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Physical characterization of cellulosic fibres from *Sesbania grandiflora* stem

K Savitha[1], Grace S Annapoorani[1], V R Sampath[2] & Desalegn Atalie[2, a]

[1]Department of Textile and Apparel Design, Bharathiar University, Coimbatore 641 046, India

[2]Ethiopian Institute of Textile and Fashion Technology, Bahir Dar University, Bahir Dar, Ethiopia

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In the present investigation, the morphology and the porosity of the *Sesbania grandiflora* fibre has been studied by SEM in order to understand their effects on the capillary structure and the hygroscopic behavior. The physical properties, such as tensile strength, elongation, density, fineness, morphological structure, water absorption coefficient and thermo-gravimetric analysis, have been examined. X-ray diffraction and Fourier transform infrared spectroscopy (FTIR) are used to identify the crystalline index and chemical groups present in the fibre. It has been found that this new vegetable material has a very low bulk density and a highest water absorption capacity. FTIR and X-ray analyses have proved that these fibres are rich in cellulosic content with crystallinity index of 51% cellulose content of 70.75 wt %, density of 1.4738 g/cc, and tensile strength of 365-11100 Mpa. The results show that *Sesbania grandiflora* fibres have comparable fibre strength, elongation and cellulose content to jute, hemp, ramie, Phoenicx sp, okra and *Prosopis juliflora*. The new fibre has better crystallinity index than banana, bagasse and sponge gourd and hence can be utilized for technical textiles application.

Keywords: Cellulose, Fibre characterization, Fibre extraction, Natural fibre, *Sesbania grandiflora*

1 Introduction

Natural coarse fibres are being increasingly used in the area of technical textiles like automotive parts and composite reinforcement. For this reason, cheaper and stronger natural cellulosic fibres need to be explored[1]. The most effective way to insure the concepts of green energy is the use of eco-efficient environment-friendly materials as thermal insulators. As a result, specialized engineers and researchers attracted to the design and develop advanced materials in order to enhance and maximize energy performance and minimize energy consumption. Consequently, this will lead to have a cost-effective, durable and ecofriendly material that will meet the global needs of thermal rehabilitation. A good example is the natural fibres reinforced composites which are of interest as a replacement of conventional synthetic fibres reinforced polymers for some applications[2], such as industrial sectors, especially automotive and aerospace[3,4]. These composites consist generally of two or more components with natural fibres in order to obtain specific characteristics, such as low cost, low density, renewable resource, low energy inputs in their production, high tensile & compressive strengths, and reduced shrinkage & cracking. In addition, they may be bio-degradable and completely or partially recyclable after the use, depending on the selected matrix[5]. The advanced research of the last few years has shown that it is possible to produce high performance natural fibre composite[6]. Manmade fibres are generally non-degradable, whereas natural fibres are degradable and ecofriendly[7].

The concept of ecofriendly and recyclable products which are highly recognized, brings natural fibres into our sharp focus. The less explored natural fibres are nothing but stem fibres. The vegetable fibres are classified according to their origin and the parts of the plant, i.e. leaf, seed, bast, stem, fruit, grass and stalk. Their properties naturally become affected by factors, such as climate, maturity, harvesting or physical or chemical treatments[8]. Different natural fibres have been investigated as possible reinforcement of composite materials. These fibres include sisal, coir, bamboo and coconut husks[9, 10].

Sesbania grandiflora is a widely available Indian medicinal plant commonly known as Agathi, belonging to family Fabaceae[10, 11]. *Sesbania grandiflora* has unique medicinal properties, as all the parts of the plant serve as a natural anti-oxidant [12, 13].

In this study, *Sesbania grandiflora* fibres have been extracted from the stem using an ecofriendly method (immersion in fresh water). In fact, the extraction

[a]Corresponding author.
E-mail: desalegnatalie@gmail.com

methods and planted area affect the fibre properties. The extracted fibres are characterized for their cellulose, lignin, ash and wax contents. Furthermore, the fibre moisture content, fibre strength, density, water absorption, microstructure, crystallinity index, functional groups and thermal properties are also characterized and compared with other natural coarse fibres.

2 Materials and Methods

2.1. Raw Material and Fibre Extraction

The stems of *Sesbania grandiflora* (SG) were collected from Erode Districts, Tamil Nadu, India. The length of the SG stem is 2-4 feet. The stems are cut at required length by knife and manually peeling process. These peeled parts are immersed in water for 7-10 days for extraction. During this water retting process, the peeled parts are completely wetted. As the gums were present in the fibres separated, the extracted fibres were washed thoroughly to remove the unwanted materials. After these fibres were dried in sunlight at least from 2-4 h to remove the water content, the bundles of dried fibres were collected for further investigation.

2.2 Characterization Methods

2.2.1 Chemical Properties

Table 1 shows various natural coarse fibres which are differing from one another. This causes the release of fibre encrusting substance (lignin, celluloses, wax, etc). The chemical composition of SG is determined by using standard test procedure[14].

2.2.2 Microscopic Structure

The fibre structure testing procedures were done according to ASTM D 276 standard. Optical microscope was used to take the image of the fibre in longitudinal direction and these images were imported to image analyzer software to measure the diameter of 20 samples at five places.

Table 1 — Chemical composition of *Sesbania grandiflora* and other natural fibres

Fibre	Cellulose wt %	Lignin wt %	Ash wt %	Moisture wt %	Wax wt %
Phoenicx sp.	76.13	4.29	19.69	10.41	0.32
Hemp	74	4	-	10.8	2.3
Sesbania grandiflora	70.75	19.49	1.12	8.33	1.4
Prosopis juliflora	61.65	17.11	5.2	9.48	0.61
Hop stem	84	6.0	2.0	-	-
Okra	60	0.6	-	7.5	0.3

2.2.3 Tensile Strength and Elongation

Tensile strength, young modulus and elongation-at-break were tested as per ASTM D 3822. Twenty samples were randomly selected from raw fibres (20 mm length) and then tested using Instron 5500 R universal testing machine for tensile strength at 65% relative humidity and 21 ± 1.5°C temperature with a cross-head speed of 10mm/min. A 1.0 kN load cell was used in the study.

2.2.4 Fineness

The fineness (tex) test was done by measuring the length of the extracted fibres, weighing on the electronic balance and then calculating the value using the following formula:

$$\text{Fineness (tex)} = \frac{Fibre\ mass\ (g)}{Fibre\ length\ (m)} \quad \ldots (1)$$

2.2.5 FTIR Study

Fourier transform-infrared spectroscopic study of *Sesbania grandiflora* fibre was executed using Nicolet smart ITR –ATRIs, 10 FTIR spectrometer. To ensure the presence of free functional group, peaks in the range 500-4000 cm^{-1} at 25° C and 65% RH were recorded in absorbance mode as a function of wave number.

2.2.6 SEM Study

Scanning electron microscopic (SEM) test was performed to observe the microstructure of various fibres. The high resolution scanning electron microscope (model JEOL JSM -6390) was used. After 72 h of drying, the sample was directly observed without any treatment. In this study, we focused particularly on the longitudinal and transversal surfaces of each fibre. SEM produces very high-resolution at three dimensional images, which are very much useful for understanding the surface morphology of fibres.

2.2.7 XRD Study

Powder X-ray diffraction (XRD) method was used to find out the crystallinity of fibre. It was analyzed at room temperature (23°-25°C) under the conditions of current 30mA, voltage 40KV and Cu anode made. Scanning mode ranging from 10° to 89° with scanning speed of 10de/ min were kept for testing. In this study, a new approach is made which is based upon the idea of extracting fibres from SG stem at a low temperature. The behavior of individual material is different as it gets polarized at room temperature (23°-25 °C). The deformation behavior at low temperature differs from that observed at the

room temperature (23°-25°C). The crystallinity index (I_{cr}) was calculated by means of following expression.

$$I_{cr} = 1 - L_{min} / L_{max} \quad \text{... (2)}$$

where L_{min} and L_{max} represent the intensity at minimum and maximum crystalline peaks respectively[15].

2.2.8 Thermo-Gravimetric Analysis

Thermal decomposition was observed in terms of global mass loss by using Jupiter simultaneous thermal analyzer (MODEL STA499 F3, NETZSCH, Germany). This apparatus detects the mass loss with a resolution of 0.1 mg as a function of temperature. The samples were evenly and loosely distributed in an open sample pan with an initial sample amount of 5 mg. The sample was exposed to nitrogen gas at a flow rate of 20 mL/min. Ten milligram of the SG was crushed and kept in alumina crucible to avoid the temperature variations measured by the thermocouple. The temperature change was controlled from room temperature (25°–1000 °C) at a heating rate of 10 °C/min.

3 Results and Discussion

3.1 Chemical Composition

Table 1 shows the chemical composition of the *Sesbania grandiflora* and other natural fibres for comparison. Cellulose content in natural fibre is considered to be main component, for strength, stiffness, and structural stability. This SG fibre has high cellulose content. The lignin content in the fibres contributes to the rigidity and its value is found greater than those of *Prosopis juliflora* and Hop stem[16]. The wax content is low in comparison with Hemp. The *Sesbania grandiflora* fibre has low ash content. The moisture content present in the fibre is found higher and comparable to existing fibres like *Prosopis juliflora* and Okra.

3.2 Physical Properties

3.2.1 Microscopic Structure

Examination of longitudinal and cross-sectional views gives detailed information with regard to the surface morphology of the fibre. Like other natural fibres, the surface morphology of the *Sesbania grandiflora* fibre has several smaller fibres (known as fibrils), which remains stick as one in the length-wise direction by non- cellulosic compound forming a bundle. In addition to this, the diameter range of SG fibre sample is calculated as 275-317mm. The average diameter of this fibre is calculated as 280mm.

3.2.2 Density

The density value of *Sesbania grandiflora* fibre was found to be 1.4738 g/cc (Fig. 1). It has been noticed that the density value of this fibre is comparable with those of flax, ramie, jute, sisal and nettle fibres. But it is higher than those of coconut, bamboo and kenaf fibres.

3.2.3 Tensile Strength and Elongation

In Table 2, the tensile strength of the SG is found 365-1100Mpa, while the tensile strengths of other natural fibres such as flax, hemp, jute and ramie are 345-2000, 368-800, 393-773 and 400-1000 MPa respectively. Tensile strength of a fibre determines the kind of technical application it can be used for. In composite reinforcement, stronger fibres impart higher strength to the manufactured composites[10].

3.2.4 Fineness

Fineness is also one the most important fibre characteristics showing how heavy a fibres. In this investigation, the fineness of the SG fibre is determined in g/km (tex unit). The range between

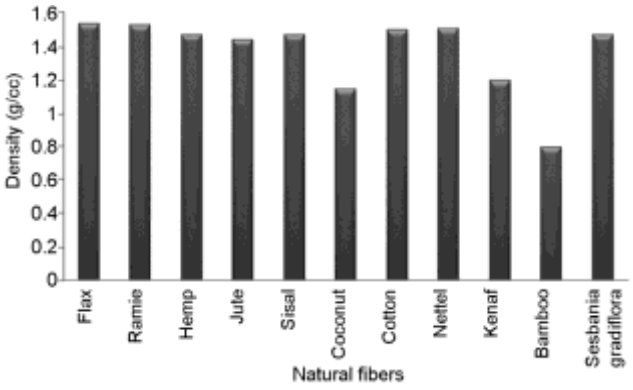

Fig. 1 — Fibre density of Sesbania grandiflora and fibres

Table 2 — Tensile strength of *Sesbania grandiflora* and other fibres

Fibres	Tensile strength MPa	Elongation-at-break, %
Flax	345-2000	1-4
Ramie	400-1000	1.2-3.8
Hemp	368-800	1.6
Jute	393-773	1.5-1.8
Sisal	350-700	2-7
Coconut	131-175	15-40
Nettel	560-1600	2.1-2.5
Kenaf	240-930	16
Bamboo	140-230	-
Sesbania grandiflora	365-1100	1.5

minimum (0.70 tex) and maximum (2.8 tex) fineness in water retted extracted fibres was wide.

3.2.5 Water Absorption Capacity

The water absorption coefficient is an important parameter. In fact, there is need to know the proportion of water absorbed by the plant fibre, to deduce the amount of water that reacts with the other nonwovens. It is found that minimum water absorption of *Sabania grandiflora* fibre (290-400%) is comparable with jute fibre (281%). However, *Sesbania grandiflora* fibre shows greater water absorption than sisal fibre (190-250%), bamboo (145%), and *Retama monosperma* (53%) fibres, since the water absorption is increasing with an acceleration of the free water infiltration in the micro-pores[17,18].

The studied fibres have a water absorption coefficient variable between 290% and 400% after 60 min immersion in water at 20 °C. These values are described as very high in comparison with those of flax and hemp. This feature is both harmful and beneficial. In the short term, a high absorption coefficient may create difficulties during the implementation of fresh material, due to the mobilization of a large part of the mixing water to the fibres.

3.3 FTIR Study

As shown from the results, the peaks which appear at 3345 cm^{-1} correspond to O-H stretching vibration and hydrogen band of hydroxyl group[19]. The band at 2920 cm^{-1} represents the characteristics of C-H stretching vibration from CH and CH_2 in cellulose components. The peak at 2852 cm^{-1} is associated to the asymmetric and symmetric stretching of methylene CH_2 main components. The wave number at 1738cm^{-1} and 1633cm^{-1} corresponds to the carbonyl group (C=O) of lignin and hemicelluloses. The peak at around 1427cm^{-1} corresponds to the (CH_2) groups of cellulose. Presence of peak at around 1371cm^{-1} is associated with (C-H) groups of cellulose and the band at 1244 cm^{-1} belongs to polysaccharide in cellulose (1157.93 cm^{-1}). It is observed that the C-OC group cellulose and hemicelluloses are exposed. The peak at 1030cm^{-1} represents the presence of C-O and OH stretching vibrations, which belong to polysaccharide in cellulose.

3.4 Morphological Analysis

The morphological analysis of *Sesbania grandiflora* fibre is shown in Fig.2 (a) and (b) at ×300 and ×500 magnifications respectively. The illustration shows that the fibrils are clean and at the same time they have smooth surface. The surface of individual cell is clearly visible perhaps due to low wax content.

3.5 X-Ray Diffraction Analysis (XRD)

As per the XRD results, Sesbania grandiflora crystallinity value calculated using the curve is 51% of fibre. It is slightly higher than other natural fibres like banana (38%), bagasse (48%) and sponge gourd (50%), and lower than that of jute (71%) and hemp (88%) (refs. 20,21)

3.6 Thermogravimetric Study

It is clearly observed that the thermal degradation of SG fibre has occurred in three stages. As a result of the evaporation of moisture, thermal degradation starts at the initial stage. Next to it, the degradation at second stage starts at 225^{0}C but the mass loss occurs

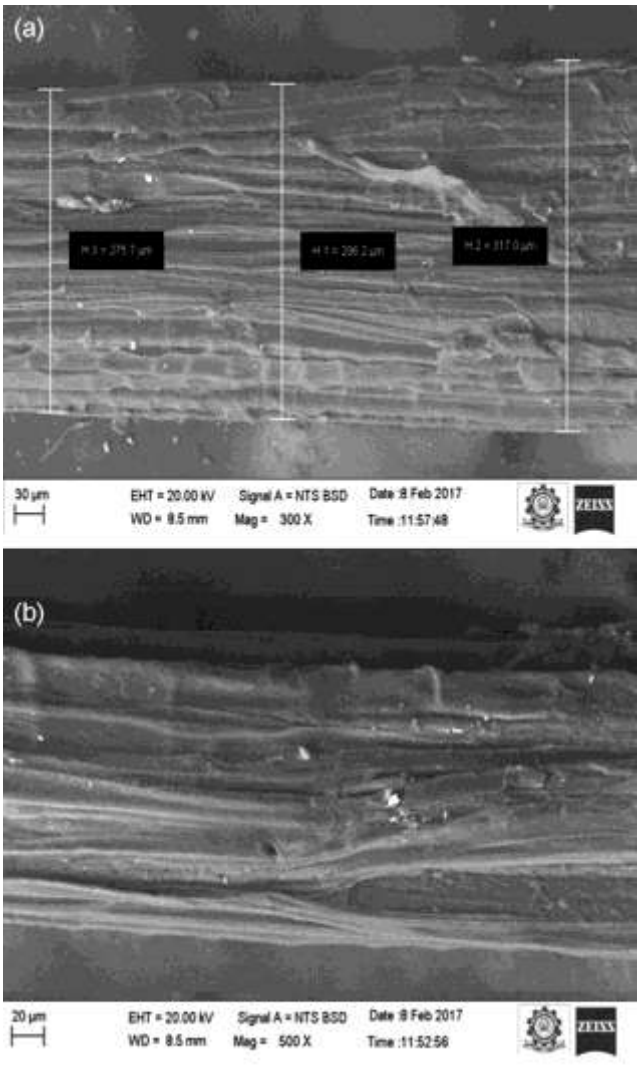

Fig. 2 — Scanning electron microscopy at (a) ×300 and (b) ×500

at 325^0C in third stage. Each stage goes on with the degradation of alpha-cellulose and lignin in SG. However, the thermal stability of SG seems to be comparable to other fibres, such as wild date palm, tamarind and bagasse. From the study of TG curve diagram, it is obviously observed that SG appears up to 225^0C. In a lingo-cellulosic material subjected to a temperature rise, generally hemicelluloses decompose first, followed by the decomposition of the cellulose and lignin.

4 Conclusion

The analysis of the study on the micro structural and physical characterization of *Sesbania grandiflora* fibre for the purpose of eventual use in insulation materials highlights the following key points:

4.1 *Sesbania grandiflora* has high cellulose content than in okra and *Prosopis juliflora* fibres, and higher tensile strength than sisal, bamboo, kenaf and coconut fibres.

4.2 Scanning electron microscopy shows soft surface of fibre.

4.3 The water absorption of *Sesbania grandiflora* is better than other natural fibres, which leads to better dimensional stability, reduced porosity and improved adhesion between the fibres.

4.4 The obtained crystallinity of the *Sesbania grandiflora* fibre is found 51%. FTIR spectrum absorbance peaks represent the presence of C-H stretching of alkenes groups and O-H stretching of alcohol groups.

4.5 *Sesbania grandiflora* fibre has been introduced as a lignocellulosic fibre for the development of an acoustic nonwoven fabric for the use of any lightweight structure.

4.6 When compared with other fibres, *Sesbania grandiflora* fibres properties like tensile strength, elongation, water absorption and degradation point are found comparable with jute, sisal and ramie fibres. For this reason, *Sesbania grandiflora* fibres can be used for technical textile application, such as composite reinforcement.

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