Material and characterization of new cellulosic fibers from *Muntingia calabura* stem

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Abstract

In line with the rising environmental concerns, the synthetic fibres are replaced with the natural fibres in the recent past. Stem fibre from *Muntingia calabura* species is extracted using water retting method and its physico-chemical properties are investigated for the purpose of this study. The tensile strength of the fibre 416-872Mpa, cellulose content 68.51%, density 1.351g/cc, crystallinity index 47% properties were identified in the *Muntingia calabura* fiber is one of the good natural fiber that can be used as reinforced nonwoven materials different application.

Keywords: *Muntingia Calabura*, Nonwoven, Scanning electron microscopy, Thermogravimetric analysis, X-ray diffraction

1. Introduction

Natural cellulose fibres have gained significant importance and usage since decades.[1] More than 1000 plant types bear fibres. Plants are made up of huge number of cells. A cell is called a fibre when its length is more than the width.[2] Chemical composition of natural fibres plays an important role in its differentiation from being classified as plant, animal or mineral fibres base on the origin. The components like cellulose (semi crystalline polysaccharide responsible for hydrophillic nature) hemicelluloses (amorphous polysaccharide partially soluble in water and alkaline solution); pectin (polysaccharide that holds fibres together), lignin, waxes and water soluble substances.[2]

Plant/vegetable fibres are classified as stem fibres, leaf, seed and fruit fibres based on their source. Some of the plants that produce usable fibres from stem are Jute, flax, hemp, Ramie and Kenaf whereas sisal, banana and pineapple are from fruit.[3]

The wide availability and ease in range of synthetic fibres have led to manufacturing of over 100 million tons every year out of which 50% is from crude oil. This poses a serious environmental issue. The biodegradability and environmental safety concern are important in the design of new natural features.[4] The material engineers are now trying a different alternative keeping the environmental issue in concern. In the recent years, natural fibres are given considerable attention by way of development and utilization, wide range of natural fibre’s applicability in automotive textile fields is because of certain efficient property such a resistance and flexibility in nonwoven materials. There are many variety of natural and man-made fibers used in automotive non-woven textiles.[5]

One of the most profuse fruiting trees! This is a very fast-growing tree of slender proportions, reaching 25 to 40 ft (7.5-12 in) in height, with spreading, nearly horizontal branches.[6] Common Namescalabur tree, calabura, silkwood, Singapore cherry, sirsen, strawberry tree, strawberry tree. The leaves are evergreen, alternate, and oblique at the base. Flowers resemble strawberry bloom, hence the name of the tree. The abundant fruits are round, 3/8 to 1/2 in (1-1.25 cm) wide, with red or sometimes yellow, smooth, thin, tender skin and light-brown, soft, juicy pulp, with very sweet, musky, somewhat fig-like flavor, filled with exceedingly minute, yellowish seeds, too fine to be noticed in eating. Fruit taste like cotton candy.[7]

2. Materials and experimental procedure

2.1 Raw Material

The stems of *Muntingia Calabura* were respectively collected from farms in Erode Districts, Tamil Nadu.
2.2 Fiber Extraction

The length of the *Muntingia calabura* stem is from 2 to 4 feet. These trees and stems as shown in (Fig 1.a) are obtained by removing the leaves and are cut at required length with knife and manually peeling process as shown in (Fig.b). These peeled parts are immersed in water for 1 day for extraction. During this water retting process the peeled parts are completely wetted. The gums were present in the fibers separated. Thus the extracted fibres were washed thoroughly to remove the unwanted materials. After these fibers were dried in sunlight at least from two to four hours to remove the water content. The bundles fiber dried fibers were collected as shown in (Fig.c) for further investigation.

![Muntingia calabura fiber extraction](image)

*Fig 1: Muntigia calabura* fiber extraction: (a) Muntingia calabura tree (b) Stems manual extraction of fibers and Peeled part immersed in water (c) bundle of extracted fiber

2.3 Characterization Methods

2.3.1. Tensile Strength

A single fiber tensile test was carried out using an INSTRON 5500R Universal testing machine to determine its tensile strength at 65% relative humidity and temperature 21±1.5 °C with a cross head speed of 10mm/min. A 1 kn load cell was used to measure. The length of 20mm were tested for randomly 20 samples selected from *Muntingia calabura* fibers, maximum strength according to the ASTM D 3822 [8].

2.3.2 Chemical properties

The composition of *Muntingia calabura* fiber is shown in Table 1. Chemical composition such as Cellulose is the major component followed by lignin and moisture [9].

2.3.3 Scanning electron microscopy

The morphology structure of the *Muntingia calabura* fiber specimen by using SEM model of JEOL JS M -6390 electron microscopes. The instrument which was handled scanned the surface with high energy electron beam. The SEM produces very high-re solution at three dimensional images, which are very much useful for understanding the surface morphology of fibres [10].

2.3.4 Fourier transform infrared (FT-IR)

Fourier transform-infrared spectroscopy studies of *Muntingia calabura* fiber was captured by using Nicolet smart ITR –ATRIs and is 10 FT-IR spectrometer. To ensure presence of free functional group on the 500-4000 cm⁻¹ at room temperature of 25°C and relative humidity of 65% was recorded in absorbance mode as a function of wave number[11].

2.3.5 X-ray diffraction (XRD)

X-ray diffraction method was accessed to know about the crystallinity of the fiber. Its analysis was under room temperature under the conditions of current 30mA and Voltage 40Kv Cu anode was made. Not only scanning mode of range from 2θ to 89θ but also scanning speed of 10de/mils was undertaken. The crystallinity index (Icr) was calculated by means of expression where Lmin and Lmax represent the intensity at minimum and maximum crystalline peaks respectively [12, 13].

\[ I_{cr} = 1 - \frac{L_{min}}{L_{max}} \]

2.3.6 Thermogravimetry (TG)

Thermal stability behavior of the *Muntingia calabura* fiber was obtained by thermogravimetric (TG) analyses using Jupiter simultaneous thermal analyzer (MODEL STA499 F3, NETZSCH, Germany) in order to measure mass and transformation. The *Muntingia calabura* was exposed to nitrogen gas at a flow rate of 20 ml/min. Ten milligram of the *Muntingia calabura* was crushed and kept in alumina crucible to avoid the temperature variations measured by the thermocouple. The step increment of heating rate was from 10 °C/min over a range of temperature i.e., from room temperature 25 °C to 500 °C [14].

3. Result and discussion

3.1. Physical properties

In Fig 2, the tensile strength of the *Muntingia calabura* was found 416-872Mpa while the tensile strengths of other natural fibers such as flax, hemp, jute, and ramie, are 500-900,690,370 -134, and 915 MPa, respectively [15]. Any natural fibers hardly possess uniform diameter throughout its length. It depends on place and condition where the plant grows. Optical microscope was used to take the image of the fibers in longitudinal direction and these images were imported to image analyzer software to measure the diameter of 20 samples of five places. The diameter range of *Muntingia calabura* fibers sample was calculated from 241.1 mm to 248.9 mm. The density value of *Muntingia calabura* fibers was found to be 1.3521 g/cc.
3.2 Chemical composition

Table I shows the chemical composition of the *Muntingia calabura* fibers which was determined by means of using standard methods, is compared with natural fibers. Cellulose content in natural fiber is considered to be main component of strength, stiffness, structural stability. This fiber has much cellulose content lower than *phoenix fiber* [8]. The lignin content in the fibers contributes to the rigidity and its value is greater than *jute fiber*. The wax content is low in comparison with *hemp*. The fiber has low ash content. The moisture content present in the fiber is higher than comparable to existing fibers like *hemp* and *okra*.[16]

<table>
<thead>
<tr>
<th>Fibre</th>
<th>Cellulose (wt %)</th>
<th>Lignin (wt %)</th>
<th>Ash (wt %)</th>
<th>Moisture (wt %)</th>
<th>Wax (wt %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Phoenicx sp</td>
<td>76.13</td>
<td>4.29</td>
<td>19.69</td>
<td>10.41</td>
<td>0.32</td>
</tr>
<tr>
<td>Hemp</td>
<td>70.2-74.4</td>
<td>3.7-5.7</td>
<td>-</td>
<td>6.2-12</td>
<td>0.8</td>
</tr>
<tr>
<td>Muntingia Calabura</td>
<td>68.51</td>
<td>15.36</td>
<td>6.27</td>
<td>9.27</td>
<td>0.43</td>
</tr>
<tr>
<td>Jute</td>
<td>61-71.5</td>
<td>11.8-13</td>
<td>-</td>
<td>12.5-13.7</td>
<td>0.5</td>
</tr>
<tr>
<td>Prosopis juliflora</td>
<td>61.65</td>
<td>17.11</td>
<td>5.2</td>
<td>9.48</td>
<td>0.61</td>
</tr>
<tr>
<td>Okra</td>
<td>60</td>
<td>0.6</td>
<td>-</td>
<td>7.5</td>
<td>0.3</td>
</tr>
</tbody>
</table>

3.3 Scanning electron microscopy

The morphological analysis of the *Muntingia Calabura* fiber is shown in fig.3 at magnification. The illustration of surface characteristics explains that the fabrils are clean and at the same time they have smooth surface. The surface of individual cell is clearly visible perhaps due to low wax content.

3.4. FTIR

In FTIR spectrum of Muntingia Calabura fiber (fig.4) the peak at 3330cm⁻¹ are associated with the x cellulose (O-H)stretching [1]. The two sharp peak 2923 & 2850 cm⁻¹ of group assigned CH & CH₂ streching [17]. The band at 1608 cm⁻¹ indicate the presence of C-O stretch of acetyl group of hemicellulose and O-Hstretch of carboxylic acid [8], 1540cm⁻¹ CH₂ bending [17]. Peak at 1325cm⁻¹ is attributed to the C-H bending [20], and the band at 1420-1430 cm⁻¹ is assigned to aromatic skeletal vibrations associated to C-H in plane deformation of cellulose [19]. The bands at 1460, 1425 and 1220 cm⁻¹ are characteristic of C-H and C-O deformation [19], The cellulose characterized by 1028.
XRD analysis
X-ray diffraction shown in Fig.5 the raw *Muntingia calabura* fiber shows two peaks at $2\theta=16^\circ$ and $2\theta=22^\circ$. The two peak are assigned to (110) and (200) crystallographic plane in the fiber samples. The crystallinity index ($I_c$) which is measure of amount of crystalline cellulose with respect global amount of amorphous materials was determined as 49%. *Muntingia calabura* crystallinity value calculated using the curve are 47%of fiber its slightly higher than other natural fiber like banana, bagasse, and 38%,48% [12].

3.6 Thermogravimetric Thermogravimetric
TG of *Muntingia calabura* fiber is illustrated in figure. 6. The thermal degradation of *Muntingia calabura* fibers occured in three stages [27], 300-350 which is due to the evaporation of moisture [1], thermal degradation happened at the initial stage. Next to it, the degradation at second stage started at 425$^\circ$ but the mass loss happened at 475$^\circ$ at third stage. Each stage went in hand with the degradation of alpha-cellulose and lignin in *Muntingia calabura* [28, 29]. However, the thermal stability of *Muntingia calabura* seems to be comparable to other fibers such as wild date palm, tamarind, boarass. From the study of TG curve diagram, it is obviously observed that *Muntingia calabura* appears up to 300$^\circ$C.
Conclusion
Researchers attempt to find a extracting the stem of *Muntingia calabura* into fiber in an eco friendly manner. The new natural fiber *Muntingia calabura* plant was extracted by way of manual peeling process. Besides their physical-chemical, mechanical, and morphological characteristics were studied. *Muntingia calabura* fiber has high cellulose content, and high tensile properties of fiber. Scanning electron microscopy has shown that the surface morphology of the fibers becomes soft. The moisture content of *Muntingia calabura* fiber is better than other natural fibers, which lead to better dimensional stability, reduced porosity and improved adhesion between the fibers. The obtained crystallinity of the *Muntingia calabura* fiber was 47%. FTIR spectrum, the absorbance peak represent the presence of C-H stretching of alkene groups and O-H stretching of Alcohol groups. The thermogravimetric analysis of fibers showed that is thermally stable up to 300°C. In this study, *Muntingia calabura* fiber has been introduced as a stem fiber for the development and production of an acoustic non-woven fabric for the use of any lightweight structure.

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