Extraction and characterization of natural cellulosic *Erythrina variegata* fiber for biocomposites

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Abstract

This paper presents study on extraction and characterization of the cellulose fiber from the bark of *Erythrina variegate* (EV) plant. Several tests were carried out on *Erythrina variegata* fibers (EVFs) to determine their properties. These included thermogravimetric analysis (TGA)/difference thermogravimetric (DTG), X-ray diffraction (XRD) analysis, Fourier transform infrared spectroscopy (FTIR) as well as morphological analysis, using scanning electron microscopy (SEM) and atomic force microscopy (AFM). From the results obtained, chemical composition of EVFs, such as cellulose, lignin, ash and wax content of 70.60, 12.70, 8.60 and 0.24 wt.%, respectively were recorded through standard chemical analysis. The maximum and average tensile strength of the EVFs was found as 6.06 ± 0.02MPa and 2.80 MPa,

respectively. The thermogravimetric analysis on the fiber showed excellent stability with a char residue of 19.23% and a maximum degradation temperature of 349 ^{o}C. The crystallinity index (CI) of 37.5% and crystalline size of 36.93 nm of EVFs were calculated through X-ray diffraction analysis. The morphological study established that EVFs possessed rough surface even in raw form. The density of EVF was obtained at 1412 kg/m^3, which was higher than that of Grewia damine of 1378 kg/m^3 and lower than that of jute of 1460 kg/m^3.

Keywords: *Erythrina variegata*fibers (EVFs), extraction, characterization, chemical composition, biocomposites, applications.

1. Introduction

Green composites are changing the assessment of properties in the field of material innovation, as they show various attractive properties, such as lightweight, higher strength-to-weight proportion, simple bio-degradability, less wear, low energy use, simplicity of manufacture of composite structures, more prominent mechanical properties and low cost in comparison with regular synthetic fiber reinforced polymeric (FRP) composites. At present, to avoid environmental damage caused due to the usage of synthetic FRP composites, many studies are focused on new ecofriendly and recyclable materials. Furthermore, synthetic fibers have some challenges, such as high density, high abrasiveness, high cost when compared to natural fibers, high energy consumption, low renewability and very low recyclability, among others. To prevail over the aforementioned drawbacks, natural fibers act as an alternate reinforcement solution. They are comparatively better than synthetic fibers. Natural FRP composites have many superior properties. These include, but are not limited to, better hydrophobicity, higher surface area, surface roughness, thermal and mechanical properties (Lila et al. 2020), although with a few fiber modifications or treatments. It has been reported that the use of alkali treatment (NaOH) reduced the hemicellulose, lignin and moisture contents present in natural fibers. Hence, it improved its surface property and enhanced the mechanical interlocking between fibers and polymer matrices during composite production(Narayanasamy et al. 2020; Ilangovan et al. 2020; Njoku et al. 2020). The tensile property of a natural fiber is influenced by the amount of cellulose content (Khan et al. 2020)and density present(Khan et al. 2019; Ravindran et al. 2020).

In automotive industry, the usage of hybrid green composites provided higher acoustic absorption frequencies (Hariprasad et al. 2020). Moshi et al. (Cited by Ravindran et al. 2020) performed a morphological study on *Grewia damine* flowering plants stem, using atomic force microscope (AFM) and analyzed the average surface roughness value of the fiber. Also, study on the shape factor for diameter, elongation strength, modulus of elasticity and percentage of strain using Weibull distribution was reported. Similarly, *Sansevieria roxburghiana*natural fiber showed presence of higher cellulose content of 78.63 wt%, as a promising reinforcement for green composites (Gopi Krishna, Kailasanathan, and NagarajaGanesh 2020). Natural cellulose fibers from *Kigelia africana* produced good mechanical properties after alkali treatment and bleaching. In addition, cellulose content in the fibers increased after bleaching for suitable manufacturing composites and non–woven applications (Ravindran et al. 2020). Nendran banana peduncle plant fibers have been analyzed to show its usefulness in lightweight automotive components and construction equipment (Manimaran et al. 2020), as their reinforcing elements. Fibers extracted from the bracts of banana exhibited longer length and reasonably good resistance against thermal degradation, as evidenced by thermogravimetric analysis (Amutha, Sudha, and Saravanan 2020). Fibers extracted from *Vachellia farnesiana* exhibited the presence of lignin, which influenced the fiber structure and morphological characteristics (Vijay et al. 2020a).

Based on the improved properties of the aforementioned natural FRP composites, using some plant fibers as their reinforcements. To further benefit from *Erythrina variegate* fibers (EVFs) as a potential natural fiber for reinforcement of biocomposites, this study therefore focuses on extraction and characterization of EVFs, using several tests and characterization methods.

2. Experimental procedures: Materials and methods

2.1. Selection and extraction of fibers

Regular fiberswere extracted from the stem bark of *Erythrina variegate* (EV) tree of Malvaceae family. This plant is known as *Mullumurungai* in Tamil. It was found in south Tamil Nadu, India. The average height of the plant was 3.90m. Different species of the plant are utilized to prepare medicines for skin infections and cough. The stem bark of the plant was collected from

Pottalpalayam village, Sivagangai District, Tamil Nadu, India. The gathered stem barks were cleaned in water to eliminate undesirable particles from outside of the stem bark. Then, the cleaned stem barks were inundated in fresh water over a period of 27 days for microbial degradation. Afterwards, fibers were extricated from the stem bark physically, using a metal brush. Then, the fibers were washed in refined water in order to eliminate the residue particles (Saravana Kumaar et al. 2019).Figs 1(a), (b) and (c) depict a typical EV tree, harvested stem bark and extricated fibers, respectively.

INSERT FIGURE 1

2.2. Physical property estimation

Optical minuscule pictures of 20 examples selected from various pieces of the fiber were taken to obtain their diameters. The fiber diameter measurement was needed, as it influenced the rigidity and density of the fiber. Density of the fiber was determined with the assistance of a pycnometer (Raja et al. 2021; Amutha, Sudha, and Saravanan 2020). The fiber test for the density estimation was arranged just by drying with intention of eliminating the dampness present in the fiber.

2.3. Characterization

2.3.1. Chemical investigation

Chemical investigation was carried out on the extracted fibers to establish the presence of their different constituents, such as cellulose, lignin, ash, wax and moisture, usually present on their outer surface level. Cellulose was determined by treating the samples with mineral acid at high temperature. The lignin content of each fraction was determined by Klason method. The wax content of each fraction was determined by hydrocarbon-based solvent extraction process. Using electronic moisture analyzer (Sartorious, model MA45) the moisture content was measured and the ash content percentage of the EVFs was determined by a method suggested by ASTM E1755-01 standard. Experiments were repeated for five times and the average value was reported.

2.3.2. Fourier transform infrared investigation

Fourier transform infrared (FTIR) investigation of the fiber test was conducted to examine the compound groups present on the fiber surface and understand relationship that occurred between the chemical compositions. This examination was performed with Perkin Elmer Red Infra Spectrometer test arrangement. The samples for this examination were set up in the powdery structure and framed as pellets by blending them with potassium bromide, with the frequency range of 4000 and 5000 cm^{-1}. Also, energy-dispersive X-ray (EDX) analysis of EVFs was similarly carried out and its results were subsequently reported.

2.3.3. X-beam diffraction examination

The X-beam diffraction technique was used to determine the crystallinity of the fiber, using a normal size of the crystallites. The glass-like behavior of the fiber was investigated, as it assumed a significant part in deciding the mechanical properties of the fiber. The estimation of the crystallinity index (CI) was determined from the in-strained quality of the greater and lower tops obtained at the most extreme and least conditions, respectively.

Therefore, CI and the grain or crystal size (CS) were calculated, using Eqs. (1) and (2), respectively.

$$CI = \frac{H_{22.53} - H_{15.38}}{H_{22.53}} \quad (1)$$

$$CS = \frac{K\lambda z}{\beta Cos\theta} \quad (2)$$

Where $H_{15.38}$ and $H_{22.58}$ in Eq. (1) represent the maximum intensity of the peaks (1, 1, 0) and (2, 0, 0), which contributed to the amorphous and crystalline fractions, respectively. From Eq. (2), K is the shape factor (= 0.89 as a Scherrer's constant), λ is the wavelength of the X-ray used for the diffraction, θ is the angle of diffraction and β is the peak's full width at half-maximum. The crystallographic plane (2, 0, 0) implied the presence of rich cellulosic substance. The (1, 1, 0) plane showed the shape that was less idea for the fiber. This was attributed to the presence of lignin constituents.

2.3.4. Thermogravimetric examination

Thermogravimetric analysis (TGA) was performed to investigate into the thermal ability of EVFs. In this technique, the dependability of the different compound constituents, and thus, the soundness of the fiber was estimated as an element of temperature increased. In this measurement, TGA was done with Mettler Toledo Analyzer, as the fiber was gradually warmed from 30 to 550 ^{o}C under pure nitrogen gas. The most extreme degradation temperature cut off of the fiber was obtained from TGA and difference thermogravimetric (DTG) plots, and later discussed.

2.3.5. Single fiber/EVF test

ASTM standard D3822-07 was followed during testing for the pliable conduct of the stem fiber or EVF. A general testing machine (Zwick/Roell**)** was used to gauge the percentage strain. Experimentation was performed with measured length and crosshead speed of 40 mm and 5 mm/min, respectively. 25 EVF samples were utilized for the estimation of ductile qualities and the mean qualities were taken.

2.3. Morphological investigation

Morphological investigation was performed on the outside parts of the fibers to examine the surface profile on them and whether the surfaces of the fibers are either harsh or smooth.

2.4.1. Scanning electron microscopic investigation

Scanning electron microscopy (SEM) was used to study EVFs and different components present on their surfaces. VEGA3 (TESCAN OXFORD) SEM was used with speeding up voltage of 20 kV for the morphological investigation. Gently gold-covered fiber samples were utilized to obtain SEM images of high-resolution.

2.4.2. Surface harshness estimation

AFM examination was conducted to obtain both quantitative and surface hardness properties of the fibers. Therefore, Nanosurf EPscan2 (Swiss make) instrument was used on EVFs. The results obtained were subsequently and concisely elucidated.

3. Results and discussion

3.1. Physical and chemical properties

The physical and chemical property values of EVFs were obtained and later compared with other natural fibers. A sample size of 20 was tested in order to get the average value of diameter, as obtained to be 2.48 ± 10 µm. Natural fibers are typically made up of cellulose, lignin and wax content. These three compositions are responsible for the rigidity of the plant cell. The chemical composition of EVFs and other natural fibers are presented in Table 1. The chemical compositions of natural fibers are always different, due to several factors, such as age, extraction method, climate conditions and type of soil, among others. EVFs possessed cellulose content, lignin, wax, moisture and ash of 70.60, 12.70, 0.24, 7.80 and 8.60 wt% as well as density of 1412 kg/m^3. Cellulose content of EVFs was higher than that of Grewia damine, *Cissusvitiginea*, *Momordica charantia*, *Vachellia farnesiana*, *Acacia nilotica* plant and it was close to that of Nendran banana peduncle plant and jute fibers.

INSERT TABLE 1

Moving forward, relevant studies have reported that the presence of cellulose content improved the reinforcement of natural FRP composites, resistance to oxidizing agents and tensile properties (Khan et al. 2020). Higher level of hemicelluloses was responsible for the degeneration of cellulose micro fibrils present in the fibers (Indran, Raj, and Sreenivasan 2014). Cellulose of EVFs was higher than that of *Cissusvitiginea*, *Momordica charantia*, *Vachellia farnesiana*, *Acacia nilotica* plant, Grewia damine, but it was lower than that of coconut leaf sheath and Nendran banana peduncle plant fibers.

Lignin content of EVFs was higher than that of *Cissus vitiginea*, *Momordica charantia*, *Vachellia farnesiana* and *Acacia nilotica* plant, but lower than that of Grewia damine and Nendran banana peduncle plant. It has been reported that higher presence of wax reduced the bondability of the fiber with the matrix during preparation of composite material (Khan et al. 2019). Significantly, the wax content in EVFs was lower than that of Grewia damine, *Cissus vitiginea* and *Momordica charantia* fibers.

3.2. Fourier transform infrared spectroscopy analysis of EVFs

FTIR spectra of EVFs are shown in Fig. 2(a). A broad U-bend was observed at3325 cm^{-1}, due to the presence of cellulose and lignin contents, which was denoted by the stretching band of O-H. The two sharp peaks at 2919 and 2854 cm^{-1}were obtained, due to the C-H functional groups, representing cellulose and hemicellulose compounds (Shyam Kumar et al. 2021; Vijay et al. 2020b). The C-C bond, showing minimizing of wax, was observed from the peak at 2350 cm^{-1}. The next significant sharp peak obtained at 1608 cm^{-1} confirmed the presence of aromatic rings of lignin (Raja et al. 2021).In addition, the small peak at 1451 cm^{-1} also established the presence of hemicelluloses and C-O group of lignin was confirmed with the peak at 1372 cm^{-1}. A strong peak observed at 1022 cm^{-1} established the presence of C-O stretching of cellulose, hemicelluloses or lignin (Pandey, Jose, and Sinha 2020). Besides, EDX analysis quantified the elements present in EVFs, such as oxygen (O), carbon (C) and calcium (Ca), which were the major peaks and confirmed the organic nature of EVFs (Fig. 2b), as previously described in the FTIR chart

INSERT FIGURE 2

3.3. X-ray diffraction *analysis of EVFs*

XRD spectrum of EVF is shown in Fig. 3. The two lower intensity peaks at 2 θ values of 15.38 and 16.20° (110) were corresponded to the crystalline contents of cellulose and the amorphous form of cellulose, respectively. The two prominent broad peak at 2 θ values of 22.53 and 24.47° (200) confirmed the crystalline phase of cellulose constitution. Segal's peak difference method was employed to find the crystalline index (CI) from the intensity of the obtained peaks. The CI

of EVF was obtained as 37.5%. Crystallite size of EVF was computed as 36.93 nm, using Scherrer's equation.

INSERT FIGURE 3

Degree of crystallinity or percentage of crystalline represents the packing factor of the crystallites in the fibers and makes the fiber less water-permeable. The mechanical strength of fibers increase with its percentage of crystalline. In this study, the percentage of crystalline of EVF was calculated as 37.5%. Therefore, it confirmed that its crystallite packing was high and structural attachment was strong enough to produce durable biocomposites.

3.4. Thermogravimetric analysis

Thermal analysis showed that the degradation of EVFs followed a three-step process with a temperature ranged from 30 to 600 ^{o}C (Fig. 4). The degradation occurred in two main stages. The initial stage took place between 30 to 125 ^{o}C and it led to about 8.13% of the degradation, due to the removal moisture existing above the surface of the fiber. Only 3.37% of weight loss was observed in the TGA after the first phase, until 225^{o}C. The thermal peak observed at 225 ^{o}C established the depolymerization of the hemicellulose of EVFs. The prominent peak at 349 °C represented the cellulose degradation with a maximum weight loss of 46.36%. Similar peaks observed from other natural or bio-fibers were *Cissus vitiginea* plant of 304.0 ^{o}C (Chakravarthy et al. 2020), Nendran banana peduncle plants of 356.0 ^{o}C (Manimaran et al. 2020), *Coccinia indica* of 376.3 ^{o}C (Bhuvaneshwaran et al. 2021) and *Albizia amara* of 330.6 ^{o}C (Senthamaraikannan et al. 2018). The final phase of cellulose degradation occurred from 350 to 592 ^{o}C and the char residue content was measured as 19.61%.

INSERT FIGURE 4

3.5. Surface morphological analysis of EVFs

The SEM images of raw EVFs in Fig. 5 present the clear details of the surface topography of the natural fibers. EVFs were observed as a long and uniform cross-section, showing a better aspect ratio. The surface of EVFs in longitudinal direction is shown in Figs 5(a)-(d). There were many micro-holes present on the surfaces of EVFs. From Fig. 5(a), the white portion symbolized the presence of hemicellulose on the surface of EVFs. It was also evident from the images that its surface was highly rough, as later observed through AFM. EVFs also possessed irregular rods and rocky morphologies, when examined at higher magnification of 419x (Fig. 5(b)), and they were loosely packed. This further showed that the water penetration was high in the fibers. EVFs possessed extremely rough surface with minimum wax content even in its raw form, as observed in Fig. 5(d). This extremely rough and less closely packed surface can help its bonding with resin during preparation of biocomposites. The porous surfaces of fibers usually support a good fiber-matrix interfacial adhesion (Subramanian, Rajkumar, and Ramkumar 2021). Since the total surface area depends on the pore size, when the pore size is reduced, the relative surface area increases. Similarly, EVFs possessed an irregular surface with more number of scaly appearance and intermittent vacuoles on their surfaces, as depicted in Fig. 5(d).

INSERT FIGURE 5

3.6. Single fiber tension properties

Fig. 6 shows the stress-strain curve of EVFs. The single fiber test indicates the maximum stress that a material can withstand without failing when it is subjected to a pulling force. The ultimate tensile strength of the fibers was 6.06 ± 0.02 MPa, as similarly reported (Saravanakumaar et al. 2018; Kumaar et al. 2020; Muthu Chozha Rajan et al. 2020; Rajan, Muthu Chozha and Senthil Kumar 2018) and the mean tensile strength was 2.80 MPa. The single fiber test values ranged from 1.51and 6.06 MPa. The deviation in the tensile strength values was attributed to the non-uniform nature of the fibers and the presence of lignin and hemicellulose.

INSERT FIGURE 6

3.7.Surface topographical analysis of EVFs

Both two and three-dimensional (2D and 3D) surface images obtained from the AFM analysis are shown in Figs 7(a) and (b). The related plots and values highlighted that the average surface roughness value of EVF was 192.620 nm, which was comparatively and significantly higher than that of *Cissus vitiginea* (Chakravarthy et al. 2020), Nendran banana penducle (Manimaran et al. 2020) and Grewia damine (Ravindran et al. 2020) plant fibers of 0.194, 13.000 and 39.000 nm, respectively.

INSERT FIGURE 7

The surface skewness value of EVFs was -0.268 and exhibited dominant peaks and lower porosity on the fiber surface. Kurtosis shows how the spikes are spread with respect to the mean surface height. Negative skewness value indicates that the surface is somewhat porous in nature and valleys are dominant on the fiber surface (Kumar et al. 2020; Md et al. 2020). A negligible negative surface skewness (Rsk) value of a material sample denotes that very fewer cracks are presented on the sample. A higher value of average surface roughness produces a lower existence of unnecessary impurities on the fiber surface (Khan et al. 2019). The ratio of RMS roughness obtained from EVF was 224.170 nm. The average roughness value was 192.620 nm, which determined whether EVF can be used for tribological applications. It was in the range of 1.163, just slightly lower than the range specified. The maximum surface roughness value (R_y) was 872.14 nm and it indicated a rough apparent surface. For this present study, EVF exhibited a surface kurtosis value of 3.446 and it was higher than 3.000. This showed that EVFs possessed a bumpy surface texture. High roughness value obtained in this study indicated the presence of less impurities on the sample surface and a larger contact area, which can further lead to better fiber-matrix interfacial adhesion. Natural fibers are often treated chemically to modify the surface characteristics for further enhanced interfacial adhesion between fibers and matrices (Thirumurugan et al. 2021).

4. Conclusions

New natural fibers from EV plant have been extracted, using water-retting technique and their properties were analyzed. Chemical composition and morphological structure of EVFs and possibility of using them as reinforcement in polymer biocomposite materials were also investigated. From the chemical analysis, the results indicated that EVFs possessed rich cellulose content of 70.60% and thus, a promising property that could support good fabrication of green composites.

From the XRD analysis, the calculated CI and crystalline size of EVFs were 37.5% and 36.93 nm respectively, which confirmed that the crystallite packing was high and structural attachment was strong enough to produce durable biocomposites. The thermal degradation performance estimated through TGA and DTG curves showed that EVFs were thermally stable up to 349 ^{o}C. The density of EVF was 1412 kg/m^3 and has the advantage of replacing some synthetic fibers in lightweight applications. The obtained results showed that EVFs have some rich cellulose content and low wax. Therefore, this could be a potential replacement for the synthetic fibers.

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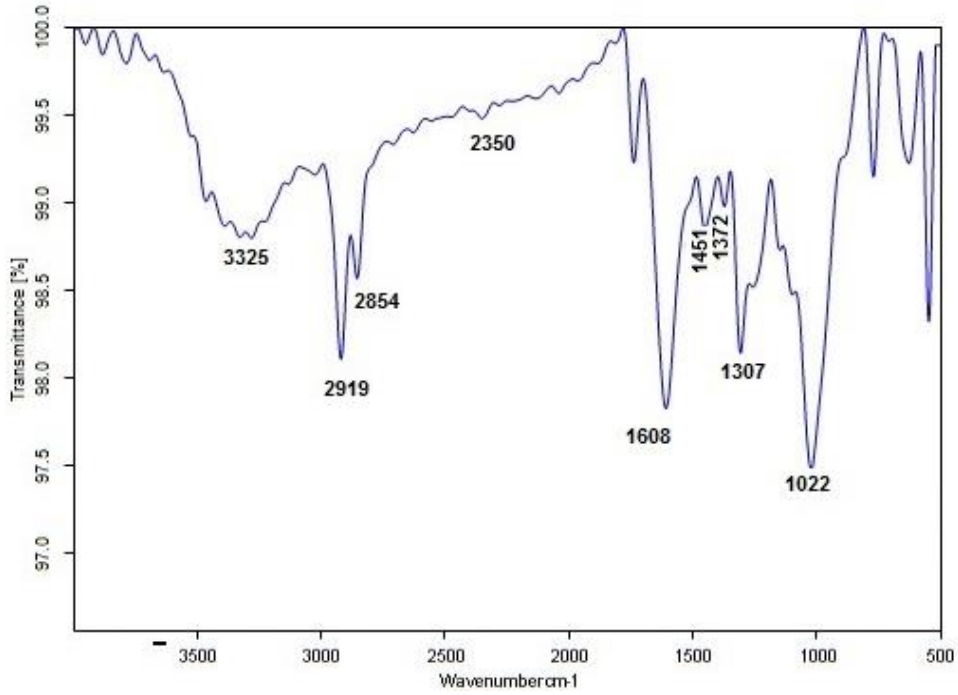
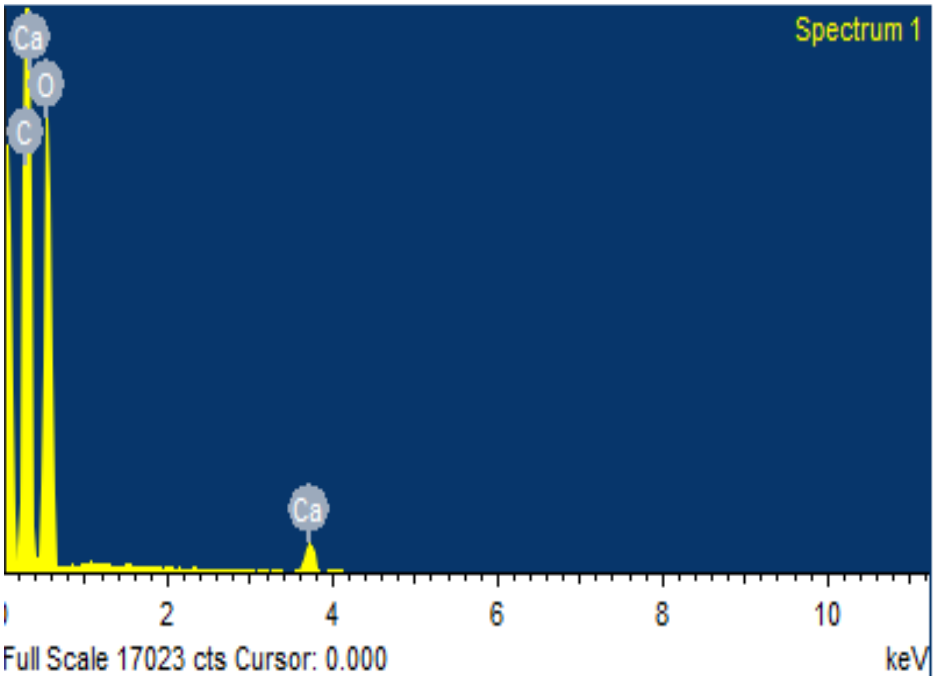
(a) (b) (c)

Fig. 1. (a) *Erythrina variegata* tree, (b) EV stem soaked in a bucket and (c) dried EVFs.



(a)



(b)

Fig. 2. (a) FTIR spectra between the range of 4000 and 500 cm^{-1} and (b) EDX analysis of EVFs.

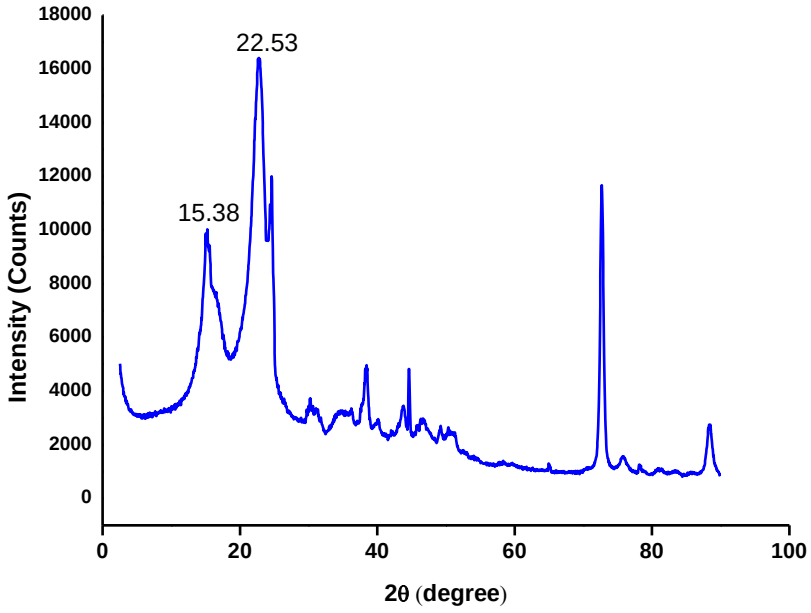


Fig. 3. XRD image of EVFs.

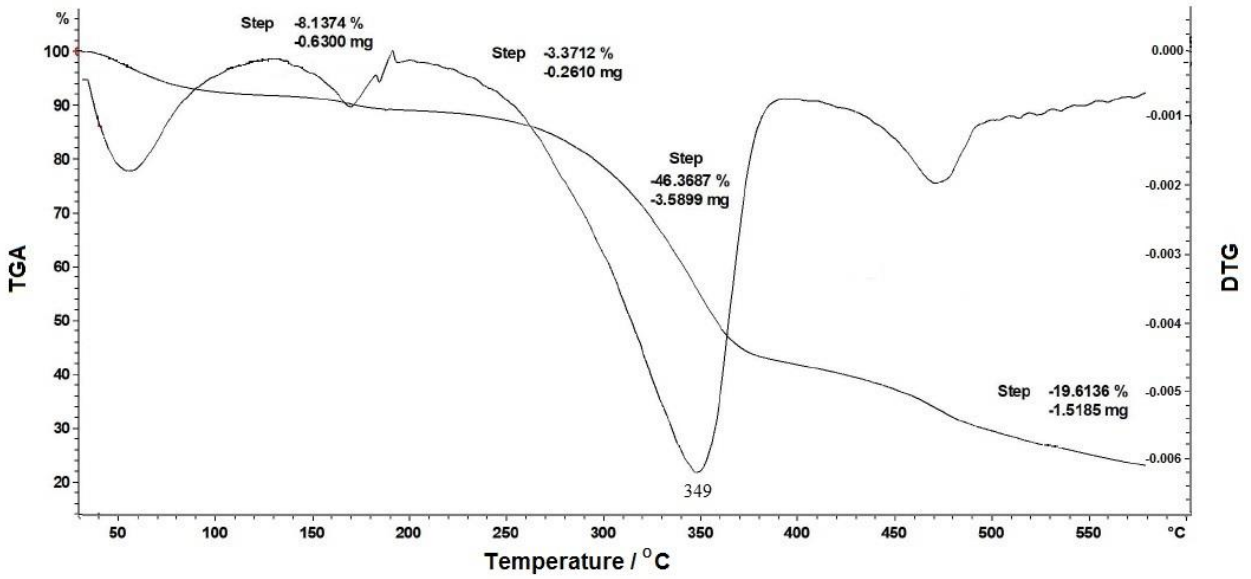
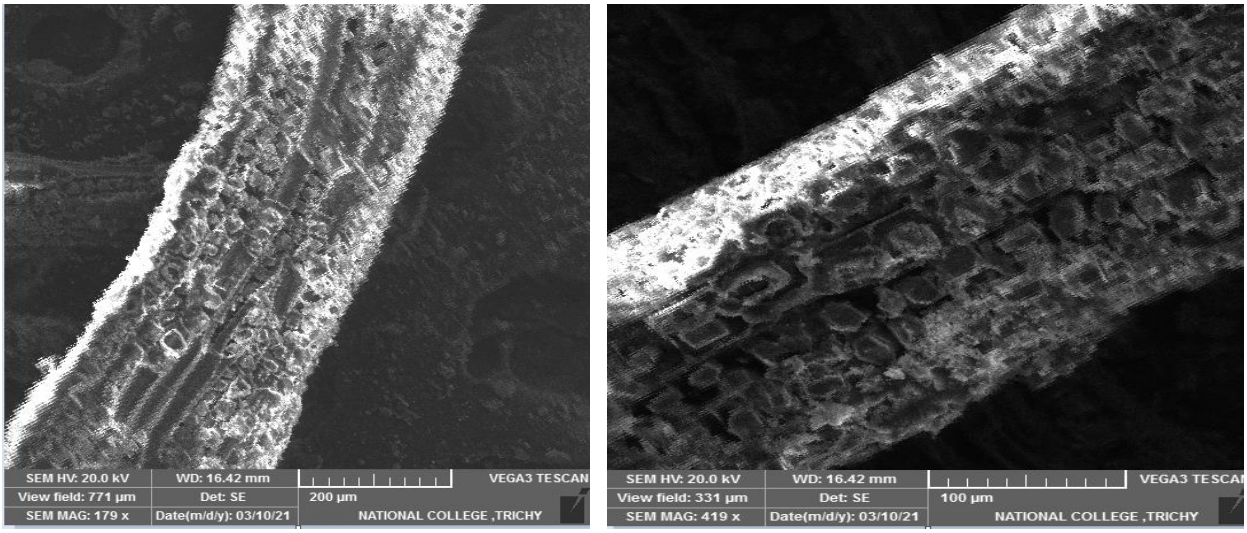
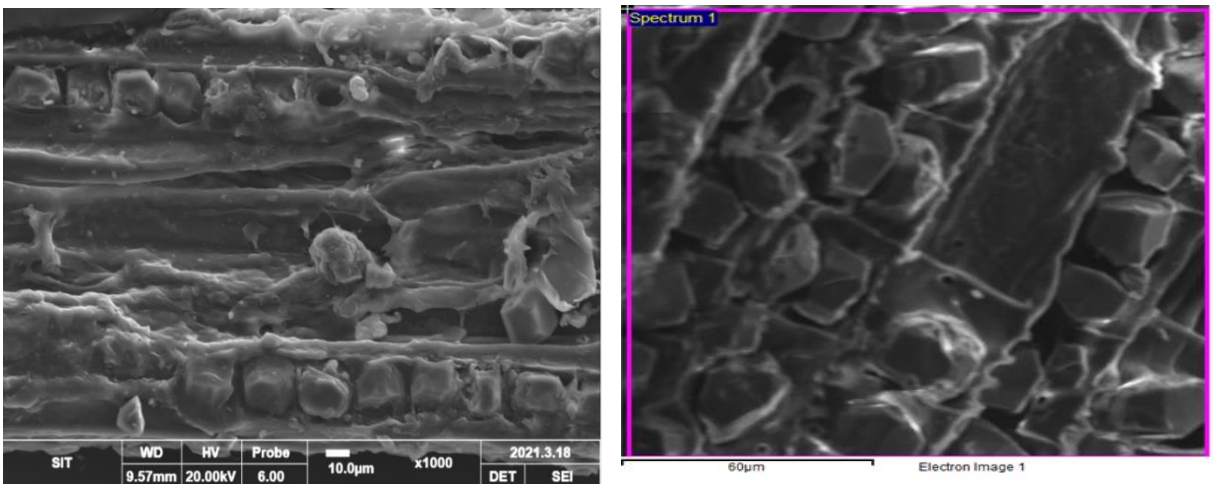


Fig. 4. TGA and DTG thermograms of EVFs.



(a) (b)



(c) (d)

Fig. 5. SEM images of the surfaces of EVFs.

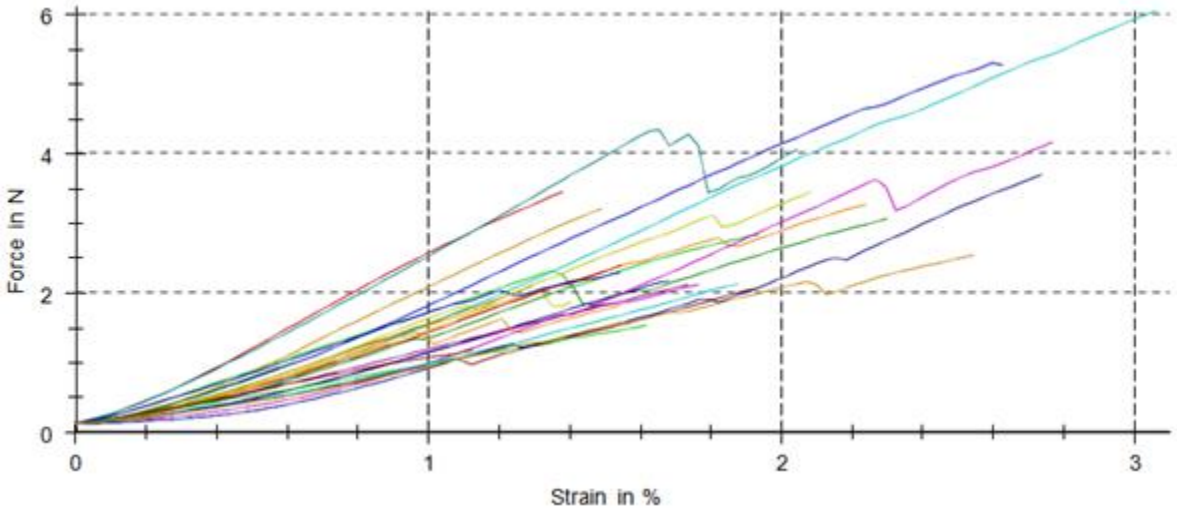
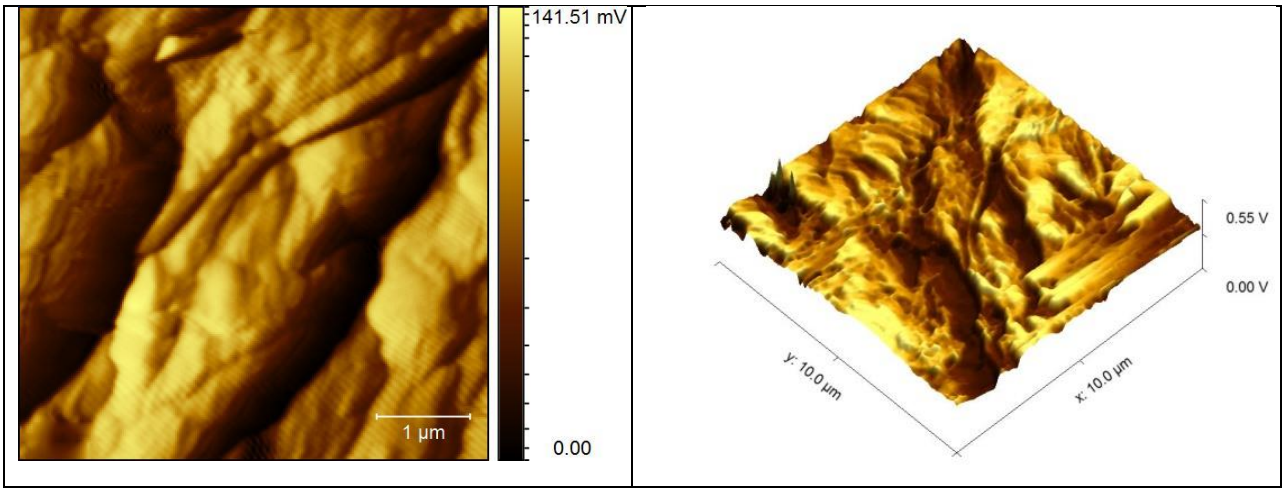


Fig. 6. Stress-strain curve of the single fiber obtained from the tension test on EVFs.



(a) (b)

Fig. 7. Surface topographical AFM images of EVFs in (a) 2D and (b) 3D.

Table 1

Comparison of chemical composition of EVFs with various similar plant fibers.

Fiber name	Cellulose (wt.%)	Hemi cellulose (wt.%)	Lignin (wt.%)	Wax (wt.%)	Moisture (wt.%)	Density (wt.%)	Ash (wt.%)	Ref.
Erythrina variegata (EVF)	81.60	26.21	12.70	0.24	7.80	1412	8.60	*Present work*
Momordica charantia	61.20	17.30	4.80	1.10	6.30	1339	2.24	[5]
Grewia damine	57.78	14.96	16.65	0.59	---	1378	7.74	[7]
Nendran banana peduncle plant	73.20	10.85	15.32	0.25	9.01	972	2.59	[10]
Vachellia	38.50	12.10	9.20	3.40	11.00	1270	6.21	[12]

farnesiana								
Cissus vitiginea	65.43	14.61	10.43	0.39	8.47	1287	12.05	[19]
Acacia nilotica plant	56.46	14.14	8.33	0.85	---	1165	4.93	[22]
Coconut tree primary flower leaf stalk fiber	71.70	6.03	11.57	0.12	9.03	1114	3.31	[30]