International Journal of Home Science 2021; 7(1): 255-260

International Journal of Home Science

ISSN: 2395-7476
IJHS 2021; 7(1): 255-260

www.homesciencejournal.com
Received: 13-11-2020
Accepted: 29-12-2020

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Extraction of bast fibres from odal (*Sterculia villosa*) and evaluation of Physical characteristics

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Abstract
Natural fibres have attracted the interest recently due to their abundance in nature, low cost and biodegradability. Odal (*Sterculia villosa*), one of the most important plant which could be utilized for extraction of fiber. Odal is one of the fast-growing plant species abundantly available in the North Eastern Region of India. This study was attempted to extract the fibres from Odal bark and to evaluate the physical properties of the fibres accordingly. Extraction of Odal fibre was carried out by decortications process. Later fibres were subjected to water retting with various time intervals. The yield of fibre after twenty one days was found highest as 2.8% with good fibre quality whereas, the lowest 2.0% was recorded in seven days of retting. The highest length of fibre was recorded in retted Odal fibre as 2.31mm. The maximum wall thickness and diameter was recorded in retted Odal fibre as 1.46 mm and 5µm respectively. The maximum tensile strength (2.92 g/tex) was observed in retted Odal fibre and the least was noticed in bleached Odal fibre (1.48 g/tex). Retted Odal fibre had showed highest elongation (2.22 %). Bleached Odal fibre posed highest density (1.40gm/cc).

Keywords: *Sterculia villosa*, natural fibre, extraction, retting, degumming

Introduction
Today's world is increasingly environment conscious and natural clothing lifestyles are advancing. The inclination towards eco friendly textile alternatives and the emergence of innovative fabrics is vivid. Today's growing concerns are health, sustainability of waste management and environmental awareness which is reflecting on renewed interest in plant fibers. New fibers are being developed and are valued for their sustainable and biodegradable characteristics. The highly competitive atmosphere and stringent ecological parameters become the prime concern of the textile industry to be conscious about ecology. Due to the performance characteristics, eco friendly fibres can be incorporated as whole or parts of materials and products of various forms for a wide range of applications. Natural fibres such as jute, bamboo and pineapple are known to have very high strength and can be effectively utilized for various applications. Natural Fibers are fibers that are produced by plants, animals, and geological processes. Natural fibers can also be matted into sheets to make products such as paper, felt or fabric. Natural fibers can be used for high-tech applications, such as composite parts for automobiles. Natural fibers can have different advantages over synthetic fibers. Most notably they are biodegradable and renewable. Additionally, they often have low densities and lower processing costs than synthetic materials. In the early part of the 19th century and the beginning of the 20th century, men started producing fibres other than natural sources, which have been called as man-made fibres. Later on, these man-made fibres rose up drastically in textile industries for producing clothing and other important materials. However, some of the synthetic fibres used for producing clothing materials for day to day use are not devoid of health hazards (Gokhale and Katli, 1995) [9]. Natural fibers are nowadays increasingly employed for making nonwoven, replacing the synthetic materials due to economic and environmental considerations.
Sterculia villosa (Odal) is one of the most important non-conventional fibres which are utilized by the people. *Sterculia villosa* is abundantly available in North-Eastern region of India. A small to moderate size spreading tree, up to 1.6m girth and found in deciduous forest throughout the country. The fibre is eco-friendly to nature. The tree having large long-stalked deeply lobbed leaves and yellow flowers.

The botanical name of odal is *Sterculia villosa* belonging to the family *Sterculiaceae*. Barks are grey and light red in colour and fibrous inside. The barks are composed of layers can be stripped off from the bottom to the top of the tree. The *Sterculia villosa* produces a strong and brown colour fibre. The bark/sheath of *Sterculia villosa* is reported to produce fibres with high strength properties (Mauersberger, 1954; Saikia *et al*, 1972; Watson *et al* 1976) [14]. The physical properties of *Sterculia villosa* such as strength, absorbency, resilience, stretch, softness and wash ability are high. Ghosh and Baruah (1997) [8] reported that the bark of Odal contained 21.87%, lignin, 15.45% pentosam, 61.78% cellulose, 2.89% ash and 9.25% moisture.

The best quality fibre is obtained when the plants are cut at flowering stages. The bark from the stem is generally removed by means of a machine or by hand. Fibres are extracted by different methods - such as water retting, chemical extraction and microbial degumming (Ghosh, 2000). As water retting effective over mild conditions of pH and temperatures and as they are easily biodegradable, they pose practically no threat to environment. Hence water retting for Odal fibre was considered to improve its pliability.

Materials and Methods
Materials
For the present study *Sterculia Villosa*(Odal) plant was selected to extract the fibres.

Collection of raw material
Sterculia villosa plant was collected from Tinsukia District, Assam.

Chemicals and solvents
AR grade chemicals like hydrogen chloride, sulphuric acid, sodium hydroxide, sodium carbonate, acetic acid, hydrogen peroxide, sodium sulphide etc were used in different stages of this investigation.

Methods
Physical characteristics of the plant
The average length and width of the Odal plant were taken with the help of measuring tape, average weight of the cut plant were weighted in weighing balance. The moisture content of the bark was determined by using TAPPI standard method. (Technical Association of Pulp and Papers Industry, USA).

Extraction of fibre
The first step of extraction of Odal fibre was the decortication process. The extractions of fibres from Odal bark were carried out with the help of decorticating machine (JETIX) (model number-09013). Initially, the barks were cut to a predetermined length. The soft parts of the bark were fed into the machine, the machine crushed the fleshy parts of the bark and then feed fibres were taken out from the machine.

Fig 1: Decorticated fibres

Optimization of retting process of Odal fibre
Retting process
In the retting process, the decorticated fibres were subjected to water retting. Retting was carried out in retting tank with a constant material to liquor ratio of 1:30 at atmospheric temperature (Jose and Salim, 2014) [11] for different time duration *viz*.10, 15 and 20 days to optimize the extraction condition. After definite time of retting, the fleshy materials were come out from the Odal barks that were adhered to the fibres. Finally, the fibres were washed properly and air dried under shade and kept ready for further use.

a) Retting of decorticated bark b) Washing of retted fibres c) Retted Odal fibres

Fig 2: Retting of decorticated bark

Degumming process
Degumming is the process of removing the gummy substance from the fibre. The retted fibre contained some amount of gummy matter. So it is essential to remove the gum content from the mass. Degumming of retted fibre was carried out by using 5% Na_2CO_3 at boiling temperature for 3 hours at a material to liquor ratio 1:40 (Gahlot *et al.*, 2011) [6]. Finally, the fibres were free from gummy matter and kept for further use.

a.

b.

Fig 3: a) Degumming of Odal fibre **b)** Degummed Fibre

Bleaching process

Bleaching can be defined as the destruction of natural colouring matter from the textile materials in order to achieve a clean white end product. Degummed fibres were subjected to bleaching in order to get the superior whiteness. It was carried out by using 2% hydrogen peroxide at boiling temperature for 60 min. at a material to liquor ratio 1:30 (Kalita B.,2004) [12].

Fig 4: Bleached fibre

Physical properties

Plant morphology

The heights of the plant, its diameter were recorded as per the method described by Chaturbedi and Khanna, (1982) [4].

The height of the plant was measured in metre corrected to the third decimal place. Measurement was taken with the help of measuring tape, from the tip of leading shoot to the ground level. The total height of a plant was measured from the top in a straight line without giving any allowance to minor curves of the stem to the base.

The diameter was measured using selective plants at random with the help of a slide caliper at its base near the ground level in case of annuals, whereas, in case of perennials, the diameter was recorded at breast height (BH) level i.e.1.37 meter (universally accepted standard) and the diameter measurements were recorded as-

DBH(Diameter Breast Height) =(D1 + D2 + D3 + D4) ⁄ 4

Where, D1 to D 4 are four measurements of diameter up to breast height.

Density (g/cc)

Density indicates the mass per unit volumes expressed as grams per cubic centimeter or pounds per cubic feet. Because density is commonly determined on balances or scales, the correct expression for density is mass per unit volume (ASTM, 1970) [3]. After degumming and bleaching, fibres were combed out and were taken for density test. The fibres were cut into fine pieces with the help of sharp scissor. Then samples were inserted separately in measuring cylinder up to a level of l0ml. After that the fibre were taken out and weighed in electronic balance. The density was expressed in gm/cc.

Length, diameter and wall thickness of the fibre

Fibre was taken and gently straightened over the slide with the help of twizer. Then medicinal paraffin was used to control the fibre to measure the individual length of the fibres. The prepared slide was mounted over an dokuval photomicroscope scale (JEOL, Japan) and the length of the fibre was recorded. The diameter and the wall thickness of the fibre were also measure at the same time.

Tensile strength (g/tex) and Elongation (%)

The tensile strength was determined by ASTM procedure (1962-1964) [2].

Procedure

The tensile strength (tenacity) of fibres was measured on stelometer by taking a bundle of fibres of 25cm long and measure of tenacity at ½ gauge length. The breaking load was indicated by the pointer which moves over the large scale graduated from 0-10kg load. The tensile strength of fibres bundle was calculated according to (Booth, 1968).

$$\text{Tensile strength (g/tex)} = \frac{\text{Breaking load}}{\text{Bundle weight (mg)}} \times 125$$

Wicking height (cm)

(Miller and Tyomkin, 1984) [15] pointed out the spontaneous uptake of liquid in a fabric has always been called wicking, also stated that when a porous material such as fabric is placed in contact with a liquid spontaneous uptake of liquid may occur.

Procedure

A length of test specimen, pre-conditioned in 25±2 °C at 65±2 per cent relative humidity was suspended vertically with its lower end immersed in a reservoir of distilled water. The height reached (at a constant time of 2 minutes) by the water in the fibre above the water level in the reservoir of distilled water was measured and recorded (AATCC, 1967) [1].

Mechanical properties of fibres

Scanning electron microscopy

SEM test was carried out in Central University, Tezpur. For the test fibres were separated and mounted on specimen holders with the help of a electro conductive tapes. The samples were coated with gold in an ion sputter coater (JFe 100, JEOL, Japan) in low vacuum with layer ISO-l0G nm thick and observed in JEOL, JSM-35M-35CF electron microscope at 15 KV an accelerating potential at different magnificient.

Fourier-transform infrared spectroscopy

FTIR test was carried out in Central University, Tezpur, Assam. Finely crushed degummed and bleached fibers were examined in a double-beam Fourier transform infrared spectroscopy (FTIR) spectrophotometer by attenuated total reflectance attachment (Bruker, model-Alpha). The transmittance spectra were recorded at wave numbers from 500 to 4000 cm^{-1}.

Results and Discussion

Morphological characteristics of odal plant

Table 3.1. Plant Morphology of Odal plant

Sl. No.	Particulars	Measurement
1	Length (m)	6
2	Diameter (cm)	
	A) Top	45
	B) Middle	60.00
	C) Bottom	80.80
3	Weight of the stem (g)	1,000.00
4	Weight of the bark/ sheath (g)	600.00
5	Weight of core (g)	400.00
6.	No. of leaves in stem	5.00
7.	Moisture content,%(bark/Sheath)	66.70

The results were mean of 5 observations.

From Table 3.1 it was clear that the average length of the Odal plant were observed as 6-8 meters. The width of the plant was taken by dividing the whole plant length into three parts *viz*. bottom, middle and top. The highest diameter of 80.80cm was noticed in bottom part of the plant followed by middle (60cm) and top (45cm) portion. The average weight of the whole plant and weight of the stem were noted as 1,000.00g and 600.00g respectively. The weight of core of the plant was recorded as 400.00g and there was 5 nos of leaves in stem of the plant. The moisture contents of the Odal plant in bark were found as 66.70%.

Optimization of extraction procedure

The extraction of fibres from barks of Odal plant were carried out by retting process, the plants were first subjected to decortications. Retting was done by retaining the material to liquor ratio constant *viz*. 1:30, at atmospheric temperature for different time duration.

Table 3.2. Retting parameter of Odal bark

Bath ratio (M: L)	Duration (days)	Atmospheric temperature (°C)	Yield of fibre (%)	Observation
1:10	10	Normal	2.0	Partial removal of gummy substance, fibres were stiff
1:10	15	-do-	2.2	Removal of gummy substance, fibres were stiff
1:10	20	-do-	2.8	Soft fibre, more yield

The results were mean of 5 observations.

Table 3.2 revealed the different percentage of yield and quality of retted Odal fibre. It can be perceived from the table that after retting, the highest yield of the fibre was noticed in 20 days (2.8%) followed by 15 days (2.2%) and 10 days (2.0%). The fibre yields in 10 days were stiff due to the partial removal of the gummy substance from the surface of the fibre whereas in 15 days the gummy substance were removed but the fibers were still stiff. The different percent in yields of fibres and their qualities were observed which might be due to the variation in removal of pectinous and non-cellulosic substances from the fibre cells. Fibres obtained in 20 days retting were seen to be smooth and soft.

Hence, on the basis of yield and fibre softness after retting procedure, 20 days duration of retting at atmospheric temperature and 1:30 material to liquor ratio was thereby considered as optimized condition for extraction of Odal fibre. The optimized fibre was utilized for further processing.

Physical properties of Odal fibre

Table 3.3: Physical properties of Odal and Jute fibres

Properties	Odal		
	Retted	Degummed	Bleached
Length (mm)	2.31	2.21	2.0
Wall thickness (mm)	1.46	1.36	1.00
Diameter(μm)	4.0	3.5	3.23
Tensile Strength (g/tex)	2.92	1.59	1.48
Elongation (%)	2.22	1.50	1.60
Density (g/cc)	1.10	1.20	1.40

The results were mean of 5 observations.

The examined data on physical properties such as length, diameter, wall thickness, tensile strength, elongation and density of the fibres were bestowed in Table 3.3.

From the Table 3.3, it was seen that among the Odal fibre the maximum length was exhibited in retted Odal fibre 2.31 mm and minimum exhibited as 2.00 mm in bleached fiber. The similar work related to the fibre length was reported by (Ghosh *et al*.; 1997) [8]. The values of fibre length are within the range as given by (Lewin, 2007) [13]. According to him, the length of bast fibres varies from 10 to 140cm.

In regards to the diameter of the fibres, maximum diameter of 4µm was registered in retted fibre as while the minimum diameter of 3.23 µm was noted in bleached fibre. Lewin, 2007 [13] described the range of diameter of bast fibres from 5 to 76µm. The retted Odal fibre registered highest wall thickness (1.46 mm) followed by degummed (1.36 mm) and bleached fibre (1.00 mm). Maximum diameter and wall thickness of retted Odal fibre might be due to the presence of extra cellular materials. The similar work related to the fiber diameter was reported by Ghosh and Baruah, (1997) [8]. Fiber diameter plays a vital role in determining the quality of yarn and fabrics. After degumming and bleaching, the wall thickness (3.0 and 2.80mm) and lumen diameter (14.10 and 12.2µm) were reduced in comparison with raw fiber (Sharma and Dhoundiyal, 1983) [17].

Similar results were also obtained by (Dey and Satapathy, 2013) [5] which showed conformity with the present investigation.

It was evident from the Table 3.3 that among the fibers, the maximum tensile strength (2.92g/tex) was found in retted Odal fiber followed by degummed Odal fiber (1.59g/tex). Highest elongation (2.22%) was observed in retted Odal fibre followed by degummed Odal fiber (1.50%). According to (Mishra 2000), the tensile strength of bast fibres ranges from 5-8 g/d. The strength loss may also be contributed by the attack of alkaline hydrogen peroxide in the cellulose portion.

From the empirical data, it was also observed that bleached Odal fibre had showed highest density (1.40gm/cc) followed by degummed Odal fiber (1.20gm/cc). It might be due to that degummed process had some positive effects on density of fiber and fineness. The increase in density may be due to the removal of hemicelluloses and lignin during bleaching. After bleaching, there is an improvement in fiber fineness, but bundle strength deteriorated from 21.5 to 15.6g/tex and it may be due to the loss of hemicelluloses content and lignin. Lignin and hemicelluloses are continuous network of the fiber and directly influence the physical and mechanical properties of fibers (Hazarika *et al*., 2017) [10].

Microscopic evaluation of Odal fibres

SEM of Odal fibre

The surface appearance of raw, degummed and bleached fibers was studied using FE SEM images as shown in **fig.5**. The gum content of the raw fibers is clearly visible in the SEM image (Figure 5(a)). The surface is found to have cracks and holes. This indicates the presence of gummy matter on fiber surface. The alkaline degumming almost removed the gummy matter (Figure 5(b)) and bleaching process removed whole of the gum from the fiber surface (Figure 5(c)). Degumming and bleaching resulted in individualization of fiber entity and smoothening of surface by removing the gum from the fiber.

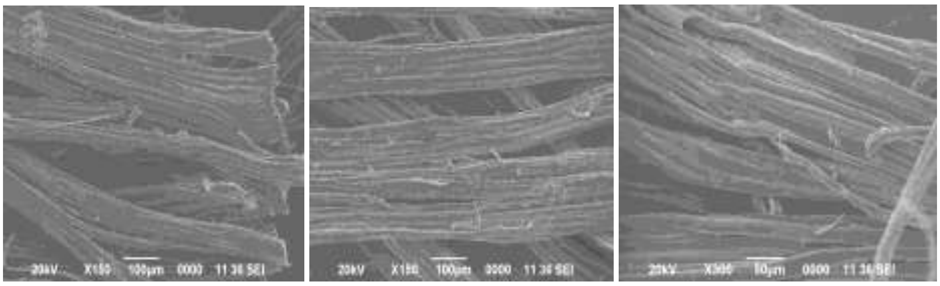

Fig 5: a) SEM image of raw Odal fibre **b)** SEM image of degummed Odal fibre **c)** SEM image of bleached Odal fibre

FTIR of Odal fibre

The figure 6 shows the FTIR images of Odal fibre. In the raw fibre, the sharp peak at 1515cm $^{-1}$ corresponds to C=C aromatic symmetrical stretching of lignin. A significant reduction in the peak was observed after degumming and bleaching. This indicates that, both degumming and bleaching removed lignin from the fibre. The carbonyl stretching vibrations of ester and carboxyl groups in hemicelluloses are attributed to the band at 1742cm^{-1}. The degumming removed the hemicelluloses to certain extent; however, in the spectrum of bleached fibre, the peak is almost eliminated, which indicate the complete removal of hemicelluloses from the fibre during bleaching. The peak at 1460cm^{-1} corresponds to CH_2 stretching vibration in lignin and xylan. The broad band at 1022cm^{-1} assigned to C=O stretching in cellulose, hemicelluloses and lignin or C=O=C stretching in cellulose and hemicelluloses. The peak at 1426cm^{-1} for the bending vibration of CH_2 that is strong in crystalline cellulose and weak in amorphous cellulose. A sharp peak at 1646cm^{-1} in raw fibre indicates the presence of pectin. It is evident from the spectra that, degumming resulted in partial and bleaching resulted in the complete removal of pectneous matter from the fibre. The peaks at 1332cm^{-1} assigned for O-H in plane deformation of cellulose rings. In the figure is it shown that, the intensity of the peak is higher in the bleached fibre than raw fibre. It indicates that, the cellulose content of the fibre got increased during bleaching due to the removal of no cellulosic components like lignin, hemicelluloses and pectin’s.

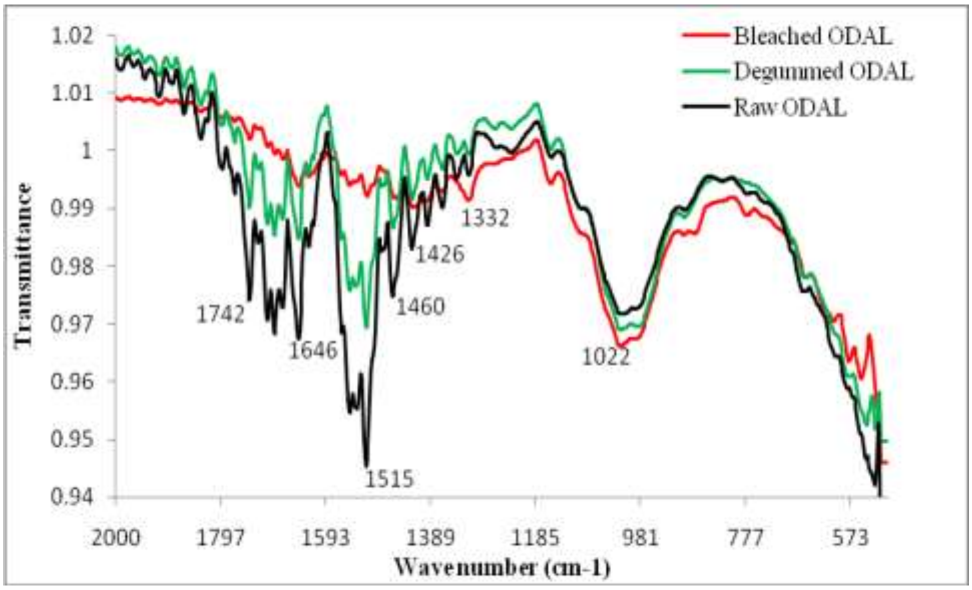

Fig 6: FTIR of Odal fibre

Conclusion

The scientific world is trying of developing new and advanced technologies and methods to treat solid wastes, particularly non-naturally-reversible polymers. The processes to decompose those wastes are actually not cost-effective and will subsequently produce harmful chemicals. Owing to the above ground, a natural fiber is the way to go. From the various findings as enumerated, it could be concluded that the non-conventional plant materials selected for the present study were ideal sources of bast fibre. With the growing scarcity of biodegradable fibres, these plant fibres could suitably be used as alternative source of synthetic fibres. Odal fibres have good potential for exports owing to their economics, aesthetic appeal and improved overall properties.

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