Journal of Materials Science Research and Reviews

2(2): 169-175, 2019; Article no.JMSRR.45087

Extraction and Characterization of Bast Fibres from Roselle (*Hibiscus sabdariffa)* Stem for Industrial Application

E. U. Akubueze[1*], C. S. Ezeanyanaso[1], O. S. Muniru[1], F. C. Nwaeche[1], M. I. Tumbi[1], C. C. Igwe[1] and G. N. Elemo[1]

[1]Department of Chemical, Fibre and Environmental Technology, Federal Institute of Industrial Research Oshodi, P.M.B. 21023, Ikeja, Lagos, Nigeria.

Authors' contributions

This work was carried out in collaboration between all authors. Author EUA designed the study and carried out the process and production. Author CSE performed the statistical analysis. Author OSM performed the chemical analysis. Author FCN performed the mechanical analysis of the material. Author MIT sourced and sorted the raw materials. Author CCI edited the first draft of the manuscript and supervised the R&D. Author GNE supervised and approved the R&D. All authors read and approved the final manuscript.

Article Information

DOI: 10.9734/JMSRR/2019/45087
Editor(s):
(1) Dr. Yang Qu, Associate Professor, Key Laboratory of Functional Inorganic Material Chemistry, Ministry of Education of the People's Republic of China, Heilongjiang University, China.
Reviewers:
(1) Carlo Santulli, Università di Camerino, Italy.
(2) Tabe Franklin Nyenty, University of Ngaoundere, Cameroon.
(3) M. K. Gupta, Motilal Nehru National Institute of Technology, India.
Complete Peer review History: http://www.sdiarticle3.com/review-history/45087

Original Research Article

Received 17 September 2018
Accepted 28 November 2018
Published 09 February 2019

ABSTRACT

Fibers extracted from Roselle (*Hibiscus sabdariffa)* stem were investigated as starting raw materials for textile industries, bio-packaging material, pulp and paper products, particle boards and composite material applications. Two extraction methods for obtaining bast fibres from *Hibiscus sabdariffa* stem such as Bacterial retting (BR) method and chemical retting (CR) method were studied. The Physico-chemical and mechanical properties of Bacterial retted (BR) and chemically retted (CR) Roselle (*Hibiscus sabdariffa)* fibre was Light brown in colour, with 16.07-20.2 Diameter(μm), 2.10-2.8 Fibre length (mm), 9.54-11.00% Moisture, 0.93-2.50% Ash, 55.55-60.40% Cellulose, 33.70-27.04% Hemicellulose, 10.50-11.40% Lignin, 145.60-177.55 MPa Strength, 20.70-24.90 GPa Modulus, 0.5-0.7% Elongation at break for bacterial retting (BR)

**Corresponding author: Email: emmanuel.akubueze@fiiro.gov.ng, emmauzic@yahoo.com;*

method while chemical retting (CR) method was Dark brown in colour, 16.07-20.2 Diameter(μm), 2.10-2.8 Fibre length(mm) 12.90-14.50% Moisture, 1.00-2.70% Ash, 48.56-56.62% Cellulose, 38.07-31.85% Hemicellulose, 12.50-11.64% Lignin, 135.15-159.05MPa strength, 19.85-23.70GPa, 0.4-0.6% Elongation at break. The results showed that fibres produced by bacterial retting (BR) has better quality than chemical retting (CR) in terms of mechanical properties and flexibility, but the time involved in bacterial retting (BR) was too long to be considered for today's industrial process integration, thus making it less attractive. More advancement in biotechnology with combined enzymatic and chemically retting will give shorter time with high quality fibres. In general, bast fibres from Roselle (*Hibiscus sabdariffa)* stem is also a unique natural fiber in the class of jute and kenaf. R&D into large scale production and industrial utilization of indigenous bast fibres will no doubt aid the revival and future development of Nigerian textile and allied industries.

Keywords: Roselle (Hibiscus sabdariffa) stem; bast fibres; mechanical properties; chemical properties; bacterial and chemical retting.

1. INTRODUCTION

Hibiscus sabdariffa is a shrub belonging to the family—Malvaceae and genus *Hibiscus*. It is an annual erect shrub with red or green stem, which is slightly hairy, leaves are serrate, lower Leaves ovate, and flowers are large yellow with dark crimson. [1]. The plant grows to a height of about 3 to 3.5 meters with a diameter of 1.0 to 2.0 cm within 130-150 days after sowing It is mainly grown for their fleshy calyxes (sepals), which have been given more commercial attention due to it medical/ pharmaceutical and food-flavoring application. In Nigeria, its use majorly in food preparation such as sauces, jams, juices, jellies, syrups and coloring agent for food and drinks. The phytochemical screening of *Hibiscus sabdariffa* for various medicinally important compounds and their quantification have been reported [2]. In China it is used to treat hypertension, pyrexia and liver damage, and in ayurvedic medicine [3]. Recently the sepal extract has been used as an effective treatment against leukemia due to its high content in polyphenols, particularly protocatechuic acid [4]. It has certain therapeutic properties, taking it in the form of herbal tea for soothing colds, clearing a blocked nose, clearing mucous, as an astringent, promoting kidney function, aiding digestion, as a general tonic, as a diuretic, and helping to reduce fever [5]. Taken as a drink made from the calyx, it is a mild diuretic and purgative, among many other effects. The drink is said to be a folk remedy for cancer and reported with no bacterial isolate [6]. The seeds are a source of a vegetable oil that is low in cholesterol and rich in other phytosterols and tocopherols, particularly β-sitosterol and γ-tocopherol. The overall characteristics of roselle seed oil allow for important industrial applications and represent added value for its cultivation (Mohamed, R. et al. 2007).

Despite all the food, pharmaceutical and medical application of the plant, the stem which contains the greater percentage by weight have not been taped commercially by various fibre application industries (pulp & paper, textiles & packaging, construction materials & bio-composites). This paper presents the effect of fibre extraction methods on fibre quality and properties that could open ways for commercial exploitation. From literature search, the resources from the stem and its application have received little attention due to lack of information regarding its properties. *Hibiscus sabdariffa* bast fibre is closely related to jute and kenaf with higher economic potential. It has been reported to be one of the important bast fibre crops which stand next to jute in production [1].

There is hope for increased industrial demand for natural fibres in the nearest future, with increase deterioration of global environment caused mainly by the extensive exploration of petro-chemical resources and expansion of synthetic product market. Among many green engineering materials, natural fibres has contributed more in partial replacement of synthetic and petro-chemical materials in many applications.

The rationale for natural fibre research and development (R&D) is based on the urgent need to improve efficiency, quality and quantity of fibres, value addition and diversified products to enhance industrial growth.

2. EXPERIMENTAL

Materials and Methods: Matured and harvested Roselle (*Hibiscus sabdariffa)* stems were sourced from Kano municipal, Kano state, Nigeria. All chemicals used in this investigation such as analytical grade sodium hydroxide (NaOH), analytical grade sodium bisulfate ($NaHSO_4$), analytical grade acetic acid, ethanol, KOH, sulphuric acid (H_2SO_4) were analytical grade reagent obtained from Silveb Scientific Nig Ltd, surulere Lagos.

Fibres extraction and processing: The Roselle (*Hibiscus sabdariffa*) fibres used in the experiments were the ones extracted by polymer and textile research team at Federal Institute of Industrial Research Oshodi. The outer skin of the bark was manually peeled from the stems and used for baste fibre extraction.

Bacterial Retting (BR) process: Bacterial retting which can also be called natural retting was used for the fibre extraction in a control system tank retting process with 1: 20 (w/v) fibre-to-liquid. The temperature of the retting water was monitored and maintain at 27 ± 2°C, the pH of the retting water was between the range of 6.5 – 7.0, and the ratio of retting water to the outer skin bark of the plant material was maintained at 10:1 (w/v) throughout the process. The retting/ decomposition took place between 15-20 days. The retted fibres were washed in clean running water, sun dried and combed with soft brush to obtain a filament and finer fibres. Retting is an important process in fibre production. The process involves immersing the stalks in stagnant or slow moving water with joint action of certain bacteria and fungi that builds up within a short time, which helps in decomposition and dissolution of non-fibrous binding material like lignin, pectin, hemicellulose etc.

Chemical Retting (CR) process: Chemical retting was performed by immersion of the stalks along the entire length in 5-7% sodium hydroxide (NaOH) and 1% sodium bisulfate ($NaHSO_4$) (w/v) solution, at ratio of 1:20, fibre-to-liquid, then boiled for 1hr 30mins and neutralized with 0.3% acetic acid for 2-5 minutes. The fibres were washed thoroughly with tap water to a neutral Ph, air dried and combed. Chemical retting is a quicker process than the bacterial retting, but the fibre quality was affected in terms of strength, stiffness, colour, and luster. The fibre product could be softened chemically via avis Mercerization or by batch emulsion process to increase some properties. In this experiment, mercerization process was used to improve the properties of fibres by immersing in 1% solution of NaOH (caustic soda) for 3 minutes. Mercerization increases strength, luster and dyeability.

2.1 Analytical Procedures

Sample preparation & conditioning: The retted air-dried samples were oven dried for 24 hours at 105°C before milling and conditioned at 65 ± 2% relative humidity (RH) and 27 ± 2°C for 24 hrs to ensure environmental equilibrium moisture content, prior to testing. (NIS: 43:1980) and (ASTM: D1776-79). Conditioning is an important stage in sampling materials because it gives the exact analytical conditions and procedures used.

Proximate chemical analysis of fibre: The chemical composition of the bacterial and chemically retted *Hibiscus sabdariffa* fibres in terms of the % cellulose, hemicellulose, lignin and ash content was determined using test methods.

Chemical analysis: Direct method of cellulose, hemicellulose and lignin (Moubasher et al. 1982) 2 g of fibre was boiled in ethanol (4 times) for 15 minutes, washed thoroughly with distilled water and kept in oven for dry weight at 40°C overnight, then divided into two parts in which one is considered as A fraction. Second part of residue was treated with 24% KOH for 4 hours at 25°C, washed thoroughly with distilled water, dried at 80°C overnight and the dry weight taken as B fraction. The same samples again treated with 72% H_2SO_4 for 3 hours to hydrolyse the cellulose and then refluxed with 5% sulphuric acid (H_2SO_4) for 2 hours. sulphuric acid (H_2SO_4) was removed completely by washing with distilled water, dried at 80°C in an oven overnight and dry weight taken as C fraction.

Calculations:

Fibre samples: banana and plantain fibre
Cellulose = B – C
Hemicellulose = A – B
Lignin = C itself

Inorganic (Ash content): The inorganic content which is referred to as ash content in the fibre after combustion at a temperature of 575 ± 25°C was determined using ASTM method E1755-01.

Fibre morphology: The experiment was conducted at Forestry Research Institute of Nigeria (FRIN), Ibadan Oyo state. Samples were prepared into slivers of 4 mm ×3 mm × 3 mm and put in test tubes for maceration in equal volumes of glacial acetic acid and hydrogen peroxide (1:1). The solution was put in the oven for 4 hours at a temperature of about 100°C for maceration. Random samples of macerated fibres were mounted on slides and examined under a light microscope. Fibres were viewed and measured using a stage micrometer under × 80 magnification. Twenty (20) fibres were measured from each respective sample. Values that were determined were fibre length, fibre diameter, lumen width etc. Magnified image view of the Bacterial retted (BR) and chemically retted (CR) Roselle (*Hibiscus sabdariffa)* fibres was also examined on the appearance, and structural defect. Chemical retted (CR) fibres were too stiff, darker, but less coarse than the Bacterial retted fibres.

Magnified Image pictures of the two extracted methods: Bacterial retting (BR) and chemical retting (CR): Fig. 1 shows magnified image appearance of the fibres extracted with bacterial and chemical methods. Longitudinal image view of the two extracted staple fibre bundles was examined. Roselle (*Hibiscus sabdariffa)* fibre was Light brown in colour for bacterial method and dark brown in colour for chemical method. The two samples show consistent in appearance, flexibility and hairiness.

Fibres remain the basic and starting raw material for textile product development. Fibres are required to have suitable length, pliability and tenacity to withstand the stress and strains involved in conversion to yarn, which will open it up for diverse industrial application. Figs. 3 and 4 show the experimental processes involved in product development. Fig. 3 shows experimental spinning process which transforms the carded fibrous material into spun yarn. In this process, carded strands of fibres in a fairly parallel arrangement were spun on a spinning wheel to form a yarn. Fig. 4 shows experimental weaving frame and woven mat. The unit consist of warp and weft yarn which are interlaced with one another according to the class of structure and desire design.

Burning test: The effect of flame in contact with fibre samples was carried out using standard method as described in technical manual of the American association of textile chemists and colorist (AATCC-1981/82).

Fig. 1. Bacterial retted (BR) Sample **Chemical retted (CR): Sample**

Fig. 2. Hand carder **Carded fibre**

Fig. 3. Spun Yarn

Weaving frame

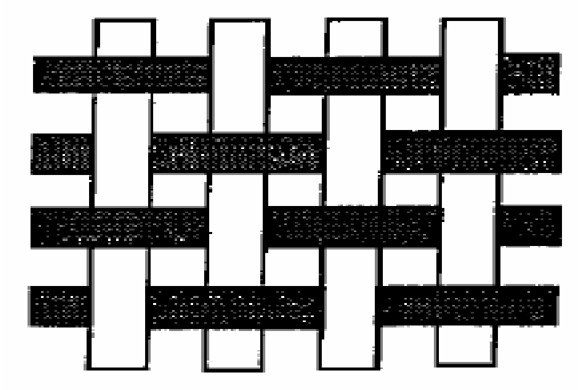

Fig. 4. Point paper design (PPD)

Woven mat (Plain weave)

Moisture content: The moisture content of fibre samples was determined using moisture analyzer (m/s-70), the % amount of moisture present in the samples, drying temperature and time was digitally recorded by the machine. The test was carried out in triplicate and average result was taken.

Fibre density: Fibres density measurement was carried out using Archimedes method of established protocol for natural fibres density measurement (2008). Fibre density measured in accordance with ASTM standard D3800-99 and D792/D276.

Fibre yield:

Yield (%) = {(Weight of the extracted Fibres/ Weight of the fibre plant material) × (100/1)}

Tensile properties of fiber: The tensile properties of cleaned and dried single fibre of both bacterial and chemically retted *Hibiscus sabdariffa* were performed using a Testometric Tensile Testing Machine (m500-25KN, S/N 500-689) according to ASTM 3822-07 Standard to obtain the breaking tenacity, % breaking elongation, and Young's modulus of the fibers. A gauge length of 50 mm and a crosshead speed of 5 mm/min were used for the testing. About 10 fibers of each sample were tested and analyzed.

3. RESULTS AND DISCUSSION

Bast Fibres of *Hibiscus sabdariffa* obtained from Bacterial Retted (BR) Process have much higher cellulose and lower lignin content than the Chemical Retted (CR) Process as seen from Table 1. The tensile strength, elongation at break and modulus of bacterial retted fibres is also higher than those chemical retted fibres. The fibres have the strength similar to kenaf and jute, the ash and moisture content also appears almost the same. The color of the fibre appears to be light gold for BR and dark gold for CR. The shapes are non-uniform and the diameter of 25 samples analyzed varies from 0.13 mm-0.24 mm fibre to fibre along the length. The fibre length ranged between 2.10 mm to 2.8 mm under light microscope examination.

Table 1. Physico-chemical and mechanical properties of bacterial & chemically of retted roselle (*Hibiscus sabdariffa)* fibres

S. no.	Properties	Bacterial retted (BR)	Chemically retted (CR)
1.	**Physical**		
	a) Moisture content (%)	9.54-11.00	12.90-14.50
	b) Density(g/cm^3)	1.35-1.46	1.35-1.46
	c) Ash Content (%)	0.93-2.50	1.00-2.70
	d) color	Light gold	Dark gold
	e) Diameter(μm)	16.07-20.2	16.07-20.2
	f) Fibre length(mm)	2.10-2.8	2.10-2.8
	g) Yield (%)	25-29	34-42
2.	**Chemical (%)**		
	a) Cellulose	55.55-60.40	48.56-56.62
	b) Hemicellulose	33.70-27.04	38.07-31.85
	C) Lignin	10.50-11.40	12.50-11.64
3.	**Mechanical**		
	a) Strength (MPa)	145.60-177.55	135.15-159.05
	b) Elongation (%)	0.5-0.7	0.4-0.6
	c) Modulus (GPa)	20.70-24.90	19.85-23.70
	d) Strain (%)	0.005-0.07	0.005-0.006

The bacterial retting process is lengthy, it takes about 15-20 days to degrade non-cellulosic materials (pectin materials, lignin and hemicellulose) but the resulting fibres have many desirable characteristics. Chemical retting is a quicker process and more attractive than the bacterial retting, but it affects several properties, including a loss in tenacity, color, and lustre when compared to the bacterial retted fibres. The combination of enzymatic and controlled chemical retting will give shorter retting time with desirable fibre quality. This also aggress with what was reported by Tahir et al. [7] that chemical and enzymatic retting offers more controls and acceptable fibre quality.

4. CONCLUSIONS

The data obtained from various studies on extracted *Hibiscus sabdariffa L*. indicated similar properties and behavior when compared with other fibres in the bast family like kenaf and jute fibres. *Hibiscus sabdariffa* has traditionally been cultivated in Nigeria for pharmaceutical and food application rather than for fibre yield. However, there are many promising industrial uses for the fibre and other fibres of similar properties which are enumerated in this paper, provided that necessary measures are taken to stop fibre importation, introduce and stabilize demand for products in the market place. It cannot be overemphasized that successful industrial utilization of natural fibres depends on close cooperation between government and public private partnership (PPP) through market driven Research and Development (R&D). *Hibiscus sabdariffa stem* fibres have strength properties required for high value textile, composite, pulp and paper and other fibrous applications.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

REFERENCES

1. Juhi Agarwal, Ela Dedhia. Current scenario of *Hibiscus sabdariffa* (Mesta) in India. Valley International Journal. 2014;1(3):129-135. ISSN: 2349-2031.
2. Arvind Mungole, Alka Chaturvedi. *Hibiscus sabdariffa* L a rich source of secondary metabolites. International Journal of Pharmaceutical Sciences Review and Research. 2011;6(1):Article-018.
3. Odigie IP, Ettarh RR, Adigun S. Chronic administration of aqueous extract of *Hibiscus sabdariffa* attenuates hypertension and reverses cardiac hypertrophy in 2K-1C hypertensive rats. Journal of Ethnopharmacology. 2003;86:181–185.
4. Tseng T, Kao T, Chu C, Chou F, Lin W, Wang C. Induction of apoptosis by hibiscus protocatechuic acid in human leukemia cells via reduction of retinoblastoma (RB) phosphorylation and Bcl-2 expression. Biochemical Pharmacology. 2000;60:307–315.

5. Ageless. The Trusted Herbal Anti-aging. Herbal remedies using Roselle (*Hibiscus sabdariffa*); 1999. Available:http://www.ageless.co.za/rosella.htm
6. El-Sherif MH, Sarwat MI. Physiological and chemical variations in producing roselle plant (*Hibiscus sabdariffa* L.) by using some organic farmyard manure. World Journal of Agricultural Sciences. 2007;3(5):609–61.
7. Tahir PM, Ahmed AB, SaifulAzry SOA, Almed Z. Retting process of some bast plant fibres and its effect on fibre quality. A review. Bio Resources. 2011;6(4):5260-5281. DOI: 10.15376/biores.6.4.5260-5281

Peer-review history:
The peer review history for this paper can be accessed here:
http://www.sdiarticle3.com/review-history/45087