



Effect of wet processing dynamics of okra fiber: Properties and processing strategies

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ABSTRACT

The earth is rich in fibrous material to cater to the needs of humans. Despite technological advancements, every source of fibre has not been fully tapped. In this regard, a study on okra fibres has been conducted. The objective is to overcome the shortage of natural fibres and utilize underutilized fibres on earth. The extraction and analysis of fibres from okra plant was carried out. The world is focusing on renewable and sustainable resources. The fibres were extracted from the stalk of the plant and subjected to extraction and evaluation of their properties such as chemical constituents, length, wall thickness, diameter, strength, and moisture content. The Scanning Electron Microscopy (SEM) study was also carried out to assess the morphological changes in the fibres on processing. Fourier transform infrared spectroscopy (FTIR) and X-Ray Diffraction test was also carried out to detect the presence of chemical groups in the fibres. The XRD spectrum of the raw, degummed, and bleached okra fibers indicated the presence of cellulose, as reflected by the sharp peak that represents the increasing portion of the crystalline structure of cellulose. The results suggest that okra fiber has the potential to be a substitute for conventional fibers in various industries such as home furnishings, automotive, civil engineering, geo-textiles, industrial filters, and medical and sanitary materials

Key words: Natural fibers, chemical properties, physical properties, SEM, XRD.

INTRODUCTION

Agricultural waste is the most prevalent type of natural fiber and is used in many sectors of contemporary industry. Depending on the region the agro-waste produces such wastes which are frequently processed in waste-to-energy plants or disposed of in landfills. The circular economy concept has offered various broad suggestions for ways to improve waste management, including using garbage to create new goods with additional value (Koul *et al.*, 12). Moving towards a more circular economy could increase competitiveness, stimulate innovation, boost economic growth and create jobs nearly about 7, 00,000 jobs by 2030. Redesigning materials and products out of agricultural waste fibers for circular use would also boost innovation across different sectors of the economy. Consumers will be provided with more durable and innovative products that will increase the quality of life and save them money in the long-term helping them to double the income (Acevedo *et al.*, 1).

Due to the increased activity in the modern agricultural sector, a large amount of waste is produced, which poses a serious threat to the environment. Natural fiber can be considered in this light as a good alternative material for the local timber sector to make value-added products, such

as bio-composites, as a result of the worry over the dwindling availability of raw materials. As a long-term plan to develop the enormous richness of natural plant fiber that are now underutilized, utilization of natural fiber, especially agricultural waste fiber, needs to be further developed. Rice husk, oil palm, bagasse, maize stalks, bamboo, pineapple, banana, and other plant parts (stem, leaf, seed, fruit, stalk, and grass/reed) can all be used to produce agricultural waste. Cellulosic fibers are the primary fiber wastes generated by agricultural activity, have potential as reinforcing materials since they are widely available, light, renewable, degradable, less expensive, and have low abrasive properties. These biomass wastes have a number of intriguing unique characteristics (Karimah *et al.*, 10).

Agricultural and plantation wastes are seen to be a viable and acceptable raw material sources for addressing the rising need for renewable resources. Among the agro-waste raw material okra and jute fibers act as one of the important sources of alternative material for the production of nonwoven. Contrary to what is possible with traditional weaving and knitting, nonwoven technologies are helpful for the development of unique materials and products at a shorter and relative faster rate. Nonwoven engineering processes also give the finished product better specialized qualities (Upadhyaya *et al.*, 9). Therefore, these natural fibers namely okra fibers

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used in this study present various properties and characterization of the fibers towards sustainable and advanced utilization.

50-65 million tons of fibres are accessible annually and the amount will rise yearly, assuming that 40% of production is available as waste and that at least 10% of the waste by weight may be collected as fibre (Gupta *et al.*, 5). There are several ways to use agricultural waste from okra fibers to improve sustainability and reduce waste. Utilizing okra fibers from agricultural waste not only lessens waste but also encourages sustainability and assists a variety of industries. The species can reach heights of up to 2 meters (6 feet 7 inches) and is perennial that is frequently grown as an annual in temperate climates. More than half of the top ten okra growers were from African countries. On the other hand, biomass from the okra plant provides a cheap, low-density, recyclable, renewable source for making bast fibers and other sustainable industrial products (Ullah *et al.*, 15).

MATERIALS AND METHODS

Okra (*Abelmoschus esculentus*) stems were procured from local agricultural farms in the district of Jorhat and Tinsukia for the present study. The average height and girth of the okra plants were measured with the help of measuring tape and the average weight of the stems were weighted in weighing balance. Depending on the morphological characteristics of the natural fibers, okra plants were chosen for the study and further extracted to separate the plant fibers. Water retting is the most common method used to extract high quality fibers (Islam *et al.*, 7). Consequently, the fibers were soaked in the water tank with a constant material to liquor ratio of 1:10 at room temperature and kept submerged in water for 10-15 days. Once the stems significantly deteriorated the extracted fibers were then washed in running water allowing the stem to get separated from the fiber. Then all of the extracted fibers were line dried for 24 hours in the shade before being exposed to additional processes. Different wet processing treatments like degumming and bleaching were carried out for the extracted fiber by using standard procedure. The physical, chemical, morphological, mechanical and color measurement properties of extracted fibers were also recorded.

Degumming is the process of removal of gummy substance from the fiber. The retted fiber contained some amount of gummy matter. So, to remove the gum content acid degumming was carried out by using 5% HCL at boiling temperature for 3 hours at a material to liquor ratio 1:40 and then neutralized by using 5% Na₂CO₃ (Gogoi *et al.*, 4). Bleaching of fibers was done to remove residual impurities from the fibers

in order to get the superior whiteness. Bleaching was done by using 4 percent of alkaline hydrogen peroxide with material to liquor ratio 1:40 and heated to a required temperature at 85-90°C for 1 hours in a close vessel. Sodium silicate was added to the bleaching bath as a stabilizing agent in the middle of the process. Then the fibers were taken out and washed properly in running water and air dried (Kalita *et al.*, 8). Fibers possess distinct physical and chemical properties. Identifying the chemical properties of fibers enables to deduce the suitability of wet processing agents and envisage the fiber's behavior under different treatments. Comprehending the physical and chemical properties of fibers is fundamental for material selection, product development, quality assurance, and sustainability efforts across various industries. It underpins the entire lifecycle of fiber-based products, from initial design and manufacturing to end-of-life considerations (Zhao *et al.*, 17).

The Scanning Electron Microscopy (SEM) were carried out to record the surface morphology of raw, degummed and bleached okra fibers. Correspondingly, Fourier Transform Infrared Spectroscopy (FTIR) and X-Ray Diffraction Analysis (XRD) method is done to provide insights into its chemical composition, functional groups and potential applications in textiles and crystallographic structure of the fiber.

RESULT AND DISCUSSION

The morphological characteristics of okra plants that was collected for the study were evaluated such as average height, weight and girth of the plant and presented in Table 1.

The experimental data presented (Table 2) evidently showed that the moisture, ash, lignin, cellulose, hemi-cellulose and fat was found maximum in raw okra fiber and maximum alpha-cellulose content in bleached okra fiber. The differences in moisture, ash, lignin, cellulose, hemi-cellulose, fat, and alpha-cellulose content between raw and bleached okra fiber can be attributed to the processing steps involved in bleaching. The bleaching process aims to purify the fiber by removing impurities and non-cellulosic components, leading to changes in the overall composition of the fiber (De Rosa *et al.*, 3). From the above results, it can be inferred that okra fiber contains the highest percentage of moisture and cellulose which in turn contributes to the strength, flexibility and durability of fibers making them suitable for various applications such as animal bedding, packaging materials etc. Different physical properties of okra fibers are given in the Table 3.

The morphological properties of the extracted fibers revealed that the highest (2.86mm) fiber length was found in raw okra fiber followed by degummed

Table 1: Morphological characteristics of okra plant.

Parameter	Measurement okra
1. Height of the plant (m)	145 cm
2. Weight of branches (without leaf)	165 cm
3. Girth (cm)	156
a. Bottom	7.6
b. Middle	5.3
c. Top	2.2
4. Moisture (%)	80

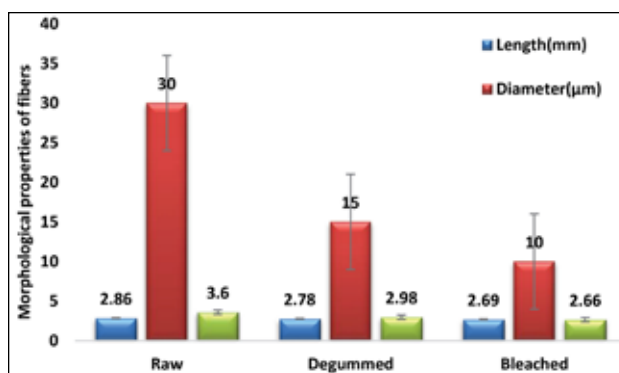
Table 2: Chemical composition of okra fiber.

Constituents	Water retted okra		
	Raw	Degummed	Bleached
Moisture (%)	14.12	9.26	8.50
Ash (%)	1.32	1.24	1.18
Lignin (%)	7.1	6.5	6.2
Cellulose (%)	58.0	52.0	50.0
Hemi cellulose (%)	16.14	16.07	15.94
Alpha cellulose (%)	62.73	63.02	65.31
Fat & wax (%)	3.93	2.91	2.44

Table 3: Physical properties of okra fiber.

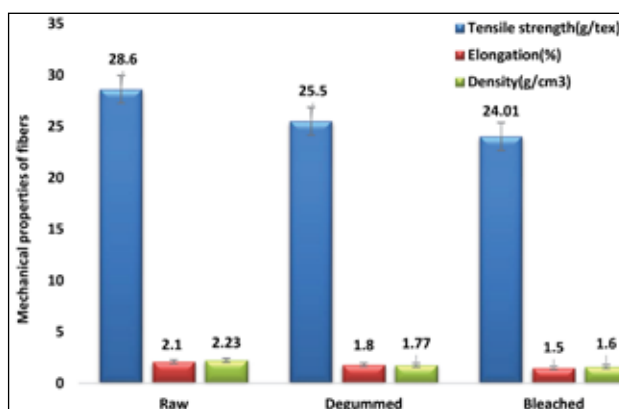
Properties	Water retted okra		
	Raw	Degummed	Bleached
Length (mm)	2.86	2.78	2.69
Diameter (μm)	30	15	10
Tensile strength (g/tex)	28.60	25.50	24.01
Wall thickness (μm)	3.60	2.98	2.66
Elongation (%)	2.1	1.8	1.5
Density (gm/cc)	2.23	1.77	1.6

(2.78 mm) and bleached okra fiber (2.69 mm) respectively. In case of cell wall thickness raw okra fiber showed maximum thickness (3.60 μm) and it was gradually decreased in degummed (2.98 μm) and bleached (2.66 μm) fiber (Fig. 1). In regards to diameter of okra fiber, raw okra fiber showed maximum (30 μm) diameter whereas minimum (10 μm) diameter was reported in bleached okra fiber. Raw okra fiber has the highest fiber length, diameter and wall thickness due to the fact that it has not undergone any processing that might reduce its length than degumming and bleaching processes that are designed to remove impurities from the fiber which lead to reduction in fiber length as some of the natural components are removed or altered during the processing (Stawski *et al.*, 14).

**Fig. 1.** Morphological properties of raw, degummed and bleached okra fiber.

The mechanical properties of raw, degummed, and bleached okra fibers were showed that the maximum tensile strength (28.60 g/Tex) was observed in raw okra fiber, while the minimum (24.01 g/Tex) was recorded in bleached okra fiber. Raw fiber also showed the highest elongation (2.1%), followed by degummed fiber (1.8%) and bleached fiber (1.5%). The density of raw okra fibers was highest (2.2 g/cm³), followed by degummed (1.77 g/cm³) and bleached okra fiber (1.6 g/cm³) respectively (Fig. 2). Due to the presence of natural components such as cellulose, hemi-cellulose, and lignin in their unaltered state contributes to the fiber's strength raw okra fibers generally exhibited higher tensile strength, elongation and density compared to degummed and bleached okra fiber. The higher physical properties of okra fibers can also influence certain factors such as strength, flexibility of the fibers (Islam *et al.*, 7; Kolte *et al.*, 11).

The effects of color measurement showed that bleached okra fiber had the highest (14.02) whiteness index, brightness index (20.4) while the bleached

**Fig. 2.** Mechanical properties of raw, degummed and bleached okra fiber.

okra fiber had the lowest (30.2) yellowness index values. Conversely, raw okra fiber has minimum (4.60) color strength and bleached okra fiber has maximum (5.32) color strength (k/s) (Fig. 3). Okra fibers are naturally lighter in color because of the microstructure and surface properties of the fibers that affect their perceived whiteness. Okra fibers may have a different surface structure that scatters and reflects light differently making them appear whiter (Dayan *et al.*, 2). From the above study it can be revealed that whiteness and brightness increased with the retting period up to a specific period and then deteriorated with further retting. While yellowness decreased till the optimum retting period and then increased with further retting period.

The surface morphology of raw okra fiber revealed rod-like structures with a serrated outer wall, and cracks on its surface exposed a visible gummy substance (Fig. 4). After degumming, the okra fiber appeared cylindrical with a thin wall thickness, as non-cellulosic components such as hemicelluloses, lignin, and pectin were removed, leaving pure cellulose fibers with a more uniform, cylindrical structure and thinner cell wall. In contrast, the bleached okra fiber had a smooth outer wall lining displaying an even smoother surface than the degummed

samples. The bleached samples displayed an even smoother surface than the degummed samples. The overlapping of cells was visible in a longitudinal view of the fibers, indicating the presence of contaminants on the surface of the okra fiber and the fibers being covered in non-cellulosic compounds (Hossen *et al.*, 6). Similar investigation was carried out by Vasugi *et al.* (16) which stated the acidic treatment remove the waxy gummy substance from the tissue of okra fiber bundle.

From the FTIR spectroscopy of the extracted fibers it was evident that the FTIR spectrum of raw okra fiber exhibits several peaks at 3842.02 cm^{-1} , 1519.91 cm^{-1} , 1234.44 cm^{-1} , 1033.85 cm^{-1} , and 686.66 cm^{-1} . The FTIR spectra of degummed okra fibers showed the intensity of the absorption band peaks at 3842.02 cm^{-1} , 1519.91 cm^{-1} , 1033.85 cm^{-1} , and 671.23 cm^{-1} . Moreover, the FTIR spectra of bleached okra fibers exhibited peaks at 3726.47 cm^{-1} , 1519.91 cm^{-1} , 1033.85 cm^{-1} and 655.80 cm^{-1} respectively (Fig. 5). Additionally, a tiny peak was noted in raw, degummed and bleached fiber of which the prominence was at 1519.19 cm^{-1} attributed to the C=O stretching vibration of the aromatic ring of the lignin decomposition. The significant absorption peak at 1033.85 cm^{-1} in raw, degummed and bleached is due to the CO and O-H stretching vibration of polysaccharide in cellulose, and peaks at 686 cm^{-1} are due to the presence of C-Br stretching halo compound, indicating good chemical bonding between the fibers and potential for use in functional applications such as insulation and antimicrobial purposes (Islam *et al.*, 7).

X-ray diffraction analysis (XRD) is a significant and non-destructive technique used in material science to analyze the crystallographic structure of the material. Also, it is used to analyze the material composition, crystalline, and phase purity for the too-small crystal size. An X-ray diffractometer with reflection mode was utilized for XRD measurements (Hossen *et al.*, 6). From the spectra, it can be seen that a broad peak in $2\theta = 20\text{--}25^\circ$ became sharper from

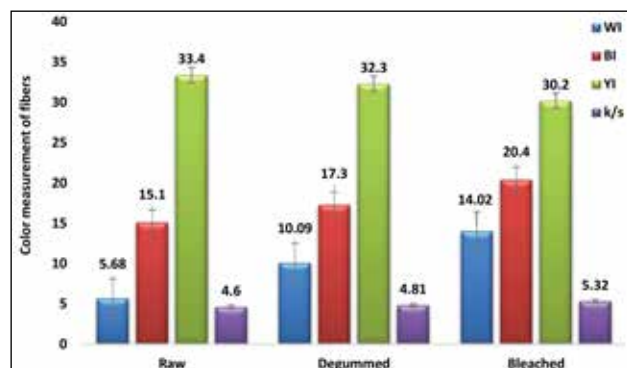


Fig. 3. Color measurement of raw, degummed and bleached okra fiber.

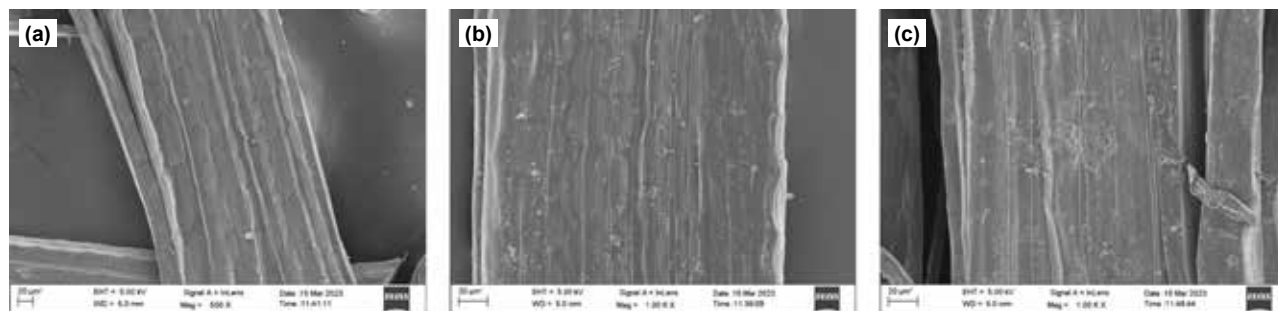


Fig. 4. SEM images of raw (a), degummed (b) and bleached okra fiber (c).

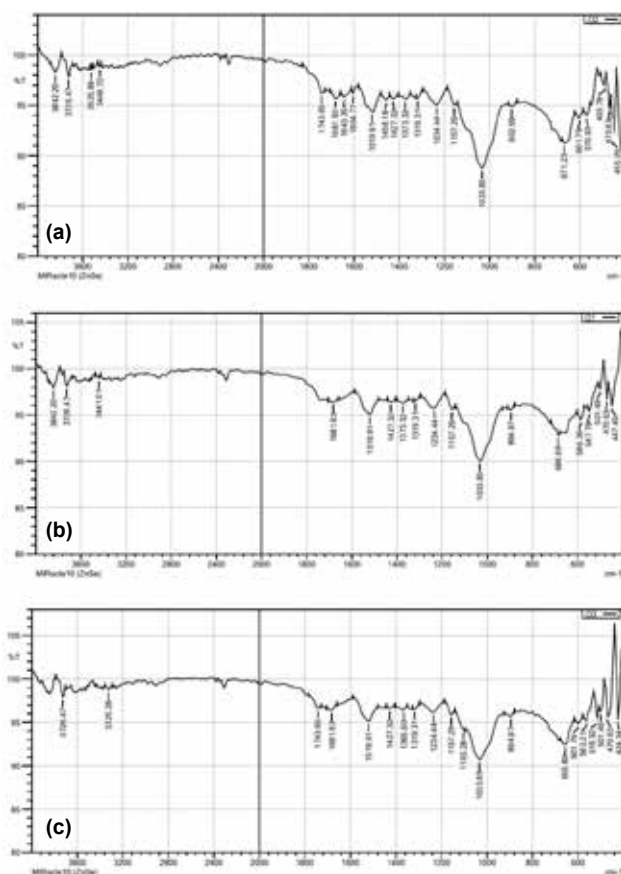


Fig. 5. FTIR graph of raw (a), degummed (b) and bleached (c) okra fiber.

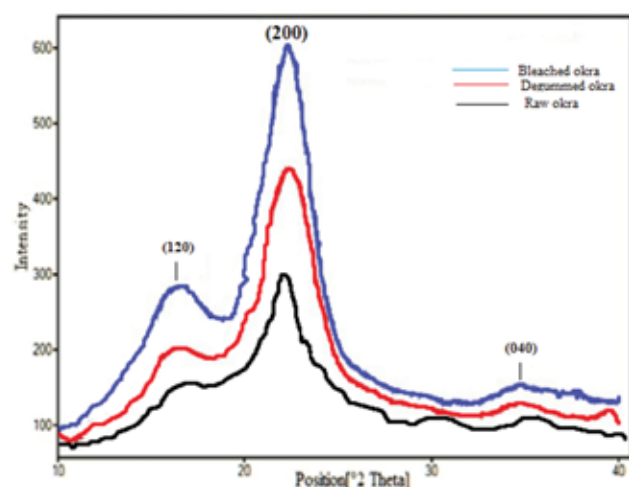


Fig. 6. XRD of raw, degummed and bleached okra fiber.

the raw okra fiber to bleached fiber. The crystallinity of the raw, degummed and bleached fiber, were ranged from 14-15% and 20-24% respectively (Fig. 6). The XRD spectra of raw, degummed and bleached okra

fiber indicates the presence of cellulose and the sharp peak reflects the increasing portion of the crystalline structure of cellulose.

On the other hand, the amorphous polymer shows weak medium peaks like structure. By comparison with the basic structure. Therefore, the okra fiber XRD pattern shows amorphous regions all over the pattern (Karimah *et al.*, 10; Kolte *et al.*, 14; Rahman *et al.*, 16). As a result, okra fiber is amorphous polymers. As it is known that cellulosic compounds show amorphous characteristics in XRD pattern and the examined sample is a cellulosic compound. So, according to the tests and evaluations, okra fiber is an amorphous polymer as it lacks regular, organized or molecular structure. The nature of okra fiber's amorphous nature gives okra fiber certain properties such as flexibility and ability to absorb water which can be useful in various applications like textiles and food products.

Okra fiber, derived from the okra plant is highly absorbent leads to significant water retention and swelling during processing. As a result, dyeing okra fibers require careful management to ensure even coloration and avoid issues like blotching. The fiber's absorbency also impacts its wettability and handling, necessitating adjustments in the use of wetting agents and drying techniques to manage the increased moisture content. Additionally, the coarse texture and rigidity of okra fiber affect its behavior in mechanical processing, potentially requiring modifications in machinery settings and processing conditions.

The study reported that the optimum duration for extracting okra fiber for further processing was 15 days using the water retting method, based on the highest yield percentage and physical properties. The results revealed that the raw okra fiber contained the highest percentages of ash, lignin, fats and waxes, hemicelluloses, and alpha cellulose. Additionally, okra fiber contains functional groups such as hydroxyl, cellulose, stretching aromatic rings, polysaccharides, aromatic compounds, and halogen compounds. The XRD spectrum of the raw, degummed, and bleached okra fibers was analyzed and indicated the presence of cellulose, as reflected by the sharp peak that represents the increasing portion of the crystalline structure of cellulose. The examined sample was found to be a cellulosic compound, and the results suggest that okra fiber has the potential to be a substitute for conventional fibers in various industries such as home furnishings, automotive, civil engineering, geo-textiles, industrial filters, and medical and sanitary materials.

AUTHORS CONTRIBUTION

Methodology, Investigation, Analysis (MH)
Writing-original draft (BBK)

DECLARATION

The authors declare no conflict of interest.

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