Physical, chemical and surface morphological characterization of single areca sheath fiber

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Abstract: The technological advance in various streams necessitated the increased demand for lesser weight and minimal cost materials. The concerns to ensure environmental sustainability, those newer materials were expected to be decomposable. In the current study, chemical, physical and mechanical and morphological aspects of areca sheath/frond fiber are investigated. It is observed that, the cellulose, hemicelluloses and lignin wt. % are 65.02, 8.26 and 18.62 respectively. The XRD study confirms the percent crystallinity of 81.33% and the crystallinity index of 0.77. The fiber further exhibited the tensile strength of 59.62±2.20 MPa. The TGA study reveals the higher degradation rate at 322.2 °C and the residual mass at 500 °C is 33.23%. The morphological study is done through scanning electron microscopy (SEM). The other properties such as density (0.9 g/cm³), the fiber length, diameter, and moisture uptake are also reported. From the very promising results, the fiber can be used as a reinforcement agent and the composites may find potential applications in automobile panels, interiors, structural light weight bearing sheets, etc.

1. Introduction:
The propensity to save the environment can be witnessed with great amount of eco-friendly approaches by the human beings. The vivid research activities in the field of natural fiber composites during the recent past few years can be attributed to it [1-3]. The inclination towards the natural fibers as a reinforcement in the polymer composites is because of its intrinsic qualities viz., reduced weight,
readiness to decompose, non-polluting, toxic free, minimal cost, renewability, and good mechanical properties [4-7]. The researchers exploited its inherent qualities in devising the composites out of these fibers and found its suitability for various applications in automobile, aerospace, sport equipment, construction and many other sectors.

The fibers used in the manufacturing of the composites are from the various parts of a plant. The concentration of fibers may be different for different plants and the common sources are bark, stem, fruit, leaf stalk, and frond. The source from where the fibers are extracted will have direct influence on its biochemical features and physical characteristics such as density, diameter of fiber, surface roughness, and thermo-stability [8, 9]. The natural fibers consist of three primary constituents, namely cellulose, hemicelluloses and lignin [10]. The cellulose has a unique geometry with respect to a fiber type. Generally, the cellulose content present in it will have a significant influence on the mechanical properties [11]. The mechanical properties of composites like tensile strength, Young’s modulus, elongation at break are dependent on the cellulose content of the fiber [12]. The fraction of components such as cellulose and lignin will affect the thermo-stability of the fiber and hence its composites [13]. The hemicelluloses, lignin and pectin present in the fiber will act as an adhesive to bind the cellulose structure in the fiber [14]. The features of the fiber that governs its mechanical properties includes its crystal structure, the degree of crystallinity, included spiral angle of fibrils, degree of polymerization, void content, lumen size and the chemical constituents [15]. Other factors such as the form of the fiber (powder, short, or continuous), inclination of the fiber, and fiber fraction will also alter the mechanical properties of a composite, when the fibers are used as reinforcement. The other aspects like the density, aspect ratio, and fibril orientation, will significantly affect the mechanical behavior [16]. For example, the aspect ratio is one of the critical functions which govern the stress transmission. If the diameter of the fiber is less, then the fiber and matrix adhesion will be good in the composites, which in turn help in the effective stress transmission. Water uptake character is also discussed. If the fiber absorbs more moisture due to the hydroxyl groups present in it, the fibers present in the composites will swell and therefore the adhesion with the matrix will fail resulting in poor mechanical properties [17].

Various perennial fibers from giant reed (Arundo donax L.), Cissus quadrangularis, Grewia tiliiifolia, banana, hemp, sisal, etc. and novel natural fibers like Saccharum Bengalense Grass (Sarkanda), Furcraea Foetida leaf fiber, Coccinia grandis L., Sida cordifolia L., etc. are being explored to find its potential in environment friendly applications [18-23]. With a quest to find the potential and its suitability as a reinforcement agent, the areca sheath lignocelluloses fibre is investigated. The other unique reasons concerned to the study on areca sheath fiber are: The fiber is abundantly available at the site of research situated in Mijar, Dakshina Kannada, Karnataka, India; To the best of authors’ knowledge, no similar literature is available on the current study. The study is concerned with physical properties, chemical constituents, thermo-stability, tensile properties, crystalline properties and morphological aspects.

2. Research Methodology
2.1 Materials
The parameters such as the fiber property, the type of polymer used as a matrix material, the strength of adhesion between the fiber and the matrix govern the different properties of the composite materials. The climatic condition and the age of the plant from which the fibers are extracted influence the chemical constituents in it which contribute to the various properties of the fibers [24]. All the fibers have cellulose in common in them as a principal structural component. Most fibers also contain the structural polymers such as hemicellulose and lignin. With these three major constituents the fibers are collectively named as lignocellulosic fibers. The cellulose structure in the fiber comprises of more hydrophilic groups. The conflict in hydrophilic nature of the natural fibers and the hydrophobic nature of the matrix interrupt the interfacial adhesion between them. Therefore, before introducing a fiber into the manufacturing of composites, the characteristics of the fibers are to be studied thoroughly and hence this study. The areca sheath fibers were procured from Go Green products, Chennai. The fiber characterization is done as per the ASTM guidelines.
2.2 Thermal studies
The composites stability associated with the temperatures above the surrounding temperatures are of practical interest [25]. STA 409 PL Luxx thermal analyzer is employed which is operated from room temperature into high temperature of 500 °C with a rate of heating maintained at 10 °C/min. Inert atmosphere of nitrogen is used.

2.3 X-ray diffraction investigation
High modulus fibers are preferred for the composite application. The crystallinity of the fibers influences the moduli of the fibers and hence to achieve the desired mechanical properties on the fiber composites, consideration of higher crystallinity is an essential factor. To study the crystallinity of the areca sheath fiber, the powdered areca sheath fiber specimen is tested under X-ray generator (Bruker D8 Advance), Ni filter and scintillation counter as 40 kV and 50 mA. The radiation source Cu-Kα line (1.54 Å) is used. The intensity of the diffraction is recorded in 2θ angle range (between 3° and 60° at 2θ scale at 1 second step size).

2.4 Morphological study
The areca sheath fibers sputtered with 10 nm gold layer are observed under JEOL JSM-T330A SEM (scanning electron microscope).

2.5 Quantification of chemical constituents:
Following methods were incorporated in the investigation of the chemical constituents present in the areca sheath fiber [26-28].

2.5.1 Quantification of extractives
The organic extractives of air dried areca sheath fibers (5 g) were removed in Soxhlet extraction heater for 5 h, using a mixture of ethanol and toluene. After taking the fibers out from the extractor, they were rinsed with ethanol and hot water. Then it is oven dried at a 60 °C. The extractives were quantified comparing the weight loss and the initial weight in terms of percentage.

2.5.2 Quantification of lignin
Certain properties like the hardness, bleachability, color etc. are connected with the amount of lignin present in the fiber. As lignin is hydrophobic in nature, it reduces the water absorption. The lignin quantification is done with the following procedure. 15.0 mL of 72% sulphuric acid incrementally added while stirring to the beaker containing 2.0 g of extracted areca sheath fiber sample. Sulphuric acid is used to destroy the cellulose and to enable the lignin isolation. To ensure the complete solution, the material is stirred frequently for 2 h at 25±1 °C. Once the specimen is dispersed, 300 mL of distilled water is added. The solution is then boiled for 2 h and allowed to get the insoluble lignin to get settled keeping the flask in an inclined position. After about 24 h, the supernatant solution is siphoned off and the lignin was transferred and washed with hot water to remove the acid residual. The crucible containing the lignin is then dried at 105±3 °C to constant weight. Then it is cooled in desiccator and weighed.

2.5.3 Quantification of holocellulose
Holocellulose is highly hydrophilic in nature and causes in more water absorption. A 3.0 g of extractive-free dry fiber samples are placed in a 250 ml Erlenmeyer flask. Further, 150 ml of distil water is added in to the flask along with the acetone acid 0.5 ml and 1.0 g sodium chlorite. The content in the flask is heated up to 70 °C in a water bath for 1 h, following an additional dosage of 0.5 ml of acetone and 1.0 g of sodium chloride were added. Heating is continued for next 1 h. Repetition of the process is done 4-6 times until all the lignin content is completely removed. Finally, the content in the flask is allowed to cool for about 24 h and the residue is washed using acetone. It is then dried in the hot air oven at 105 °C for 24 h and solid residue left out is weighed.

2.5.4 Quantification of α-Cellulose
Approximately 3 g holocellulose sample is kept in a 250 cc beaker and 35 ml of 17.5% carbonate-free solution of sodium hydroxide (20 °C) is added to it. The solution is allowed to stand for about 5 minutes. The sample is macerated by gently stirring with a glass rod. The sodium hydroxide is intermittently (once in 5-6 min) added in 10 ml portions and a total of 40 ml of sodium hydroxide is added during this interval. Then a 35 ml of distil water is added and the beaker is covered with a watch
glass for an hour. The residue is filtered and successively rinsed with 100 ml of 8.5 % sodium hydroxide, distil water, 10% acetic acid and finally with water. The α –cellulose is then oven dried up to constant weight and allowed get cooled in desiccator and then weighed.

2.5.5 Quantification of hemicellulose

The difference between the fractions of holocellulose and the α-cellulose is the fraction of hemicellulose.

3. Results and Discussions

3.1 Chemical constituents of areca sheath fiber

The experimental results of the major constituents of areca sheath fibers are in agreement with various other studies reported. The experimental values of cellulose, hemicelluloses and lignin are observed to be in wt.% of 65.02, 8.26 and 18.62 respectively. The result is very much compromising with other studies [29, 30]. The cellulose content is very much closer to the Jute (61-71 wt.%), Hemp (68 wt.%), Ramie (68-76 wt.%), Abaca (53-63 wt.%), and Sisal (65 wt.%)[31].

3.2 Fiber length

Fiber length in a composite is very significant parameter which imparts certain properties to it. Generally, the parameters pertaining to a fiber such as the higher crystallinity, higher cellulose content, higher aspect ratio and lower micro-febrile angle will cause in enhanced mechanical properties of the composites [32]. The fiber length is performed with a hundred areca sheath fibers. The fibers were extended and the length is measured between the ends, ensuring no stretch outs incurred. The lengths of the fibers are ranging between 800 mm and 1400 mm.

3.3 Determination of linear density and the diameter of the fiber

The measurement of fineness, linear density of the fiber is the amount of mass per unit length and determined in line with ASTM-D1577–01. A single fiber which is long enough is cut in to small pieces of known length and it is then weighed. By measuring the weight (0.2352 g) and length of specimen fibers (110 cm), linear density is calculated with the following Equation 1 and found to be 0.2138 g/m (213.8 Tex) [33].

\[
\text{Linear Density (in Tex)} = \frac{\text{Weight of fiber (in gram)}}{\text{Length of fiber (m)}} \times 1000
\]  

(1)

The optical microscope (LEICA DM750M) is used to determine the fiber diameter. The average of 10 known length single fibers and average of 100 fiber diameters are used to determine the volume density (\(\rho_f\)) with the Equation 2 [34]. With the lengths varying from 80 cm to 140 cm and diameter varying between 400 μm to 700 μm the fiber density of areca sheath fiber is estimated to 0.90 g/cm³.

\[
\text{Volumetric Density (}\rho_f\text{)} = \frac{M}{L\left(\frac{\pi d^2}{4}\right)} \text{g/cm}^3
\]  

(2)

Where, \(M\) is the weight of the fiber (g), \(L\) is the length of the fiber (cm) and \(d\) is the diameter of the fiber (cm).

The density of areca sheath fiber is very closer to Hemp (1.1-1.6 g/cm³), Kenaf (0.6-1.5 g/cm³), Bamboo (1.2-1.5 g/cm³), and Coir (1.15-1.6 g/cm³) [35].

3.4 Thermal analysis

The areca sheath lignocellulosic fiber is subjected to various techniques of thermal analysis and the physical property and its reaction products are determined as a function of temperature. Consequent change in mass versus the temperature (TGA curve) will provide facts on its thermal stability and/or composition of the specimen/ intermediate compound and of any residue left out. The DTG curve depicts the decomposition reaction rate for the weight losses over time. The analytical technique DTA
which provides supplementary information such as transformations occurred during the heating program such as glass transition, crystallization, melting and sublimation is included. TGA and DTA curves pertaining to the thermal degradation of areca sheath fiber is shown in figure 1. The DTG curve of raw areca fibers is shown in figure 2.

![TGA and DTA curves](image1)

**Figure 1.** TGA and DTA curves.

![DTG analysis](image2)

**Figure 2.** DTG analysis.

The analysis of thermal stability of the areca sheath fiber through TGA, DTA and DTG is presented in the table 1, 2, and 3 respectively. The thermal stability of fiber was analyzed by using TGA plots and the outcomes are tabulated in table 2 and table 3. The thermal stability of fibers is complemented by the DTG curves and are tabulated in Table 4.

| 1<sup>st</sup> level of degradation | Weight loss (%) | 2<sup>nd</sup> level of degradation | Weight loss (%) |
|----------------------------------|-----------------|-----------------------------------|-----------------|
| Temperature range 1(°C)          |                 | Temperature range 2(°C)          |                 |
| 241.5 to 297.0                   | 17.13           | 297.0 to 353.0                    | 29.54           |
Table 2. Analysis of decomposition temperature and weight loss percentage of Areca sheath fibers.

| Decomposition temperature, °C | Mass change at, % | Residual mass at 500 °C (%) |
|-------------------------------|------------------|---------------------------|
| 25% mass change | 50% mass change | 150 °C | 300 °C | 500 °C | 33.23 |
| 289.5 | 328.5 | 8.04 | 21.39 | 37.32 |

Table 3. DTG results of Areca fibers.

| Decomposition temperature, °C | Peak 1 | Peak 2 |
|-------------------------------|--------|--------|
| 84.8 | 322.2 |

As reported by other similar studies on natural fibers [36, 37, 13], areca sheath fibers also exhibited the decomposition of its contents at major two levels. The organic extractives and the moisture got vapourised completely at 241.5 °C and the early weight loss of 10.96% can be witnessed from the TGA curve. From this temperature onwards the areca sheath fiber started losing its thermal stability. In the first level of content decomposition between the temperature range 241.5 °C and 297.0 °C, due to the depolymerization of hemicellulose and pectin through hydrous pyrolysis, dissociation of links of cellulose and lignin, 17.13% of weight loss is observed. In the second level of decomposition between the temperature range 297 °C and 353 °C, due to the decomposing of α-cellulose as well as lignin, weight loss of 29.54% can be noticed [36].

The very complex structure of lignin with various branches of aromatic rings results in its gradual degradation starting from 241.5 °C till the end. Similar trend of lignin degradation can be observed in the curve from 353.0 °C to 500 °C resulting in a 9.16% weight loss. The overall weight loss due to the decomposition of 25% is recorded at 289.5 °C and 50% at 328.5 °C and is presented in Table 3. The weight losses of 8.04%, 21.39% and 37.32% are observed at 150 °C, 300 °C and 500 °C respectively.

At 499.4 °C, the final residual weight percentage of areca sheath untreated fiber is 33.23% and contain carbonaceous left outs and un-degraded fillers if any [38].

The DTG curve presented in Figure 2 complements the aforesaid results. The decomposition rate corresponding to the weight loss experienced by the specimen is depicted by DTG curve. The first peak is observed at 84.8 °C and the latter peak at 322.2 °C. The first peak is correlated to the evaporation of the organic extractives and the moisture content present in the untreated areca sheath fiber. Similarly, the latter peak can be correlated to the degradation of cellulose content. The second bigger peak indicates the higher degradation rate of the content took place at 322.2 °C and hence the change in the weight loss.

3.5 XRD analysis

The structural information of the specimen under test is studied by X-ray powder diffraction method. X-ray diffraction is an excellent tool to notice the difference between amorphous and crystalline material present in the natural fiber. Most common general diffraction method employs the diffraction of monochromatic X-rays from a powder specimen under investigation and the intensity of the scattered rays yield the great deal on structural information. The examinations were done by the diffractometer with the intensity of Cu-Kα radiation wavelength of 0.154 nm. The percentage crystallinity and crystallinity index (CI) of the fiber was determined by using following equations [39].

\[
\text{%Cr.} = \left( \frac{I_{\text{crystalline}}}{I_{\text{crystalline}} + I_{\text{amorphous}}} \right) \times 100 \tag{3}
\]

\[
\text{C.I.} = \frac{(I_{\text{crystalline}} - I_{\text{amorphous}})}{I_{\text{crystalline}}} \tag{4}
\]
Figure 3. XRD spectrum.

The XRD pattern of the areca sheath is displayed in Fig. 3. The two enlarged diffraction peaks at 18° and 22° are clearly seen in most of the bio-fibers. The peak at 20 = 18° point out the existence of amorphous constituents in fiber. The peak at 2θ = 22° denotes the crystalline content present in the cellulose. The percentage crystallinity and crystallinity index of areca sheath was calculated as 81.33 and 0.77. The crystallinity index 0.77 is higher than that of other bio fibers, such as C. grandis (0.52), F. foetida (0.53), Saharan Aloevera (0.57), S. cordifolia (0.57), C. pangorei (0.41), Acacia leucophloea (0.51), Acacia Arabica (0.52) and almost equal to that of Jute (0.71) and Hemp (0.88). The good crystallinity index indicates the lower amount of amorphous materials like hemicellulose, lignin, and some other non-cellulosic material in the areca sheath fiber.

3.6 Tensile properties of areca sheath single fiber

The areca sheath fiber of 25mm length is investigated for its tensile properties on digital universal testing machine (Mecmesin 2.5 Xt) at a cross head speed of 2 mm/min. The test is done in accordance to ASTM D3822-07 standards. Average of 20 specimens under test is reported. Following results are observed: Tensile strength: 59.62±2.20 MPa; Young’s modulus: 1.24 ± 0.12 GPa and the Elongation at break: 3.02 ± 0.36%.

3.7 SEM image analysis

SEM micrographs display a general view of cellulosic fibers with a rough surface suitable for a good bond with matrix polymer. The areca sheath fiber under SEM is shown in the figure 4. The fibrils are exhibiting a network structure and interlinked by the hemicellulose and lignin. The fibrils are parallel to each other and oriented in the direction of the fiber. The adhesives, lignin fatty substances are populated in the intercellular spaces and attribute to the firm bonding of the fibrils. Further, it can be clearly seen in the image, there is no cracks, micro voids and helical fibrils on the surface of fiber. So it is revealed that these fibers can be used as reinforcement in polymer composite manufacturing.
3.8 Water uptake study
The hydrophilic character of areca sheath fiber is studied using desiccators with distilled water. The hot air oven at 60 °C is used to dry the fiber specimens prior to the investigation. It is then weighed using a precise 4-digit balance and placed in the distilled water set up in the humidity chamber. The specimens were taken out after 24 h and wiped with the filter paper and weighed immediately after wiping with a filter paper. The difference in weight is quantified in terms of water absorption by the specimen. The %water uptake is calculated using the following equation 3 and resulted to 60%.

\[
\text{% of Water absorption} = \frac{\text{Final weight} - \text{Initial weight}}{\text{Initial weight}} \times 100
\] (5)

4. Conclusion
The properties of the fiber studied such as density, length and diameter, chemical composition, crystallinity, tensile strength, surface characteristics, thermo-stability and moisture uptake will serve as catalyst in further studies on fiber composites. The density of 0.9 g/cm³ will show its potential use in light weight load bearing applications. The crystallinity index is 0.77 and the cellulose content of 65.02% is very promising which imparts higher strength to the fiber. The lower lignin content of 8.26 wt.% is contributing the increased tensile strength. The surface study shows smoother surface indicating lower microfibrillar angle which will also supplement to the strength of the fiber. Thermal stability of 322.2 °C and the moisture uptake behaviour (60%) and above mentioned qualities will qualify the fiber as a reinforcement agent in the manufacturing of environment friendly composite materials. The composites may find applications in light weight load bearing applications, automobile panels or interiors, packaging sheets, seat rear supports, etc.

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