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Investigation on morphological and tensile properties of chemically treated Borassus Flabellifer Lontar fruit fibers

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ABSTRACT

Natural fiber reinforced composite materials are used to make the best outcome materials due to their ease of availability, ability to be recycled, and environmental friendliness. The items made from this substance can help both urban and rural locations. Borassus flabellifer L fruits, leaves, and stems are commercially used, although some are discarded as scrap. This byproduct of Borassus-Flabellifer L can be utilized as a fiber source and as the principal component of natural fiber polymer composites with reinforcement. Apart from conventional methods of water retting or dew retting and mechanical decortication to extract the fiber, in this study new novel methods of extraction take place. An examination is conducted on Borassus Flabellifer fruit fibers that have been treated with alkaline and peroxide solvents. The purpose of this inquiry is to assess the possibility of using these fibers as reinforcement in composites. The presence of cellulose, hemicellulose, and lignin can be determined by conducting a chemical analysis. The effect of various chemical treatments on morphological attributes is assessed using SEM and UTM techniques. The X-ray diffraction examination reveals that the crystallinity of the fibers is altered by various chemical treatments, as observed in both the untreated fibers and those subjected to chemical treatment (i.e.,) Alkali (NaOH), Hydrogen peroxide (H_2O_2) and NaOH + H_2O_2. The effectiveness of Borassus Flabellifer fruit fibers as a component of natural polymer composites is examined.

Keywords: Borassus Flabellifer L fruit fibers; Surface treatment; Tensile strength; XRDA; FeSEM analysis.

1. INTRODUCTION

The use of natural fiber has been widespread since before the invention of manmade materials. They were admired in the communities for their strength and flexibility. Utilizing these natural fibers as their reinforcement has become popular in recent fiber reinforced composite developments. Compared to glass fibers, the natural fiber offers less strength and durability. Yet low specific gravity signifies more stiffness and specific strength than glass. It is easy to process without the need for equipment and has good thermal, dielectric, and insulation qualities [1–5]. The possible application of renewable materials in numerous industries is receiving increased attention as a result of the current ecological concern. In order to meet design requirements as well as attain a high level of customer satisfaction, material selection is crucial with regard to sustainable product development. The global market's current situation indicates a growth in the usages of natural fiber reinforced composites (NFRC), which were formerly predominately made of synthetic fibers like glass fiber [6, 7]. This is mostly attributable to natural fibers' benefits of availability and lower weight, which make the resulting composite physically and financially comfortable. When compared to fibers made from petroleum, natural fibers are not significantly denser. NFRC

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may take the place of petroleum fiber based composites in various applications due to their minimal density, recycling ability, availability, good strength and modulus, cost comfort, accessibility, and low consumption of energy along with characteristics of natural fibers. These kinds of fibers may be taken from several plant categories, including hemp, trunk, leaves, seeds, fruits, and roots using any method of extraction. Numerous studies have demonstrated the use of natural fibers in NFRC products, including sisal, coir, hemp, jute, banana, and pineapple leaf. Because of its low cost, abundance, eco friendliness, biodegradability, sustainability, and sanitary qualities, NFRC are replacing many petroleum based fibers [8–10].

Fiber is a substance which is longer than wide. Fibers are available naturally and extracted from plants. Synthetic fibers are also made from animals that are cheap and stronger than the natural fibers. Fibers are mostly used in the manufacturing sectors to make other materials. Vegetable fibers and wood fibers are natural fibers. Cellulose is the major content of the natural fibers. The strength of the natural fiber is depending on the cellulose content of the fibers. Silk and wool are the animal fibers that contain proteins. Mineral fiber such as asbestos and man-made synthetic fiber like glass fibers are the other types of fibers used in the field of manufacturing sectors [11–14].

For composites reinforced with fibers that the fibers are included inside the matrix. The composite is endowed with a high specific strength and a high specific modulus by NFRC. The stiffness and stability of the material depends on the bond between the fiber which has been used as reinforcement and the matrix. Good adhesion means better stress transfer is the result. Further, the strength of the material depends on various factors like orientation of the fiber, composition and others. Three types of fiber orientation are practiced viz. aligned, random and woven [15, 16]. Laminates reinforced polymer composites composed of fibers bonded together layer by layer to improve the strength in both longitudinal and lateral directions. The strength is depending on the number of layers in the materials. When load is applied, the matrix fails first and micro crack forms [17–19]. The cracks propagate layer by layer and withstand the load for a certain time period. The laminate materials and number of layers are chosen based on the specific application of the material to satisfy the engineering requirements [18–22]. Fiber as reinforcement influences a major role in polymer composite material. For that the identified fiber has to be preprocessed to attain maximum strength and eliminate the unwanted contaminants in the extracted natural fiber through the chemical treatment [23–25]. In this research, the fibers characteristics, properties and morphology have been compared among untreated fibers and chemically treated fibers (i.e.,) Alkali (NaOH), Hydrogen peroxide (H_2O_2) and NaOH + H_2O_2.

The novelty of this research lies in its pioneering exploration of Borassus flabellifer (palmyra palm) fruit fibers for polymer matrix composites, employing innovative sodium hydroxide (NaOH), hydrogen peroxide (H_2O_2), and combined NaOH + H_2O_2 treatments to enhance fiber morphology and surface characteristics. By integrating X-ray diffraction (XRD), scanning electron microscopy (SEM), and single fiber tensile testing, this study comprehensively evaluates how these treatments influence crystallinity, morphology, and mechanical properties, filling a significant gap in current literature [26–28]. This research not only introduces a sustainable and underexplored natural fiber source but also offers insights into optimizing fiber treatment processes for applications in lightweight, environmentally friendly composite materials across various industrial sectors.

2. MATERIALS AND METHODS

2.1. Materials

Both thick and thin fibers were taken from Palmyra palm fruits and for chemical treatment Hydrogen Peroxide and pellets of commercial-grade sodium hydroxide were utilized. NaOH solution was used for the purpose of alkali treatment of fibers in this investigation. H_2O_2 solution was used for the purpose of peroxide treatment. The Chemicals were purchased from Synthesis Chemical Lab, Coimbatore, Tamil Nadu, India.

2.1.1. Natural fibers

The fibers extracted from the plants that are used in polymer composites to reduce the weight as density of these fibers are low when compared to other materials. Natural fibers are attractive due to being environmentally friendly, abundantly available, low cost and biodegradable. The Borassus genus L is a member of the Arecaceae family of palm trees, and is helpful to people. All the tree components, from the roots to the fruits, are used to make domestic furniture, food, and items for culture and art. Based on the biological structure and tropical regions where they are growing, it has more than seven species [11]. The Southern region of India has Borassus Flabellifer L. (Lontar, Asian palmyra palm), the fiber extracted from fruit of this kind of palm is the natural fibers taken for this research.

2.2. Extraction of fiber from palmyra palm fruit

The species of the Borassus Flabellifer L is a member of the Aceraceae family. Fruits that were dried and matured were used to create the Borassus fibers. The black skin was removed after the fruits had been submerged in water for two weeks. The fruit nut was edge to edge with coarse fibers, while the fruit shell was covered with fine fibers. Both kinds of fibers were properly cleaned with normal water as shown in Figure 1 and then boiled before being dried for several days in sunlight. The moisture was then removed from the fibers by placing them in a hot air furnace for more than 20 hours at 105–110° C.

2.3. Surface Treatment for fiber reinforcement

2.3.1. Alkali treatment of fibers

The most popular NaOH treatment for natural fiber is this one. This covers three possible techniques, including using a constant NaOH concentration for a fixed amount of time, using various NaOH concentrations for a certain amount of time, and maintaining a constant NaOH concentration throughout a range of NaOH concentrations.

The 2nd and 3rd practice methods are the ones that are most frequently employed to identify the appropriate conditions for natural fiber change. The liquor ratio should be 20:1, which enables for the removal of more fatty compounds, and the fibers should be treated with 2%, 5% and 10% (weight/volume) NaOH solutions at various NaOH concentrations for an extended period of time. The fibers are bleached, washed, and oven after this chemical procedure [18–20]. The Borassus fibers are kept in a 5% NaOH solution for maintaining a constant NaOH concentration. The fibers from the Borassus fruit were treated within 8 hours in a 5% sodium hydroxide solution to assist them in overcoming hydroxyl bonding, enhance fiber-matrix bonding, and get rid of surface impurities. They were then rinsed with normal water and dried for more than 20 hours at 105° C in a furnace (Figure 2a).

2.3.2. Hydrogen peroxide treatment

Hydrogen Peroxide (H_2O_2) treatment can improve the fiber and matrix interface properties. The surface of the fiber becomes adhered to the polyethylene grafting brought on by peroxide. The matrix and the hydroxyl group of the fiber are also affected by the interactions of free radicals generated by peroxide. Good fiber matrix adhesion along the contact therefore happens. Additionally, this treatment increases thermal stability and lessens the fiber's propensity to absorb moisture [21]. The Borassus fruit fiber with fiber to Liquous ratio of 1:30 was added into a bleaching bath prepared by 0.2 mol of H_2O_2 with 3 g/L of Na_2SiO_3 and the initial pH value was maintained at 10.5 using anhydrous Na_2CO_3. The whole bleaching process was carried out in a 250 mL sealed glass container using a vibratile laboratory dyeing machine. After bleaching, the samples were rinsed thoroughly with distilled water and dried in an oven (Figure 2b).

2.3.3. Alkali and peroxide treatment

The composites enhanced mechanical characteristics were demonstrated by pre-treating the fiber with peroxide. The fiber is first given an alkali pretreatment before being treated with peroxide (at a concentration of about

Figure 1: Fibers of Borassus Flabellifer L fruit.

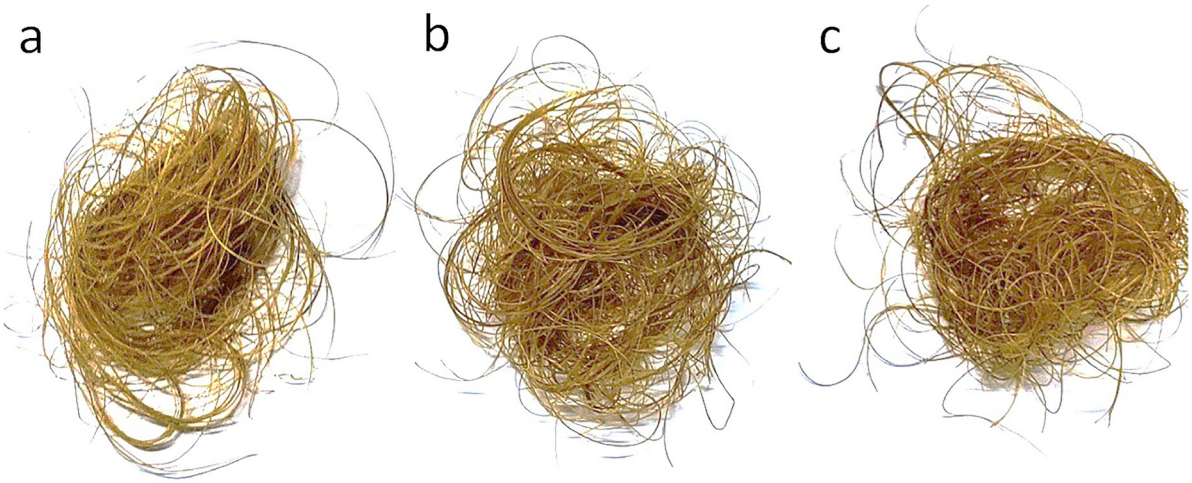

Figure 2: (a) Fiber treated with NaOH (b) Fiber treated with Peroxide and (c) Fiber treated with both NaOH and H_2O_2.

6% in acetone solution) for 30 minutes. By heating the solution to a higher temperature, the peroxide might completely decompose (Figure 2c). In short BFF fiber the peroxide treatments and they showed increased tensile strength qualities.

2.4. Characterizations

2.4.1. Physicochemical characterization

The diameters of BFL Fibers were meticulously assessed using an optical microscope at 4 randomly selected locations on 16 fibers, and the resulting average value was recorded. The density of the BFL Fibers was estimated using the Archimedes or Buoyancy technique. The fiber's length was determined using a Measuring Tape. The cellulose and lignin content in the fiber of the BFL fruit were assessed using the Kushner and Hoffer methods. The Hemicellulose content of BFL Fiber was determined according to the specifications outlined in the NFT 12–008 standards [29]. The wax content was deliberated with XRD method and moisture content was measured with an electronic moisture analyzer (Sartorious, modelMA45), whereas The ash content of the fiber was calculated utilizing ASTM E1755-01 [11–13].

2.4.2. Single fiber tension

A single fiber tensile test was used to determine the maximum tensile strength of the fruit fiber from Borassus Flabellifer L. According to the standard procedures, the testing was done with the Instron UTM. The Borassus Flabellifer Fruit Fiber's ultimate tensile strength was assessed as per ASTM C1557 standard using a crosshead speed of 0.1 cm/min for a gauge length of 50 mm. The uniformity of the results for the samples was evaluated, and the fair samples were shown in the graphs [15, 16].

2.4.3. X-ray diffraction analysis (XRDA)

The crystallinity index of Borassus Flabellifer Fruit Fiber was determined using X-ray diffraction (XRD) analysis. The samples were tested using the diffractometer, namely the Bruker D2phaser. It produces Cu K alpha monochromatic radiation with a precise wavelength of 0.162 nm. The measurement was taken for two values ranging from 15 to 82 degrees. The equation (1) is used to compute the crystallinity index [19].

$$CI = \frac{H_{22.22} - H_{16.82}}{H_{22.22}} \tag{1}$$

The crystal size calculated using the equation (2)

$$CS = \frac{K\lambda}{\beta Cos\theta} \tag{2}$$

Where Scherrer's constant K = 0.89, λ – known for the wavelength and β - represent the peak of full-width at half-maximum.

2.4.4. Morphological analysis

The surface morphology, cell pores of the Borassus Flabellifer Fruit Fiber were studied using a ZEISS Sigma 360VP Scanning Electron Microscope (SEM) with an accelerated voltage of 30 kilo volt and attainable vacuum level of 0.0015 Pa, possessing a Tungsten electron gun and To prevent the buildup of electrical charges during the testing, the specimens were covered in a thin coating of gold [20, 21].

3. RESULTS AND DISCUSSION

3.1. Physicochemical analysis

The Borassus Flabellifer Fruit Fiber has a diameter of 0.28 mm (Figure 3) and a density of 1141 kg/cm3, making it 8.84 times and 1.07 times larger than Agave sisalana fiber while being smaller than Gossypium herbaceum fibers [18]. Applications that require light weighting can employ this lower density.

According to the chemical study as tabulated in the Table 1, the cellulose percentage was 1.4 times greater than the fiber from Agave sisalana and 1.1 times lower than the fiber from Gossypium herbaceum. More cellulose helps to enhance tensile strength, modulus, and crystalline index. The lignin concentration was 9.12 percent; the rise in lignin will cause the material to become more brittle. The ash content was 4.38 percent, When wax and moisture concentration were lower the fiber was better able to lock into the matrix.

3.2. Tensile properties of borassus flabellifer

The average value of tensile strength and modulus of 98.81, 143.92, 113.48 and 156.37 MPa and 1.21, 1.35, 1.53 and 1.79 GPa is founded from untreated and treated fiber with NaOH, H_2O_2 and both NaOH & H_2O_2 respectively.

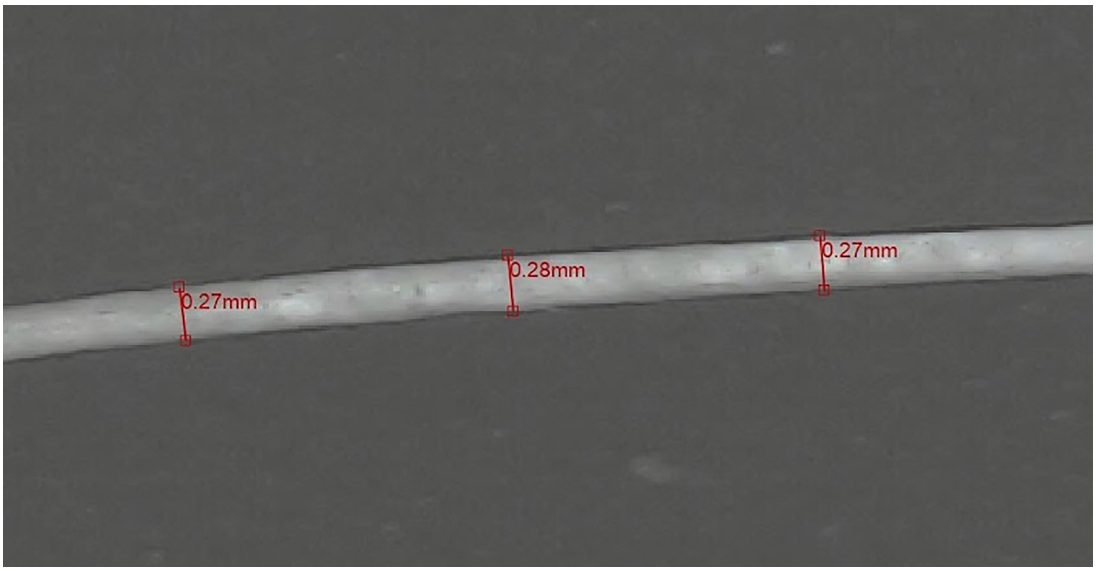

Figure 3: Diameter of the Borassus Flabellifer fruit fiber.

Table 1: Chemical composition of untreated and chemically treated fibers.

FIBER	CELLULOSE	HEMICELLULOSES	LIGNIN	WAX	MOISTURE
Untreated	51.4	29.62	9.12	0.71	6.52
NaOH Treated	63.56	18.12	5.8	0.8	6.89
H_2O_2 Treated	54.7	28.63	8.83	0.85	7.21
NaOH + H_2O_2	68.62	13.05	5.41	0.62	6.64

Figures (4) and (5) represents the tensile strength and modulus contrary to specimens, where the chemical treatment of the fibers increased each specimen's tensile characteristics. Comparing the tensile properties of single fibers from various plant-based sources provides insights into their potential as reinforcement materials in composite applications. Here's a general comparison table based on typical values reported in literature for different plant fibers – Table 2, deliberates the Tensile Strength which represents the maximum stress the fiber can withstand before breaking, comparatively higher than the banana fiber and lesser than others. Young's Modulus which Indicates the stiffness of the fibers, It seems lower values than other fibers it has low rigidity and resistance to deformation under tension. Elongation at Break which represents the ability of the fiber to stretch before breaking. Fibers with higher elongation percentages exhibit greater flexibility, here BFF fiber posses high flexibility because it has the high elongation percentage.

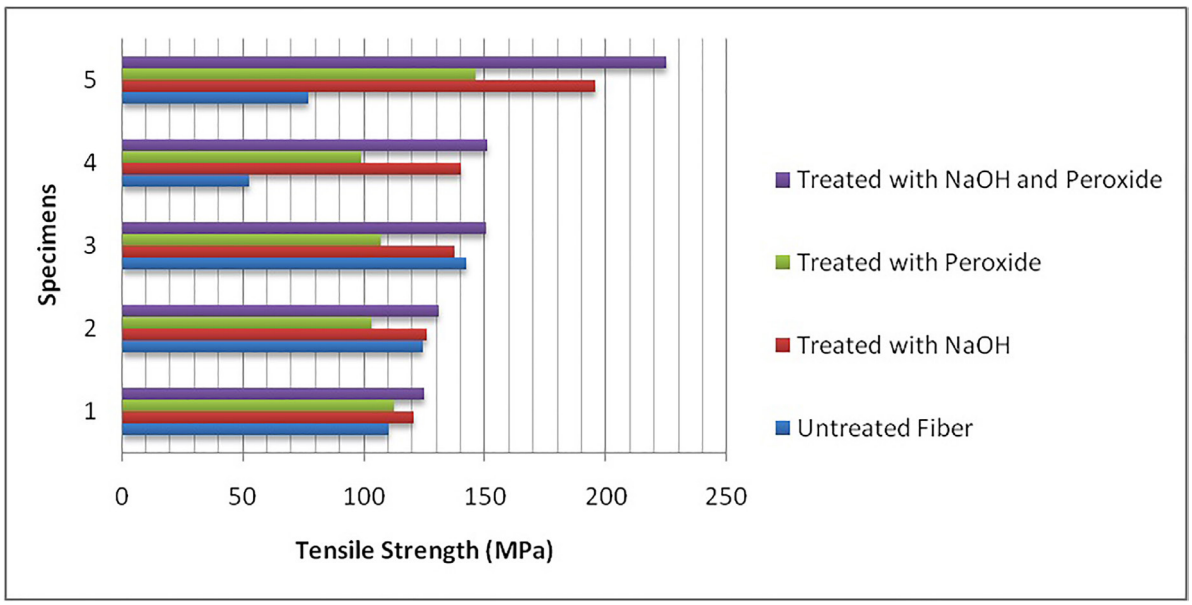

Figure 4: Tensile strength of UT & CT BFF fibers.

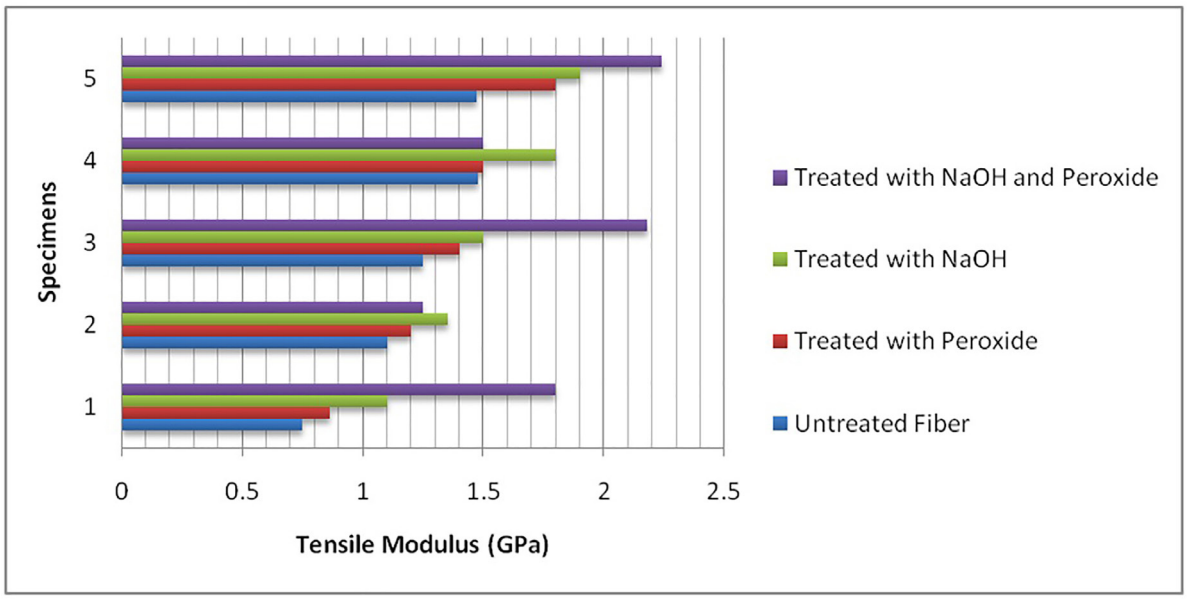

Figure 5: Tensile modulus of UT & CT BFF fibers.

Table 2: Comparison of physical properties of Borassus Flabellifer Fruit fiber (BFF) with various types of fibres.

FIBERS NAME		ρ (g/cm³)	σ (MPa)	E (GPa)	σ/ρ (MPa/g/cm³)	E/ρ (GPa/g/cm³)	ε (%)	REFERENCE
BFF	UT	0.7	132	1.25	188	1.78	25.3	–
	NaOH	0.76	146	1.34	192	1.76	35.2	–
	H_2O_2	0.73	138	1.36	189	1.86	32.6	–
	NaOH+ H_2O_2	0.80	154.8	1.78	193	2.23	41.9	–
Banana		1.36	12–30	12	8.8–22	8.8	1.5–9	[26]
Jute		1.3–1.4	392–770	13–27	392–550	10–19.3	1.16–1.5	[1, 4]
Flax		1.5	346–1100	28	230–733	18.67	2.7–3.2	[6]
Sisal		1.45	465–640	9–22	320–441	6.2–15	3–7	[21]
PALF		1.52	410–1625	34–84	269–1069	22–55	1.6	[17]
Cotton		1.5–1.6	285–800	5.5–12.5	190–500	3.6–7.8	7–8	[9]

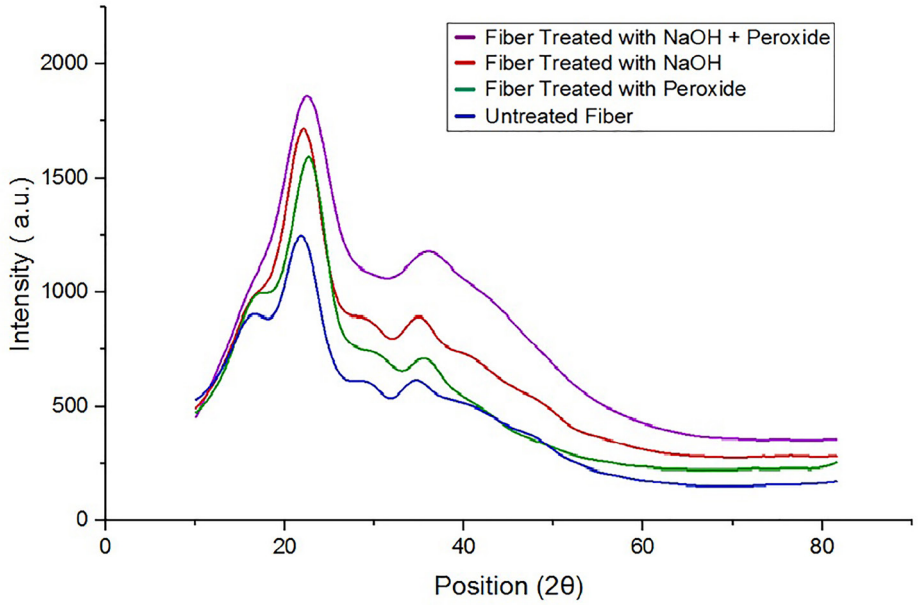

Figure 6: Result comparison of XRDA of untreated fiber & various treated fibers.

3.3. XRD analysis

The XRDA diffractograms of the Borassus Flabellifer are vividly displayed in Figure (6). Two separate peaks may be seen in Figure 3.3. The lattice plane (2 0 0) is the location of the first prominent peak, which is seen at 2θ = 21.22°. This shows that Cellulose I or alpha Cellulose is present. The subsequent peak, which indicates the availability of amorphous components of hemicellulose, cellulose, lignin, pectin, and so on, is seen at 2θ = 16.82° at the lattice plane (1 1 0). The distinguishing characteristic of natural fibers is these two peaks. Equation 1 is used to determine the Borassus Flabellifer's crystallinity index (CI). In comparison to untreated Borassus Flabellifer FF 8.8%, the CI of the Borassus Flabellifer FF treated with 5% of NaOH is determined to be 32.21%, which is 3.64 times greater. The natural fiber's chemical reactivity and moisture absorption are decreased by the larger crystallite size.

From Table 3, The XRD analysis reveals that treating Borassus flabellifer fruit fibers with NaOH, H_2O_2, and a combination of both significantly affects their crystallinity and crystallite size. The NaOH + H_2O_2 treatment appears to be the most effective in enhancing the structural properties of the fibers, making them potentially more suitable for reinforcing polymer matrix composites.

3.4. Morphological characterization

The scanning electron microscope (SEM) may be used to picturize the surface morphology of the BFL fruit fiber. The Borassus fiber indicates the significant changes in surface that was treated with the NaOH and Hydrogen Peroxide treatment. The surface of the BFL fruit fibers which were treated and untreated is investigated. In SEM pictures (Figure 7), they indicate that whereas the untreated raw Borassus fruit fibers were smooth, perfect, the surfaces of alkali-treated fibers showed roughness and fibrils of the surface. The relationship between the polymer matrix and Borassus fiber has enhanced as a result of the taking away of hemicellulose and contaminants from the fiber, according to an examination of the SEM analysis of the fruit fibers.

To access the fiber's suitability as reinforcement has been acknowledged by using SEM. By scanning an electron beam over the surface of the fibers and picking up the secondary or backscattered electrical signal, scanning electron microscopes (SEM) produce precise, high-resolution pictures of the fibers. To avoid the potential buildup of electrical charges following analysis, the sample utilized for the SEM must have a thin

Table 3: XRD results for untreated, NaOH treated, H_2O_2 treated, and NaOH + H_2O_2 treated of Borassus Flabellifer fruit fibers.

FIBER	PEAK POSITIONS (2Θ)	CRYSTALLINITY INDEX (CI)	CRYSTALLITE SIZE (nm)
Untreated	22°, 16°, 34°	0.5	17
NaOH Treated	22°, 16°, 34°	0.72	24
H_2O_2 Treated	22°, 16°, 34°	0.63	19
NaOH + H_2O_2	22°, 16°, 34°	0.78	26

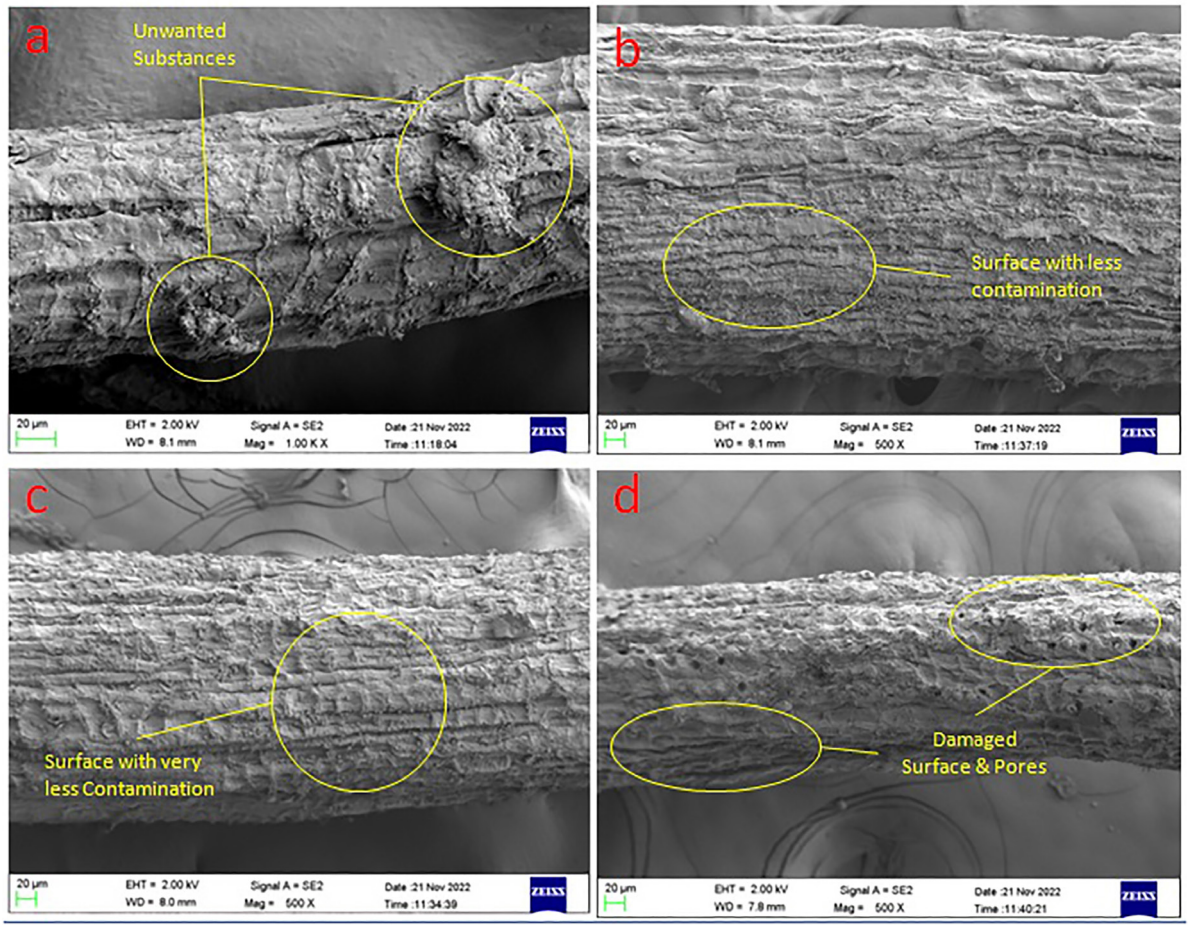

Figure 7: SEM result of Borassus Flabelifer L. fiber (a) Untreated fiber, (b) Treated with NaOH, (c) Peroxide and (d) NaOH + peroxide.

layer of gold coating applied. The shape of composites reinforced with fibers from the Borassus fruit was also assessed using SEM examination. At various magnifications, SEM analysis was utilized to evaluate the surface of handled and unhandled coarse and fine fibers. In contrast to the cross section of the fiber, which shows multicellular structure, the SEM images of the treated fibers show roughening of the surface. Each fiber unit cell is made up of tiny cellulose particles that are bound together by lignin and hemicelluloses.

4. CONCLUSIONS

By performing surface treatment, the impurities from the fibers of the Borassus Flabellifer L Fruit were removed. Based on their size, the extracted fibers are separated into coarse and fine fibers. Chemical analysis is used to identify the fibers composition and the results show the presence of cellulose, hemicelluloses, and lignin. Because hemicelluloses was solubilized during the alkali and peroxide treatment of the fibers, the hemicelluloses concentration in the fibers was decreased after surface treatment. Through the integration of X-ray diffraction (XRD), scanning electron microscopy (SEM), and single fiber tensile testing, the study offers a comprehensive evaluation of these treatments on fiber crystallinity, morphology, and mechanical properties. Morphological changes are seen in the SEM analysis of treated and untreated fibers. The decrease in hemicelluloses content resulted in a rough surface with pores formation, as seen in the treated fibers. The spectrum analyses were further validated by wide-angle X-ray diffraction, which revealed a rise in the intensity of I002 (crystalline domain) for the fiber treated with both alkali and Peroxide is comparatively very high rather than individual treatment of Alkali and Peroxide. This work not only introduces a sustainable and underexplored natural fiber source but also provides valuable insights for optimizing fiber treatment processes. Consequently, it supports the development of lightweight, environmentally friendly composite materials, addressing a significant gap in the current literature and advancing applications across various industrial sectors.

5. ACKNOWLEDGMENTS

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