Extraction and Characterization of Ethiopian Hibiscus Macranthus Bast Fiber

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Abstract

Hibiscus macranthus is one of the Malvaceae family and genus Hibiscus plant, which grows mainly in the western part of Ethiopia. Hibiscus macranthus is the most adaptable and abundant plant in the nation. It is used as an ornamental plant, often a hedge or fence plant. It is also used as firewood after harvesting the stem together with the bark. The plant is also used as a fiber in the form of a rope for tying different kinds of things. However, Hibiscus macranthus plant fiber has not been commercially exploited and extracted properly. This study describes the possibility of mechanical and retting methods of Hibiscus macranthus fiber extraction and characterization. Hibiscus macranthus fiber is a bast fiber obtained naturally from the stem or stalks of dicotyledonous plant since it is a natural cellulose plant fiber. And the fiber was characterized by studying its physical and chemical properties. The physical characteristics were investigated as follows: including length of 100-190mm, fineness of 1.0-1.2Tex, diameter under X100 microscopic view 16-21 microns, moisture content of 12.46% and dry tenacity of 48-57 cN/Tex along with breaking extension of 0.9-1.6%. It was observed that out of 12-18% of the stem, more than 65% is primary long fibers, which is an indication of Hibiscus macranthus' fiber productivity. The fiber separation methods prove decreasing of non-cellulose ingredients in the order of mechanical, water and chemical methods. The color measurement also shows that the raw Hibiscus macranthus fiber has a natural golden color according to YID1925 and paler look under both retting methods than mechanical separation. Finally, it is suggested that Hibiscus macranthus fiber can be used for manufacturing of natural and organic crop and coffee packages as well as super absorbent, fine and high tenacity textile products.

Keywords: Hibiscus Macranthus; Bast Fiber; Extraction; Characterization; FT-IR

I. INTRODUCTION

Natural cellulose fibers have successfully proven their qualities when also taking into account an ecological view of fiber materials [1]. Nature in its abundance offers us a lot of materials that can be called fibrous. Plant fibers are obtained from various parts of plants, such as the seeds (cotton, kapok, milkweed), stems (flax, jute, hemp, ramie, kenaf, nettle, bamboo), and leaves (sisal, manila, abaca), fruit (coir) and other grass fibres. Fibres from these plant parts can be considered to be totally renewable and biodegradable [2]. Bast fibers, i.e. flax, jute, hemp, ramie, kenaf, and nettle are soft woody fibers, which are obtained from stems or stalks of dicotyledonous plants. The fibers occur in bundles or aggregates [1]. The bundles consist of 10 up to 25 elementary fibers, with the length of 20 up to 25mm and a diameter of 10 up to 50µm. The bundles are connected by lateral ramification, which forms a three-dimensional network. The elementary fibrils and bundles are cemented by lignin and pectin inter-cellular substances, which must be removed during the processing of fibers extraction [3]. More intense retting separates the fiber bundles in the elementary fibers that can be several centimeters long. Often bast fibers have higher tensile strength than other kinds, and are used in high-quality textiles blended with natural or synthetic fibers. Currently, bast fibers are raw materials not only used for the textile industry but also for modern environmentally friendly composites. They are also used in different areas of applications like building materials, particle boards, insulation boards, food, cosmetics, medicine and are a source for other bio-polymers [1].

Family *Malvaceae* is one of the most important families consisting of 82 genera and 1,500 species with *Hibiscus* over 200 species, *Sida* 200 species, *Ablution* 190 species and *Malva* 40 species. The family is present across the globe, but is mostly represented in the tropical and subtropical regions [4]. *Hibiscus macranthus* is just one of many hundreds of plants that are found within the *Hibiscus* genus, and it is native to West-Central Africa, more particularly, Cameroon. In many cultures throughout West Africa, *Hibiscus macranthus* has been used to help boost male fertility. It has also been used to help regularize the menstrual cycle for women as well as address infertility in female subjects. It is essentially a supplemental herb which is derived from the calyxes of the plant, i.e. the collection of sepals that separate the stem from the flower [5]. There have been other studies that have focused on usages of the plant for increasing body weight, strength, and even energy levels [6]. Many research studies were conducted on the parts of the plant used primarily for medicinal purposes including fresh and dried epicalyxes and

calyxes. Unfortunately, the stem of *Hibiscus macranthus* plant currently has no commercial value, despite the fact that it contains bast fibers. There is little reported about *Hibiscus macranthus* bast fibers.



Figure 1: Hibiscus macranthus plant grown in Ethiopia

Ethiopian *Hibiscus macranthus* belongs to the *Malvaceae* family. Its genus name *Hibiscus* is used for the production of bast fiber. Its vernacular name in the Amharic speaking region is 'Tug' while in Affan Oromo, it is known as 'Inchiny'. The plant is an annual erect, mostly branched, woody, herbaceous sub-shrub that grows mainly in all warm and humid climatic regions of the nation. It grows to 1.5-2.5m in height at maturity and has a deep penetrating root. It has a smooth or nearly smooth, cylindrical, typically dark or light green to red or brown stems. The leaves are green with reddish veins and long or short petioles. Leaves of young seedlings and upper leaves are deeply lobed (five to seven or even nine lobes) and the margins are toothed. Flowers are borne singly in the leaf axis and yellow or buff with a deep purple center. The typically red calyx consists of large sepals with a collar of slim pointed bracts around the base.

In general, it is a crop grown, adaptable and abundant plant in the western part of Ethiopia around Wellega in rivers and wet areas as compared to other regions in the nation. Most of the local farmers use the plant as an ornamental plant, often a hedge or fence plant. It is also used as a fiber in the form of a rope for tying different kinds of things and for making nets and baskets since it has good strength. It is also used as a firewood after harvesting the stem together with the bark. *Hibiscus macranthus* fiber is produced from the stem of *Hibiscus macranthus* plant as natural bast fiber; however, the fiber has not been commercially exploited and extracted properly. Traditionally, local farmers use two different methods to extract the fiber; the first method is putting the stem in water for two weeks and the other method is putting the stem in mud for three weeks to completely separate the fiber from the plant stem. In both separation methods, drying takes place using sunlight, but the fiber changes its color and the degree of whiteness depends on the type of separation methods. Once separated, the bast fibers are ready for spinning and weaving into textiles, or for pulping into high quality pulp. Bast fibers are ideal for specialized paper products such as industrial filters, currency paper, tea bags or cigarette paper [7, 8].

A. Fiber Extraction

The fibers are embedded in the cells of bast that lies between the outer bark and woody core of the stem. The spinnable fibers are obtained by decortication and degumming. It is not normally possible to spin the fiber with 20-30% adhered gum, which therefore, needs to be removed by the process of degumming [9]. For textile purposes, only 6% residual gum is recommended, which makes the filament well separated and fiber characteristics fully realized [10]. Several techniques are used for extraction of conventional bast fiber. In mechanical separation, fibers are separated by hammer mill or the dried stem stalk is decorticated mechanically to separate the skin from the wooden material. Scutching and hackling will be the next operations for complete separation of fiber from epidermic constituents. However, mechanical separation doesn't involve any retting processes and it also produces massive quantities of short fiber in a short time [11]. On the other hand, retting is controlled rotting to remove gummy

(pectinous) substances which glue fibers together. Retting is sometimes termed as degumming because degumming is done to remove gums and pectins from decorticated fibers either by chemical or by microbial means [10]. Retting is defined as the process of separating the embedded fiber from the stem stalk or it is a process of separating fibers from non-fiber tissues in plants through a number of ways [8].

Various methods of degumming or retting have been reported in the literature for removal of gums from crude fibers by microbial or biological means: One is dew retting which relies on indigenous soil fungi to colonize the stem or bast and to degrade pectin and hemi-cellulose (particularly the arabinose) by releasing of polygalacturunes and xylanase. The resulting fiber is often coarse and of variable quality. Dew retting has the advantage of pectin material which could be removed easily by bacteria. However, it has some drawbacks such as lower strength, low and inconsistent quality, restriction of certain climatic changes and product contaminated with soil. Despite good quality of fibers, dew retting is usually replaced by other more economical methods because the process is very time-consuming and weather dependent [11]. The second method is water retting, which is performed in an aqueous environment and anaerobic, pectinolytic bacteria are responsible for the decomposition of pectin substance and the subsequent release of fiber by conducting in rivers or pools through bacterial action within two up to three weeks. This process consistently yields high quality and uniform fiber, but also has some disadvantages such as extensive stench and pollution arising from anaerobic bacteria fermentation of the plant [11]. The last, but not the least, is enzymatic retting. It is a process is the most suitable method to reduce the amount of lignin in the fiber and it is a faster and cleaner process. The drawback of enzymatic process is low fiber strength [11].

The other method of degumming or retting is chemical retting for removing non-cellulose material attached to the fibers to release individual fibers. It is performed by placing the stem stalks in chemical solutions like sodium hydroxide, sodium carbonate, soaps or mineral acids. The process takes only a few hours of retting, but it has a drawback of low quality, low strength of fiber and unfavorable color; it also has a high processing cost.

Chemical retting is most efficient and can produce clean and consistent long and smooth surface fiber within a short time [11]. Instead of atmospheric retting, chemical methods or enzyme retting with pectinases, hemi-cellulose and cellulose are used. However, fiber properties depend on extraction conditions significantly. The differences between the procedures are not only in expenses and process duration, but the most important thing is quality and uniformity of retted fibers.



Figure 2: Plant stems preparation for fiber extraction

Most bast fibers are cemented to the adjacent cells inside the stem with pectin (a form of carbohydrate), which can be extracted by retting processes. After harvesting, the stems are usually kept either in the field or under water for up to three weeks, during which the pectinous substances that bind the fiber with other plant tissues are softened and degraded by microorganisms. A quality of fiber is largely determined by the retting condition and duration [8]. Following retting, a sequence of processes to remove the fibers from the woody stalks is carried out in the sequence of breaking the stem stalks, then scraping or scutching them off. Finally, hackling is a combing process to separate long fibers from short fibers and to remove the remaining woody material.

II. MATERIALS AND METHODS

A. Materials

- 1) Plant stem: The stem stalks used in this research works were obtained from the plant of *Hibiscus macranthus* grown in Wellega area, which is 370 km from Addis Ababa, Ethiopia. Stem stalk harvesting was done immediately when the plants were three months old. As time passes, the lignin part will grow fast and the fibers become coarse and inflexible [8, 10, 12]. Accordingly, the plant stem was collected in the first week of October 2016 by cutting the stem 10 cm above the ground level and 20 cm below the tip to obtain good fiber quality. The collected stems were laid down in an open field to sundry for 10 consecutive days and then stored under moderate room temperature.
- **2)** Chemicals and reagents: All chemicals, reagents and solvents used are listed as follows: water, caustic soda (NaOH), sodium sulphate (Na₂SO₃), ethylenediaminetetraacetic acid (EDTA), benzene, sulfuric acid, ethanol and methanol.
- **3)** Equipment: The apparatus utilized during the study included thermometer, flasks, digital balance, drying oven, beakers, glass stirring rods, conditioner chamber, ruler, vibroscope, ETADRY, biological microscope (video analyzer), shirley trash analyzer, FT-IR, reflectance spectrophotometer, K1HS 0265 single fiber strength tester with the speed of 0.01 to 1000 mm/min, soxhlet extractor, filtering flasks, crucibles, siphon tube and 10 mesh stainless steel sieves.

B. Methods

In this study, mechanical separation, water and chemical retting were done to separate fiber from the stem of the plant. In mechanical separation method, the plant stems were harvested and collected in the field after three months from its germination period. The collected stems were exposed to sunlight for 10 consecutive days in an open field until they completely dried. However, the drying conditions were not too prolonged because the fiber gets damaged by sunlight. As a result, its strength is reduced. After drying, the stems were rubbed by a rough material like soft emery paper, leather or thick plastic gloves in order to make the surface smoother and to remove needles on the surface. Then, the bast fibers were separated from the stem by either pilling from top to bottom or decorticating the stem using a hammer. Scutching is followed for further separation of skin from woody material and then hackling or combing to completely separate the long fiber from the short one.

In water retting, water is prepared in a bath for laying and dissolving the epidermic layers of the stems after harvesting the plant stems. The natural decay process removes the bark and separates the long bast fiber from the core or stick. In this method, 10 kg plant stem stalks were prepared and placed in 500L retting water using bath for 14 consecutive days; at the end of this period, the stems get rinsed. The fibers were then extracted from the stem after consecutive washing and completely dried under sunlight, but in a shaded area. In order to get good results, the minimum ratio of plant material weight in kg to liquor volume in liter is **1:20** by volume. Some dried fibers were dyed and used to weave baskets, and the remaining dried fibers were characterized.

In chemical retting, the plant stem stalks were immersed in a chemical solution of 1.0% NaOH, 0.5% Na₂SO₃ and 0.05% EDTA at 10-10.5 PH range after the stem bark is scraped out and cut into pieces. This batch retting method was used as a combination of methods applied by Kundu *et al.*, (1996) [10] and Ramaswamy *et al.*, (1994) [13] which follows no pre-treatment and treating in 1.0% NaOH, 0.5% Na₂SO₃ and 0.05% EDTA at 100°C for 60 min and rinsed for 10 min at 60°C, then after treatment in 2.0% CH₃COOH for 10 min at 60°C. After the batch retting process, the epidermis, wax, color and woody materials were removed by two dunks in hot water at 60° C [14]. The fibers were then submerged under the water and dunked 10 times by hand before being removed and hydro extracted to remove the excess water. After the two dunks the fibers were exposed to dry under sunlight for two hours. The dried batch was then subjected to beating to remove significant lignin, bark and woody portions of the stalk. Whereas, opening of extracted fibers to separate short fibers and long fibers is done to remove short fibers and straighten long fibers [15]. The fibers were then characterized, and some of the dried fibers were dyed and used to weave baskets.

The fibers were removed from the clean fiber box and left open in a standard atmosphere laboratory for three days to be conditioned. Tests were then carried out on the fiber. Fifty numbers of fibers were selected randomly and tested for each fiber's physical property tests as per extraction methods. Five samples of fiber were also tested for moisture content by taking 50g sample in each test. The mean value of every test is described in *Table 1* and all tests were done in Bahir Dar University, EiTEX laboratory rooms at 65% relative humidity (R.H%) and 21°c room temperature (T°).

The fiber characterization process includes the investigation of fiber length, fiber fineness, fiber strength, moisture content, color values, morphology and chemical constituents. The main objective of this study is to evaluate the physical fiber properties of genotype of the same plant species. The fineness was determined by using vibroscopic methods and measurement of fiber diameter was done using a video analyzer biological microscope, while the fiber strength and elasticity properties were investigated using a single fiber tensile testing machine by measuring breaking tenacity and elongation at break of the fibers in the standard methods of SFS-EN ISO 5079 (ISO 5079 1995 (E)), which has been designed for measuring tenacity of textile fibers. Moisture content of the fiber was determined using an oven ETADRY machine as per the standard of ASTM 2654-76 at 105°c heating temperature until constant dry mass of fiber was achieved. The color values of the fibers were also indicated by D1925 yellowness index. The measurement was done using color eye 3100 instrument where the sample is measured 10 times using optimatch 6.5 color software and then averaged the color data.

A study of the chemical functional groups was done using Perkin Elmer Spectrum to Fourier Transform Infrared Spectroscopy (FT-IR) as illustrated in *Figure 5*; however, a brief mention of molecular structure and position of infrared absorption bands serves to orient the reader to the useful areas of the electromagnetic spectrum. Much information about the structure of a compound was deduced from its infrared spectrum since many of the individual bands occurring in the infrared spectrum are characteristics of specific pairs or groups of atoms [16]. Determination of the amount of cellulose, hemi-celluloses, wax and pectin content present in the fiber was also undertaken using ASTM 1695-77 as a chemical constitute of the fiber. Likewise, TAPPI T222 in accordance with ASTM D1106 was used for acid insoluble lignin determination [10,16,17], and the hemi-celluloses are then dissolved out of the remaining holo-cellulose by treatment with dilute alkali. The final insoluble residue is the α -cellulose constituent, which invariably contains traces of sugar residues other than glucose [18].

III. RESULTS AND DISCUSSIONS

A. Use and Productivity of the Fiber

Hibiscus macranthus bast fiber can be dyed with different colors and forms cords after it's spun into yarns which can be used to weave ladies' bags or baskets as shown in *Figure 3*. Stem yield and fiber content in stem determine the bast fiber yield. The overall bast fiber content is 12-18% of the stem. *Hibiscus macranthus* bast fiber consists of more than 65% primary fiber and the rest is valuable secondary short fiber.



Fig 3: Use of fibers to make cords and baskets

B. Longitudinal View of the Fiber

The longitudinal view showed some crack-like lines along the length of the fiber, which could be an indication of fiber clusters being held together by gummy material or clusters of fibres are held together by some non-fibrous materials. The fiber exhibits fibrilliar structure as other bast fibers like jute, hemp and ramie [8, 10]. The surface morphology of the fiber was viewed using video camera integrated microscope; the result is presented in *Figure 4*. The diameter of the fiber can be measured directly from the captured picture and it is found that the fiber has a mean diameter of 16.61-20.77 microns.



Fig 4: Longitudinal view of fiber (x100)

C. Physical and Mechanical Characteristics of the Fiber

Hibiscus macranthus fibers extracted, as explained in the experimental section, were investigated for three different retting methods of the following properties. The fiber length is measured manually using a simple rule and it ranges from 100mm up to 190mm. The ETADRY test reveals that the fiber has 12.46% moisture content and hemicelluloses are mainly responsible for moisture sorption. Hence, the accessible cellulose, non crystalline cellulose, lignin, and surface of crystalline cellulose also play major roles [18].

The fiber fineness was determined using video analyzer biological microscope with respect to the fiber diameter and using vibroscopic method regarding the fiber fineness in Tex. The fineness of the fiber seems to be high in mechanical separation as compared to other methods, which could be due to the fact that the fiber is not degummed and the remaining adhering bark increases the linear density of the fiber [18].

The dry tensile strength and breaking extension of fibers were measured using single fiber strength testing in cN/Tex and its result seems to be high in mechanical separation. But extensive extraction of lignin on chemical retting resulted in increasing flexibility and decreasing young's modulus of the same fibers [18].

The color strength value (K/S) of the fiber measured and its result decreases respectively as illustrated in *Table 1*. This indicates that the fibers became whiter, so does yellowness. The color changes are associated with the lignin content of the fiber because the isolated α -cellulose and hemi-cellulose fractions are unaffected by exposure to UV light of the correct wavelength band. And it is evident that the intensity of yellowing became more pronounced as the lignin content increased [18].

Parameter	Mechanical Separation	Water Retted	Chemical Retted
Fiber Fineness (Tex)	1.2	1.13	1.08
Tenacity (cN/Tex)	57.2	50.97	48.6
Elongation at Break (%)	0.91	1.22	1.64
YID 1925	59.9	44.52	35.16
K/S Values (at 420 nm)	3.432	2.032	0.643

Table 1: Physical properties of fibers obtained through different methods

D. Chemical Composition and Characteristics of the Fiber

The lower percentage of cellulose and higher hemi-cellulose may affect some physical and chemical characteristics of the fiber. Due to degumming, hemi-cellulose, lignin, pectin, ash, fat and wax decrease. However, holo-cellulose and α -cellulose are found to increase [10, 16, 17]. Hemi-cellulose has a lower degree of polymerization. As a result, it has poor resistance to acids and alkalies. However, cellulose has good resistance to alkalies, but can be attacked by strong acids. Lignin, binding cellulose and hemi-cellulose together, has an adverse effect on mechanical properties [9, 16, 17].

Ingredients	Mechanical Separation	Water Retted	Chemical Retted
Cellulose Content (%)	51.92	64.67	86.42
Hemi-Cellulose Content (%)	18	15.28	8.81
Lignin Content (%)	17.18	12.16	3.02
Pectin Content (%)	1.04	0.58	0.32
Fat and Wax Content (%)	0.53	0.375	0.18

Table 2: Chemical composition of fibers obtained through different methods

The chemical contents of a bast fiber can affect the mechanical properties of the fiber [19] and is exemplified in *Table 1. Hibiscus macranthus* fibers, like other bast fibers, are considered to be a natural composite material where cellulose is the main weight bearing component and hemi-cellulose and lignin are the matrix. In particular, the fiber is consisting of fibrillate in a matrix of lignin [12]. The chemical composition of fiber is given in *Table 2*. The cellulose content of chemically retted fiber seems to be high as compared to the other methods. FT-IR analysis, illustrated in *Figure 5*, revealed that *Hibiscus macranthus* fiber is a mixture of cellulose, hemi-cellulose, lignin, gum and other components. Pectin is a heterogeneous group of acidic structural polysaccharides that have complex structures. The spectra are recorded in the absorbance mode as a function of wave number given in cm⁻¹. The spectra of *Hibiscus macranthus* fibers exhibited as follows, peaks at 3326.7 cm⁻¹ could be attributed to bond of O-H stretching. The peak at 1024 cm⁻¹ could be attributed to the bending groups. The peak at 1735.2 cm⁻¹ could be ascribed to C=O stretching for acetyl groups in hemi-cellulose and for aldehyde groups present in lignin according to Favaro *et al.*, (2010) [20]. While, the vibration peak at 1248.4 cm⁻¹ is to C-O stretching vibrations. Other minor peak at 2888.3 cm⁻¹ could be ascribed to C-H stretching. Also, minor peak at 1380 cm⁻¹ could be ascribed to bending vibrations.

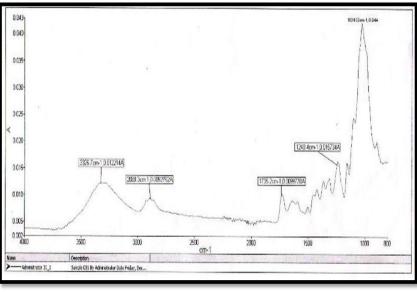


Fig 5: FT-IR results for Ethiopian hibiscus macranthus fiber

IV. CONCLUSION

Extraction and characterization of *Hibiscus macranthus* bast fiber grown in Wellega, Ethiopia were undertaken. From the above results, it can be concluded that the different *Hibiscus macranthus* fiber extraction or separation processes lead to fibers, which exhibit different physical, mechanical and chemical characteristics. The physical and mechanical properties of *Hibiscus macranthus* fiber suggests that it can be used as a bast fiber, which is adequately supported by the presence of chemical functional groups, which are also present in another bast fiber. The fiber is strong enough to meet the needs of textile processing such as spinning and weaving. Some of the chemical functional groups were similar to those found in kenaf fibers. It is also necessary to suggest that the *Hibiscus*

macranthus fiber can be an ideal raw material for making eco-friendly organic packages of crop and coffee as well as super absorbent, fine and high tenacity products; however, the retting method is the predominant challenge in the application of *Hibiscus macranthus* bast fibers.

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