

**Characterization of New Natural Fiber Extracted from *Abelmoschus
esculentus* stem****S. Yamuna Devi and Dr. S. Grace Annapoorani***Ph. D Research Scholar and Associate Professor
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Abstract: Developing an eco-friendly composites in structural and automotive applications is more essential in natural materials. The aim of this work is to characteristic analysis of new natural unconventional fiber extracted from *Abelmoschus esculentus* stem (AESF). AES fibers show cellulose content of 67 wt% with a range of tensile strength from 2020 to 5820 Mpa and elongation of 0.62%. AES fibers has crystallinity index of 62% which is compared with other unconventional fibers. Furthermore, the degradation of cellulose up to 375°C resist in AES fiber.

Keywords: Unconventional fibers, FTIR, SEM, XRD and Thermal gravimetric analysis.

INTRODUCTION

In recent years, increasing warming and environmental hazardless, most of researchers are concentrating on eco-friendly materials to develop the environmental quality of products. ^[1, 2] Natural fibers are sustainable and biodegradable materials, which are extracted from bast of trees, stem, leaves, and roots of plants. Some of agro waste fibers are Roselle, sun hemp, okra, *Prosopis juliflora*, Banana. ^[3] The agro waste fiber reinforcement green composites materials are utilized in applications like automobile, aerospace, marine, civil structures, sports equipment's and industrial applications due to natural fiber has biodegradable, rarity, low health hazards, high specific strength, excellent thermal properties, and are economical and renewable. However, accelerating the substitution of man-made fibers by natural fiber requires the large availability of such fibers and current production level does not meet today's demand. New plants should be found that modify easily and low cost extraction methods that do not impair the properties of the fiber.

Most of the researchers investigated the physico-chemical, mechanical, and thermal properties of natural new cellulosic fiber-like *P. juliflora* bark fiber, *Cissus quadrangularis* root fiber, *Phoenix reclinata*, snake grass fiber, *Acacia leucophloea* bark, and so on, using chemical analysis, single fiber tensile testing, Fourier-transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), and thermo-gravimetric analysis (TGA) and SEM analysis. The newly identified *Abelmoschus esculentus* stem fiber extracted from *Abelmoschus esculentus* plant stem has not been investigated from the viewpoint of the physico-chemical, mechanical, and thermal properties till now. The *Abelmoschus esculentus* plant which belongs to the family *malvaceae*, native to tropical and subtropical regions and sometimes cultivated.

Concerning the above, this investigation deals with the extraction of new natural fibers as *Abelmoschus esculentus* (AES) fiber from the stem of *Abelmoschus esculentus* plant and analysis of the physico-chemical, mechanical, and thermal properties of AESF using XRD method, FT-IR spectroscopy, TGA, and single fiber tensile test in comparison with other natural fiber.

Methodology:**2.1 Materials:**

Abelmoschus esculentus plant were collected from Erode district, Tamil Nadu, India. *Abelmoschus esculentus* is the only vegetable crops significance in the *malvaceae* family and is very popular in the indo-pak sub continents. A native of the plant is South America, North America, Africa, India and Eastern mediterranean area ^[4]. It is a tropical to subtropical crop and is sensitive to frost, low temperature, water logging and drought condition ^[5].

2.2 Extraction of *Abelmoschus esculentus* stem fiber:

The mechanical process was adopted for extraction of fiber from *Abelmoschus esculentus* stem fiber ^[6]. The fiber were separated by using traditional combing process done with a metal brush to remove fleshy surface layer of the stem and long uniform fiber were obtained ^[7].

2.3 Characterization of AESF

2.3.1 Chemical Analysis:

(A) Solvent Analysis: The chemical compositions such as cellulose, hemicellulose, and lignin content of the AESF were determined at KCT (Kumaraguru College of Technology, Coimbatore, India) laboratory by the standard test procedures^[8]. The AESF of Cellulose content was determined according to Kushner and Hoffer's method^[9]. The AESF content of the lignin was measured according to the klason method^[10]. The AESF, ash content was measured as per ASTM E1755 – 01 standard^[11]. The holo cellulose of AESF was determined according to the method described by wise et.al^[12]. The cellulose content and holo-cellulose content difference is measured to identify the hemi cellulose content. The cellulose, lignin, hemi cellulose, holo-cellulose and ash content were found in percentage. The average of five samples with standard deviation value has been reported.

(B) FTIR:

The free functional groups present in the AESF and their unique chemical bonds were identified by TENOXO 27 using an infrared spectrometer in a PR Mode. The fiber sample was analyzed with a scan rate of 32 scans per minute at a resolution of 2 cm^{-1} in the wave number region of $500\text{--}4000\text{ cm}^{-1}$ ^[13].

(C) SINGLE FIBER TENSILE TEST:

The tensile properties of single AESF were determined using a Universal Tensile Tester with the aid of 1 kN load cell in accordance with ASTM D 3822 standards. To ensure the accuracy of the results, tested a minimum of 20 single AESF at a 50-mm gauge length with a crosshead speed of 10 mm/min ^[14]. The entire test was carried out at an ambient temperature of $21\text{ }^{\circ}\text{C}$ and the relative humidity was maintained at 65% ^[15].

2.4 MORPHOLOGICAL STUDIES

2.4.1 SEM ANALYSIS

The surface morphology of AESF was examined using a scanning electron microscope (SEM) FEI Quanta 200. The fiber sample was coated with a thin gold layer to make its surfaces conductive and to avoid electron charge gathering. The SEM studies were conducted by scanning the fiber samples with a high-energy electron beam at an accelerating voltage of 25 kV in a vacuum level of $1.5 \times 10^{-3}\text{ pa}$. Then the sample surface was observed at different magnification and the resulting image were capture^[16].

2.4.2 X-RAY DIFFRACTION SPECTROSCOPY

The crystallinity index (CI) and crystallite size of AESF were studied by X-ray diffraction (XRD) spectroscopy (Shimadzu) with monochromatic Cu K α radiation of 0.154 nm wavelength at a current of 30 Ma with an accelerating voltage of 40 kV. The analysis was carried out in the 2θ ranges from 10° to 90° at a scanning speed of 10° per min in order to obtain an acceptable diffraction pattern.^[17]

2.5 Thermogravimetric Analysis (TGA) of AESF

The thermal stability of AESF was determined from the thermograph obtained by heating the powder samples (10 mg) in a thermal analyzer (Model NETZSCH STA449F3) at a rate of $30\text{ }^{\circ}\text{C/min}$ and its ranges from RT to $1000\text{ }^{\circ}\text{C}$. The experiment was carried out in the nitrogen atmosphere with a flow rate of 20 mL/min and the samples were kept in an alumina crucible to avoid the temperature variations measured by the thermocouple^[18].

2.6 Physical Properties of AESF

2.6.1 Fiber Length

The *Abelmoschus esculentus* stem Fiber is analyzed for its length manually using a calibrated metal scale. The fiber was stretched on the flat table and straighten with care to avoid elongation while measuring. The results are expressed in centimeters.

2.6.2 Fiber Diameter

The *Abelmoschus esculentus* stem Fiber diameter is analyzed using a Scanning Electron Microscope (SEM). The average value can be calculated by analyzing the ten different areas of an individual fiber.

2.6.3 Moisture Regain and Moisture Content

The moisture regain and moisture content of the *Abelmoschus esculentus* stem Fiber is analyzed manually using BIS and ASTM D 629 methods. The predetermined amount of fiber (A) is conditioned in oven at 1050 C and the constant mass of the fiber is obtained (B). Thus moisture properties are calculated from the measured values using (1) for moisture regain and (2) for moisture content.

Moisture regain = $A - B / B \times 100$ (1)

Moisture content = $A - B / A \times 100$ (2)

2.6.4 Fiber Fineness

The *Abelmoschus esculentus* stem Fiber fineness is analyzed according to ASTM D 1577 test method. The fibers of selected length (2 inches) were cut and bundled to the nearest weight of 0.001 mg and the number of fibers in the bundle were counted. Randomly twenty bundles are selected for testing and the average was calculated.

3. RESULT AND DISCUSSION:

3.1 FTIR Analysis:

The summarized functional group of the AESF spectrum as illustrated in Fig 2 has were numbers from 4000 to 500 cm^{-1} . The unique features of the AESF spectrum are due to its contents such as cellulose, hemicellulose and lignin^[15]. FTIR Analysis spectra show 12 well define peaks of AESF at 4629, 4486, 4467, 4281, 4206, 3462, 3363, 3107, 2970, 677, 536 and 520 cm^{-1} . One of the most noticeable peaks in the AESF spectrum appeared at 3462 cm^{-1} corresponding to O-H stretching and O-H bending frequencies, presence of cellulose respectively^[20]. A broad absorption band at 3363 cm^{-1} is due to O-H stretching vibrations of cellulose and hemicelluloses^[21]. The peak at 3107 cm^{-1} indicates the presence of cellulose is absorption at C-H stretch of aromatics.

The peak at 2970 cm^{-1} in the AES fiber indicates the presence of C-H stretching of cellulose^[22].

A peak found in 677 cm^{-1} is C-OH out-of-plane bending denote the presence of cellulose^[23]. The peak at 536 cm^{-1} indicates the C-X stretching of organic halogen compounds^[24] and a band region at 520 cm^{-1} were observed because of out of plane bonding of OH^[25].

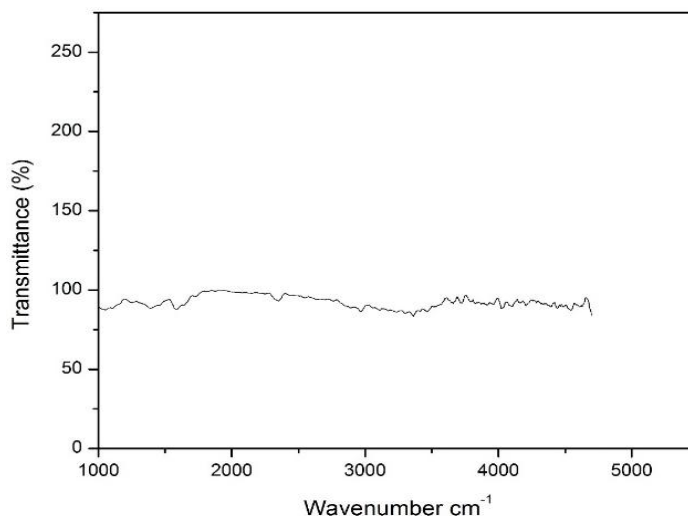


Fig 2: FTIR analysis of AESF

3.2 CHEMICAL COMPOSITION OF AESF

The chemical composition of AESF were summarized and compared with those of other common agro waste fibers in Table 1 [13, 27, 28]. The variance existing between the chemical compositions of unconventional fibers is because of age and part of the plant where the fiber was extracted, extraction procedure, and soil and weather conditions^[26]. The rich content of cellulose (67wt%) tends to improve the mechanical properties of the AESF whereas the low hemicellulose content (15.4wt%) tends to reduce the moisture absorption capacity and in turn increase the thermal stability of the fiber. The presence of lignin content (7.1wt%) acts as a bonding agent between the cell wall structures to improve the rigidity and

strength of the fiber. Less amount of wax content (3.9wt%) in the fiber is desirable because it reduces the bonding characteristics between the fiber and polymer matrix in composites. The ash and moisture contents of the AESF were found to be 2.44wt%, respectively. The AESF have low density (1.365 g/cm^3) as compared to the E-glass (2.5 g/cm^3) and carbon fibers (1.7 g/cm^3), which is favorable for making lightweight composite components ^[13].

Table 1: Chemical composition analysis of *Abelmoschus esculentus* fiber compared with other Agro Residues

Fiber Name	Cellulose	Hemicellulose	Lignin	Ash	Holocellulose
<i>Abelmoschus esculentus</i>	67	15.4	7.1	2.44	82.4
Pineapple Leaf	70-83	19.5	10.5	2.0	80.5
Banana	56-63	20-25	7-9	1.5	65.2
Palmyra	42-52	40-43	18.5	0.6	68.5
Wheat Straw	51	26	16	7.4	77
Oil Palm	65	10.12	17.5	2.4	83.5
Cotton stalk	45.5	19.3	18.2	2.52	75.6
Rice straw	70.9	30.7	17.2	16.6	70.9
Rya straw	74.1	16	15.4	3.2	74.9
Corn stalk	39.0	42.0	7.30	24.9	82.1

3.3 SINGLE FIBER STRENGTH OF AESF

After that fiber length and fineness, the fiber strength is considered in order of importance amongst fiber properties ^[24]. Fiber strength denotes the maximum tension, the fiber is able to with stand before breaking. It can be expressed as breaking strength and tenacity etc. The mechanical properties of AES fiber depend largely on the chemical composition, especially the cellulose percentage and cell wall structure ^[30]. The pre conditioned of fiber was at $21 \pm 1^\circ\text{C}$ and $65 \pm 2\%$ relative humidity. The single fiber was mounted in the jaws of the clamps. All slacks was removed without really stretching the specimen and care was taken to keep the specimen straight within the jaws and ensured that the fiber sample lay on the line of action between the force and measuring device and the point where the fiber left from the moving jaw face. Twenty samples were randomly selected for test condition and a graph was plotted as load as strain to test the fiber samples, the mechanical properties of the AES fiber we determined ^[31]. The maximum tensile force was found to be 2.35N, young's modulus of AES fiber exhibit Mpa and the percentage of elongation at break of AES fiber 0.62% shown in fig(3).

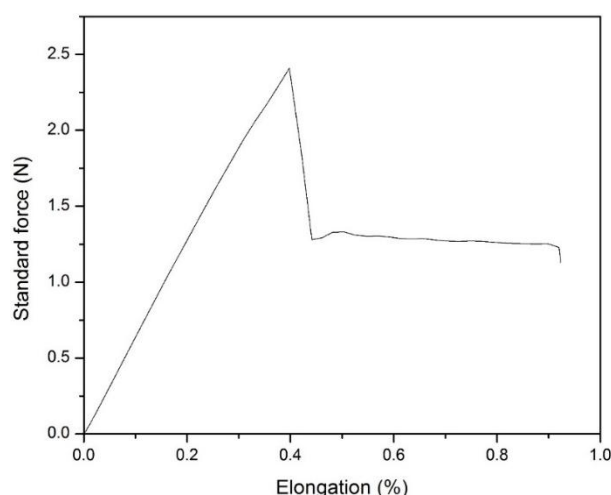


Fig 3: Single fiber strength analysis of AESF

3.4 SCANNING ELECTRON MICROSCOPE OF AESF

The scanning electron microscope is used to analysis the surface of the fiber sample and the AESF shown in fig 4 is similar to other natural fibers. SEM micrographs of AESF, which it is clear that the fibers contained surface impurities such as wax and fatty substance, the internal fibrils can be seen in higher magnification at 1000x, it is clear that the fibrils bound

together by hemicellulose can be seen in fig 4 (a). The fiber has a spotless and even surface and appears as a thick layer of uniform deposits over the entire length that is composed of hemicelluloses and lignin. These layers could enhance the interfacial bonding between fiber and matrix in the composites and the image capture in the magnification 500x is shown in fig 4(b). The surface morphology of the *AESF* shows in fig 4(c) that the fibers had multicellular structure. The *AES* fiber are used as absorption materials and filters and in polymeric composites^[32].

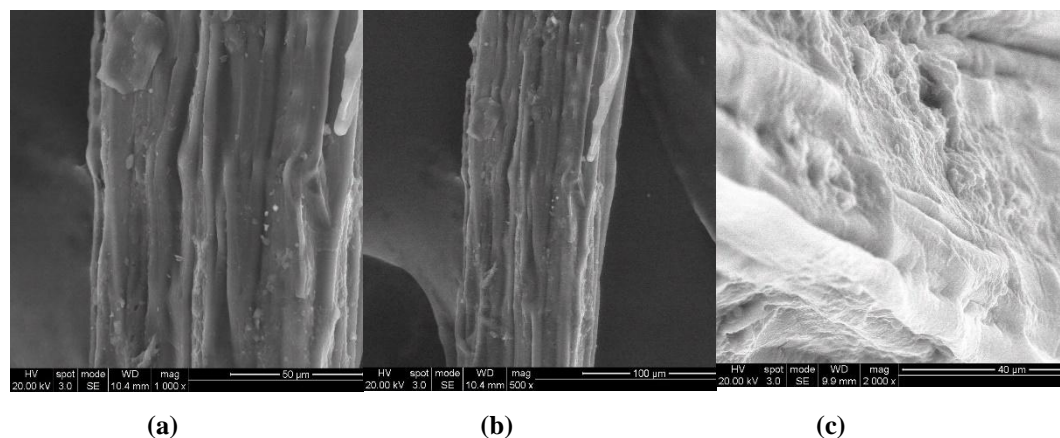


Fig 4: Scanning electron micrographs of AES fiber – (a) 500x, (b) 1000x & (c) 2000x.

3.5 X – RAY DIFFRACTION OF *AESF*

The highest intensity peak observed at $2\theta = 15.92^\circ$ represents the (1 0 1) crystallographic plane. This peak shows the presence of hemicellulose and lignin in an amorphous form^[33]. The relative amount of ordered crystalline material in cellulose was calculated by using Segal's empirical Equation (2)^[34].

The obtained CrI of the *AESF* is 62%, which is greater than that of *Ipomoea staphylinifera* fibers (43.96 %) and smaller than that of jute (71%) and hemp (88%) [35]. The average crystalline size (D) of the *AESF* was computed by substituting the X-ray wavelength value as 0.154 nm in λ , K value as 0.94 (Scherrer constant), FWHM (full-width at half maximum) value at (1 0 1) crystallographic plane in β (radian), and θ value as Bragg's angle at peak intensity count in Scherrer Equation (3)^[36].

The computed crystalline size (D) of the *AESF* is 2.68 nm, which is lower than that of Napier grass fiber (2.83 nm) and rice straw (3.75 nm)^[37]. The CrI and crystalline size influence the chemical reactivity and water absorption capacity of the fiber^[8].

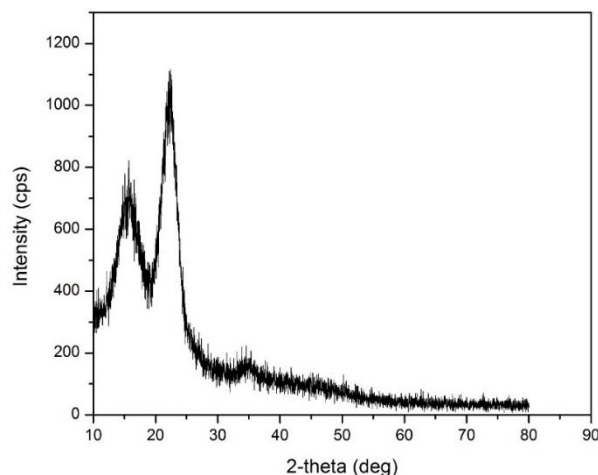


Fig 5: X-Ray Diffraction of *AESF*

3.6 THERMAL ANALYSIS OF *AESF*

The primary thermo gram of *AES* fibers is shown in fig (6). It is evident that the thermal degradation of *AES* fibers occurred in three stages. The thermal degradation of the first stage was due to the evaporation of mixture, the thermal degradation of second stage started at 375°C , and the final stage of mass loss occurred at 998.8°C . This stage corresponds to the degradation of alpha-cellulose and lignin in *AES* fibers. The thermal stability of *AES* fibers is compared with the natural

fibers like Napier grass fibre, Tamarind and Borassus. From this TGA curve, it can be clearly seen that AES fibers are thermally stable up to 375°C. It has higher thermal resistance. So used for acoustic composite panels.

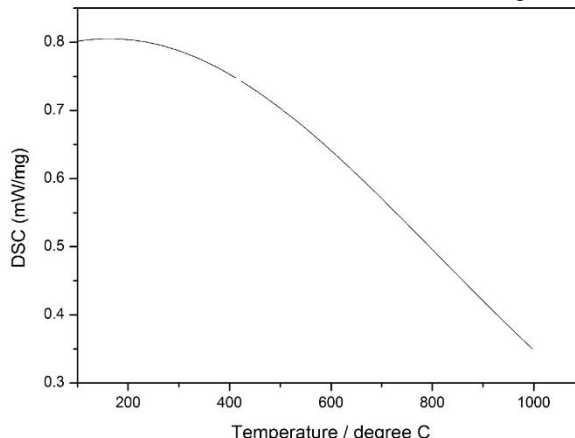


Fig 6: Differential Scanning Calorimeter of AESF

3.7 Physical Properties of *Abelmoschus esculentus* stem Fiber

The length of the fibre depends upon the plant selected for extraction. *Abelmoschus esculentus* stem Fiber has the length vary from 42 cm – 50 cm and diameter of 530 µm. The moisture regain and moisture content of the *Abelmoschus esculentus* stem Fiber is found to be 17.64 % and 15 % respectively. *Abelmoschus esculentus* stem Fiber has the fineness of 17 tex which shows the fibre is least bulk. (See Table I).

Table I Mechanical properties of *Abelmoschus esculentus* stem Fiber

Single fiber length	42-50cm
Single fiber diameter	530µm
Moisture regain	17.64%
Moisture content	15%
Fiber fineness	17 tex

CONCLUSION

In this study, broad characterization of AESF was carried out. The obtained results showed that the fiber has high cellulosic content, which provides better mechanical strength. The chemical composition, density, and tensile strength of the AESF were comparable with those of the other available unconventional fibers. Further, the tiny lined surface morphology and the high thermal stability of AESF ensure better bonding with polymer resins for making composites under elevated temperature without degradation of the fiber. Hence, promising results of the AESF showed that it could be a potential alternative to synthetic fibers for making light-weight composite materials and good-strength composites in a wide-ranging of industrial applications.

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