2$  is the mass of the pycnometer filled with chopped fibers (kg),  $m_3$  is the mass of the pycnometer filled with toluene (kg), and  $m_4$  is the mass of the pycnometer filled with chopped fibers and toluene solution (kg).

### Chemical analysis of PDFs

The aim of chemical analysis is to calculate the percentage of cellulose, hemicelluloses, lignin, wax, ash and moisture content in the PDF by the following appropriate methods. The cellulose content of PDFs has been calculated by Kurshner and Hoffer's method [3]. The hemicellulose was decided as per NFT 12-008 standard (Sathishkumar et al. 2013). The lignin content of the PDFs was calculated according to the Klason method (Sreenivasan et al. 2011) . The PDFs wax content was fixed by Conrad method (Segal et al. 1959). Ash content was appraised through the ASTM E1755-01 standard procedures. The moisture content of PDFs was analyzed with an electronic moisture analyzer (Sartorius, model MA45) (Senthamaraikannan et al. 2016).

### Fourier Transform-Infrared (FTIR) Analysis

FTIR is ~~the~~ a economical, rapid and non-destructive, easy and suitable tool to finalize the chemical and functional groups of existing in the natural fibers. A SHIMADZU 8400S

spectrometer was used to acquire the spectra of the PDFs. The PDFs were crashed into fine powder and mixed with KBr and the mixture was compacted into plates for FT-IR analysis (Jayaramudu et al. 2010) . The FT-IR spectrum of the sample was attained in the wavelength range of  $4000-500^{-1}$  with spectrare solution of  $4\text{cm}^{-1}$  at room temperature.

### X-Ray Diffraction (XRD) Analysis

XRD is the precise simple technique to determine the crystallinity index (CI) and crystallite size (CS) of natural fibers. The powder X-ray patterns was measured by using 'XPERT-PRO diffractometer equipment with a  $\text{CuK}\alpha$  radiation source ( $\alpha = 1.54060\text{\AA}$ ) that works in the generator Settings voltage of 40 kV and filament current of 30 mA. The specimen is scanned from  $2\theta=10^{\circ}$  to  $80^{\circ}$ , with the step size of 0.05 in order to attain a standard diffraction pattern. The peak height method used by Manimaran et al for Red Banana Peduncle fiber was applied to determine the crystallinity index (CI) of the PDFs (Manimaran et al. 2018b; Segal et al. 1959).

$$I_c = \left(1 - \frac{I_{am}}{I_{002}}\right) \times 100\% \quad (2)$$

Where  $I_{002}$  is the intensity of crystalline peak, and  $I_{am}$  is the intensity of amorphous peak in the XRD spectrum. The crystallite size (CS) was calculated by using Scherrer's formula denoted in equation 3 (Kumar et al. 2017).

$$CS = \frac{K\lambda}{\beta \cos \theta} \quad (3)$$

Where  $K=0.89$ -Scherrer's constant,  $\beta$ -peak's full-width at half-maximum, and  $\lambda$  - wavelength of the radiation.

### Thermogravimetric Analysis

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2  
3 It is necessary to examine the thermal stability of the natural fibers to confirm the suitability of  
4 fibers for in high temperature applications. The thermal stability of PDFs was established by a  
5 thermogravimetric analyzer (NETZSCH STA 449F3). The measurement was executed in an inert  
6 gas environmental (nitrogen) with a gas flow rate of 20 mL/min and heated from 30 to 1000 °C  
7 at a rate of heating of 10 °C/min. The analyzer contains heater in which the specimen is placed  
8 on Alumina crucible with lid aided by a precision balance.  
9

### 19 Tensile Testing of PDFs

20  
21 ASTM D3822-07 is the standard way to check the tensile properties (tensile strength, Young's  
22 modulus and % elongation) of single fiber. The single fiber tensile tests for PDFs were  
23 performed by INSTRON 5500R Universal Testing Machine.. Tests were done for different  
24 gauge lengths of 10 mm, 20 mm, 30 mm, 40 mm and 50 mm with 25 specimens for each length  
25 group and mean values were taken into the account. A fixed cross head speed of 0.1mm/min was  
26 maintained for the entire testing and it were executed at room temperature of around 25°C.  
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### 38 Surface Morphological Analysis

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40 The SEM analysis of a bio-fiber helps to verify the surface roughness of the fiber. Scanning  
41 electron microscope (VEGA3 TESCAN) was utilized to study the surface morphology of  
42 PDFs. The surface morphology of PDF was scanned at various magnifications and results were  
43 displayed. The investigations were succeeded with an electron beam accelerating voltage of 20  
44 kV  
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## 51 RESULTS AND DISCUSSION

### 54 Physical Analysis of PDFs

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3 The cross-sectional SEM image and Weibull distribution of diameter of PDFs. as are shown in  
4 Figure 2 (a) and (b). From the existing literature, the cross-sectional area of the fiber was  
5 assumed as circular cross-section (Senthamaraikannan et al. 2018; Gurukarthik babu et al. 2018).  
6  
7 The fiber cross-sections varied with varying fiber length, were determined by SEM at various  
8 locations. Like other natural fibers, the variability in diameter of fiber-cells and lumen has a  
9 great influence on the physical and mechanical properties of PDF (De Rosa et al. 2010) . The 25  
10 samples were used to determine the diameter of the fiber at four points with equal spacing  
11 through a 100 mm of 1000 mm long fibers (Sathishkumar et al.2013). The fiber diameter  
12 is  $144.3 \pm 46.63 \mu\text{m}$  approximately. The diameter is in the range but there are some diameters  
13 which are outside the range. Table 1 shows the diameter of some other natural fibers. The  
14 density of fibers vary due to climatic conditions, plant growth rate, and plant tissue. The density  
15 of PDF is  $0.865 \pm 0.026 \text{ g/cm}^3$  as measured. PDF can be used as reinforcement material in natural  
16 fiber composites for light-weight applications due to its low density.  
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### 35 Chemical Analysis of PDFs

36  
37 Table 2. presents the chemical compositions of PDF in comparison with other natural fibers. The  
38 assessed PDFs cellulose ( $75.15 \pm 0.26 \text{ wt.}\%$ ) is higher than other natural fibers such as *Acacia*  
39 *leucophloea* (68.09 wt.%), *Acacia planifrons* (73.01 wt.%), *Grewia tilifolia* (61.8 wt.%),  
40 *Cordia dichotoma* (59.7 wt.%), *Cyperus pangorei* (68.5 wt.%), *Prosopis Juliflora* (61.65 wt.%).  
41  
42 But it is low when compared to *Cissus quadrangular is* (77.17 wt.%) (Mayandi et al.2016;  
43 senthamaraikannan et al.2016; Jayaramudu et al.2010; Saravanakumar et al.2013; Indran et  
44 al.2014). Generally, high cellulose content of fiber has considerably contributed for the  
45 enhancement of tensile strength and young's modulus (Prithiviraj et al.2016; Kathiresan et  
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3 al.2016). Hemicellulose content of PDF is  $10.23 \pm 3.45\%$ , which is very low when compared to  
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5 *Acacia leucophloea*, *Cissus quadrangularis*, *Grewia tilifolia*, *Cordia dichotoma*, *Prosopis*  
6  
7 *Juliflora*. The hemicellulose content will reduce the strength of the fiber as it leads to degradation  
8  
9 and disintegration of micro fibrils (Indran et al.2014) . Lignin content of PDF is  $12.14 \pm 1.56\%$ .  
10  
11 This will also influences the fiber structure, properties, and morphology. The wax content of  
12  
13 PDF is very less ( $0.22 \pm 0.08\%$ ), that enhances the proper bonding between the fiber and the  
14  
15 matrix during composition fabrication (Maheshwaran et al.2017).  
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### 21 **Fourier Transform-Infrared (FTIR) Analysis**

22  
23 Chemical functional groups PDFs were investigated by a non-destructive technique namely  
24  
25 FTIR. An FTIR spectrum was detected in the  $4000-500\text{cm}^{-1}$  and the wave number range is as  
26  
27 shown in Figure 3. The strong extensive band is detected around  $3395\text{cm}^{-1}$  because of the  
28  
29 hydrogen bonded O-H stretching of  $\alpha$ -cellulose (Jayaramudu et al.2010) . A moderate intensity  
30  
31 band at  $2914\text{cm}^{-1}$  is accredited to C-H stretching frequency of  $\alpha$ -cellulose. The band at  $1735\text{cm}^{-1}$   
32  
33 recognized C=O stretching of hemicelluloses (Fiore et al.2011). The band at  $1653\text{cm}^{-1}$  is an  
34  
35 outcome of OH stretching of absorbed water content in the non-crystalline region of cellulose.  
36  
37 The peak at  $1500\text{cm}^{-1}$  is initiated due to C=C stretching of lignin. The peak at  $1019\text{cm}^{-1}$  is  
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39 allotted to symmetric C-OH stretching of lignin (Thamae et al.2007).  
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### 47 **X-Ray Diffraction (XRD) Analysis**

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49 Properties of the natural fibers depend on orientation of the crystalline and amorphous  
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51 constituencies relating to the fiber axis, crystalline size and presence of crystalline regions.  
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53 Crystallinity Index (CI) measures the orientation of the cellulose crystals in a fiber to fiber axis.  
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3 Commonly the natural fibers are semi crystalline **in common**. The X-ray diffraction pattern of the  
4 PDFs is displayed in Figure 4. It shows two perfect diffraction peaks at around  $2\theta=15^\circ$  and  $22^\circ$   
5  
6 which specifies the existence of both crystalline and amorphous regions in the PDFs  
7  
8 (Jayaramudu et al.2010). **The calculated CI for PDFs was  $49.2\pm 2.45\%$ , which was greater than**  
9  
10 **that of *Cyperuspangorei* (41%) and *Prosopisjuliflora* (46%), lesser than that of *Acacia Arabica***  
11 **(51.72%) and *Acacia planifrons* (65.38%) (Manimaran et al.2016; Manimaran et al.2017).**  
12  
13 Higher crystallinity index tend to be brittle at the same time higher crystallinity index gives  
14  
15 higher strength to fibers. The crystallite size (CS) of PDFs was assessed by Scherer's formula as  
16  
17 detailed in Equation (2) and the CS value was establish to be 14 nm which is significantly larger  
18  
19 than that of *Azadirachta indica* bark fiber (2.75nm) and nearer to the *Prosopis juliflora* bark fiber  
20  
21 (15nm). A large crystal size of the fiber means condensed surface area as a result lower water  
22  
23 and chemical absorption of fibers (Manimaran et al.2016; Manimaran et al.2017)..  
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### 33 **Thermogravimetric Analysis**

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35 Thermal stability of the PDFs was explored by the thermogravimetric analysis showed in Figure  
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37 5. The lesser weight loss around  $170^\circ\text{C}$  spotted which is relates to moisture elimination by  
38  
39 evaporation. The major degradation occurred between  $170^\circ\text{C}$  and  $320^\circ\text{C}$  which is relates to the  
40  
41 degradation of cellulose and lignin in PDFs (Senthamaraikannan et al.2016). The final stage of  
42  
43 thermal decomposition initiated from around  $500^\circ\text{C}$  to  $600^\circ\text{C}$  indicated degradation of the  
44  
45 charred residue. The thermal analysis result prove that PDFs can be consumed as reinforcement  
46  
47 of polymers matrix composites such as the thermoplastics resin working temperature below  
48  
49  $170^\circ\text{C}$  (Belouadaha et al.2015). The thermal stability of PDFs is similar to that of other bio-fibers  
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3 such as *Dichrostachys cinerea*, *Azadirachta indica*, *Prosopis juliflora* and *Acacia Arabica*  
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5 (Manimaran et al.2017; Baskaran et al.2017).  
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### 10 **Surface Morphological Analysis**

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12 Scanning electron microscopy (SEM) provides an excellent method for examination of surface  
13 morphology of fibers. Figure 6(a) and (b) shows the SEM micrograph of PDFs (SEE IMAGE  
14 file), it is clearly contain impurities, wax, fatty substances and globular protrusions. The removal  
15 of surface impurities on fibers by chemical treatment is advantageous for fiber–matrix adhesion  
16 as it **facilitates** both mechanical interlocking and the bonding reaction (Ghali et al.2009) . On the  
17 other hand, figure 6(c) and (d) shows the **rough** surface of the fiber indicating that the chances of  
18 **to** increase the bonding in a polymeric matrix–fiber interface. Figure 6(e) and (f) shows a surface  
19 **with** unwanted impurities. **From the above, it is clear that the presence of some impurities is also**  
20 **evident on the surface of the fibers.** To eliminate these impurities enhancing interfacial adhesion  
21 with polymer matrices, natural fibers are often treated chemically **to** the changes mechanical  
22 properties **that are** probably caused by changes in the physical or chemical structure of the fibers  
23 (Indran et al.2014).  
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### 44 **Tensile behavior of the PDFs**

45 The chemical composition of the natural, especially cellulose content, create an influence on the  
46 tensile strength. Table 4 displays the tensile strength and Young’s modulus. Tensile strength and  
47 Young’s modulus amplifies with increase in specimen lengths from 10 mm to 40 mm. The  
48 deviation from the average values is relatively high, anyway which was acceptable limit for bio-  
49 fiber (Sreenivasan et al.2011).  
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## CONCLUSIONS

The physico-chemical, Structural, thermal, tensile and morphological properties of *Pithecellobium Dulce* fibers were studied. The conclusions observed from the experiments are:

- The chemical analysis and XRD results exposed that the PDFs has a high amount of cellulose content ( $75.15 \pm 0.26$  wt. %) with better crystallinity index ( $49.2 \pm 2.45\%$ ).
- The FTIR analysis confirms the cellulose, hemicellulose, lignin and other functional group present in the PDFs.
- The thermogravimetric analysis shows that the PDFs are withstand upto  $170^\circ\text{C}$ .
- The SEM analysis encounter that PDF has rough surface.
- The tensile strength of the PDFs is around 600 MPa and Young's modulus is about 7 MPa.

From the above results it can be concluded that PDF may be used as a better reinforcement for green composites in light weight applications. A next step as a progress of this research is to fabricate the *Pithecellobium Dulce* fibers reinforced polymer matrix composites and to study their properties.

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3 **Table Captions**  
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5 **Table 1.** Diameter of other natural fibers.  
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7 **Table 2.** Comparison of chemical compositions of raw PDFs with various natural fibers.  
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9 **Table 3.** FTIR Peak Positions and Allocations of chemical stretching in the PDFs.  
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11 **Table 4.** Mechanical Properties of PDFs.  
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**Table 1.** Diameter of other natural fibers.

Type of fiber	Diameter ( $\mu\text{m}$ )
<i>Pithecellobium Dulce</i>	<b>144.3<math>\pm</math>46.63</b>
<i>Sisal</i>	205 $\pm$ 4
<i>Banana</i>	120 $\pm$ 6
<i>Bundle of bamboo</i>	88–125
<i>Jute</i>	30–50

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

Fiber name	Cellulose (wt.%)	Hemicelluloses (wt.%)	Lignin (wt.%)	Wax (wt.%)	Moisture Content (%)	Density (kg/m <sup>3</sup> )	Ash (wt.%)
<i>Pithecellobium dulce</i>	75.15 ±0.26	10.23 ±3.45	12.14 ±1.56	0.22 ±0.08	6.24 ±1.26	865±26	2.13 ±0.56
Jute	59–71	12–13	11.8–12.9	-	-	1460	0.7
Sisal	60–67	10–15	8–12	-	-	1400	0.14– 0.87
Banana	60–65	6–8	5–10	-	-	1350	1.2
Flax	79.0	11.0	3.0	1.5	-	1540	-
Hemp	72.0	10.0	3.0	-	-	860	-
<i>Oil palm empty fruit bunch</i>	65	29	17.5	4	-	946	-
<i>Acacia leucophloea</i>	68.09	13.6	17.73	0.55	8.83	1385	0.08
<i>Acacia planifrons</i>	73.1	9.41	12.04	0.57	8.21	660	4.06
<i>Cissus quadrangularis</i>	77.17	11.02	10.45	0.14	7.3	-	-
<i>Grewia tilifolia</i>	61.8	21.2	14.4	-	2.3	-	-
<i>Cordia dichotoma</i>	59.7	23.6	14.7	-	-	-	-
<i>Cyperus pangorei</i>	68.5	-	17.88	0.17	9.19	1102	-
<i>Prosopis Juliflora</i>	61.65	16.14	17.11	0.61	9.48	580	5.2

Table 3. FTIR Peak Positions and Allocations of chemical stretching in the PDFs.

Peak positions (Wavenumber (cm <sup>-1</sup> ))	Allocations
3395	OH-stretching of $\alpha$ -cellulose
2914	CH stretching of $\alpha$ -cellulose
1735	C=O stretching of hemicelluloses
1653	OH (Absorbed water)
1500	C=C stretching of lignin
1019	Symmetric C-OH stretching of lignin

Table 4. Mechanical Properties of PDFs.

Length group	Tensile strength (MPa)	Young's modulus (GPa)	Strain to failure %	Cross sectional area (mm <sup>2</sup> )
50mm	654.28 ± 36	6.81 ± 1.7	10.52 ± 1.4	0.021 ± 0.005
40mm	622.42 ± 37	6.67 ± 1.3	10.35 ± 1.9	0.022 ± 0.003
30mm	618.49 ± 28	6.54 ± 1.8	10.48 ± 2.2	0.024 ± 0.006
20mm	609.78 ± 32	6.36 ± 2.4	10.23 ± 2.1	0.023 ± 0.002
10mm	598.63 ± 18	6.14 ± 1.3	10.64 ± 1.6	0.026 ± 0.004

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3 **Figure Captions:**

4 **Figure 1.** PDF tree and fibers.

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6 **Figure 2.** SEM images of the longitudinal section of PDFs.

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9 **Figure 3.** FT-IR Analysis of PDFs.

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11 **Figure 4.** X-ray diffractogram of PDFs.

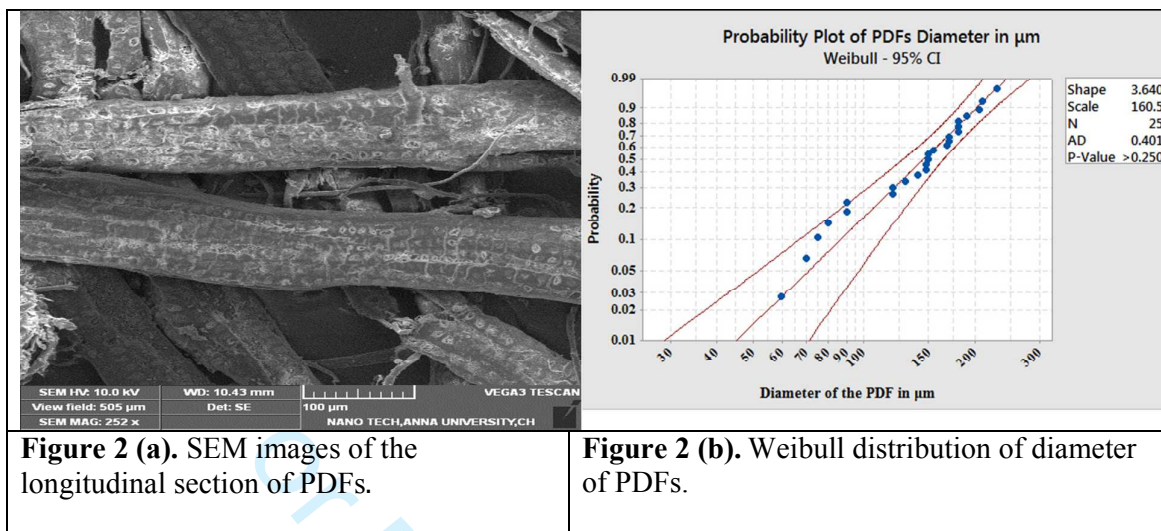
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13 **Figure 5.** TG and DTG curves of PDF.

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15 **Figure 6.** Cross-sectional SEM micrographs of PDFs  
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**Figure 1.** PDF tree and fibers.



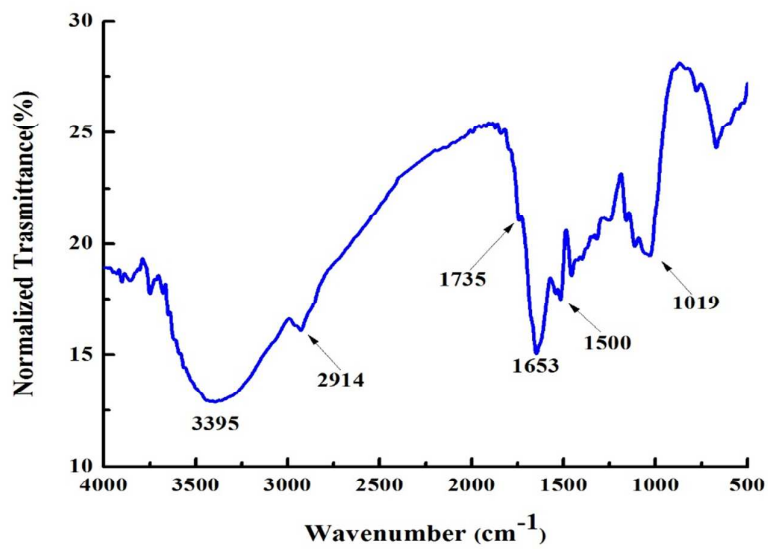
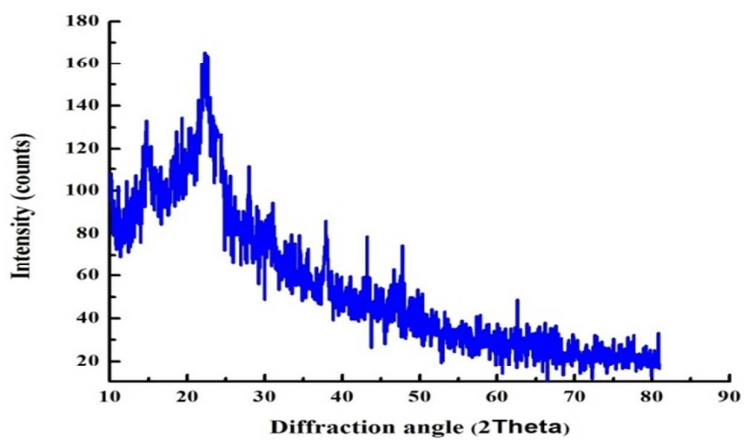


Figure 3. FT-IR Analysis of PDFs.



**Figure 4.** X-ray diffractogram of PDFs.

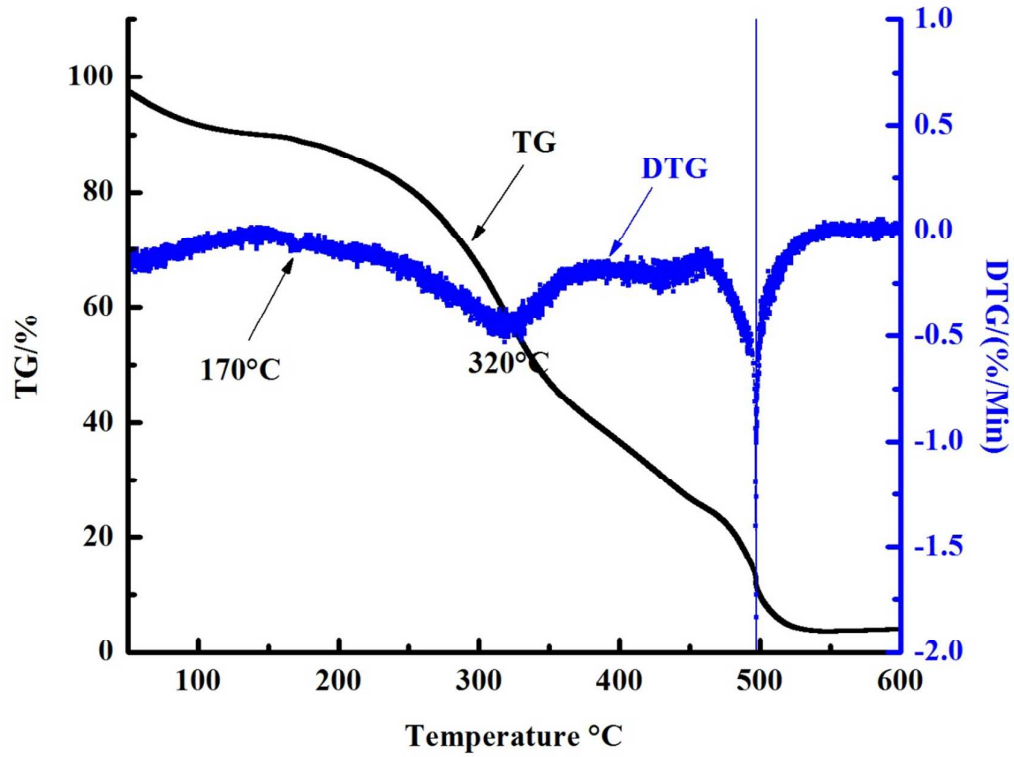


Figure 5. TG and DTG curves of PDF.

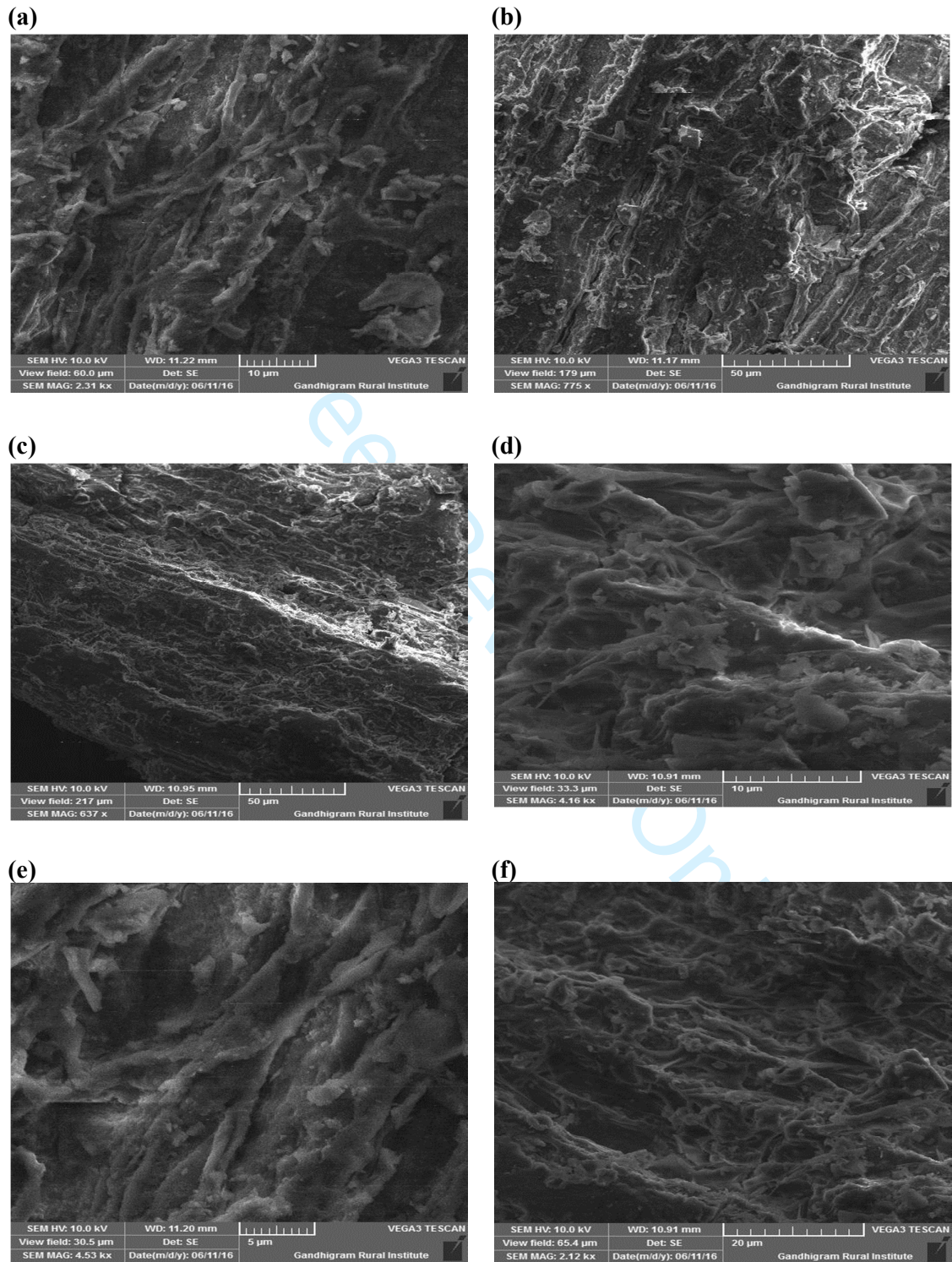


Figure 6. Cross-sectional SEM micrographs of PDFs