



Extraction and Characterization of New Cellulose Fiber from the Agro-waste of *Lagenaria Siceraria* (Bottle Guard) Plant

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ABSTRACT

This article explores the extraction and characterization of natural fiber from the agro-waste of *Lagenaria siceraria* (LS) plant stem (commonly known as 'bottle guard') for the first time. The extracted fiber from the waste stems has high cellulose content (79.91 %) with good tensile strength (257–717 MPa) and thermal stability (withstand up to 339.1°C). The immense percentage of crystalline index (92.4%) with the crystalline size (7.2 nm) as well as low density (1.216 g/cm³) of the LS fiber renders their possibility to use as an effective reinforcement material in lightweight eco-friendly composites for various industrial applications.

Indexing terms/Keywords

Natural fiber; *Lagenaria siceraria*; TGA analysis; FTIR; XRD; Crystalline index

Academic Discipline And Sub-Disciplines

Mechanical Engineering, Chemistry, Composites

SUBJECT CLASSIFICATION

Natural fiber composites

TYPE (METHOD/APPROACH)

Analysis and Characterization

1. INTRODUCTION

The global economy is increasingly dependent on 'go green' concepts in industrial and technological sectors. Of late, the world community is highly apprehensive of the depletion of fossil fuels and is concerned about the carbon footprints. Our developmental life style is left behind and is hard pressed for going for alternative sustainable materials. In every nook and cranny of the world, Research and Development is in full swing to chance upon bio-based substances and environmental friendly sustainable materials for industry producers and consumers^[1]. In this context, a variety of natural fibers such as sisal, flax, jute, coconut and banana are being effectively utilized as reinforcement phase in composite materials since it has a host of advantages over its synthetic counterparts such as low-priced, easily separated, and are abundantly available.

Lagenaria siceraria (Bottle Guard) originates from southern Africa and commonly cultivated in tropical and subtropical areas of the world. It is a biennial/annual crop which can be harvested young and used as a vegetable. After harvesting the vegetables of the plant, the stem of the plants is remained as agro-waste. Extensive research is being carried out at the LS plant to explore the various pharmacological activities such as anti-diabetic, cardio-protective, hepato-protective so on, and so forth besides been used in the green synthesis of nano particles for various industrial applications^[2-6].

The present study is aimed at a better understanding of physical-chemical, thermal, and mechanical properties of *Lagenaria siceraria* fibers (LSFs) extracted from their agro-waste stem by the comprehensive characterization for the first time.

2. MATERIALS AND METHODS

2.1 *Lagenaria siceraria* fiber and its extraction

The *Lagenaria siceraria* (LS) plants were collected from the fields of the plantation after harvesting the vegetable in Therukkuppallam village, Erode District, Tamil Nadu, India. The agro waste stems of LS plant were separated by removing

their leaves and tendrils using knife and washing it with water to remove the impurities and dirt. The fibers can be extracted from the skin of the collected stems of water-retting process [7]. The long and continuous stems were cut into pieces at the joints having an approximate length of 10-20 cm each and kept immersed in water for fourteen days to facilitate microbial degradation. Then, the fibers were separated from the stem piths and washed thoroughly with distilled water to remove their impurities. The obtained fibers were sun-dried for 72 h to remove the moisture. The LSFs plant and extracted fiber from LSFs is shown in Fig 1.



Fig 1: *Lagenaria siceraria* plant (Left) and Extracted LS stem fiber (Right)

2.2 Characterization of the PFFs

2.2.1 Determination of chemical composition of LSFs

By opting standard analytical procedures, the chemical compositions of LSFs such as cellulose, hemi-cellulose, lignin, and ash content were determined [8]. The Mettler Toledo XS 205 balance method was adopted to estimate the density of the LSFs [9]. Conrad procedure was followed in the determination of the wax content of LSFs [10]. According to the ASTM E1755-01 standard, the ash content of LSFs was determined [11] and the moisture content was determined by standard method described elsewhere [12].

2.2.2 Fourier Transform-Infrared Spectroscopy

The existences of various major functional groups present in the LSFs were ascertained by using an FTIR spectrometer (Perkin Elmer RXI). The LSFs powder was made into a pellet by mixing with Potassium bromide (KBr) followed by scanning in the wavelength range of 500 to 4000 cm^{-1} with 30 scans per minute. The resultant peaks were recorded.

2.2.3 X-ray Diffraction assay

The X-Ray Diffraction (XRD) assay was performed to assess the Crystalline Index (CI) and crystal size of the extracted LSFs. The assay was carried out by using XPERT-3 diffractometer fitted with $\text{CuK}\alpha$ radiation (wavelength 1.5406 Å nm) source at a current of 30 mA, with an application of 45kV tension. The sample was examined at a temperature of 25° C in the 2θ ranges between 10° and 80°. The value of the peak intensities of the LSFs was used in the calculation of crystalline index, CI (%) with the help of Segal's equation (1) [13]

$$CI(\%) = \left[\frac{I_{002} - I_{AM}}{I_{002}} \right] \times 100 \quad (1)$$

Where I_{002} is the maximum intensity of crystalline peak and I_{AM} the intensity of amorphous peak. By applying Scherrer's formula as shown below (Equation 2), the average crystal size (CS) of the LSFs was determined. [14]

$$D = \frac{K\lambda}{\beta \cos \theta} \quad (2)$$

Where D is the crystal size, K the Scherrer's constant ($K = 0.94$), λ the wavelength of the $K\alpha$ radiation (1.5406 Å), β the full peak width at half of the maximum, and θ the Bragg's diffraction angle.

2.2.4 Thermo gravimetric Analysis (TGA)

A thermo gravimetric analyzer (Model STA 449 F3, NETZSCH, Germany) was used to examine the thermal stability of the LSFs. The 10mg of the powdered LSFs sample was taken in an alumina crucible and heated continuously at a rate of 10°C min^{-1} from 30 to 550°C in a nitrogen atmosphere at a flow rate of 20 ml/min.

2.2.5 Single fiber tensile testing

INSTRON 5500 universal testing machine was used in the examination of tensile properties of the LSFs at a crosshead speed rate of 10 mm min^{-1} . A gauge length of 100mm was maintained with a load cell capacity of 1 kN. As per the ASTM D3822/D3822 M-14 standards, 20 samples were analyzed for the study with 65% of relative humidity at 21°C [15].

2.2.6 Morphological analysis

The surface morphology of the LSFs was studied with the help of scanning electron microscope (JEOL JSM-6390) by applying high energy beam of electrons with an accelerating voltage of 10 kV and an attainable vacuum level of 1.5×10^{-3} Pa. The images were recorded at different magnifications.

3. RESULTS AND DISCUSSION

3.1 Chemical composition of the LSFs

The major components of lignocellulosic fibers are cellulose, lignin, hemicellulose, pectin, wax, and ash. The percentage of each component varies for different type of fiber. The amount of these components in a fiber depends on the geographical area, climate, age, source of the fiber and methods of extraction. The quantity of the fiber constituents directly influences the physicochemical properties of the fiber [16]. The chemical composition and density of LSFs were compared with a few natural fibers and are shown in Table.1.

Table 1. Chemical compositions of the raw LSFs compared with the other common fibers.

| Fiber | Cellulose (%) | Hemicellulose (%) | Lignin (%) | Wax (%) | Moisture (%) | Ash (%) | Density (g/cm ³) |
|--------------------|---------------|-------------------|------------|---------|--------------|---------|------------------------------|
| LSFs | 79.91 | 12.69 | 7.62 | 0.31 | 9.96 | 0.92 | 1.216 |
| Jute [17] | 64.4 | 12 | 11.8 | 0.7 | 1.1 | - | 1.3 |
| Flax [17] | 64.1 | 16.7 | 2.0 | 1.5–3.3 | 3.9 | - | 1.5 |
| Hemp [17] | 68 | 15 | 10 | 0.8 | - | - | 1.47 |
| Kenaf [17] | 31–72 | 20.3–21.5 | 8–19 | - | - | - | 1.45 |
| Ramie [17] | 68.6–85 | 13–16.7 | 0.5–0.7 | 0.3 | 7.5–17 | - | 1.5 |
| P.Foetida [17] | 77.96 | 12.63 | 10.47 | 0.35 | 9.54 | 0.94 | 1.328 |
| A.leucophloea [20] | 68.09 | 13.60 | 17.73 | 0.55 | 8.83 | - | 1.385 |
| I. staphylina [25] | 72.76 | 13.6 | 19.56 | 1.51 | 8.28 | 1.4 | 1.40 |

The LSFs have significantly high cellulose content 79.91 wt %, to withstand the hydrostatic pressure gradient of the fiber [17]. The LSFs have a considerable amount of lignin 7.62 wt %, which wind α - cellulose micro fibrils, into a bundle of cellulosic fiber and as well serves as a chemical adhesive between cellulose and hemicelluloses and ensure fiber rigidity and thermal stability. The high percentage of hemicellulose 12.69 wt % of content in LSFs strongly influences the moisture absorption, bio and thermal degradation of the fiber. The wax content 0.31 wt % LSFs may render poor interfacial binding between fibers and polymer matrices while preparing the composites [18]. The LSFs consists of moisture 9.96 wt % and ash 0.92 wt %. Furthermore, the density of the LSFs was measured and found to be 1.216 g/cm³, which render it suitable for lightweight composite materials.

3.2 FT-IR Analysis of LSFs

The FT- IR spectrum of LSFs is shown in Fig 2.

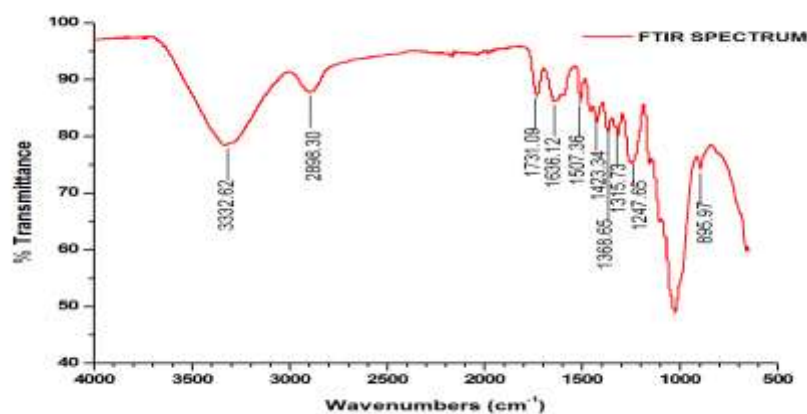


Fig 2: FTIR spectrum of the LSFs

The absorption peaks at 3332, 2898, 1731, 1636, 1507, 1423, 1368, 1315, 1247, 1155, 1058 and 896 cm^{-1} revealed the presence of three major components of LSFs such as cellulose, hemi-cellulose, and lignin. The absorption peaks at 3332, 2898, 1423, 1315, 1155, 1058 and 896 cm^{-1} were due to free and hydrogen bonded O – H stretching, asymmetric stretching of the methylene group ($> \text{CH}_2$), $-\text{CH}_2$ scissoring, $-\text{OH}$ bending, C – O antisymmetric bridge stretching, C – O – C stretching and C – O stretching vibrations of α - cellulose component of LSFs [19-20]. The absorption peaks observed at 1731 and 1247 cm^{-1} were due to C=O stretching and C – O – C stretching vibrations of acetyl group of hemi-cellulose component of LSFs [21-22]. The absorption peaks that appeared in 1636, 1507, 1423, 1368, and 1058 cm^{-1} were due to C=C stretching, C – O stretching, $-\text{CH}_3$ asymmetric stretching, $-\text{CH}$ symmetric stretching, and aromatic plane deformation of lignin components of LSFs [23]. The peak observed at 896 cm^{-1} corresponds to the presence of β (1-4) - glycosidic linkages between the monosaccharide in cellulose and hemi-cellulose [24-25].

3.3 XRD Analysis of LSFs

The amount of cellulose and non-cellulosic content of fibers determines its crystallinity. The X-ray diffractograms of LSFs is shown in Figure 3.

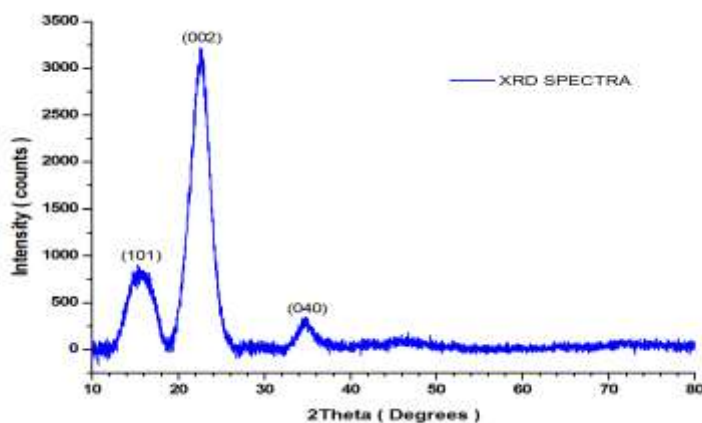


Fig 3: XRD spectrum of LSFs

The XRD clearly indicates three well-defined peaks at $2\theta = 22.68^\circ$ (002 plane), $2\theta = 17.28^\circ$ (101 plane), $2\theta = 34.66^\circ$ (040 plane), which correspond to crystalline and amorphous peaks of the cellulose of the natural fiber.

The crystalline index (CI) of LSFs 92.4% is calculated from equation (1), which is abnormally higher than that of the other reported natural fibers such as hemp (80%), jute (65.8%) and flax (70%), cotton (68%), mending grass (58.6%), sisal (55%), and Wheat straw (45.57%). The average crystal size of LSFs calculated by using Scherrer's formula (equation 2) is found to be 7.2 nm lower than that of jute (29.25 nm), sisal (16.99 nm), mending grass (14.3 nm) but higher than that of ramie (6.6 nm), wheat straw (6.4 nm) and cotton (5-7 nm) [8, 26-28].

3.4 Thermo gravimetric analysis of LSFs

The thermal stability of LSFs examined by TG and DTG curves is shown in Fig 4.

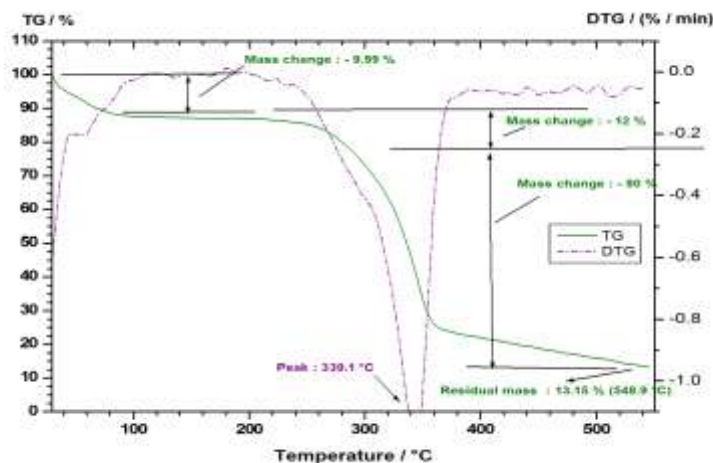


Fig 4: TG/DTG spectrum of LSFs

The initial minor degradation of the fiber noticed at 88.4°C with a weight loss of 9.9% due to the evaporation of the moisture content present in the fiber [29]. The second major degradation observed in the temperature range between 240° and 400°C with a weight loss of 12 % is due to the thermal depolymerisation of hemicellulose [30]. A very prominent peak at 339.1°C with a major weight loss of 80% is due to the degradation of cellulose. The results were significantly comparable with that of the other natural fibers such as bamboo, hemp, jute, and kenaf fibers were observed at 321°, 308.2°, 298.2°, and 309.2°C respectively [31]. Later, there is a minor degradation after 400°C with a weight loss of 12 %, which is due to the decomposition of lignin and wax content of the fiber [32].

3.5 Tensile properties of LSFs

During the single-fiber tensile testing, the tensile properties such as strength, modulus, and elongation at the break of the LSFs were found to be 257–717 MPa, 7–42 GPa, and 1.38–4.67% significantly comparable with that of the other natural fibers such as flax (345–1500 MPa, 27.6 GPa, and 2.7–3.2%), hemp (690 MPa, 70 GPa, and 1.6%), jute (393–773 MPa, 26.5 GPa, and 1.5–1.8%), and kenaf (930 MPa, 53 GPa, and 1.6%) [33]. The higher cellulose content of the lignocellulosic LSFs with a lower micro fibril angle significantly influences the tensile strength of the LSFs.

The micro fibril angle (α) of the LSFs was calculated using Equation (3), which is as follows: [21]

$$\varepsilon = \ln \left(1 + \frac{\Delta L}{L_0} \right) = -\ln(\cos \alpha) \quad (3)$$

Where ε is the global deformation (or) strain (mm/mm), α the micro fibril angle (deg.), L_0 the gauge length (mm), and L the elongation at break (mm). The calculated micro fibril angle (α) of the LSFs was in the range of 8.52 – 25°.

3.6 Surface morphological analysis of LSFs

The scanning electron microscopic image of LSFs is shown in the Fig 5.

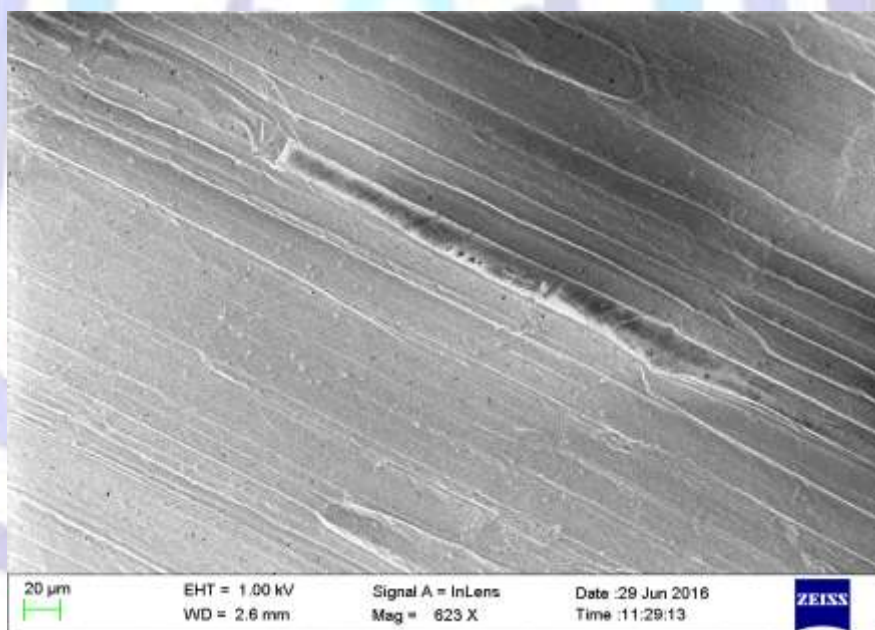


Fig 5: SEM image of LSFs

The surface morphology of the LSFs consists of several fiber cells covered with non-cellulosic components, wax and other impurities over the entire length of the fiber. It has a clean and smooth surface and appears as a thick layer that facilitates both mechanical properties and binding efficiency with matrix materials [34].

4. CONCLUSIONS

Owing to the increasing significance of the use of natural fibers in the field of biodegradable composite materials, the agro waste of *Lagenaria siceraria* stem was chosen for the study of extraction, characterization, mechanical and thermal behavior of the fiber. The fiber from the waste stem has high cellulose content (79.91%) with an abnormal high crystalline index (92.4%) and crystalline size (7.2 nm) playing an imperative role in the tensile strength (257-717 MPa) of the fiber, which improves the mechanical properties of the composite materials. The high thermal stability (withstand up to 339.1°C) renders the fiber to be processed with various thermo polymer resins. The light density (1.216 g/cm³) of the lignocellulosic LSFs from the agro waste renders the possibility of using it as an alternative reinforcement for lightweight eco-friendly composite materials for various industrial applications.



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