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ORIGINAL ARTICLE

Extraction and characterization of novel cellulosic biofiber from peduncle of *Areca catechu* L. biowaste for sustainable biocomposites

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Abstract

The use of readily available natural resources and wastes has become critical for achieving industrial sustainability. The current study focuses on the extraction and characterization of cellulosic *Areca catechu* fruit peduncle fibers (ACFPFs) from the peduncle of industrially discarded agro-waste after harvesting its economic value. To ensure ACFPF's potential and competitiveness in increasing the sustainability of the automobile industry, a number of comparisons were made between ACFPF and other commonly used natural fibers. To validate its claims, the major cellulose content (50.1 wt.%), less wax (0.3 wt.%), low density (1.05 g/cm^3), high crystallinity index (49%), tensile strength (88–184 MPa), and Young's modulus (1.1–5.5 GPa) of ACFPF were estimated. Besides that, thermal analysis (TGA/DTG) ensures ACFPF thermal stability up to 228 °C. Furthermore, when exposed to higher temperatures, the differential scanning calorimetry (DSC) analysis of ACFPF revealed endothermic and exothermic reactions, whereas the Fourier transform infrared spectroscopy (FTIR) spectra revealed chemical bonds in ACFPF. Besides, technical qualities, performance, environmental, economic, and societal factors all contribute significantly to the sustainability and productivity of eco-friendly fiber composite industries. Also, the estimated values and rough surface observed with a scanning electron microscope (SEM) validate the use of sustainable cellulosic ACFPF as reinforcement in polymer composites for lightweight structural applications. Moreover, its implementation has a significant environmental impact in terms of developing an effective, sustainable waste management approach.

Keywords Discarded agro-waste · *Areca catechu* L. fruit peduncle · Cellulosic fiber · Biocomposites · Characterization

Highlights
- Biowaste *Areca catechu L.* fruit peduncle fiber (ACFPF) characterized for reinforcement.
- Low wax (0.3 wt.%) and density (1.05 g/cm^3) of ACFPF ensure better bonding.
- Thermal analysis (TGA and DTG) certifies the thermal stability of ACFPF to 228 °C.
- The rough surface texture of ACFPF confirms good interfacial bonding features.
- Specific properties attained favors ACFPF reinforcement for sustainable biocomposites.

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1 Introduction

At the moment, businesses are concentrating on natural fiber-reinforced polymer composites because, compared to artificial fibers, they are lighter, cheaper, non-toxic, and break down over time [1]. Natural fiber-reinforced polymers were used to make door panels, windows, furniture, and appliances for homes, as well as seat covers, door panels, dashboards, clutch plates, and backrests for cars. Natural fibers from different parts of plants, like stems, leaves, stalks, roots, seeds, fruit peels, and animal fibers, are used as renewable assets to reinforce polymer composites that are used to make industrial products [2, 3]. Natural fibers have also become an important part of designing and making products.

In the recent industrial environment, biodegradable raw materials are given more importance due to environmental regulations and to improve the sustainability of every commercial product from industries [4]. These norms are owing to the concern over the environment and the depleting

natural resources. In this way, in the composite industry, man-made synthetic fibers, which are non-biodegradable, consume more energy during the manufacturing process and question the sustainability of products are thought to be replaced by an eco-friendly alternative [5, 6]. Natural fibers from plants are considered to be the main counterpart owing to their plenty of availability, low cost, ease of processing, and biodegradable nature. But the challenges of using natural fibers by composite industries are its assurance of continuous supply, non-standard forms like size, shape, and its hydrophilic nature [7]. The assurance of a continuous supply of natural fibers can be addressed by concentrating on the utilization of natural fibers from agro-waste which remains in landfills in agro-farms.

Lignocellulosic fibers made from discarded agricultural waste are used as the main reinforcement in polymer matrix composites because they are strong and last a long time [8]. Also, these lignocellulosic fibers have a wide range of properties that depend on their inner structure, chemical composition, density, age, microfibril angle, defects, and cell size. These assets need to be studied in detail for each natural cellulosic fiber [9, 10]. Characterization of fibers from *Sansevieria ehrenbergii* reveals that it was thermally stable until 333.02 °C within a diameter range of 10–250 µm and a density of 0.887 g/cm^3 [11]. The single fiber tensile test reveals its strength as 50–585 MPa, extension at failure as 2.8–21.7%, and tensile modulus as 2.5–7.5 GPa. Fibers from Tunisian *Agave americana* L. investigated for their mechanical characteristics reveal their sufficient tensile strength of 384.66 MPa and elongation at break of 4.96% which is promising to be used as reinforcement in polymer composites [12].

Cellulosic fibers from borassus fruit, after treatment with 5% alkali solution, exhibited a fiber diameter of 226.35 µm and density of 1.265 g/cm^3 with a cellulose content of 82.85%, hemicelluloses content of 3.02%, lignin content of 5.02%, wax content of 0.49%, and moisture content of 8.04% which is revealed through their investigation [13]. In the present work, eco-friendly fibers were extracted from the cerebral peduncle of the *Areca catechu* tree which is left out as agro-waste after harvesting the *Areca catechu* nuts and investigated for its suitability to be used as a reinforcement in polymer composites. The *Areca catechu* fruit peduncle fibers (ACFPFs) were characterized chemically, physically, and thermally to know their chemical composition, mechanical properties, and thermal stability, respectively. The surface nature of the sustainable ACFPFs was understood from the scanning electron microscopy (SEM) pictures of the fibers. Furthermore, Fourier transform infrared spectroscopy (FTIR) and X-ray diffraction (XRD) were done to ascertain the operational group and crystal structure of ACFPF. The above outcomes designate that eco-friendly ACFPFs reinforced in polymer composites not only advance the thermo-mechanical characteristics of biocomposites but also promote a major solution to industrial agro-waste causing ecological issues, thereby leading to a cleaner and more sustainable environment.

2 Materials and methods

2.1 Collection and extraction of cellulosic ACFPF

The inflorescence of the *Areca catechu* tree was collected from the Areca plantation in the Mettupalayam village located near Coimbatore, Tamilnadu, India. The peduncle of the collected inflorescence was isolated from the remaining part, and the outer skinny greenish layer was removed manually. Then, the *Areca catechu* tree peduncle was soaked in water for 2 weeks to loosen the fibers [14]. During the soaking process, the peduncles were rinsed in running water 2 to 4 times a day to remove the dirt and dust particles. After the soaking process, the fibers in the peduncle get loosened from the pulp and the ACFPFs were extracted from the peduncle by mechanical means [15]. The extracted ACFPFs (Fig. 1) were sun-dried in the open air for 3 days to diminish the vapor content before carrying out further investigations. These ACFPFs were investigated for its physico-chemical, thermo-mechanical, and morphological properties to know the prospect of employing them as an alternative reinforcement in polymer composites.

2.2 Characterization of cellulosic ACFPF

Standard test techniques portrayed in the existing works were adopted to identify the wt.% of cellulose, hemicellulose, lignin, and wax content in cellulosic ACFPFs [16]. The mass-volume method and standard weight loss method were followed to know the density and moisture content of ACFPFs, respectively. The tensile test of a single fiber was conducted as per ASTM D3822-07 standard [17]. The tests were repeated thrice to avoid experimental errors. To understand the crystalline nature and the presence of functional groups in the ACFPFs, X-ray diffraction spectroscopy (XRD) analysis and Fourier transform infrared (FTIR) spectroscopy analysis were carried out, respectively. The FTIR analysis was carried out at a resolution of 4 cm^{-1} with a scan rate of 32 scans per minute, whereas the XRD study was conducted in the 2θ angle range of 10° to 80° with 5°/min goniometer speed [18]. Also, the surface morphology of ACFPFs was observed utilizing a scanning electron microscope of model SUPRA 55 Zeiss with an accelerating voltage of 21 kV [19]. Furthermore, the thermogravimetric analysis (TG & DTA) and differential scanning calorimetry (DSC) analysis were performed on ACFPFs at a heating rate

Fig. 1 Extraction of cellulosic *Areca catechu* fruit peduncle fibers. (**a**) Collected peduncle of *Areca catechu* fruit and (**b**) extracted *Areca catechu* fruit peduncle fibers

of 10 °C/min in the temperature range of 30 to 550 °C and 30 to 450 °C, respectively [20].

3 Results and discussions

3.1 Chemical analysis of cellulosic ACFPF

The chemical nature of the natural fibers is highly influenced by the maturity, topographical, and weather conditions in which the plant is cultivated [21]. This, in turn, affects the mechanical, bonding nature, morphological, and moisture absorption characteristics of the fibers. These fibers when used as reinforcement in polymer composites highly govern the functionality of the materials. The results of the chemical analysis displayed in Table 1 reveal that the ACFPFs exhibit a higher cellulose content of 53.1 wt.% which is comparably higher compared to the other natural fiber reinforcements used in polymer composites [22]. The mechanical characteristics of ACFPFs highly depend on the wt.% of cellulose in fibers. The presence of a higher weight percentage of cellulose content in ACFPF improves the crystalline nature of the fiber, thereby resulting in better hydrophobic nature. The lesser wt.% of hemicellulose amounting to 11.4 wt.% in the ACFPFs favors the usage of ACFPF as probable reinforcement in polymer composites. The 23.62 wt.% of lignin present in ACFPF improves the binding nature of the fibers with polymer matrix when applied as reinforcement. The thermally stable nature of ACFPF is ensured by the ash content of 1.5 wt.% which also reflects the firefighting capacity of the fiber. This is also important for the ACFPF to withstand polymerization temperature when used as reinforcement in polymer composites. The acceptable wt.% of wax (0.28 wt.%) and moisture content (10.1 wt.%) in the ACFPF further confirm its usage as viable reinforcement in polymer composites.

3.2 FTIR analysis of cellulosic ACFPF

The FTIR spectrum displayed in Fig. 2 presents the existence of major functional groups in cellulosic ACFPF. The sharp crest identified in the wavelength of 563.79 cm^{-1}, 874.56 cm^{-1}, and 1037.63 cm^{-1} is attributed to the stretching of the alkyl halide organic functional group [25]. The presence of twisting alkene organic operating group = C-H is indicated by peaks noticed at the wavelength of 1298.73 cm^{-1} and 1374.53 cm^{-1}. These vibrations noticed in this waveband are due to the cellulose polysaccharides in ACFPF [26]. The peak noticed at 1413.57 cm^{-1} is designated to the extending vibrations of carbonyl functional group C-O. The subsequent peaks at 1508.12 cm^{-1} and 1687.32 cm^{-1} ensure the presence of lignin and hemicellulose in ACFPF, respectively. The crest at 2879.31 cm^{-1} corresponds to the C-H symmetric bending of the alkaline functional group [27]. The wider crest noticed at 3370 cm^{-1} is attributed to OH stretchings in the cellulose. The crests of the FTIR spectrum endorse the existence of biochemical constituents identified in the ACFPF.

3.3 Anatomical and morphological examination of cellulosic ACFPF

Table 2 portrays the physical properties of cellulosic ACFPFs. The dimensions presented for the cell wall, lamellae, lumen, diameter, and density of ACFPF are obtained by repeating the experiments thrice to reduce the errors. The incidence of cellulose and lignin in the primary and secondary cell wall, respectively, highly influences the mechanical properties of fibers [28]. Lamellae is composed

Table 1 Properties of cellulosic ACFPF in comparison with other counterparts used as reinforcement in polymer composites [23, 24]

Fiber name	Cellulose (%)	Hemi cellulose (%)	Lignin (%)	Wax (%)	Density (g/cm^3)	Elongation at break (%)	Tensile strength (MPa)	Young's modulus (GPa)
ACFPF*	**53.10**	**11.40**	**23.62**	**0.28**	**0.80–1.10**	**3.90–8.10**	**107–182**	**1.70–6.20**
Jute	61	20.40	13	0.70	1.30–1.49	1.16–1.50	393–800	13–26.50
Flax	64.10	16.70	2	1.50–3.30	1.50	2.70–3.20	500–1500	27
Cissusquadrangularis stem	82.73	7.96	11.27	0.18	1.22	3.75–11.14	2300–5479	56–234
Raffia textilis	148–660	-	-	-	0.75	2	148–660	28–36
Areca Fiber	57.35	13–15.42	23.17–24.16	0.12	0.7–0.8	10.23–13.15	147–322	1.12–3.15
Coir	32–43	0.15–0.25	40–45	-	1.20	30	593	4–6
Sisal	60–78	10–14.20	8–14	2	1.50	2–2.50	511–635	9–22
Hemp	74.40	17.90	3.70	0.80	1.47	2–4	690	70
Bagasse	55.20	16.80	25.30	-	1.20	1.10	20–290	19–27
Acacia Leucophloea	76.69	3.81	13.67	0.13	1.43	1.91–5.88	357–1809	10.45–87.57
Sansevieria cylindrica	79.90	10.13	3.80	0.09	0.91	12.30–13.70	666–706	6–8
Grewiatili folia	67.90	17	15	-	-	2.0	75.30	5
Alfa	45.40	38.50	14.90	2	0.89	5.80	350	22
Ramie	76	15	0.70–1	0.30	1.50	2–3	220–938	44–128
Kenaf	45–57	14.33	8.18	0.80	1.31	1.60	427–519	23.1–27.1
Aramid	-	-	-	-	1.40	3.30–3.70	3000–3150	63–67
Carbon	-	-	-	-	1.40	1.40–1.80	4000	230–240
E-glass	-	-	-	-	2.50	0.50	2000–3500	70

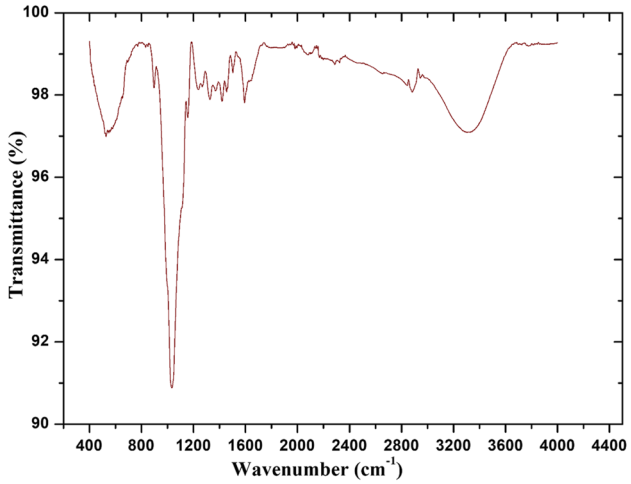

Fig. 2 FTIR spectra of cellulosic *Areca catechu* fruit peduncle fiber

of hemicellulose and lignin which acts as an interlayer and connects the cells. Lumen is a hollow space in the cell which contributes to the density of fiber when utilized as a prospective reinforcement in polymer composites.

The surface and cross-sectional image of the cellulosic ACFPF obtained through SEM is presented in Fig. 3. The surface of ACFPF is found to be rough with more ups and downs,

Table 2 Physical properties of cellulosic ACFPF

Property	Value
Thickness of primary cell wall (μm)	1.451 ± 0.0012
Thickness of secondary cell wall (μm)	0.408 ± 0.001
Thickness of middle lamellae (μm)	3.843 ± 0.002
Thickness of cell lumen (μm)	8.970 ± 0.0025
Fiber diameter (μm)	421.5 ± 0.052
Fiber density (g·cm^{-3})	1.050 ± 0.0031

as shown in Fig. 2a which is a positive sign of obtaining better bonding characteristics between the ACFPF and matrix when employed as reinforcement in polymer composites [29]. If the rough surface of ACFPF when used as reinforcement in polymer composites, the matrix will have more surface area with the ACFPF reinforcement to interact, thereby achieving better interfacial bonding characteristics. The presence of lignin which acts as binding material and micro fibrils in attachment with parenchyma cells was also ensured through the obtained SEM images of ACFPF. The angle of micro fibrils also influences the mechanical properties of ACFPF which are calculated using Eq. (1) as 6.58° ± 29°.

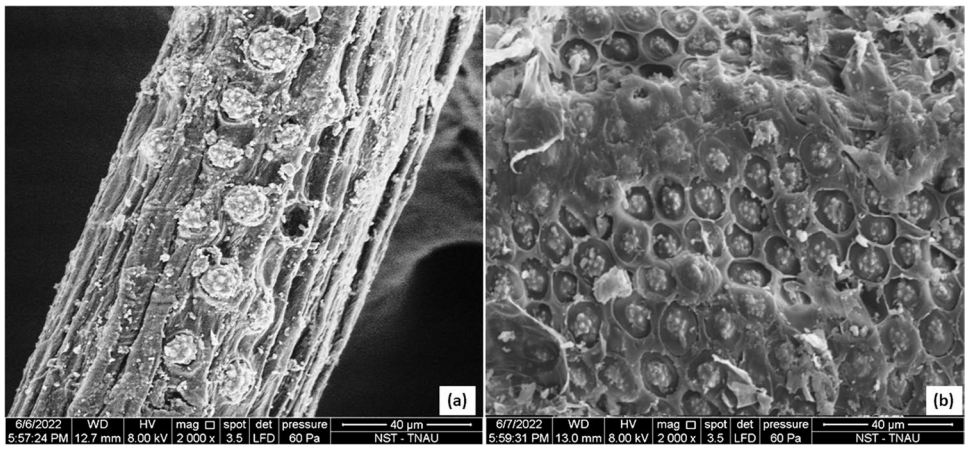

Fig. 3 SEM image of cellulosic *Areca catechu* fruit peduncle fiber: (a) parallel view 2000 × and (**b**) sectioned view 2000 ×

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

where ε is the strain, α is the micro fibril angle (degree), ΔL is the elongation at break (mm), and L_o is the gauge length (mm). Natural fibers with lesser micro fibril angle when reinforced in polymer composites ensure improved stress transfer between the matrix and the reinforcement.

3.4 XRD analysis of cellulosic ACFPF

Figure 4 portrays the XRD spectra of cellulosic ACFPFs. The semi-crystalline nature of ACFPFs is demonstrated by the strong peak at Bragg angle 2θ = 22.8° obtained due to the incoherent scatter of X-rays [30]. Another minor peak noticed at Bragg angle 2θ = 17.01° is related to the amorphous contents in ACFPFs. These identified peaks are attributed to the crystallographic planes (2 0 0) and (1 1 0) for Bragg angles 22.8° and 17.01°, respectively [31]. The crystallographic plane (2 0 0) fits to cellulose-I having a monoclinic arrangement. Other fluctuations noticed in the XRD spectra of ACFPF are due to the availability of impurities in the ACFPF sample.

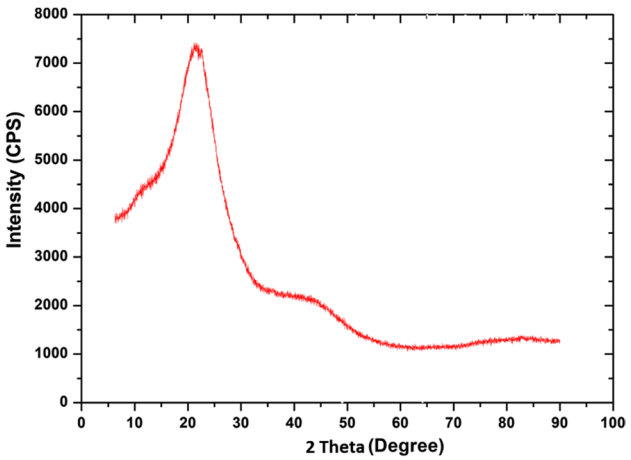

Fig. 4 XRD spectra of cellulosic *Areca catechu* fruit peduncle fiber

The empirical Eq. (2) [32] was applied to calculate the crystallinity index (CI) of ACFPF as 49%.

$$\mathrm{CI} = \frac{(I_c - I_{am})}{I_c} \tag{2}$$

where I_c is the maximum intensity of crystalline peak at 2θ = 22.8° and I_{am} is the intensity of amorphous peak at 2θ = 17.01°. The obtained CI value reflects the better mechanical characteristics and compactness of the ACFPF when reinforced in polymer composites. The crystallite size (CS) of the ACFPF was assessed using Scherrer's Eq. (3) as 1.29 nm [32]. The higher value of CS obtained for ACFPF indicates the hydrophobic nature of fibers.

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

where $K = 0.89$ is Scherrer's constant, λ is the wavelength of the radiation, β is the peak's full width at half-maximum (FWHM) expressed in radians, and θ is Bragg's diffraction angle.

3.5 Physico-mechanical analysis of cellulosic ACFPF

Table 1 portrays the physico-mechanical properties of cellulosic ACFPF in contrast with its other natural and synthetic counterparts. An optical microscope along with image J software helped in obtaining the diameter of ACFPF which ranged between 360 and 483 µm. Even though the cross-sectional shape of ACFPFs deviated from the standard circular shape, in order to estimate its mechanical properties, it is presumed to be circular, and the diameter was estimated using Eq. (4) [33].

$$D_f = \sqrt{\frac{L_f}{9000 \times M_d \times 0.7855}} \quad (4)$$

where D_f indicates the diameter of ACFPF, L_f indicates the linear mass density of ACFPF in denier which is a quantity of fineness of the ACFPF assessed as per ASTM D1577-07 standard, and M_d indicates the mass density in g/cm^3. Powdered ACFPF samples were jam-packed in a cylindrical container of known weight and volume to estimate the density of fiber on the mass-volume basis as 1.05 g/cm^3. The minimum density of ACFPF safeguards its use as viable reinforcement in polymer composites used in automobile sectors. Moreover, ACFPF with the obtained density when reinforced in polymer composites helps to achieve better specific characteristics. The diameter of the ACFPF samples was calculated using Eq. (4) as 406.53 µm and cross-checked with the values obtained from image J software.

The tensile strength of the ACFPF depicted in Table 3 ranges between 89 and 186 MPa which is in the nominal range of its counterparts used as reinforcement in polymer composites. The variation in physical properties of ACFPF observed over the change in gauge length is due to the dissimilarities in the physical structure of ACFPFs. The higher strain-to-failure values and the lower Young's modulus values of ACFPF indicate its load-carrying capacity and failure-resistant nature of fibers. The calculated and measured physico-mechanical values of ACFPF indicate its adequacy to be used as likely reinforcement in polymer composites. In polymer composites, the reinforcements are considered to be the main load-carrying members. The ACFPF with better tensile characteristics, when reinforced in polymer composites, will result in composites of enhanced load-carrying capacity.

3.6 TGA analysis of cellulosic ACFPF

Figure 5 demonstrates the thermal nature of cellulosic ACFPF. The vaporization of humidity content in ACFPF is suggested by the weight loss until 114 °C. The ACFPF was observed to be thermally stable until 228 °C which is indicated by the minor weight loss observed at this temperature.

Table 3 Mechanical properties of cellulosic ACFPF obtained as per ASTM standards

Gauge length (mm)	Tensile strength (MPa)	Young's modulus (GPa)	Strain to failure (%)	Diameter (µm)
10	89 ± 22	1.2 ± 0.2	4.7 ± 1.6	386 ± 0.031
20	123 ± 32	3.3 ± 0.4	3.8 ± 1.4	442 ± 0.052
30	155 ± 39	4.3 ± 0.6	3.7 ± 0.8	423 ± 0.081
40	186 ± 43	5.6 ± 0.8	3.4 ± 0.6	411 ± 0.073

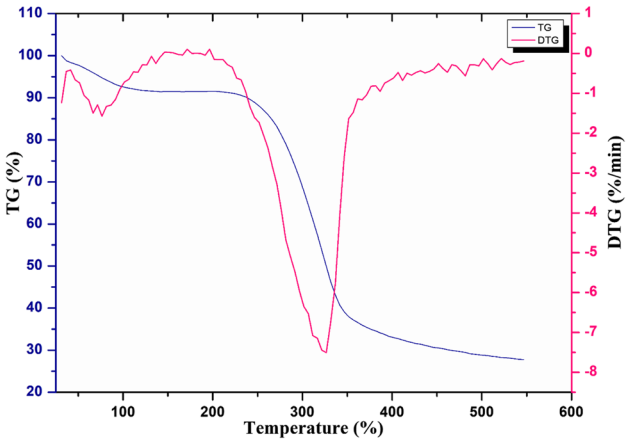

Fig. 5 TGA-DTG curve of cellulosic *Areca catechu* fruit peduncle fiber

Increasing the temperature further starts the degradation of cellulose, hemicellulose, and lignin along with depolymerization in ACFPF which is indicated by the weight loss of 14.7% until 283 °C [34]. A considerable weight loss noticed from 283 to 389 °C indicates the complete deprivation of cellulose in ACFPF. An additional increase in temperature reads some weight loss which is the degradation of wax and bits and pieces of lignin in ACFPF. The infliction point seen at 326.8 °C in the DTG curve with a mass variation of 52.95% denotes the pyrolysis and fragmentation of molecular structure in the cellulose [35]. The residual char content of 14.72% noticed at 999.7 °C indicates the firefighting capacity of ACFPF. Broido's Eq. (5) was used to determine the dynamic activation energy (E) of the ACFPF as 74.67 kJ/mol.

$$\varepsilon = \ln\left[ln\left[\frac{1}{y}\right]\right] = -\left(\frac{E}{R}\right)\left[\left(\frac{1}{T}\right) + K\right] \quad (5)$$

where R is the universal gas constant (8.314 J/mol K), T is the temperature in Kelvin, and y is the normalized weight. The obtained kinetic activation energy value helps to understand that ACFPFs are capable of withstanding the polymerization temperature when used as a credible reinforcement in polymer composites.

3.7 DSC analysis of cellulosic ACFPF

Figure 6 portrays the DSC plot of cellulosic ACFPF, which demonstrates a sequence of exothermic and endothermic reactions with an increase in temperature. The endothermic region of ACFPF indicates the evaporation of water molecules which are free as well as linked to cellulose. The water molecules that are linked to cellulose evaporate at a relatively higher temperature above 100 °C compared to the free

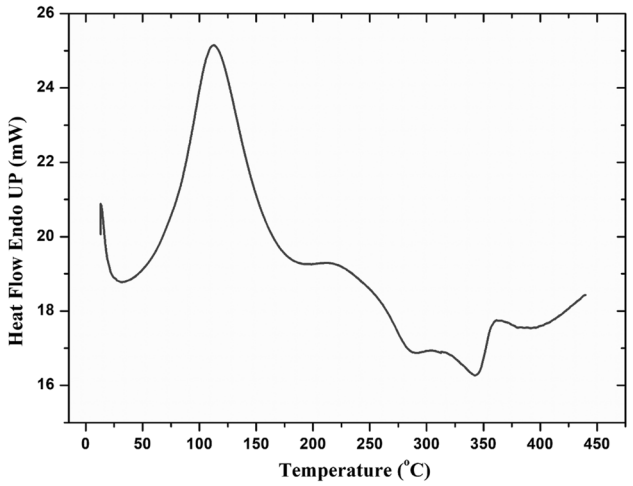

Fig. 6 DSC plot of cellulosic *Areca catechu* fruit peduncle fiber

water molecules in ACFPF [36]. The evaporation of both free and linked water molecules is observed in the endothermic region until 128.31 °C. The exothermic and endothermic processes observed at 172.87 °C and 218.52 °C, respectively, indicate the degradation of wax from ACFPF. Another endothermic peak noticed at 342.46 °C indicates the degradation of cellulose, whereas the exothermic peak observed at 348.57 °C indicates the desiccation and pyrolysis of lignin and hemicellulose in ACFPF. The remaining exothermic and endothermic processes seen above 350 °C indicate the degradation of lignin and hemicellulose in ACFPF. The observed exothermic and endothermic processes in ACFPF indicate that ACFPF-reinforced polymer composites can be used for low-density applications where geographical locations record higher ambient temperatures.

4 Conclusions

The estimated outcomes of eco-friendly agro-waste ACFPF characterization ensure its use as bio-reinforcement in polymer composites to improve the sustainability of material and widen the scope of application in fiber composite industries. The comparable cellulose content of 50.1 wt.% with the coarse surface texture of cellulosic ACFPF helps to obtain better mechanical and interfacial bonding characteristics with the matrix when reinforced in polymer matrix composites. Also, the adequate tensile strength of 184 MPa and lesser density of 1.05 g/cm^3 of cellulosic ACFPF showcases excellent specific strength when reinforced in a polymer matrix requiring lightweight structural applications. The hydrophobic nature of cellulosic ACFPF revealed through XRD analysis is sufficient enough to use ACFPF-reinforced polymer composites in applications requiring materials that come into contact with moisture. In addition, the thermally stable nature of eco-friendly ACFPF exposed through TGA and DSC analysis ensures its stability in polymerization temperature and can be used as reinforcement in polymer composites deployed in high ambient temperature industrial environments. Also, using eco-friendly ACFPF in the locomotive industry will be a step toward making the industry more sustainable and productive, as well as helping to solve and manage agro-waste generated in farms.

Author contribution Joseph Selvi Binoj: investigation, writing – original draft. Mariatti Jaafar: review and editing. Bright Brailson Mansingh: writing – original draft. Govindarajan Bharathiraja: resources and supporting.

Data availability This is an ongoing research work, and hence, the data cannot be shared at this moment.

Declarations

Ethics approval and consent to participate All the authors demonstrate that they have adhered to the accepted ethical standards of a genuine research study. Also, individual consent from all the authors was undertaken to publish the data prior to submitting it to the journal.

Consent for publication Written formal consent ensures that the publisher has the author's permission to publish research findings.

Competing interests The authors declare no competing interests.

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