

Research Article

Investigation of Physicochemical Properties and Characterization of Leaf Stalk Fibres Extracted from the Caribbean Royal Palm Tree

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Synthetic fibres (SF) are replaced by natural fibres (NF) and are utilized as polymer reinforcement owing to their eco-friendliness. The composite has been introduced in the current development by employing NF as reinforcement and stuffing in the polymer matrix (PM). The advantages of using natural resources are being eco-friendly, having plentiful natural availability, higher strength, lower cost, and a simple extrication process. When heated to a specific temperature, certain synthetic products create noxious materials. Therefore, replacing these synthetic substances with natural substances has greater advantages for the environment. In this study, a novel NF extricated from the Caribbean royal palm (CRP) along with its features is determined to replace the harmful SF effectively. The CRP's leaf stalks, termed CRP leaf stalk fibres (CRPLSFs), are extricated and categorized by (i) thermogravimetric analysis (TGA), (ii) scanning electron microscopy (SEM), (iii) Fourier-transform infrared (FT-IR) spectroscopy, (iv) physical-chemical analysis, (v) X-ray diffraction (XRD), and (vi) tensile test (TT). The physical-chemical characteristics of CRPLSFs, cellulose content (CC), tensile strength (TS), density, and hemicelluloses correlate with other NF characteristics. The CRPLSFs' chemical components include hemicelluloses (14.52%), lignin (9.15%), and cellulose (61.67%). The TGA shows that the CRPLSFs are thermally stabilized up to 326°C. The XRD proved that the CRPLSFs are enriched with a cellulose fraction comprising a crystallinity index (CI) of 30.27%. The outcomes recommended that the biodegradable coconut peduncle leaf stalk fibres (CPLSF) could be exploited as possible reinforcement in the PM composite structure and can be engaged in making composites.

1. Introduction

NF has been replaced over several SF like Kevlar, carbon fibres, glass (mainly S or E glass), etc, on account of their features such as less weight, reduced density, biodegradable, reasonable cost, and availability in abundance [1, 2]. The inclusion of NF as a reinforcing agent in both the thermoset and thermoplastic polymer composites (PC) has attained an increasing application in numerous fields of engineering as well as technology [3]. By employing modified synthetic techniques, a range of NF-based PC materials have evolved to enlarge their usage from automotive to biomedical fields [4]. Natural cellulose fibres (NCF) are disengaged from several bioresources such as birds, vegetables, animals, or minerals. Recently, the deployment of fibre-reinforced composite (FRC) has displayed an enhancement in numerous applications [5]. Presently, these compounds have been deployed in new application fields such as civil structures and biomedical devices [6]. Figure 1 displays the classification of NCF. The NCF's physical and mechanical properties are constrained by the plant's age, the chemical composition, the region where it is grown, and the extrication methodologies [7].

The NF is more beneficial than glass fibre so that it possesses the specific factors of lower density and higher volume, offers better thermal resistivity, and better skin protection over radiation [8]. SF, such as carbon, Kevlar, glass, etc possesses higher strength and stiffness. However, the challenges related to them include recyclability, health hazards, biodegradability, and initial processing cost to discover a substitute source for the development of composites [9]. These factors of the NF prepare it qualified for reinforcement by the polymeric materials (applied in biopolymer composites) [10] that are inputted for manufacturing the thermal insulator, functional prototypes, and irradiation shield [11, 12]. The success of these fibres relies on their structural along with mechanical properties. These factors establish the classification features for fibres [13]. These features are impacted by the region in which these substances originated, climate conditions, plant age, and extrication methodologies utilized. Several NF is attained as reinforcements of PC. The NFs like hemp, jute, sisal, flax, and kenaf have extensively utilized an account of their characteristics along with attainability [14, 15].

NFs' PC has been the focus of research attempts worldwide for the past few years. Now, in numerous fields like automotive components, interior decorating, and biomedical appliances, the practical utilization of NF reinforced composites is increasing rapidly [16, 17]. The differentiations between NF have been summarized by numerous researchers regarding the mechanical properties along with the appliances of those fibres. Numerous researchers stated the consumption of NF such as flax, bamboo, okra, coir, jute, Arundodonax (giant reed), alfa, and wheat straw as reinforcement in PC materials [18]. The physicochemical features of leaf stalk fibres extricated from the CRP (Roystoneaoleracea) plant are illustrated in this paper. CRP is a genus of palm that inhabits in the Lesser Antilles, northern South America, and Guatemala. The CRP's leaf stalks are categorized by FT-IR spectroscopy, physicalchemical evaluation, TGA, single fibre TT, and XRD, and the outcomes are contrasted with other prominent NF.

The paper's remaining sections are structured as follows: the related works are explained in section 2; the materials and the methodologies of the present study are illustrated in section 3; and section 5 winds up the paper.

2. Literature Survey

Manimaran et al. [19] handled the Sidacordifolia fibres (SCFs) categorized by single fibre TT, chemical analysis, XRD, FT-IR analysis, TGA/DTG, and atomic force microscopy (AFM). The SCFs' chemical compositions included hemicellulose (17.63%), lignin (18.02%), and cellulose (69.52%). The TGA evaluation proved that the SCFs were thermally constant up to 338.2°C. The XRD established that SCFs were wealthy in cellulose, comprising a CI of 56.92%. Taslima Ahmed Tamanna et al. [20] coped with the NFs that were extricated from the Coryphataliera fruit (CTF), and its features were described for the substitution of noxious SF. XRD and chemical composition classification assured a high quantity of crystallinity (62.5%) in conjunction with cellulose (55.1 wt %) in the CTF fibre. The FT-IR evaluation ensured the various functional classes of cellulose, lignin, and hemicelluloses were comprised in the fibre. The highest TS was achieved at 53.55 MPa for GL 20 mm and Young's modulus of 572.21 MPa for GL 30 mm. The thermal evaluation assured the CTF's thermal sustainability up to 230°C. Overall, the abovementioned results confirmed that the state-of-the-art CTF fibre possibly strengthened the FRC materials. Vijay et al. [21] managed the classification of Partheniumhysterophorus fibres (PHF) extricated from its stem by executing a retting process manually. Subsequently, extricated fibres were alkali-treated, and their features were correlated with those not treated. The varied activities of the alkali-treated together with nontreated PHF were reviewed utilizing FT-IR spectroscopy, TT, physical-chemical evaluation, XRD, SEM, and TGA. The outcomes displayed that the alkali-treated PHF established an elevation in CC by 8.9% more than the nontreated PHF, while the char deposit was 38.5%. It also revealed higher CI, surface features, and better TS. In the sliding direction, the debonding and breaking of fibres were observed to be very high in the composites. During the wear, the thermomechanical loading and shear stresses are caused in the sliding direction, resulting in breaking of the fibres from the matrix.

Prithivirajan et al. [22] handled Musa paradisiaca L. blossom petal (MPBP) fibres of banana agro-based domestic waste. It was the first time; the MPBP fibres' characteristics were documented. XRD evaluation displayed a CI of 56.71% along with a size of 16.38 nm. The TGA indicated that the MPBP fibres were thermally constant up to 220°C. Differential scanning calorimetry evaluation provided two thermal deterioration temperatures of 368.1 and 476.8°C, comprising a kinetic activation energy of 62.43 kJ. TS, Young's modulus, together with strain at failure, was recorded as of the single fibre TT as 108 MPa, 1.05 GPa, and 11.15%, correspondingly. Arvinda Pandian et al. [23]



FIGURE 1: Classification of natural fibres.

described and identified the appropriateness of calotropisprocera fibres (CPF) to reinforce PMs from FT-IR spectroscopy and XRD studies. Composite models were made utilizing different weight % of comprising epoxy resin as the matrix. The consequence of fibre loading (FL) on the tensile, impact, flexural, and water absorption features was performed and documented. It was recognized that FL enhanced the composites' mechanical properties and was endorsed by the strong connection between the fibre reinforcement and the PM. The FL above 40 weight % might mitigate the mechanical strength, whilst no vital variation in water absorption is perceived when the FL exceeds 30 weight %. The work recommended that CPF be capable of reinforcing polymers so they could be consumed for the composite fabrication.

3. Materials and Methods

3.1. Materials. (See Figure 2) shows Roystoneaoleracea (R.oleracea), also called CRP, imperial palm, cabbage palm, or palmist, is a palm species native to Colombia, Antilles, Trinidad and Tobago, and Venezuela. Roleracea is a tall palm that grows up to a height of 40 meters (130 ft). Its stems are either grey or whitish-grey with a diameter of 46-66 centimetres (18-26 in). The stem's top region is surrounded by leaf sheaths, forming a greenish part termed the crown shaft, which is generally around 2 m (6.6 ft) long. A single tree is recorded to possess 16-22 or 20-22 leaves. Leaves comprise a 60-100 cm (24-39 in) long petiole and a 4-4.6 m (13-15 ft) rachis. It is greatly esteemed as a decorative and multiuse tree in its native region. The variety has been extensively commenced for landscaping uses, along with the amalgamation of higher productivity; higher seed germination rates; and seed dispersion by animals contribute to its invasiveness.



FIGURE 2: Caribbean royal palm tree.

3.2. Methods

3.2.1. Fibre Extraction. The CRP's leaf stalks were cut down from the CRP tree in Coimbatore District, Tamil Nadu, India. The leaves on the stalks were taken off manually. The leaf stalks being collected were left for water absorption for 3 weeks meant for microbial degradation conducted by the water retting procedure, which takes off the gum-like substances between the fibres. The fibres were extricated by beating the retted stalks utilizing a wooden mallet. Lastly, the extricated fibres were cleansed with purified water; after that. They were dried out at room temperature for 6 days to remove the wetness that occurs in the fibres.

3.2.2. Physical Analysis

(1) Diameter Measurement. Using a Carl Zeiss Optical Polarizing Microscope, the CRPLSF's diameter was computed. The diameter was calculated for approximately 20 varied sections at 5 varied locations of a single fibre to consider their variation in thickness together with single units.

(2) Density Measurement. The traditional pycnometer methodology was utilized to appraise the CRPF's density. Before the execution, the CRPLSFs model was sliced into 5–6 mm in length and set aside in desiccators comprising silica gel for 5 days to remove the wetness. After that, the CRPLSFs were wrapped up in a container with toluene for two h to eliminate the tiny gaps in the outer region. Lastly, CRPLSFs were positioned in the pycnometer and density was established utilizing the analytical relation [24]as given below.

$$D_{s} = \frac{\beta_{2} - \beta_{1}}{(\beta_{3} - \beta_{1})(\beta_{4} - \beta_{2})},$$
(1)

wherein, β_1 specifies the unfilled pycnometer's mass in kg, the pycnometer together with fibre's amalgamated mass is signified as/in kg, β_3 specifies the mass of toluene filled in the pycnometer in kg along with the combined mass of toluene, and fibre, along with the pycnometer in kg, is specified as β_4 .

3.2.3. Chemical Analysis. The NFs' characteristics have relied on their chemical opus. The fibres' chemical opus was highly persuaded by the region, physiognomies of soil, extrication situations, the plant age, and the methodologies were utilized to recognize the composition. The chemical compositions of CRPLSFs, namely hemicelluloses, cellulose, and lignin, were established using standard analytical methodologies. The CRPLSFs' ash content percentage was recognized by the methodology of the ASTM E1755-01 standard [25]. The moisture content was calculated utilizing an electronic moisture analyzer such as Sartorius, model MA45; together, the wax was detected as stated by a typical methodology produced by Conrod [26].

3.2.4. X-Ray Diffraction Method (XRD Analysis). The CRPLSF's CI was reviewed utilizing XRD. The extent of structural arrangement is established via the CI, whose quantity is highly vital as it impacts the alkali treatment in tandem with the mechanical features of NCF. The XRD evaluation of AIFs was reported on a Bruker Eco D8 Advance AXS process. The monochromatic strength of Cu Ka rays in the range of 10° -80° at a rate of 5°/min is developed in an X-ray tube. It operates at 40 kV together with 30 mA. Using the following formula , the CI was appraised.

$$C_I = 1 - \frac{I_{ap}}{I_{cp}} \times 100\%,$$
 (2)

wherein, I_{ap} specifies the amorphous peak's intensity and I_{cp} indicates the crystalline peak's intensity in the XRD spectrum. The CRPF's CS was operated by formula

$$C_{\rm S} = K_{\rm n}/\psi \,\cos\,\theta,\tag{3}$$

wherein, Scherrer's constant is signified as K = 0.89, the peak's full width at half-maximum is specified as ψ , and η specifies the radiation's wavelength.

3.2.5. Fourier Transform Infrared Spectroscopy. The potency of an NF to be exploited as reinforcement is determined by the FT-IR spectrum that reveals the subsistence of biopolymers. FT-IR spectroscopy is performed on the fibre obtained utilizing a SHIMADZU FT-IR spectroscopy in the range of $4000-500 \text{ cm}^{-1}$ with a mean of 32 checks in conjunction with a resolution of 4 cm^{-1} at room temperature.

3.2.6. Thermogravimetric Analysis (TGA). TGA and differential TGA explored the CRPLSF's thermal stability by employing a Jupiter simultaneous thermal analyzer like Model STA 449 F3, Netzsch, Germany. The computation was implemented in a highly pure nitrogen environment at a rate of 20 mL/min, and the loss in weight was reported at a heating range of 10° C/min at 30° - 500° C. The extensions were produced utilizing an alumina melting pot to uphold a better connection between the model and the thermocouple.

3.2.7. Tensile Strength. The CRPLSF's chemical opus influences mechanical aspects like TS. The NF's TS is a consequence of its quantity of CC. The universal testing machine (UTM) INSTRON was utilized to execute TT with a power of 3 KN. Over 20 individual fibres were extricated, and the experimentation was conducted in accordance with the ASTM D 3822-07 standard at a rate of 0.5 mm/min.

4. Results and Discussion

CRPLSFs' physical-chemical properties are correlated with other NFs, as shown in Table 1 and Table 2. The CC in NF is regarded as the primary substance that results in the elevation of stability, TS, stiffness, and resistance to hydrolysis, together with the economical manufacturing of fibres for various purposes. The cellulose available in the CRPLSFs is 61.67%, which is higher than various NFs, namely, (a) bamboo (26%) [27], (b) coir (43%) [28], (c) kenaf (53.14%) [29], (d) coconut (32–43%) [30], (e) Althaea officinalis *L* (44.6) [31], (f) Arundo donax (43.2) [14], (g) Pergulariatomenstosa L. seed fibre (43.8) [32], (h) Carica papaya fibres (58.71%) [33]. In fibres, the elevation in CC provides a smarter performance as a reinforcing agent in polymers.

The other substances in the CRPLSFs contain 9.15% lignin, 14.52% hemicelluloses, 0.21% wax, 12.12% ash, and 2.48% moisture. In fibre, the lignin supports maintaining water and provides shelter against bioattack. The drawback in NF is the larger quantity of wax, which provides lower interfacial bonding between the polymer and fibre whilst utilizing it as reinforcement in the fibre-reinforced synthetics along with the wax in CRPLSFs was merely 0.21%. The CRPLSF's density is 1468 kg/cm3, which is high when correlated with other NFs, namely, coconut (1.2), Arundo donax (1006), Kenaf (1400), Althaea officinalis *L* (1180), Carica papaya fibres (943), and Pergularia tomenstosa L. seed fibre (1168). The CRPLSF's diameter is 387 μ m, more than the other fibres used for comparison.

TABLE 1: Diameter and density of CRPLSFs with other NF.

Fibre name CRPLSFs	Diameter (μm) 387	Density (kg/m ³) 1468	References Current study
Bamboo	240-330	_	[27]
Coir	100-450	_	[28]
Kenaf	65–71	1400	[29]
Coconut	100-450	1.2	[30]
Althaeaofficinalis L	156–194	1180	[31]
Pergulariatomenstosa L. seed fibre	35.6	1006	[14]
ArundoDonax	_	1168	[32]
CPFs	_	943	[33]

TABLE 2: Comparison of physical and chemical compositions of CRPLSFs with other NF.

Fibre name	Cellulose (wt. %)	Hemicelluloses (wt. %)	Lignin (wt. %)	Wax (wt. %)	Moisture content (%)	Ash (wt. %)	References
CRPLSFs	61.67	14.52	9.15	0.21	2.48	12.12	Current study
Bamboo	26	31	30		9.16	_	[27]
Coir	43	1.7	45	—	11.36	—	[28]
Kenaf	53.14	14.33	8.18	0.8	12.02	2-5	[29]
Coconut	32-43	0.1-0.2	40-45	—	10	—	[30]
Althaeaofficinalis L	44.6	13.5	2.7	_	_	2.3	[31]
Arundo donax	43.2	20.5	17.2	—	—	1.9	[14]
Pergularia tomenstosa L. seed fibre	43.8	16	8.6	1.88	8.5	2.74	[32]
Carica papaya fibres	58.71	11.8	14.26	0.81	9.73	4.7	[33]

4.1. FT-IR. The CRPLSF's FT-IR spectrum in Figure 3 demonstrates the biopolymers' existence in the fibres in the fingerprint area between 3500 and 500 cm^{-1} as of the bands. α - Cellulose is detected as the peak at 3330 cm⁻¹ which is answerable for the O-H stretching [34]. The wellbalanced peak at 2915 cm⁻¹ displays the availability of cellulose in association with hemicelluloses in the fibres that are accountable for C-H stretching together with vibration as of CH and CH2. Organic compounds and lignin are detected from the peaks at 1692 cm^{-1} and 1323 cm^{-1} , which are liable for CH₂ symmetrical and C=C bonding. The peak at 1151 cm⁻¹ creates the aromatic stretching vibration ring inhaling with C-O stretching in polysaccharides. Lignin's C-O stretching vibration and symmetric C-OH stretching are recognized as the peaks at 1027 cm^{-1} . The peak at 446 cm⁻¹ corresponding to the glycosidic acid existing in cellulose, is accountable for C-O stretching [35].

4.2. XRD. CRPLSF's X-ray spectrum model is represented in Figure 4. It is demonstrated that the fibres' diffractogram demonstrates a primary peak accompanied by a secondary peak. The prime peak at 22.25° is established as the uppermost intensity peak that comprises the subsistence of crystalline CC in the CRPLSFs. The secondary peak at 15.27° equates to the unstructured content in the fibres [36]. It is a consequence of the availability of polysaccharides such as lignin, hemicellulose, and pectin appearing in the fibre's cell wall. The cellulose CI was deliberated as 30.27%, which is more than that of coconut fibre (19.9%), and less than that of Cocosnucifera (33.63%),

Juncus effuses (33.4%), and Calotropis gigantea (56.08%). Furthermore, the CRPLSF's crystalline size (CS) was detected as 6.15 nm, decreasing the fibre's water absorption capability and chemical reactivity [37].

4.3. Tensile Properties. The CRPLSF's TS was detected to be 237.23 MPa. The CC's percentage and the CI were primarily offered to the CRPLSF's TS. Nevertheless, the CRPLSF's elongation was appraised at 1.91%.

The result exhibited that the normal load and treated fibre increased the tensile strength. The increased temperature was also observed in between the interfaces due to an increase in tensile load. The contact failure increased in between the neck and holder [38]. The maximum tensile strength in the CRPLSFs indicates that the temperature was increased in between the interface [39].

4.4. Thermal Analysis. The composite's processing temperature is essential to appraise the NF's thermal activities before its utilization as reinforcement in PC. Figure 5 represents the TGA's outcomes along with the differential thermal analysis of CRPLSFs. In Figure 5(a), the TG curve displayed a 3-stage degradation in the CRPLSFs [40]. The 1st degradation occurred at 65°C, and the 2nd stage of degradation occurred at 326°C. The last degradation exists at 547.9°C with a mass loss of 26.25%, which is endorsed by the disappearance of wetness in the fibre. Additionally, the DSG curve in Figure 5(b) specified that the fibres degradation temperature and thermal stability were augmented [41]. The outputs of the thermal analysis displayed that the CRPLSFs



FIGURE 3: FT-IR spectrum of Shwetark fibres.



FIGURE 4: XRD diffractogram of CRPLSFs.

possess the highest degradation temperature and the lowest char residue; consequently, they are appropriate for highertemperature appliances [42].

4.5. SEM Studies of Fibres. It is vital to appraise the bio-fibres surface morphology to verify the fittingness of the fibre to be utilized as a better reinforcement in polymer resin [43]. The SEM micrographs of treated and untreated CRPLSFs are specified in Figure 6(a). The closure view of treated and untreated CRPLSFs is indicated in Figure 6(b). The treated

CRPLSF's SEM image displays the exterior portion of fibre having no breaks along with contaminations in contrast to nontreated CRPLSFs' augmenting exterior unevenness observed in Figure 6(c) [44]. Figure 6(d) shows the closure view of treated CRPLSFs displays that the treated CRPLSFs appear extremely uneven with several slots in conjunction with grooves that augment the resistance to humidity absorption together with enhanced connection when exploited as reinforcement for PC [45].

The alkali treatment and hybridization of CRPLSFs have increased the fibre matrix adhesion and reduced the fibre







FIGURE 6: SEM images of treated and untreated fibre. (a) Untreated fibre. (b) Treated fibre. (c) Closure view of untreated fibre. (d) Closure view of treated fibre.

pullout, resulting in increased properties of the composites [46]. Treated fibre has high tensile properties and a better stress transfer rate compared to CRPLSFs. In hybrid composite systems initially [47, 48], the load will carry by treated fibre, then it will transfer to APLSF without affecting the matrix [49]. The maximum strain rate is achieved by treated fibre [50]. Multiple failures occur in the treated fibre after, which the CRPLSFs will take the load and effectively transfer

the load, resulting in increased properties of the composites [51].

5. Conclusion

The CRPLSFs' physical-chemical composition was correlated with other NFs such as coir, bamboo, kenaf, Althaea officinalis *L*, coconut, Arundo donax, Carica papaya fibres, and Pergularia tomenstosa L. seed fibre. The physical evaluation's outcomes uncovered that CRPLSFs possess a density and a diameter of 1468 kg/m^3 and $387 \mu \text{m}$, correspondingly. Although the fibre possesses a dense structure, the fibre's lightweight feature can be deployed to the highest consequence, which can direct to the replacement of the commercially attainable nonorganic fibres in composite structures. The CRPLSFs achieved higher CC (61.67%) in correlation with other NF, which is completely evident for a better tensile characteristic. The CRPLSFs have a lower wax substance (0.21%), a limitation for a better interfacial connection between reinforcement and matrix in the composites. The XRD outcomes proved that CRPLSFs possess the CI and CS of 30.27% and 6.15 nm, correspondingly. The TGA outcomes displayed that the CRPLSFs can endure up to 326°C with a lesser residual mass of 26.25%. FT-IR outcomes proved the subsistence of several chemical compounds (cellulose, hemicelluloses, and lignin) in conjunction with their corresponding functional classes. SEM provided proof that CRPLSFs possess a closely structured composition and the attainability of contamination on the fibre surface. The fibre's TS was appraised utilizing a single fibre TT and was determined to be 294 ± 1.62 237.23 Mpa, along with its respective elongation at break, which was esteemed as 1.91%. The outcomes proved that the CRPLSFs could be used as NF compounds' reinforcement. The composites will be structured in the future by deploying numerous volume fractions of treated and untreated CRPLSFs and will illustrate their thermomechanical activity.

Data Availability

The data used to support the findings of this study are included within the article. Should further data or information be required, these are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that they have no conflicts of interest regarding the publication of this paper.

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