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Extraction and Characterization of Natural Cellulosic Fibers from *Carissa Edulise* Plant Stems

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Abstract— *Studies on Natural fibers are intensive these days due to their eco-friendly nature and special properties. Possibility of discovering certain non-conventional sources for natural fibers is being explored. Plant fiber is one of the lignocellulose fibers that use in textile industry. However natural fiber not enough for any textile application and has to compete with synthetic fibers. In this present research, a cellulosic fiber was extracted from the trunk of Carissa Edulise plant by two different methods such as chemical and water retting. The extracted fiber was examined for its diameter, fineness, tensile strength and elongation by using the ASTM method. The diameter of the fiber was found to be 16.85 -17.95 microns with a fineness of 0.58 - 0.65 Tex .The tensile strength and elongation of the fiber were found to be 135 gm/ Tex and 37.8%. with Moisture content and moisture regain of fibers 8.3% and 9.09% respectively. From the result, it is observed that the fiber had properties like those of other cellulosic fibers such as jute and sisal. Since, it is believed to be a potential source for natural cellulose textile fiber that can be used for apparel textile and paper application in textile industries*.

Keywords— *Natural fibre, cellulosic fibers, Carissa Edulise plant, water retting, Chemical extraction*.

I. INTRODUCTION

Nowadays, the development of environmentally friendly products is at its core point, in order to the constant study for practical solutions to environmental problems and the urgent need to change from non-renewable products. Multiple investigations have been developed in this regard trying to find new materials in order to replace the conventional ones such as plant fibres which have so many advantage like renewable natural resource, bio degradable, cheap skate in cost, good specific mechanical properties and low density [1-4]. Most of the natural reinforced composite textile materials focus on jute, Flax and sisal fibers those fiber they have own disadvantage or drawback for example, Kenaf fiber is brittle and difficult to process [6]. Ramie fibers are extracted from the stem of the plant and it's difficult to extract by water retting, even by a combined microbial and chemical treatment [7]. Jute, one of the cheapest natural fibers, is also the most versatile, eco-friendly, natural, durable and antistatic fiber available but low extension at break (1-2%) is characteristic weakness [8, 9, 10]. Flax fibre has the problem of more Crystalline in nature. Twisting of flax fibers [11], [12] posses problem beside not enough supply for world markets and not fully applicable for industrial applications because of the dominance of synthetic fiber. [13].

In this research, the fiber that was successfully extracted from natural abundantly available plant in Ethiopia, Carissa Edulise from its stems by using microbial retting by water and chemical retting using sodium hydroxide (NaOH) alkali is reported and it can be used for textile and other applications. It is the microbial decomposition of pectin that is needed for separation of fibre from stalk. This process has major effect on the end product quality; the water retting procedure is of crucial importance to the processing ability of the fibers [14, 15]. Alkali treatment leads to fibrillation which causes the breaking down of the composite fiber bundle into smaller fibers, which mean that alkali treatment reduces fiber diameter [16.17].

Extraction by retting and chemical processing once completed, the non-fibers are removed and washed, and subjected to mechanical processing to remove the soft tissues and then dry to obtain the fibers. It was found that non-cellulosic substances were completely removed or reduced after both microbial (water) retting and alkalization treatment. The final product indicated that the fibers have good properties; therefore, it can be alternative source for natural cellulose fibers that can be used for textile and composite application.

II. MATERIALS AND METHODS

Materials

Carissa Edulise Plant branch of stems were collected or harvested from rural area of Ethiopia. The outer skin of the bark or stem was peeled from the stems by hand and used for fiber extraction. The inner bark was very tough and not suitable for extracting fibers. They were subsequently subjected to the removal of non-fiber content from the skin surface by both chemical and water retting methods.

Fiber Extraction

Based on experiences in obtaining fibers from stems byproducts, several conditions to extract fibers from the Carissa Edulise stems were used. We observed that the Carissa Edulise stems were sensitive to extraction conditions. Strong alkali conditions resulted in the disintegration of the bark into small fibers. After several trials, the most optimum conditions of fiber extraction were developed based on the yield, length, and strength of fibers obtained. Under the optimum conditions, the peeled bark (trunk) was dipped in 3% sodium hydroxide solution with a solution to bark ratio of 30:1 at boiling temperature. The water retting which is a microbial process of retting process, was conducted by placing trunk in water at room temperature for 26 – 28 days in a coated metal container or plastic container.

Methods

Experimental procedure

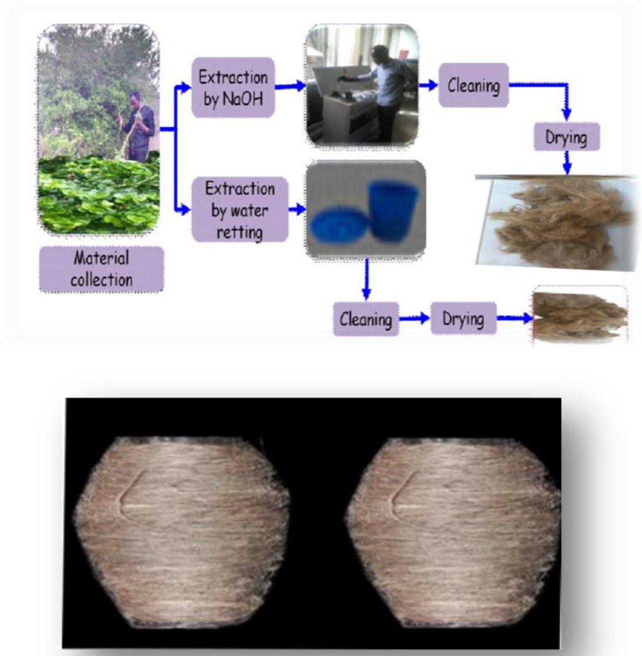

Fig. 1. Experimental procedure.

Characterization

The fiber properties of Carissa Edulise were investigated for their potential as a new source of textile fibers. The following tests were carried out.

i. *Fiber diameter*

Electron Microscope was used to measure the diameter of the fiber and to capture its surface morphology.

ii. *Fiber fineness*

The fiber fineness was determined according to ASTM D-1577:07 test standard. It was done by single-fiber weighing method. This test method is recommended for measurement of the linear density of single fibers and is not suitable for fibers shorter than 30 mm. The length of a single fiber was measured and the fiber was weighed.

iii. *Tenacity and elongation*

Tenacity of the fibers was tested using ASTM D- 3822:07 standards. The gauge length between the jaws was 5cm. The fiber was preconditioned at, 20 +/- 2°C and 65 +/- 2 % relative humidity. The fiber specimen was mounted in the jaws of the clamps.

All slack was removed without really stretching the specimen. Care was taken to keep the specimen straight within the jaws and it was ensured that the fiber sample lay on the line of action between the force-measuring device and the point where the fiber left the moving jaw face.

iv. *Fiber length*

The simplest direct way of measuring single fiber length by hand and the fiber alongside a rule tension applied should be sufficient to remove fiber crimp without stretching the fiber too much. 30 random samples from the extracted fibers for each extraction method were measured using meter scales.

v. FTIR *characterization*

IR spectra of the samples were recorded using the Perkin Elmer FTIR instrument in the frequency range 4000 - 500 cm-1 using 20 scans and recorded in the transmittance mode as a function of wave number.

vi. *Thermal characterization*

To analyze the thermal stability of extracted fibers, the Thermogravimetry (TG) technique was used. Tests were carried out using Perkin Elmer TGA 4000 model.

Measurements were conducted from ambient temperature to 50°C at a heating rate of 20°C/min in a nitrogen atmosphere to 500°C. Samples weights were approximately between 2 – 4 grams. To verify the accuracy of the results, two sample runs were performed under same experimental conditions.

III. Results and Discussion

Fiber Diameter

The morphology of fibers obtained from the single cells was observed using a Projection Microscope. The widths of the single cells were measured using a Projection microscope. The diameter or width of fiber is one of its most mandatory parameter of the fiber. Fiber diameter is usually measured with a Projection Microscope.

TABLE I. Fiber diameter of fibers by different extraction methods.

Parameter	Water retting	NaOH (3%) treated
Diameter (μm)	17.95	16.85

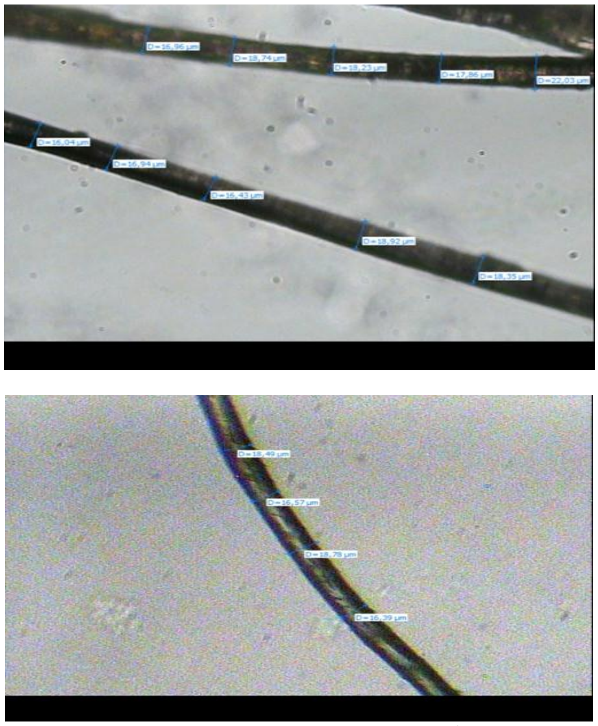

Fig. 2. Both water retting and chemical extraction fiber diameter.

The diameter of the fiber was measured as 17.95μm by water retting and 16.85μm by alkalization extraction. From the result, it can be seen that in alkalization treatment removable of non-fiber parts and some fiber parts is more than the water retting. The fibres were generally of almost equal diameter from end to end .The uniformity of fiber width is an important aspect in spinning quality and flexibility.

Fineness

Fineness or micronaire affects both the strength and irregularity of yarn. Fiber fineness also influences the Twist for maximum strength, luster, the drape and handle of fabrics.

TABLE II. Fiber Fineness of fibers by different extraction methods.

Parameter	Water retting	NaOH (3%) treated
Fineness (Tex)	0.65	0.58

"The fineness determines the number of fibers present in the cross-section of a yarn of given thickness". The linear density of the fiber was then calculated in Tex units. It was found to be 0.65Tex by water retting methods but in alkalization extraction fineness was 0.58Tex. The reason for this could be the chemical extraction method that removes non-fiber parts and some fiber parts which results in increasing fineness of the fiber.

Tensile Strength and Elongation

Tensile Properties or strength can be defined as the maximum stress a material can withstand without failing (breaking). Tensile strength is the maximum stress caused by a pulling force that a material can withstand without failing. Compression strength is the maximum stress caused by a pushing force that a material can withstand without crushing.

TABLE III. Fiber Strength and Elongation of fibers by different extraction methods.

No.	Parameters	Water retting	NaOH (3%) treated
1	Strength (g/Tex)	13.78	13
2	Elongation (%)	37.8	34.44

The Carissa Edulise stem fibers were conditioned in a standard testing atmosphere of 21°C and 65% relative humidity for at least 24 h before performing the tensile tests. The tensile tests were performed on bundle fiber. From the result, we can observe that increasing the concentration of the chemical decreases the tensile strength and elongation due to increase in the removal of some parts fiber and non-fiber parts but they give additional strength by integration within fiber.

Fiber Length:

TABLE IV. Fiber length of fibers by different extraction methods.

Parameter	Water retting	NaOH (3%) treated
Fibre length (mm)	21.97	19.49

The fiber length is the main quality parameter of raw textile material because length to width ratio is the primary requirement of any textile fiber. The fiber length range is usually suitable for producing for any count. From Table 4, we can understand that the length of fiber was decreased by the chemical method used which mean that the length not remained the same for both water retted and NaoH extracted fibers.

Moisture Content and Moisture Regain

Moisture content and Moisture regain were always determined at moisture equilibrium in standard atmosphere for textile materials.

TABLE V. Moisture content and moisture regain of fibers by different extraction methods.

No.	Parameters	Water retting	NaOH (3%) treated
1	Moisture content (%)	8.3	8.3
2	Moisture Regain (%)	9.09	9.09

It helped determine the percentage moisture regain by a dry sample of Carissa Edulise stem fibers.

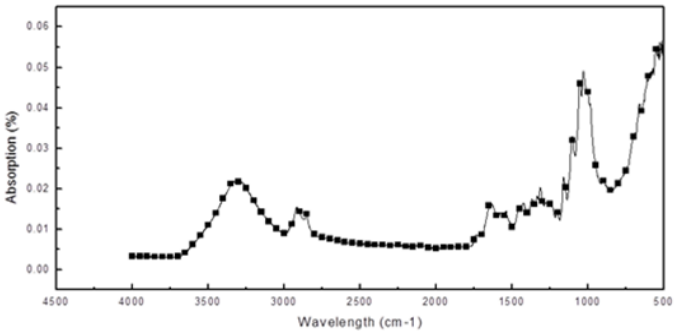

Fig. 3. FTIR curve of NaOH treated fiber.

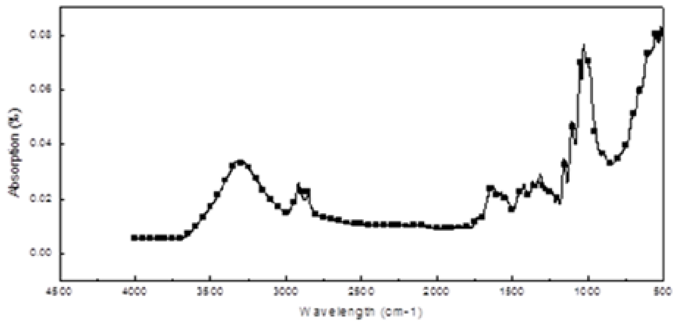

Fig. 4. FTIR curve of Water Retted fiber.

The moisture regain of the fibers was determined according to ASTM standard method 2654 using standard conditions of 21°C and 65% relative humidity. From the table, it can been seen that these moisture properties do no change with respect to retting method.

Fourier Transform Infrared Spectroscopy (FTIR)

FTIR - Fourier Transform Infrared - spectroscopy analysis and testing identifies chemical compounds in a wide range of capacities. The FTIR identifies chemical bonds in a molecule by producing an infrared absorption spectrum. The spectra create a profile of the sample, a characteristic molecular fingerprint that can be used to screen and scan samples for many diverse constituents. FTIR is an effective analytical instrument for detecting functional groups and characterizing covalent bonding information.

FTIR spectra with the standard peaks of Carissa Edulise fibers are shown in Fig. 3&4. A broad absorption peak at around 3350 cm-1 corresponds to the O-H stretching vibrations of alpha cellulose and hydrogen bond of the hydroxyl groups.

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It can be seen that water retted fibers had a higher peak than the alkaline extracted fibers. The peak at 2850 cm^{-1} is the characteristic band for alkyl C-H symmetric and asymmetric stretching vibrations of α-cellulose. Additionally, the peak at 1735cm^{-1} can be attributed to the carbonyl C=O stretching vibration mainly due to hemicellulose group, whilst the peak of C=C aromatic skeletal vibrations in lignin shows around 1550cm^{-1}. The peak at 1450 cm-1 is associated with the CH2 symmetric bending found in cellulose. The peaks at 1375 cm^{-1} and 1246 cm^{-1} correspond to the C-O stretching vibration of the acetyl group in lignin and hemicellulose component, respectively. Around 1050-1160 cm^{-1}, the absorptions can be attributed mainly to the carbohydrates (cellulose and lignin)[10]. On the whole, the FTIR peaks shows that these fibers compare well with other reported values for natural cellulosic fabric.

Thermal Characterization DSC and TGA

Thermo gravimetric analysis (TGA) is one of the members of the family of thermal analysis techniques used to characterize a wide variety of Materials. TGA provides complimentary and supplementary Characterization information to the most commonly used thermal technique; DSC.TGA measures the amount and rate (velocity) of change in the mass of a Sample as a function of temperature or time in a controlled atmosphere. The measurements are used primarily to determine the thermal of materials as well as their compositional properties. The technique can analyze materials that Exhibit mass loss or gain due to this indicates the behavior of the fibres at various temperatures. Burning characteristics of a fiber are important in determining care and use. [23] States that burning of small quantities may be used as a means of differentiating one fiber group from another. For example some fibres scorch and flame, others melt and flame or shrink. Some fibres are self-extinguishing, others are completely non-combustible. Fibres which are self-extinguishing are good for children, sleepwear and protective clothing.

IV. Conclusion

Natural cellulose Carissa Edulise stem fibers were investigated for their suitability for textile applications. Fibres were extracted from Carissa Edulise bast by chemical and water retting methods, and then the morphology of the fibres obtained and the Carissa Edulise bast fiber were all characterized. In terms of the extraction methods the non-cellulose contents in the Carissa Edulise bast were effectively removed by both water retting and Alkali retting. The Carissa Edulise stem fibres were comparable to other natural fiber such as cotton and flax in terms of some physical properties such as reasonably high tenacity and elongation at break. Also, since the Carissa Edulise plants were relatively easy to grow, they could be considered as an alternative source for natural cellulose fibres, and the fiber can be used to make products like apparel and packaging material (bags), carpet backing, ropes, yarns, paper application and wall decoration.

V. Acknowledgement

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