



Mechanical extraction and physical characterization of Agave Angustifolia v. Marginata lignocellulosic fibre

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Abstract: *Agave angustifolia v. marginata* is a leaf fibre which was mechanically extracted from the green leaves of the plant using water retting extraction method. The chemical composition of *Agave angustifolia* fibre was also determined. The composition mainly consisted of cellulose, hemicellulose, lignin and ash. Structural analysis was carried out by fourier transform infrared spectroscopy (FTIR), SEM, TGA, X-ray diffraction, tensile and moisture regain properties are studied.

Keywords: *Agave angustifolia*, extraction, chemical composition, FTIR analysis, X-ray diffraction, thermal degradation, tensile properties.

I. Introduction

There are at least 1000 types of plant that bear usable fibers [1]. Natural plant fibres are easily obtained in many tropical parts and are available throughout the world. Today these fibres are considered as environment friendly materials owing to their biodegradability and renewable properties [2]. Natural fibres have applications in fields of textile, paper manufacturing, and bioenergy industries owing to their broad availability and properties. Natural fibres can generally be classified based on their origin (e.g., plant, animal, or mineral). Plant/vegetable fibres can be further classified into subgroups according to their source (e.g., stem fibres, leaf fibres, seed fibres, or fruit fibres) [3, 4]. Natural fibres that are obtained from different parts of the plants, to name few, for example, jute, ramie, flax, kenaf and hemp are obtained from the stem; sisal, banana and pineapple from the leaf; cotton and kapok from seed; coir from the fruit.

All plant species are built up of cells. When a cell is very long in relation to its width it is called a fibre. The components of natural fibres are cellulose, hemicellulose, lignin, pectin, waxes and water-soluble substances. The cellulose, hemicellulose and lignin are the basic components of natural fibres, governing the physical properties of the fibres. The composition depends on the type, age, and origin of the fibre, as well as the method of fibre extraction. The properties of natural fibres also depend on their chemical composition [5-7]. Over 100 million tons of different fibres are manufactured every year out of which 50% of that are derived from crude oil [8]. Therefore, many efforts are made to replace at least a small portion of synthetic fibres with cellulose fibres which are obtained from plants or agro-waste materials. Day by day awareness is increasing in Consumers about environmentally friendly products and more and more people want to buy it [9]. In recent years there has been a growing interest of various industries in renewable plant materials. Recently studies have been carried out on agro-waste materials to obtain natural cellulose fibres from wheat straw, soyabean straw, rice straw, corn stalks leaves and stalks of sorghum, banana leaves, sugar cane [10- 12].

In this present work the plant which is used for fibre extraction and physical and chemical characterization is *agave angustifolia v. marginata* commonly called the "Banded Carribean Agave" *Agave angustifolia* belongs to the Agavaceae family. This is a very rugged, attractive, eye-catching plant with symmetrical narrow, stiff bayonet leaves liberally margined in creamy white. The rosettes can be 1 m in diameter with several leaves 50-80 cm long ending in 18 mm long terminal spine. It is grown as an ornamental plant worldwide. Native from Costa Rica to Mexico. Each rosette develops a trunk 40 cm high and produces offsets around the base, eventually forming clumps .A single plant has 20 to 30 offsets spreading to 15 feet away from the parent plant. This is one of the few agaves that forms much of a stem. Fairly tropical, it grows quickly, but will not tolerate much frost. It grows best in full sun but can adapt to some shade [13]. The fibres were mechanically extracted, characterized using chemical analysis, FTIR, XRD and TGA techniques. The surface structural characterization of the fibres were examined by using scanning electron microscope. Tensile testing

was used to characterize the mechanical properties. The moisture regain and moisture content properties were also examined.

II. Materials and methods

A. Materials

The fibre was extracted from the leaves of the agave angustifolia plant which was harvested from (Mumbai, India). The chemical reagents of analytical grade used are sodium chlorite (NaClO_2), acetic acid glacial, sodium hydroxide, sodium bisulphate, sulphuric acid, ethanol, toluene, benzene, acetone were procured from SD Fine Chemicals Ltd., India. Buffer solution was freshly prepared.

B. Methods

B.1. Fiber extraction

The fibres were extracted from the matured leaves of the agave angustifolia plant. The leaves were directly subjected for water retting for 15 days. After this the leaves were removed from the water and beaten with a wooden hammer to loosen the fibres. These fibres were washed continuously under running tap water to get clean fibres. These fibres after washing were sun dried, combed and hand brushed to get uniform fibres and make them suitable for physical, chemical and morphological analysis.

B.2. Determination of chemical composition

The chemical composition of the agave angustifolia raw fibres was determined by chopping the fibres into small pieces. These chopped fibres were weighed and Dewaxing was carried out in a mixture of toluene/ethanol (1:1 v/v) in a soxhlet apparatus for 6 h. The acid-insoluble lignin content was determined according to TAPPI T222 om-02 (2002). In this method of determination, lignin (also known as "Klasons lignin") is defined as constituents fibres insoluble in 72% sulphuric acid. The α -cellulose content was determined by TAPPI T203 cm-99 (2002). Ash content was determined by standard method of TAPPI T211 om-02 (2002) [14].

B.3. Morphological studies

The scanning electron micrographs of the fibre surface were recorded on a JEOL JSM 820 microscope (Akishima, Japan), from Institute of Chemical Technology. The fibre samples were sputter coated with gold before recording the micrographs.

B.4. Fourier transform infrared spectroscopy (FTIR) analysis

The IR spectra of raw fibre sample was recorded using FTIR spectrophotometer (Shimadzu 8400s, Japan) using ATR sampling technique by recording 45 scans in % transmittance mode in the range of 4000-600 cm^{-1}

B.5. X-ray Diffraction (XRD)

The crystallinity of raw Agave angustifolia fibre was studied using an X-ray diffractometer (Shimadzu 6100, Japan) equipped with CuK_α radiation ($\lambda = 1.54 \text{ \AA}$) in the 2θ range 2-50°. The experiment were performed in the reflection mode at a scan speed of 2°/min in steps of 0.02°. The crystallinity index (CrI) of the fibre was calculated according to the Segal empirical method as shown in equation 1, [15].

$$\text{CrI}\% = (I_{002} - I_{\text{am}}) / I_{002} \times 100 \quad (1)$$

where I_{002} and I_{am} are the peak intensities of crystalline and amorphous materials, respectively.

B.6. Thermogravimetric analysis

A sample of Agave angustifolia fibres was cut into small pieces and thermal gravimetric analysis (TGA) was carried out. The thermograms were recorded on Shimadzu 60H DTG machine using aluminium pan between temperature range 30-500 °C under the inert atmosphere of nitrogen at a flow rate of 50ml/min.

B.7. Tensile properties and moisture regain

The tensile properties of the Agave angustifolia fibres are measured in terms of the breaking load, percentage elongation at breaking using Tinius Olsen tensile testing machine. A gauge length of 20 mm with a speed of 5 mm/min were used for the testing. Approximately 15 fibres were tested for their tensile properties. To identify the moisture content and moisture regain values, the fibre sample was tested according to ASTM standard method 2495.

III. Results and discussion

A. Determination of fibre composition

The chemical composition of agave angustifolia fibre by mechanical extraction method is reported in Table 1. The raw fibre is composed of cellulose, hemicelluloses, lignin, ash and extractives.

Table 1. Chemical Composition of agave angustifolia fibre

Sample	α -Cellulose (%)	Hemicellulose (%)	Lignin (%)	Ash (%)	Extractive (%)
Raw Agave angustifolia fibre	65 \pm 0.1	23 \pm 0.1	6.5 \pm 0.1	2 \pm 0.1	2.5 \pm 0.1

B. Morphological studies

Scanning electron microscopy (SEM) showed the influence on agave angustifolia fibre morphology which was mechanically extracted (fig. 2). Like other lignocelluloses fibres agave angustifolia fibres obtained by mechanical extraction process have a thick layer of deposits on the fibre surface which mainly composed of wax, lignin, pectin and hemicellulose, which protects the cellulose fibres inside [16].

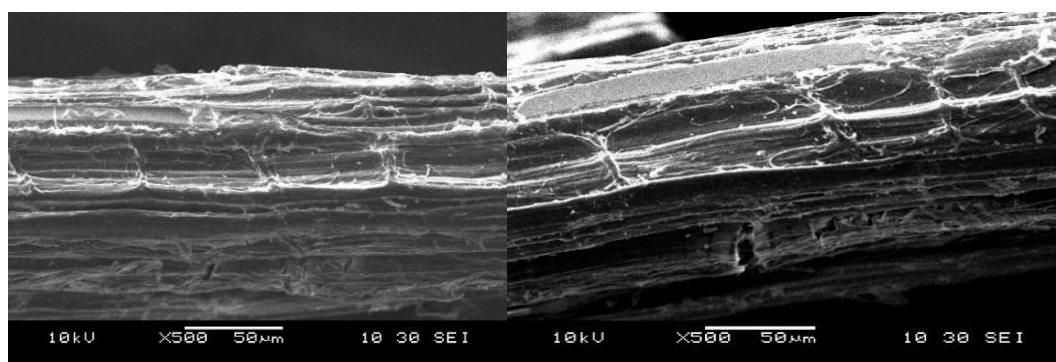


Fig 2. SEM images of raw agave angustifolia fibre

C. Fourier transform infrared spectroscopy (FTIR) analysis

The FTIR spectra of the raw Agave angustifolia fibre is presented in Fig. 3. The spectra exhibited a broad band in the region of 3400 to 3300 cm^{-1} are assigned to the adsorbed water which indicates the free O-H stretching vibration of the OH group in cellulose molecules. A peak at 1731 cm^{-1} for attributed to the C=O stretching vibration of the acetyl and uronic ester groups, from pectin, hemicelluloses, or the ester linkage of the carboxylic group of ferulic and p-coumaric acids of lignin and/or hemicelluloses [17, 18]. The aromatic ring C=C stretching vibrations of lignin skeleton was observed at 1605 cm^{-1} . The absorption peak at 1234 cm^{-1} in the raw fibre is attributed to the C-O out of plane stretching vibration of the acetyl group in the lignin [19]. The peak at 896 cm^{-1} attributes C-H rocking vibrations assigned to β -glucosidic linkage. Similarly the peaks at 1423, 1367 and 1323 cm^{-1} present in raw fibre are associated with the bending vibrations of $-\text{CH}_2$, C-H, and C-O of cellulose [20, 21]. Similarly the peak at 1020 cm^{-1} assigned to aromatic C-H in plane deformation and a peak at 1157 cm^{-1} C-O-C asymmetrical stretching in cellulose and hemicellulose.

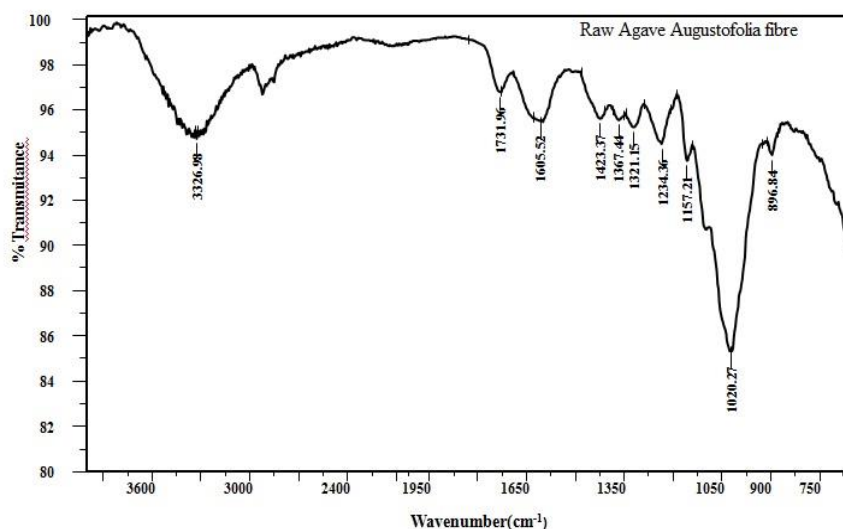


Fig 3. FTIR spectra of raw agave angustifolia fibre

D. XRD analysis

The X-Ray diffraction pattern of raw fibre is shown in Fig 4. The diffractogram showed two reflections, corresponding to 2θ values of around 16° and 22.5° respectively. Among these, the low angle reflection (16°)

was of low intensity, representing $I_{(am)}$ of amorphous material and the other reflection (22.5°) had higher intensity, and it represented $I_{(002)}$ of crystalline material in cellulosic fibre. The degree of crystallinity (CrI%) was calculated using Eq. (1). Crystallinity index of fibre is described below in Table 2.

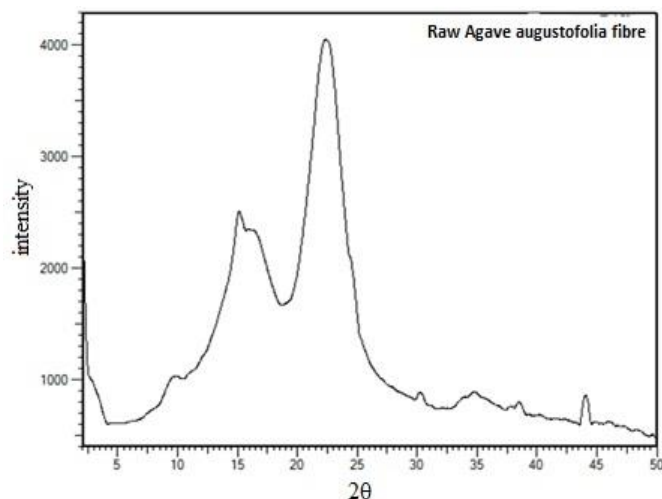


Fig 4. XRD diffraction pattern of agave angustifolia fibre

Table 2. Crystallinity index of agave angustifolia fibre

Sample	2θ (am) (°)	2θ (002) (°)	% crystallinity
Raw agave angustifolia fibre	15.12	22.36	55.71

E. Thermogravimetric analysis

The thermograms of agave angustifolia fibre is shown in Fig.5. and it can be observed that the thermal degradation of the fibre exhibited a three - step process. This behaviour was attributed due to differences in the chemical structures, of different components of wood fibres which degrade at different temperatures [22].

Agave angustifolia fibre being lignocellulosic material is a composite structure of different types of components, and it will degrade below 400 °C with wax, pectin, and hemicelluloses degraded at 180 °C, cellulose at around 300 °C, and lignin at 400 °C [23].

The first, second and third stages were found in the temperature ranges of 30–120 °C, 200–270 °C and 270 – 400 °C respectively. The first decomposition temperature range of 30– 120°C corresponded to the evaporation of moisture and the weight loss was 8.62%. The second decomposition step of raw fibres in the temperature range of 200 – 270°C indicated the loss of hemicellulose and some part of the lignin taking the weight loss to 16.41%. The third degradation step corresponding to the decomposition of cellulose was observed in the range of 270 –400 °C causing maximum weight loss of 62.89% [24].

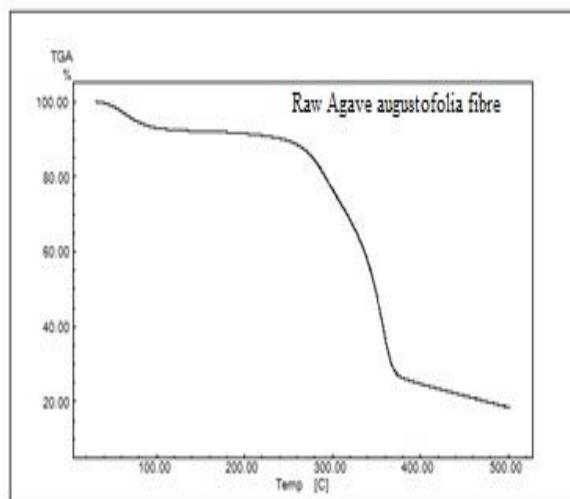


Fig 5. Thermogram of raw agave angustifolia fibre

G. *Tensile properties and moisture regain*

The fibres showed good tensile properties exhibiting 406.9gf with elongation at break at 4.4411% and moisture content 7.5-8% and moisture regain of 8.5-9%.

IV. Conclusion

The natural lignocellulosic fibre which was mechanically extracted from agave angustifolia plant has cellulose content around 65% enabling it to be suitable for the textile applications. The XRD analysis showed that the fibers have a crystallinity of 55% which is similar to cotton. TGA analysis showed that the fibre had a very good thermal stability upto 270° C. The tensile strength was also good. These properties show that, the fibre has a great potential to be used as a textile material. Although this plant is mostly grown in wild, if this fibre gets commercialized then there is likely hood that the farmers can also get a new source of income by growing this plant on a large scale, as it requires very less maintenance and it is easy to grow. The properties of the agave angustifolia fibres make them suitable for advanced textile applications and hence show promising potential.

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