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Characterization of natural fiber extracted from *Abelmoschus esculentus*: an alternative potential for unconventional fibers

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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%. The *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

1. 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, 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*, and 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 *malvacceae* native to tropical, 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.

2. 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 *malvacceae* 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 mediterr area [4]. It is a tropical to subtropical crop and is sensitive to frost, low temperature, water logging and drought condition [5].

Fig 1: Abelmoschus esculentus Plant

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 TENXO 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 500–4000 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 °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 x 10^{-3} 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 Kα 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 °C/min and its ranges from RT to 1000 °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 Fiber Length and Diameter

Advanced instrument was not possible due to brittle nature of the fiber to analysis the length of the fiber. Calibrated steel scale is manually used measure the length of the fiber. The fiber diameter was tested using SEM photography of individual fiber taken along longitudinal direction. Ten fiber strand samples were scanned in different longitudinal position to get accurate result [19].

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 spectr a show 12 well define peaks of *AESF* at 4629, 4486, 4467, 4281, 4206,3462,3363,3107,2970,677,536 and 520cm^{-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 3107cm^{-1} indicates the presence of cellulose is absorption at C-H stretch of aromatics.

The peak at 2970cm^{-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].

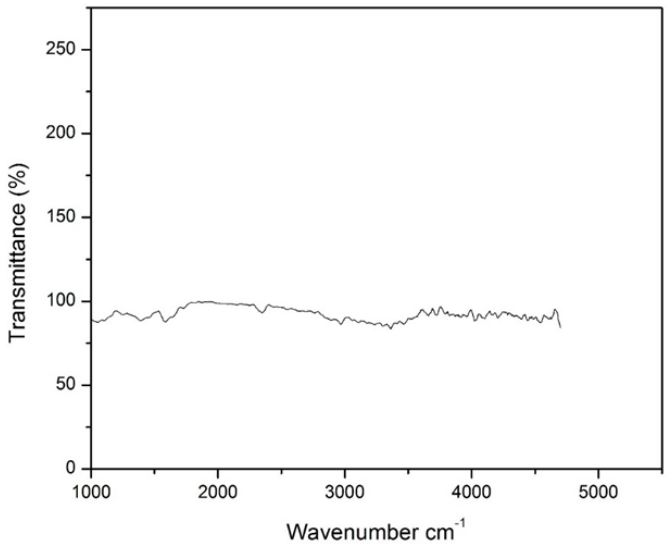

Fig 2: FTIR analysis of *AESF*

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 (67 wt%) tends to improve the mechanical properties of the *AESF* whereas the low hemicellulose content

(15.4 wt%) tends to reduce the moisture absorption capacity and in turn increase the thermal stability of the fiber. The presence of lignin content (7.1 wt%) acts as a bonding agent between the cell wall structures to improve the rigidity and strength of the fiber. Less amount of wax content (3.9 wt%) 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 2.44 wt%, 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
Abelmoschus esculentus	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±1˚C and 65±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).

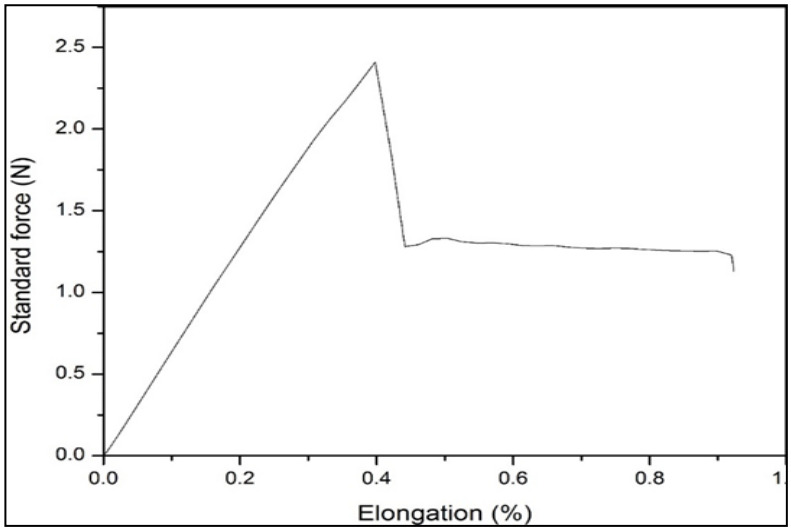

Fig 3: Single fiber strength analysis of *AESF*

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 matrials and filters and in polymeric composites [32].

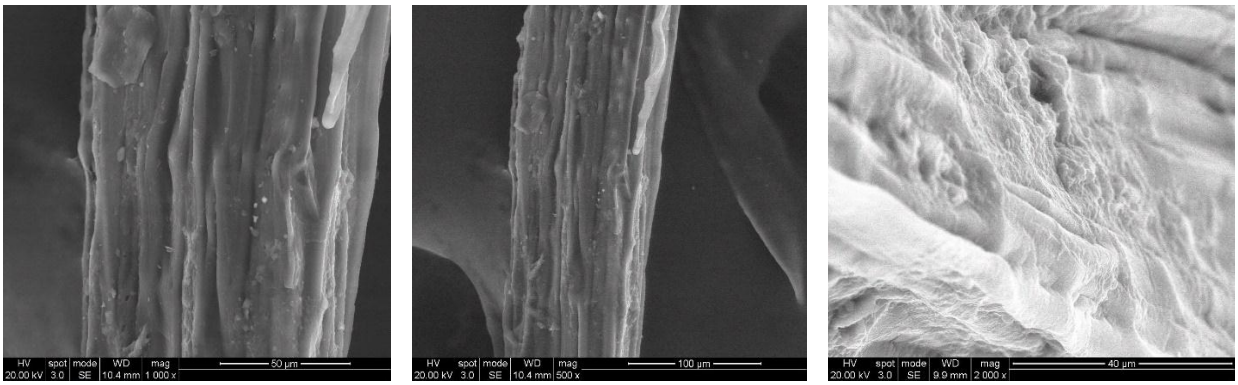

Fig 4: Scanning electron micrographs of *AES* fiber – (a) 500x, (b) 1000x & (c) 2000x.

3.5 X – Ray Diffraction of AESF

The highest intensity peak observed at 2θ = 15.92° 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 staphylina* 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 Scherer 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].

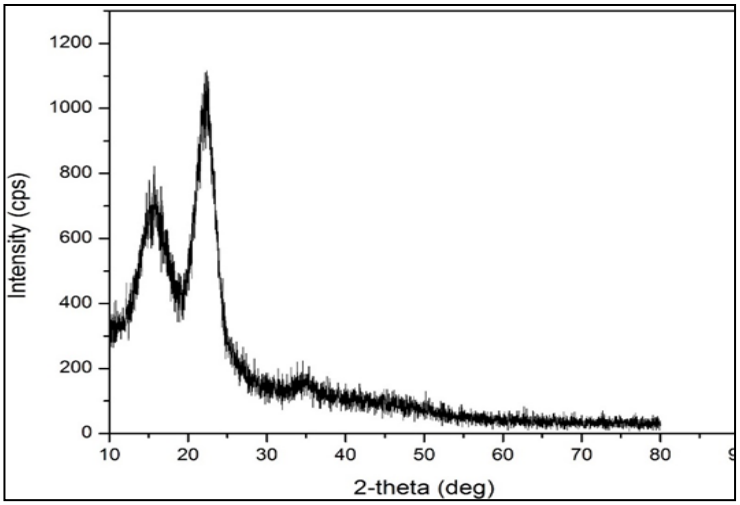

Fig 5: X-Ray Diffraction of *AESF*

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 is thermally stable up to 375˚C.It have higher thermal resistance. So used for acoustic composite panels.

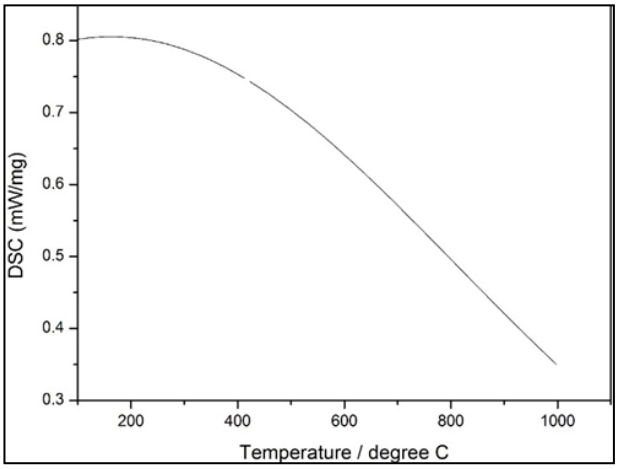

Fig 6: Differential Scanning Calorimeter of *AESF*

3.7 Fiber Length and Diameter of AESF

The low value may be attributed to the hollow nature and entrapped air on and among the fibrils due to poor wetting of the fiber by xylene [38]. The average length of the fiber from the particular plant grown in land is 42.05cm and it's Standard Deviation and Co-efficient of variation (CV) of 50.17 and 16.8% respectively. The fiber diameter observed in Scanning Electron Microscopy was found to be range 121.65µm with a mean value around 480.50µm and Co-efficient of variation is 18.01%.

4. 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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