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Characterization of a novel natural cellulosic fiber from *Prosopis juliflora* bark

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ABSTRACT

Natural fibers from plants are ideal choice for producing polymer composites. Bark fibers of *Prosopis juliflora* (PJ), an evergreen plant have not been utilized for making polymer composites yet. Hence, a study was undertaken to evaluate their suitability as a novel reinforcement for composite structures. PJ fiber (PJF) was analyzed extensively to understand its chemical and physical properties. The PJF belonged to gelatinous or mucilaginous type. Its lignin content (17.11%) and density (580 kg/m^3) were relatively higher and lower, respectively in comparison to bark fibers of other plants. The free chemical groups on it were studied by FTIR and XRD. It had a tensile strength of 558 ± 13.4 MPa with an average strain rate of 1.77 ± 0.04% and microfibril angle of 10.64° ± 0.45°. Thermal analyses (TG and DTG) showed that it started degrading at a temperature of 217 °C with kinetic activation energy of 76.72 kJ/mol.

1. Introduction

Nowadays much importance is being given to the development of recyclable and environmentally sustainable composite materials than ever before due to increasing environmental awareness worldwide (Cheung, Ho, Lau, Cardona, & Hui, 2009). Plant fiber reinforced polymer composites have attracted increased research interests owing to their potential to serve as alternative for artificial fiber composites that are associated with ecological hazards (Bledzki, Sperber, & Farunk, 2002; Mishra, Mohanty, Drzal, Misra, & Hinrichsen, 2004). Natural fibers derived from bark of various plants such as jute, artichoke, okra, hemp, *Grewia tilifolia*, kenaf, ramie and flax have fascinated the scientists and technologists because of their specific properties and availability. However, bark fiber derived from the PJ plant which may presumably has all or most of the qualities of the bark fibers known till date has not been studied yet despite its presence globally. PJ belongs to the family *Leguminosae*, genus *Prosopis* which included 44 species, grouped in 5 sections and 6 series (Burkart, 1976). *Prosopis* species have been widely introduced in several countries around the world over the past 150 years for fuel wood, fodder and their ability to grow in the poorest soils and survive in areas where no other plants can survive (Pasiecznik et al., 2001). It is highly recognized for windbreaks, soil binders and sand stabilizers, moreover provide food and shelter to animals that feed on its nectar, pollen, leaves and fruits (Golubov, Mandujano, & Eguiarte, 2001), tolerant to very high temperatures (e.g. 48 °C), annual rainfall range of 150–750 mm (Darke, 1993; Geilfus, 1994) and heavy metals (Sinha, Rai, Bhatt, Pandey, & Gupta, 2005). Its stem is green-brown, sinuous and twisted, up to 6–9 m in height and 45 cm in diameter; bark somewhat rough and dull-red (Valdivia, 1972).

Generally, all natural fibers have several attractive features that include low cost, light weight, moderate strength, high specific modulus, renewability, biodegradability, freedom from health hazards and amenability to chemical modification (Manikandan, Velmurugan, Ponnambalam, & Thomas, 2004). These superior properties of fibers are gaining more attention as reinforcement in various technical applications which include the food packaging, automotive components, furniture as well as sports (Anuar & Zuraida, 2011). This study aims to understand the bark anatomy, chemical, mechanical and thermal properties of the PJFs in comparison to other natural bark fibers known. To the best of our knowledge, the bark fibers of PJ as novel reinforcements have been

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studied for the first time. From the experimental data, it is clear that the PJFs can act as a better reinforcement for the polymer matrices and add significant value to the research fraternity.

2. Materials and methods

2.1. Extraction of bark fiber from Prosopis juliflora plant

The PJ plant has a twisted stem and flexible branches with long and strong thorns (Azevedo, 1955). The roots penetrate to great depths in the soil and can grow in wide range of soils, such as saline, alkaline, sandy and rocky soils (George, Venkataraman, & Parrida, 2007). It occurs worldwide in arid and semiarid regions (Burkart, 1976). The stems (approximately 25 mm diameter) of PJ plant were collected from Aruppukottai, Tamil Nadu, South India. Bark of this plant acted as the source of natural fiber. The barks of the PJ plant were immersed in water to allow microbial degradation for a maximum period of two weeks (Fiore, Valenza, & Di Bella, 2011) such that they became soft and both inner and outer layers got detached. The outer layer was removed and disposed off while the inner layer was retained for subsequent separation of fibers by employing a traditional combing process with the aid of fine, long metal teeth.

2.2. Characterization of Prosopis juliflora fiber

2.2.1. Bark anatomy of Prosopis juliflora

The anatomy of sliced PJ bark was studied using a polarized microscope (Nikon, Japan) essentially following the methods described elsewhere (O'Brien, Feder, & McCull, 1964; Sass, 1940).

2.2.2. Tensile and single fiber pull-out tests

The maximum strength was measured in a single fiber tensile test using a universal testing machine according to the ASTM D3379-75 standards. Tensile testing of PJFs ($n = 30$) was performed at a cross head speed of 1 mm/min for 50 mm gauge length. For the single fiber pull out test ($n = 10$), the fibers were embedded in an unsaturated isophthalic resin (1.12 g/cm^3 density) and the test was carried out at a cross head speed of 0.1 mm/min for 20 mm gauge length. A 5 kN capacity load cell was used uniformly for all the tests. The debonding force ($F_{\max}$), the diameter (d), and the embedded length of the fibers (l_e) were determined and the interfacial shear strength (τ_d) (Bozaci et al., 2009) was calculated from Eq. (1).

$$\tau_d = \frac{F_{\max}}{\pi d l_e} \qquad (1)$$

2.2.3. Determination of chemical composition

Cellulose, hemicellulose and lignin contents of PJF were determined using the standard test methods (Doree, 1950; Pearl, 1967). Density of PJF was evaluated employing the meltbertoledoxsz05 balances method (Sathishkumar, Navaneethakrishnan, & Shankar, 2012) while its wax content was quantified as per the standard protocol (Contrad, 1944). The PJFs were analyzed for ash content as per ASTM method E1755-01 (Reddy & Yang, 2008) and moisture content following a method described elsewhere (Liu, Cao, Huang, Cai, & Yao, 2010). The chemical composition of other bark fibers was obtained from the published literatures (Alam & Arifuzzaman Khan, 2007; Arifuzzaman Khan et al., 2009; Jayaramudu, Guduri, & Varada Rajulu, 2010; Mwaikambo & Ansell, 2002; Reddy & Yang, 2009; Sathishkumar et al., 2012; Yan & Pickering, 2008) and used for the purpose of comparison as shown in Table 1.

2.2.4. Fourier transform infrared (FTIR) spectroscopy

FTIR spectrometer (Model FTIR-8400S spectrum, SHIMADZU, Japan) was used to determine the presence of free functional groups on the PJF. Spectral outputs were obtained in the range of 4000–400 cm^{-1} using 32 scans and recorded in the transmittance mode as a function of wave number.

2.2.5. X-ray diffraction (XRD) analysis

The crystallinity of the PJF was determined by using a powder X-ray diffraction (PXRD) method. This analysis was essentially carried out using X'pert PRO-PANalytical system with the settings viz. current 30 mA, tension of 40 kV generator setting with Cu anode material at temperature of 25 °C with step size (2θ) 0.05. The diffracted intensity of CuKα radiation wavelength of 0.1542 nm was recorded between 10° and 80°.

2.2.6. Thermogravimetric analysis (TGA)

Thermal stability behavior of the PJF was assessed by thermogravimetric (TG and DTG) analyses using Jupiter simultaneous thermal analyzer (Model STA 449 F3, NETZSCH, Germany) for measuring the mass and transformation. The PJFs were exposed to nitrogen gas at a flow rate of 20 ml/min. Ten mg of the PJFs was crushed and kept in alumina crucible for good coupling between the sample and the temperature measured by the thermocouple. The step increment of heating rate was from 10 °C/min over a range of temperature i.e., from room temperature to 1000 °C.

2.2.7. Surface morphology of the PJF by SEM

The surface of the PJFs was visualized using a scanning electron microscope (Model SU1510, HITACHI, Japan) with an accelerated voltage of 30 kV and attainable vacuum level of 1.5×10^{-3} Pa. The specimens were coated with a thin gold layer to avoid the accumulation of electrical charges during examination.

3. Results and discussion

3.1. Anatomy of Prosopis juliflora bark and fiber

Examination of the PJ bark and fiber under a polarized microscope revealed the existence of several thin dark tangential layers of collapsed phloem alternating with several successive cylinders of phloem fibers as shown in Fig. 1. PJFs were cleaved into thick blocks by wavy dilated phloem rays. There were about 15 successive cylinders of fibers in the bark. The fiber blocks were smaller and less prominent in the outer zone and they became gradually wider and thicker in the inner zone. The outer and inner blocks of the fibers are

Table 1
Comparison of chemical compositions of the PJF with bark fibers of other plants.

Name of fiber	Cellulose (%)	Hemicelluloses (%)	Lignin (%)	Wax (%)	Moisture content (%)	Density (kg/m^3)	Ash (%)
PJF	61.65	16.14	17.11	0.61	9.48	580	5.2
Jute	72	13	13	–	12.6	1460	–
Flax	81	14	3	–	10	1500	–
Ramie	76	15	1	–	8.0	1500	–
Hemp	74	18	4	2.3	10.8	1480	–
Kenaf	53.14	14.33	8.18	–	–	1400	–
Hop stem	84 ± 1.6	–	6.0 ± 0.2	–	–	–	2.0 ± 0.1
Okra	60–70	13.1–16.7	0.6–0.7	0.3	7.5–17	–	–

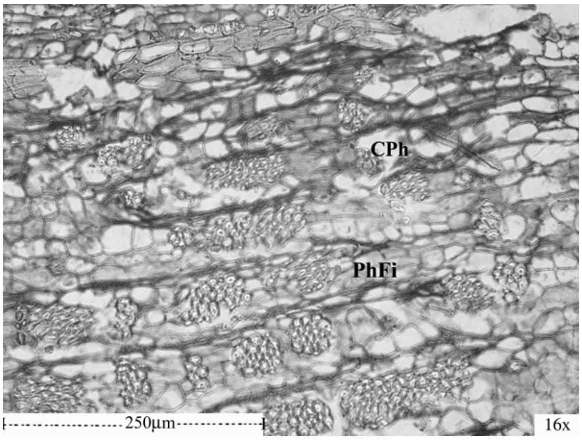

Fig. 1. Transverse section of the PJ bark showing several successive cylinders of fibers (CPh—collapsed phloem, PhFi—phloem fibers).

30 µm and 40 µm thick, respectively. Outer blocks of the PJF were 60–100 µm wider whereas the inner blocks were 200–300 µm wider. PJFs belonged essentially to gelatinous or mucilaginous type that consisted of an outer primary wall which was highly lignified while the secondary wall had mucilaginous substance. When the PJF was stained with toluidine blue, its outer primary wall appeared dark green in color while the inner secondary wall appeared purple in color. However, the primary wall of the PJF appeared red in color because it took up the phloroglucinal stain due to the presence of lignin while the secondary wall remained unstained. When the PJFs were stained and observed under a polarized microscope, the primary walls appeared bright blue in color while the secondary walls remained either colorless or differently colored. The diameter and thickness of the primary and secondary walls of the single PJF were 20 µm, 5–8 µm and 10–12 µm, respectively. Whereas the diameters of single fibers derived from other bark are 17.8–21.6 µm, 17–22.8 µm, 15.9–20.7 µm, 17.7–21.9 µm and 28.1–35 µm for flax, hemp, jute, kenaf and ramie, respectively (Perry, 1975). The S_1, S_2 and S_3 layers of the PJF were not very distinct and the cell lumen was approximately 8 µm in diameter as shown in Fig. 2.

3.2. Determination of the mechanical properties of the PJFs

Mechanical properties of bark fibers depend largely on the cell wall structure (S_1, S_2, and S_3) and the chemical composition. Tensile strength of the PJFs was found to be 558 ± 13.4 MPa with 1.77 ± 0.04% of strain rate, while the tensile strengths of other bark fibers viz. flax, hemp, jute, and ramie are 500–900 MPa, 690 MPa, 370 ± 134 MPa, and 915 MPa, respectively (Bolton, 1994; Perry, 1975; Saha et al., 2010; Seki et al., 2011). The microfibril angle (α) (Charlet, Eve, Jernot, Gomina, & Breard, 2009) of the PJF was 10.64° ± 0.45° as derived from the global deformation Eq. (2).

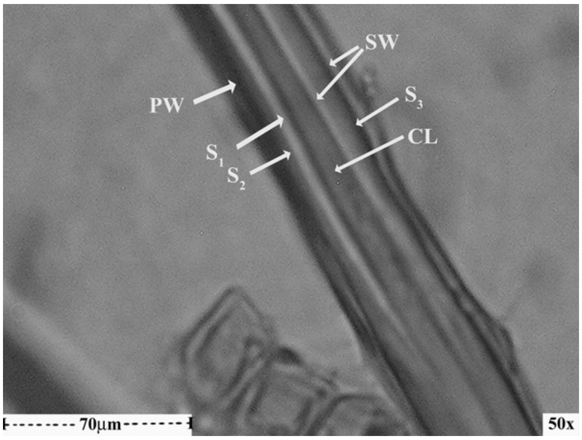

Fig. 2. The PJF as viewed under a polarized microscope (CL—cell lumen, PW—primary wall, SW—secondary wall, S_1, S_2, and S_3—wall layers).

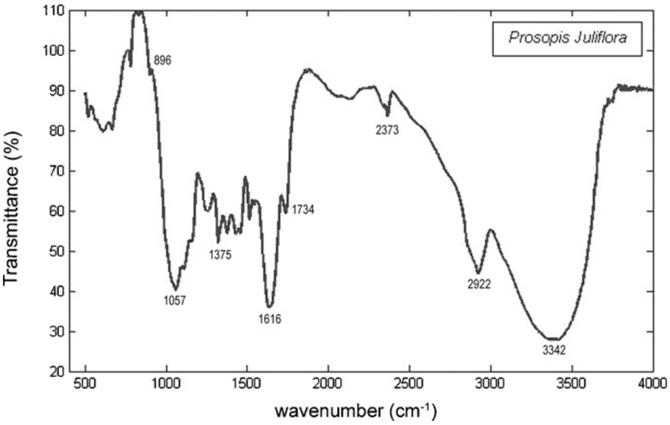

Fig. 3. FTIR spectrum of the PJF in the frequency of 400–4000 cm^{-1}.

$$\varepsilon = \ln\left(1 + \frac{\Delta L}{L_0}\right) = -\ln(\cos\alpha) \tag{2}$$

It is worth mentioning that jute, flax, hemp and banana fibers had a microfibril angle of 8.1°, 5°, 6.2° and 11–12°, respectively (Kulkarni, Satyanarayana, & Rohatgi, 1983; Perry, 1975). Finally, the fiber-matrix interfacial shear strength was determined as 5.3 ± 0.26 MPa. This is the primary factor that indicates the stress transfer from matrix to fiber in a composite structure.

3.3. Chemical analysis of the PJFs

The cellulose content of PJFs was 61.65% which is believed to act as fasten which can withstand hydrostatic pressure gradients of the fibers. PJF had hemicellulose content of 16.41% whose degradation usually leads to disintegration of fibers into cellulose microfibrils resulting in lower strength because of the linking effect (Morvan, Jauneau, Flaman, Millet, & Demarty, 1990). The wax content of the PJF was 0.61% as opposed to 2.3% and 0.3% in the cases of the hemp and okra bark fibers, respectively which may generate poor interfacial bond between fibers and polymer matrices. Higher lignin content (17.11%) of the PJF can potentially contribute to the excellent rigidity when compared to other bark fibers.

3.4. FTIR analysis

FTIR spectra with the classical peaks of PJF are presented in Fig. 3. It is noteworthy that the peaks of whole PJF appeared at 3342 and 896 cm^{-1} while the peak of the whole kenaf fiber (core and bast) projected at 3390 and 1190 cm^{-1} corresponding to O—H stretching and O—H bending frequencies (Khalil, Yusra, Bhat, & Jawaid, 2010). In PJF, the O—H stretching peak was seen at 3342 cm^{-1} and C—H stretching peak existed at 2922 cm^{-1}. For PJF a peak that appeared in 3342 cm^{-1} was due to cellulose; O—H stretching of α-cellulose was represented by the band of 3432 cm^{-1} for *Grewia tilifolia* (Jayaramudu et al., 2010). The 1057 cm^{-1} peak indicate the C—O—C (ester) group in the PJF. Carbonyl stretching C—O for acetyl groups in hemicellulose and aldehydic group of lignin are represented by the peaks at 1734 cm^{-1} to 1616 cm^{-1}. The carbonyl region was predicted at 1710 cm^{-1} in the case of hemp fiber (Pracella, Chionna, Angusillesi, Kulinski, & Piorkowska, 2006). Two bands at 1643 and 1742 cm^{-1}, respectively were assigned to the free and esterified carboxyl groups in mulberry bark (Liu et al.,

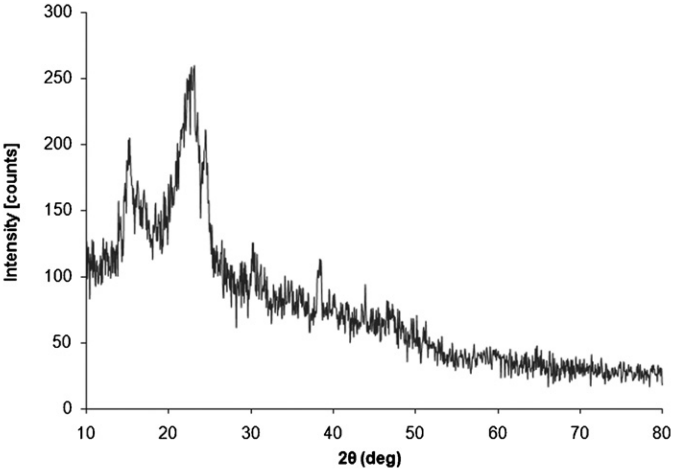

Fig. 4. X-ray spectrum of the PJF.

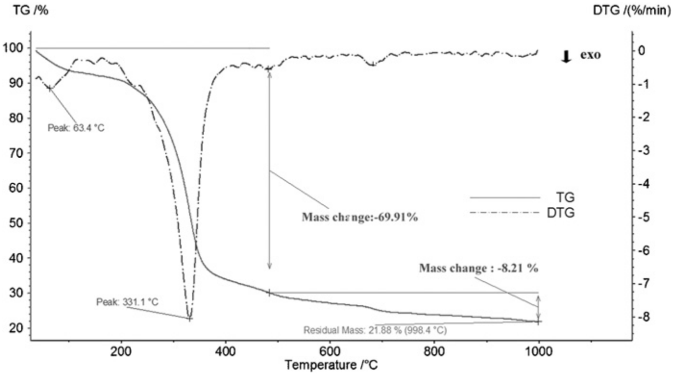

Fig. 5. TG and DTG curves of the PJF.

2010) whereas a small peak at 1517 cm^{-1} indicated C═C stretching of aromatic ring of the lignin in okra fiber (De Rosa et al., 2011).

3.5. XRD analysis

The spectrum corresponding to the PJF shows a well-defined diffraction peaks at the following angles viz. $2\theta = 18.12^\circ$ and 22.67° as shown in Fig. 4. The observation of a major crystalline peak of PJF originate at 2θ diffraction angles of 18.12° (1 1 0) and 22.67° (2 0 0), which corresponds to the crystallographic plane family of cellulose-I. Crystallinity Index (CI) which is a measure of the amount of crystalline cellulose with respect to the global amount of amorphous materials was determined using Segal empirical method (Segal, Creely, Martin, Jr., & Conrad, 1959).

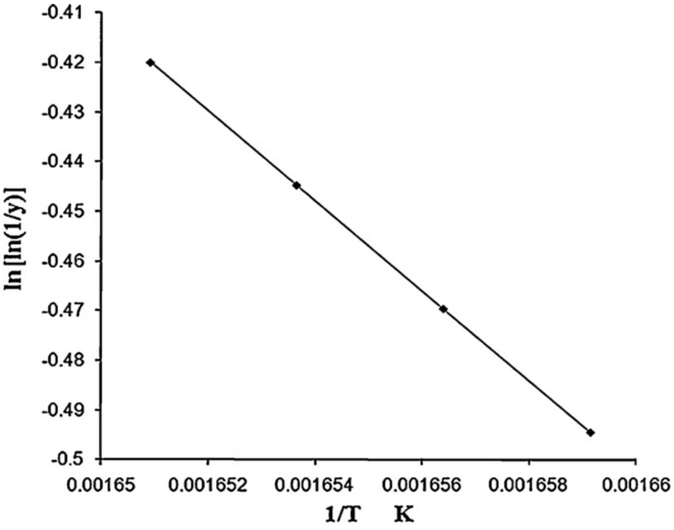

Fig. 6. Broido's plot of the PJF.

The CI value for the PJF was found to be 46%, which is much lower than that determined for jute (71%) and hemp (88%) (Mwaikambo & Ansell, 2002). Using Scherer's equation (3), the crystallite size (L) of PJF was found to be 15 nm, which is much greater than that determined for the flax fiber (2.8 nm) and quite closer to the ramie fiber (16 nm) (Reddy & Yang, 2005).

$$L = \frac{K\lambda}{\beta \cos\theta} \tag{3}$$

where K is Scherer's constant (0.89) β is the peak's full-width at half-maximum and λ is wavelength of the radiation.

3.6. Thermal analysis of the PJF

Thermal analysis of the natural fibers gives a good description of their thermal stability. Initial degradation of PJF started at 110 °C which resulted in a weight loss which is presumably due to the vaporization of water in the fiber. The PJF underwent 10% degradation at a temperature of 149 °C when the initial temperature was set at 33 °C. It was thermally stable at around 217 °C in that a significant peak observed at 219 °C fell in the range of 200 °C to 300 °C TG and DTG curve as shown in Fig. 5 that corresponded to the thermal depolymerisation of hemicellulose. A prominent peak at 331.1 °C indicated the possible decomposition of α-cellulose. However, the similar peak was observed at 321 °C, 308.2 °C, 298.2 °C and 309.2 °C for bamboo, hemp, jute and kenaf fibers, respectively (Yao, Wu, Lei, Guo, & Xu, 2008). Structurally lignin has a complex composition of aromatic rings with several branches and hence its decomposition generally occurs at a very low weight loss rate within the whole temperature range from ambient to temperatures higher than 700 °C (De Rosa, Kenny, Puglia, Santuil, & Sarasini, 2010; Yang, Yan, Chen, Lee, & Zheng, 2007). Remaining two peak values viz. 480.6 °C and 676.6 °C may be a consequence of oxidative degradation of the charred residue. The apparent activation energy E_a was determined to understand the detailed kinetic parameter of the fiber (Das & Chakrabarty, 2008) from the Broido's equation (4).

$$\ln\left[\ln\left(\frac{1}{y}\right)\right] = -\left(\frac{E}{R}\right)\left[\left(\frac{1}{T}\right) + K\right] \tag{4}$$

where R is the gas constant (8.32 J/mol K), T is the temperature in Kelvin, y is the normalized weight (w_t/w_o), w_t denotes the weight of

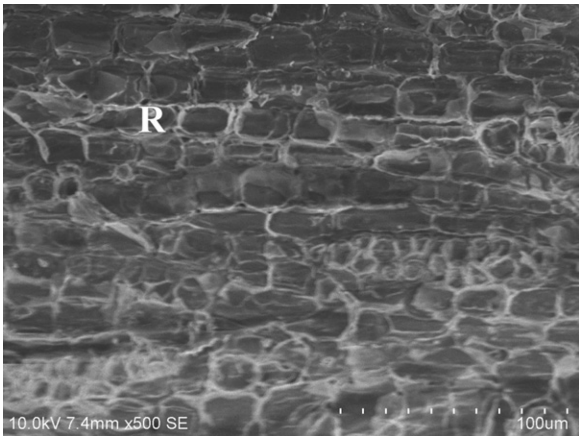

Fig. 7. Ultramicroscopic structure of the PJF as viewed under a scanning electron microscope (R—rays).

the sample at any time t, while w_o indicates the initial weight. The kinetic activation energy (E_a) 76.72 kJ/mol was interpolated from the plot of $\ln[\ln(1/y)]$ vs. $(1/T)$ as illustrated in Fig. 6. This general range might offer a comparatively narrower range of activation energy for understanding fiber decomposition for polymer composite with respect to previously reported values i.e., 60–170 kJ/mol for wood decomposition (Di Blasi, 2002).

3.7. Morphological analyses

Morphological analyses of the PJFs were essentially conducted using the phloem tissue. The phloem parenchyma and rays are clearly shown in Fig. 7 and the PJF cells assumed irregular circular configuration with the intact cell walls running in both directions.

4. Conclusions

This study emphasizes the importance of natural bark fiber that can be used as reinforcement in the preparation of polymer composite and adds value to the existing knowledge on natural bark fibers. The PJF consisted of phloem fibers that belonged to gelatinous or mucilaginous type as evidenced from the testing for bark anatomy. It also consisted of a highly lignified outer primary wall and a secondary wall that consisted of mucilaginous substance and a cell lumen. Higher lignin content of the PJF can offer relatively higher rigidity in comparison to the existing bark fibers and relatively lower density of the PJF may obviously enable light weight applications for e.g. textile and automobile industries. The PJFs possess desirable tensile properties that make them an ideal alternative reinforcement material to the conventional fibers such as glass and carbon in polymer matrices. Relatively higher strength and lower elongation will essentially be conferred to the PJFs by virtue of CI and crystallite size, respectively. Thermogravimetric analysis indicated that the PJF was thermally stable up to 217 °C and its kinetic activation energy was 76.72 kJ/mol which has been estimated within the temperature range of 200–350 °C. Further, the study will be extended to identify the suitability of the PJFs as reinforcements for preparation of biocomposite laminates and evaluate their mechanical and thermal properties.

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