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Separation and Characterization of Ethiopian Origin Nettle Fiber

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***Abstract*— This study of work describes the possibility of mechanical and retting methods of nettle fiber extraction and characterization. Wild stinging nettle, binomial name, *Urtica Dioica*, is most adaptable and abundant plant in the nation. Nettle fiber productivity was observed that 19-21% of the stem out of which 70% are primary long fibers. It exhibits the following physical properties such as a length of 5-75 cm, fineness 1.0-1.2 tex, diameter under X400 microscopic view 5- 6 microns, a moisture content of 12.3% and dry tenacity of 0.4-0.6 N/tex along with breaking extension of 2.0-2.5 %. The fiber separation methods prove decreasing of non cellulosic ingredients in the order of mechanical, water and chemical methods. The colour measurement also shows the raw nettle fiber has a natural goldish color according to YID1925 and paler look under both retting methods than mechanical separation. Finally, it is suggested that nettle fiber can be utilized for manufacturing of natural and organic crop and coffee packages as well as super absorbent, fine and high tenacity textile products.**

Keywords—Nettle, Retting, Cellulose, Tenacity, Fineness

I. INTRODUCTION

Bast fibre or skin fibre is a plant fibre collected from the phloem (the "inner bark" or the skin) or bast surrounding the stem of certain, mainly dicotyledonous, plants. They support the conductive cells of the phloem and provide strength to the stem. In the phloem, bast fibres occur in bundles that are glued together by pectin and waxes [1,2]. More intense retting separates the fibre bundles in to elementary fibres that can be several centimeters long. Often bast fibres have higher tensile strength than other kinds, and are used in high-quality textiles (sometimes in blends with cotton or synthetic fibres), ropes, yarn, paper, composite materials. A special property of bast fibres is that they contain a special structure, the fibre ***node***, which represents a weak point. Seed hairs, such as cotton, do not have nodes .

Stinging nettle or common nettle, *Urtica Dioica*, is an herbaceous perennial flowering plant, 1 to 2 m tall in the summer and dying down to the ground in winter. The plant has many hollow stinging hairs called *trichomes* on its leaves and stems, which act like hypodermic needles that inject histamine and other chemicals and possibly formic acid, that produce a stinging sensation when contacted by humans and other animals [1-2].

Stinging nettle is a powerhouse of nutrients. It contains on average 22% protein, 4% fats, 37% non-nitrogen extracts, *9-21% fiber*, and 19-29% ash. The leaves contain about 4.8 mg chlorophyll per gram of dry leaves, depending on whether the plant was grown in the sun or shade. Surprisingly, more chlorophyll and carotenoids are found in plants that have been grown in the shade. The dried leaf of nettle contains 40% protein [1-2, 9].

Nettle stems contain a bast fibre that has been traditionally used for the same purposes as linen and is produced by a similar retting process. Unlike cotton, nettles grow easily without pesticides. The fibres are coarser however. In recent years, a German company has started to produce commercial nettle textiles. Nettles may be used as a dye-stuff, producing yellow from the roots, or yellowish green from the leaves [1-3].

The production of the primary bast fibres has traditionally been a very labour intensive process, but recently an alternative fibre separation process has been developed using technologies such as *ultrasound* and *steam explosion*, which are much less labour intensive. Once separated the bast fibres are ready for spinning and weaving into textiles, or for pulping into high quality pulp. Bast fibres are ideal for specialized paper products such as industrial filters, currency paper, tea bags or cigarette paper [2,3].

II. FIBER SEPARATION

Mechanical separation doesn't involve any of retting processes. 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 epidermical constituents.

On the other hand, retting is a controlled rotting to remove gummy (pectinous) substances which glue fibres together. Retting is sometimes termed degumming. It is a chemical process for removing non cellulosic material attached to the fibres to release individual fibers. So, retting is defined as the process of separating the embedded fibre from the nettle stem, which can be carried out in a number of ways [3]: water retting, enzyme retting when some enzymes can digest the outer cover of the stem and fibers can be separated easily [6-7] and in chemical retting, stem stalks are placed in chemical solutions [3].

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 2 to 3 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 [3].

Following retting, a sequence of processes to remove the fibres 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 fibres and to remove the remaining woody material.

III. METHODOLOGY

Nettle stems, harvested after three months from germination, are exposed to sun light for 10 days and become dried completely. But, this drying condition has not been too prolonged because the fiber is damaged by the sun light and ultimately its strength is reduced. Dried nettle stems are rubbed by leather material or thick plastic glove. This is done to remove surface stinging needles. Bast fibers are separated from the stem by either pilling from tip to bottom or decorticating the stem and followed by scutching then hackling (or combing).

Water retting dissolves epidermical layers and followed by washing to remove easily. 10 kg nettle stem stalks were retted in 500L water for 10 days and at the end of the day stems get rinsed. The minimum ratio of plant material weight in kg to liquor volume in liter is **1:20** by volume to ensure good results.

Whereas, NaOH retting is immersing stem stalk in 1% NaOH, 0.5 % Na_2SO_3 and 0.05% EDTA and PH 10-10.5. The bark of the stem is first scraped out and cut into pieces. So, this batch retting method was used a combination of Kundu et al and Ramaswamy et al [5, 6] which follows no pretreatment and treating in 1.0% NaOH, 0.5 % Na_2SO_3 and 0.05% EDTA at 100°C for 60 min and rinsing for 10 min at 60°C, then after treatment in 2.0% CH_3COOH for 10 min at 60°C.

Drying is necessary to prevent further fermentation. In chemical retting process, it becomes important to establish the uniformity of the fibre quality from the base to the tip of the plant.

After the batch retting process that removes epidermis, wax and colour were removed by two dunks in hot water at 60°C. The fibre was submerged under the water and dunked 10 times by hand before it was removed and hydro extracted to remove the excess water. After the two dunks the fibre was subjected for drying exposing under sun light for 2 hrs.

Then, the dried batch is subjected to beating step to remove significant lignin, bark and woody portions of the stalk. Whereas opening of extracted fibers to separate short and long fibers is done so as to remove short fibers and straighten long fibers [4]. The fibre was opened twice after drying using Shirley Trash Analyzer MK2 F102.

Tests was then carried out on the fibre removed from the clean fibre box from both runs. The opened fibre was conditioned in a standard atmosphere laboratory for 2 days before any testing was carried out.

The functional group analysis was done by using PerkinElmer spectrum two Fourier Transform Infrared Spectroscopy (FTIR) as illustrated in fig.2. Correspondingly, chemical constituents of nettle fiber are determined using ASTM 1695-77 to estimate %weight of cellulose, hemi cellulose, waxes and pectin content. Likewise, TAPPI T222 in accordance with ASTM D1106 was employed for acid insoluble lignin determination [5, 7-9].

Moisture content of the nettle fiber was determined using an oven ETADRY as per the standard of ASTM 2654-76 at a temperature of 105°C heating to constant mass. Since the main objective of the present study was to evaluate the mechanical fibre properties of genotypes of the same plant species, the parameters fibre strength and fibre elasticity were determined by measuring breaking tenacity (cN/tex) and elongation at break (%) of the fibres using the standard method SFS-EN ISO 5079 (ISO 5079 1995(E)), which has been designed for measuring tenacity of textile fibres. For each fiber batches 100 fibers were investigated though only the mean value is described in table 2. The nettle fiber has been also indicated by the D1925 Yellowness Index.

Fiber characterization includes laboratory tests conducted for moisture content, fiber length, fiber fineness, fiber strength, colour values, morphology and chemical constituents. Four samples of nettle fiber each 50 g are tested for fiber moisture content and 50 fibers are tested for each fiber physical property tests. All these tests are done in Bahir Dar University, EiTEX laboratory rooms where the relative humidity (R.H %) and temperature (T°) are estimated as 65 % and 21°c, respectively.

IV. MATERIALS FOR EXPERIMENTS

A. Plant Stem

Stem stalk harvesting should be done immediately as the plants are three months old. As time is gone, the lignin part will grow fast and the fibers become coarse and inflexible [3, 5, 8]. Accordingly, the stinging nettle was collected in the first week of October 2013 from different regions of Ethiopia by using local crop harvesting tool ‘Machid’. Stems are cut 10cm above the ground level and 20 cm below the tip as the fibers beyond this range are poor quality. They were left for sundry for 10 days and then stored under moderate temperature room.

B. Chemicals and Reagents

All chemicals, reagents and solvents used are listed as follows: water, caustic soda (NaOH), sodium sulphite (Na_2SO_3), ethylenediaminetetraacetic acid (EDTA), benzene, sulfuric acid, ethanol and methanol.

C. Equipments

Thermometer, flasks, digital balance, drying oven, beakers, glass stirring rods, conditioner chamber, ruler, vibroscope, ETADRY, biological mmicroscope (video analyzer), FTIR, reflectance sspectrophotometer, K1HS 0265 single fiber strength tester with the speed of 0.01 to 1000mm/min, soxhlet extractor, filtering flasks, ccrucibles, siphon tube and 10 mesh stain less steel sieves were apparatuses utilized during the study.

V. RESULTS AND DISCUSSION

A. Fiber Productivity and Content

Stem yield and fibre content in stem determine the bast fibre yield. The overall bast fibre content is 19-21% of the stem. Nettle bast fibre consists of 70.0% primary fibre and the rest valuable secondary short fibre [2,3].

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Fig. 1 Longitudinal view of nettle fiber (X400)

B. Longtudinal View

The fiber exhibits as fibrillar structure as other bast fibers like hemp, jute and ramie [3-5]. Video camera integrated with microscope gives the following morphology which is illustrated below in the fig.1. The diameter can measured directly from the pictured captured. It is found that nettle fiber has a mean diameter of 5-6 microns.

C. Chemical Composition of Nettle Fiber

FTIR analysis, illustrated in fig.2, revealed that nettle fiber is a mixture of cellulose, hemicellulose, lignin, gum and other components. Pectin is a heterogeneous group of acidic structural polysaccharides that have complex structures.

It is commonly recognized that nettle fiber consists of linear macromolecules formed by β- D-anhydroglucose units linked by 1, 4-glucosidic bonds [7, 11]. The IR spectra of mechanically separated nettle fiber is shown in fig. 2.

The spectra are recorded in the % transmission mode as a function of wave number given in cm^{-1}. The spectra of the nettle fibers exhibited O-H absorption around 3300 cm^{-1}**,** C-H stretching around 1450 cm^{-1} and 2900-3000cm^{-1}, and C-O-C stretching around 1100cm^{-1}. There are also a series of peak in the 1300 to 1600 cm^{-1} which can specify the stretching carbonyl (C=O) for acetyl groups in hemicellulose and for aldehyde groups present in lignin according to Favaro et al and Abdul Khalil et al., 2010.

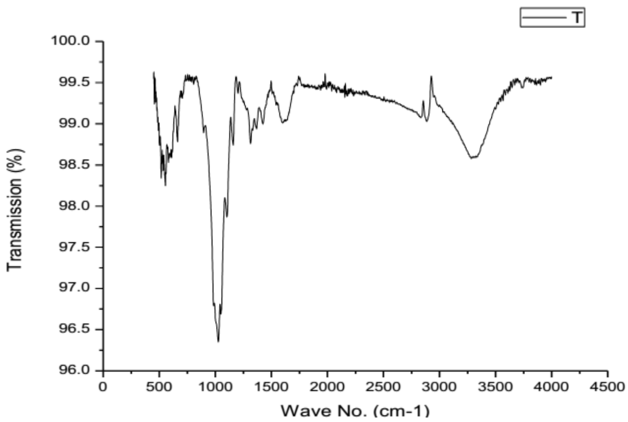

Fig.2 FT-IR transmission spectra of nettle fiber

These absorption bands are consistent with those of a typical cellulose backbone. In the structure of not retted nettle fiber, there are some other non-cellulose compositions such as hemicellulose, pectin and lignin which contain chemical bonds such as O–H, C–O–C, C–H etc.

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, holocellulose and α-cellulose are found to increase [5, 7, 9]. See also table1.

Hemicellulose has lower degree of polymerization. As a result, it has poor resistance to acids and alkalis. However, cellulose has good resistance to alkalis but can be attacked by strong acids. Lignin, binding cellulose and hemi-cellulose together, has adverse effect on mechanical properties [7-9, 11].

Table 1: Chemical composition of nettle fiber (%w/w)

Ingredients	Not retted	Water Retted	NaOH Retted
Cellulose (%)	80.27	95.35	96.42
Hemicellulose (%)	9.53	2.84	2.01
Lignin (%)	1.16	0.38	0.03
Fats and waxes (%)	0.36	0.27	0.21
Pectin (%)	1.82	0.63	0.41

The chemical contents of a bast fiber can affect the mechanical properties of the fiber (Mukherjee and Satyanarayana, 1984) and exemplified in table 2. Nettle fiber like other bast fibers is 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 fibrillae in a matrix of lignin [8].

D. Fiber Quality

- The fiber length is measured manually using simple ruler and it ranges from 5 to 75 cm.
- The ETDRY test reveals that raw nettle fibers unveil a moisture content of 12.3%.
- The fiber fineness is determined using video analyzer biological microscope with respect to the fiber diameter and using vibroscopic method regarding the fiber fineness in Tex as illustrated in table 2.
- The colour strength value (K/S) goes on decreasing and this indicates that the fibres become whiter, so does yellowness.

Table 2: Physical properties nettle fibres obtained in different methods

Parameter	Not Retted	Water retted	NaOH retted
Fiber fineness, tex	1.17	1.08	1.05
Tenacity (N/tex)	0.553	0.473	0.434
Elongation at break (%)	2.159	2.410	2.510
YID1925	59.9	45.25	34.56
K/S values (at 420 nm)	3.432	2.043	0.654

VI. CONCLUSION

Thus study confirmed that the possibility of nettle fiber separation from the stem by employing various methods. Besides, nettle fiber exhibits good chemical composition, physical and mechanical properties to be used as a textile raw material for making clothing, paper, bags and other eco-friendly organic packages. Now, it is also the time to suggest that the nettle fiber can be an ideal raw material for making suitable packages of crop and coffee as well as super absorbent, fine and high tenacity products. Composite and paper industries can develop their eco friendly products from nettle fiber, too [10].

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