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RESEARCH ARTICLE

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Compressive characterization of different chemically treated *Borassus flabellifer* fruit fibers

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Abstract

In this current work, the *Borassus flabellifer* (BF) fruit fiber's surface has been modified using a variety of chemical processes in order to enhance the fiber properties. Investigations were done into the morphological, thermal, mechanical, and water absorption properties of BF fibers. Scanning electron microscopy (SEM) micrographs were used to visualize the effects of the surface alterations made to the BF fibers. These images demonstrate rough texture of the fiber surfaces with tiny holes and grooves as a result of the removal of lignin and hemicellulose from the fiber. To investigate the effects of surface changes on the fiber composition, Fourier transform infrared (FTIR) spectroscopy of BF fibers was performed. The enhancement in the crystallinity of the surface-modified BF fibers is confirmed by the energy-dispersive x-ray (EDX) and x-ray diffraction analyses. The thermogravimetric analysis demonstrates that the surface-modified fibers exhibit increase in thermal stability as well as the tensile strength. The water absorption test confirms the reduction of the hydrophilic nature of the chemically treated BF fibers.

Highlights

- Benzoyl chloride-treated BF fiber have shown the best surface modification.
- Chemically treated BF fibers show better crystallinity, as per XRD, SEM and EDX.
- Benzolization of BF fiber, increases the hydrophobic nature of the fiber.

KEYWORDS

Borassus flabellifer fruit fiber, composite, FTIR, SEM, TGA, XRD

1 | INTRODUCTION

Researchers have been inspired to create new green materials as a result of today's growing environmental concerns. Environmental friendliness, light weight, and biodegradability have emerged as the three most crucial factors in the creation of these novel materials. Because of its desirable qualities, such as eco-friendliness, renewability and user safety, natural fibers are considered acceptable and justifiable materials which can replace synthetic fiber.[1,2] Researchers are turning their focus to natural fibers as a result of the many drawbacks of

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synthetic fibers such as their inability to degrade, to be recycled, high initial processing costs, health hazards.[3] From previous studies it is found that the natural fibers are suitable substitute to the synthetic fibers due to their appreciated characteristics such as low density, widespread availability, good specific modulus, nontoxicity, and low cost. Few of the natural plant fibers, including hemp, cotton, flax, and jute, have gained recognition as qualitative reinforcements for fiber reinforced polymer (FRP) composites. Natural fiber reinforced polymer (NFRP) composites are used in a variety of non-structural applications, including furniture, packaging, decking, interior panels, vehicle components, and many others.[1] Natural fibers are distinguished based on their sources such as plants, animals, or minerals. Typically, plant fibers are natural fibers those are gathered from the stem, seeds, roots, and leaves of the plant. In composite materials, plant fibers are typically employed as the reinforcing component. The traditional uses of natural fibers include ropes, cushions, mats, caps, and strings. These fibers are typically lignocellulosic in composition, with cellulose as the main component and lignin and hemicellulose acting as binding agents.[4] The crystalline cellulose maintains the structural stability of the fiber by giving it strength and rigidity. The branching polymeric structure of the amorphous hemicellulose is what allows the microfibrils to link together to form a fiber network. Compared to hemicellulose, cellulose has a higher degree of polymerization. Hydrophilicity is a property shared by hemicellulose and other types of cellulose.[5] Another amorphous substance, lignin, which is naturally hydrophobic and is found in plant cell walls, acts as a natural glue and gives plant cells their structural stiffness. Pectin, lipid, and waxy compounds are also parts of plant fiber. Because natural fiber is hydrophilic and comprises of impurities such waxy substances, the main disadvantages of natural fiber include inadequate interfacial adhesion between the fibers and matrix, low thermal stability, and moisture sensitivity. As a result, one of the common strategies to overcome these constraints is to modify the surface of the fibers using various chemical treatments,[6] which has been used by many studies up to this point.

Several chemical processes are used to modify the natural fibers' surfaces, supporting the interaction of reagent functional groups with the fiber surface.[6,7] As a result, the fiber surface's texture changes, enabling good interaction between the fiber and the matrix. Alkaline, permanganate, anhydride and benzoyl chloride treatments, among others, were carried out to strengthen the interfacial interaction between the fibers and matrix, which results in better mechanical and thermal properties of the fabricated composites.

However, the main disadvantages of using natural fibers in composites are their hydrophilic character, poor interfacial strength, poor moisture sensitivity, weak heat stability, and inconsistent mechanical properties. These obstacles lessen the appeal of reinforcing with natural fiber. Numerous fiber surface changes have been made to address these issues in order to enhance the mechanical properties of the fiber and composite as well as the interfacial bonding.[6] As the structural constituents of natural fibers—wax, lignin, hemicellulose, cellulose, and pectin—absorb moisture from the atmosphere, their hydrophilic nature causes weak adhesive connections to form between the fiber and matrix in composite materials, as a result the stress transfer across the composites' interface is inefficient. Therefore, chemical surface modification of natural fibers is required to address these drawbacks. Chemical functional groups reacting with the surface of the fiber is one method of surface modification that alters the composition of the fiber.[8] The fibers become less hydrophilic as a result, which enhances their compatibility with the polymer matrix. Chemically treated natural fibers more successfully couple well with the matrix.

Borassus flabellifer (BF) tree is widely available in south and southeast Asia. The BF tree has tremendous economic and social implication. Each part of the tree are used to make home furniture, food, and items for culture and the arts. *Borassus* fibers are collected from the waste of the ripened *Borassus* fruits. Previous studies about the properties of BF fibers have been already reported by many researchers[9–12] regarding the investigation of the effect of alkali treatment on the mechanical, morphological, thermal properties and chemical compositions.

The concentration of alkali, treatment duration, and temperature are the factors that influence the surface modification of BF fiber.[9–12] Various NaOH treatments ranging from 0.5% to 29%; 20 min to 8 h; and from room temperature to 120°C, have been employed by various investigators. Alkali concentration should not be higher than the desired; doing so will cause excessive delignification, which will weaken and damage the fibers.[8] That means only delignification is not only the solution to enhance fiber properties, The alkaline pretreated fiber, when undergoes chemical treatments like maleic anhydride and benzoyl chloride, new functional chemical groups react with the fiber surface, thus enhancing the properties of fiber including the reduction in hydrophilicity and thermal properties. All the previous studies that have been till reported were based on the characterization of raw and alkaline treated BF fiber[9–12] at different concentration and treatment duration. However, 5% alkali treated BF fiber exhibits the best mechanical properties.[9] The present work is based on the effect of

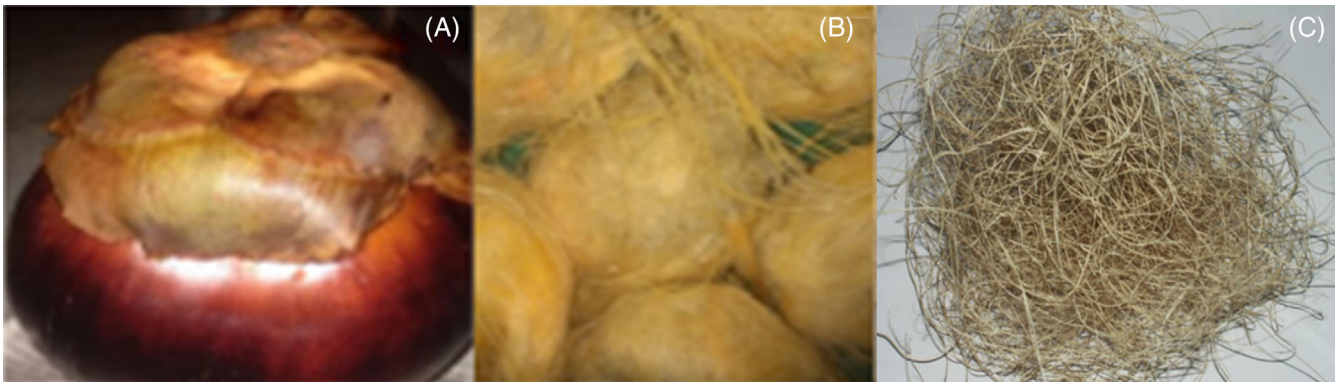


FIGURE 1 Images of (A) *Borassus flabellifer* fruit, (B) Extracted seeds, (C) extracted fibers.

chemical treatment such as alkaline (5%) as well as other chemical treatments such as maleic anhydride and benzoyl chloride on the morphological, mechanical, thermal, water absorptivity properties of BF fiber.

2 | MATERIALS AND METHODS

2.1 | Materials

Ripe BF fruits were gathered from the rural areas of Cuttack district, Odisha, India. The fruits were submerged in a tank of water for 2 weeks in order to separate the flaccid area from the fibers. Then the loosened flaccid area is removed in a subsequent phase using a water retting process, and the coarse and fine fibers are separated. The fibers are submerged in distilled water and washed to remove impurities. Finally, the fibers were dried initially in the sun for 48 h and then in a hot oven for 5 h at a temperature of 60°C. Finally, the fibers were cut into chopped pieces of about 3 mm length. Figure 1 presents the images of BF fruit, extracted seeds and finally the extracted fibers.

2.2 | Surface treatment of BF fibers

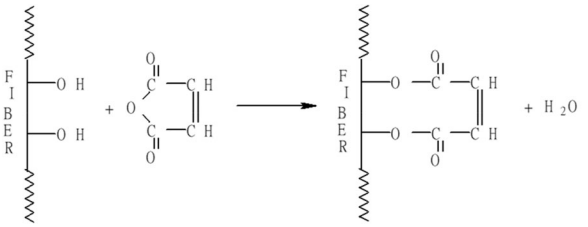
2.2.1 | Alkaline treatment

Alkaline treatment has been employed to modify the surfaces of natural fibers, Equation (1) describes how the hydrogen bond in BF fibers is disrupted by alkali (NaOH) treatment and draws off water molecules (H_2O).[13] As a consequence of the chemical treatment a certain amount of lignin, and hemicellulose are eliminated, resulting in the rough surface of fiber. The degree of polymerization is increased by this chemical process, enhancing the cellulose crystallinity, result in improved thermo-mechanical properties of BF fibers.[14] The BF fibers were submerged for roughly an hour at 60°C in a 5% aqueous solution of NaOH in this process. Following that, BF fibers were washed in distilled water for around 15 min and then the fibers are dried for 10 h in an oven set at 60°C and then sun dried for 48 h in order to de-moisturize the fiber.

$$\text{Fiber} - \text{OH} + \text{NaOH} \rightarrow \text{Fiber} - \text{O}^- - \text{Na}^+ + \text{H}_2\text{O} \quad (1)$$

2.2.2 | Maleic anhydride treatment

Maleic anhydride treatment of fibers results in the removal of the —OH functional group due to the production of water, as shown in Equation (2). The hydrophilicity of the fibers is decreased when a long chain polymer coating, or layer, is created on the surface of the fiber. The covalent bonding of the hydroxyl groups of BF fiber and the anhydride groups are effectively interlocked[6,7] The alkaline pre-treated fibers were submerged for 3 h at 50°C in a 5% maleic anhydride in acetone solution. After a distilled water wash to get rid of any remaining chemical residue, the treated fibers were kept for 10 h at 60°C in an oven before being sun dried for 48 h in order to demoisturize the fiber.

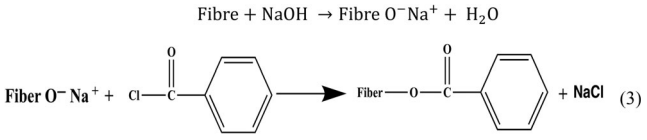


(2)

2.2.3 | Benzoylation treatment

After being treated an alkaline treatment, the BF fibers were then subjected to 10% NaOH and benzoyl chloride solution for 15 min as presented in Equation (3).

OH groups of the fibers are replaced with benzoyl group attached to the cellulose mainstay, thus increases the hydrophobic nature of the fiber.[6] Following that, the treated fibers were absorbed in ethanol for an hour to eradicate the benzoyl chloride that had become stuck to the surface of the fiber. The fibers were then rinsed in distilled water and dried for 24 h in an oven set at 60°C and then sun dried for 48 h so as to de-moisturize the fiber. The chemical process carried out as per the following reaction presented below as per Equation (3).



3 | CHARACTERIZATION OF THE BF FIBER

3.1 | Physical characterization

Using a pycnometer, the density of the BF fiber was determined. The experiment followed ASTM D-792 guidelines. The immersion liquid was kerosene.[15,16] Equation (4), which is provided below, was used to calculate the density of BF fiber.

$$\rho = \frac{(m_2 - m_1)}{(m_4 - m_1) - (m_3 - m_2)} \times \rho_k \quad (4)$$

where ρ signifies the density of the BF fiber, ρ_k signifies the density of kerosene, m_1 signifies the mass of the vacant pycnometer in kilograms, m_2 stands for the mass of the pycnometer that included chopped BF fibers in kilograms, m_3 stands for the mass of the pycnometer that had both chopped BF fibers and kerosene in kilograms, and m_4 stands for the mass of the pycnometer that contained kerosene in kilograms.

3.2 | Scanning electron microscopy analysis

The scanning electron microscopy (SEM) analysis of all treated and raw BF fiber samples was done to determine the morphology of the fibers. A 10 nm gold overlay was applied to all of the BF fiber samples. The surface morphology of gold coated fibers was carried out in a Leo Supra 35VP SEM machine operating at 20 kV and the SEM graphs were generated from the results.

3.3 | Fourier transform infrared spectroscopy

Each BF fiber sample underwent Fourier transform infrared (FTIR) spectroscopy, which was conducted in the spectral region (wavenumber) of 4000–500 cm^{-1}. A total of 14,000 scans for each BF fiber samples were obtained for the investigation, with the resolution of 4 cm^{-1}. A Nicolet AVATAR 360 spectrophotometer equipped with Germanium ATR technique was used to get the spectra in KBr pellets.

3.4 | X-ray diffraction

The x-ray diffraction (XRD) of the BF fibers were conducted to detect the amorphous and crystalline region of the fibers. To obtain the XRD patterns, Cu-K radiation in reflection mode operating at 40 kV and 150 Ma (XRD 7000 model) was used. The angle (2θ) varied from 10° to 30° at a rate of 0.05° advance per second of measuring period.

3.5 | Thermogravimetric analysis

All of the raw and surface-modified BF fiber samples, weighing roughly 5 mg to 9 mg, underwent thermogravimetric analysis (TGA) at a heating rate of 10°C/min from ambient temperature to 700°C in a (NETZSCH STA 449F3) apparatus.

3.6 | Tensile test

The BF fiber specimens for the tensile test were prepared. Each of the BF fiber with a gauge length of 50 mm was supported by thick paper sheet, was bound at the top and bottom position of the paper sheet by using glue, a hole was made centring at the centre of the paper sheet, according to the fiber gauge length. The fiber's total gauge length was exposed to ambient temperature (22°C) and relative humidity (55%). According to ASTM638B, the tensile characteristics of the fiber specimens were tested in a UTM (Instron 3369), at a feed rate 0.5 mm/min.

4 | RESULTS AND DISCUSSION

The surface of natural BF fibers is modified chemically to alter their morphological (SEM, FTIR), thermomechanical (XRD, TGA), and water absorption properties. In different

sections, the effects of each chemical treatment on untreated *Borassus* fiber are examined and explained below.

4.1 | Scanning electron microscopy

SEM is used to analyze the morphology of BF fibers is presented in Figure 2. Untreated fiber in Figure 2A has a smoother surface due to the presence of fatty and waxy materials. The lignin acts as the cementing substance which binds the cells and holds the whole units. In the case of NaOH-treated fiber, due to the exclusion of hemicellulose and lignin, as presented in Figure 2B, it has a rough texture (grooves and pits).[17] Figure 2C represents a rough surface with numerous tiny pits and holes. Following pre-alkali treatment, the fibers were generally treated with maleic anhydride, resulted in removal of fatty deposits from the fiber surface and made the fiber surface rough.[18] still, in both treatments, some sticky substances, such as lignin, were found on the fiber surface. Benzoyl chloride-treated fibers have shown the best surface modification, as indicated in Figure 2D, because of the elimination of much of the lignin as well as hemicellulose. Further, due to treatment with benzoyl chloride, the benzoyl group replaces the OH group resulting in the hydrophobic nature of the fiber.[4]

4.2 | FTIR spectroscopy

The FTIR spectra of BF fibers are displayed in Figure 3. The band intensity in between 3000 and 3600 cm^{-1} is due to the stretching O—H group in hemicellulose, lignin, along with cellulose. A strong and intense peak is observed at about 3320 cm^{-1} in the case of all the treated and untreated fibers. Within the aforementioned band, that ranges from 2500 to 3000 cm^{-1}, there is another prominent peak at 2907 cm^{-1} which displays the stretches of the —CH group in hemicellulose and cellulose.[14] The vibration of the water molecules (H—O—H) found in cellulose results in an aromatic ring of lignin and a peak at 1618 cm^{-1} in the spectrum,[17,19] which is found more intense in case of chemically treated fiber as compared to the raw BF fiber. The peak at 1318 cm^{-1} caused by the C—O stretching and aromatic bending of lignin, A peak is found at 1260 cm^{-1} in the band range of 1232–1270 cm^{-1} is attributed to the C—O—C stretching vibrations, indicating the presence of cellulose and lignin, which is more prominent in case of benzoyl chloride

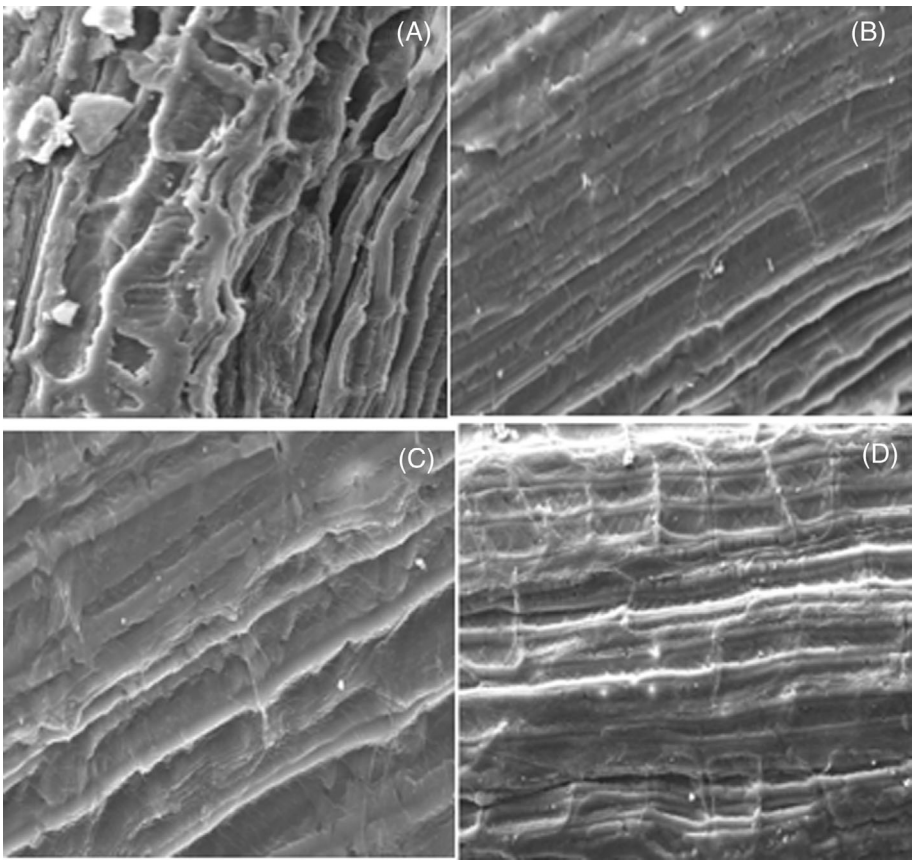


FIGURE 2 Scanning electron microscopy image of the *Borassus flabellifer* fiber before and after treatment.

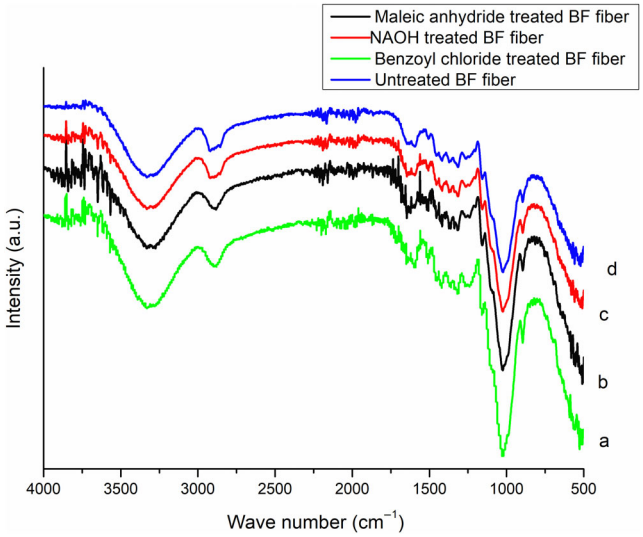


FIGURE 3 Fourier transform infrared of *Borassus flabellifer* (BF) fibers.

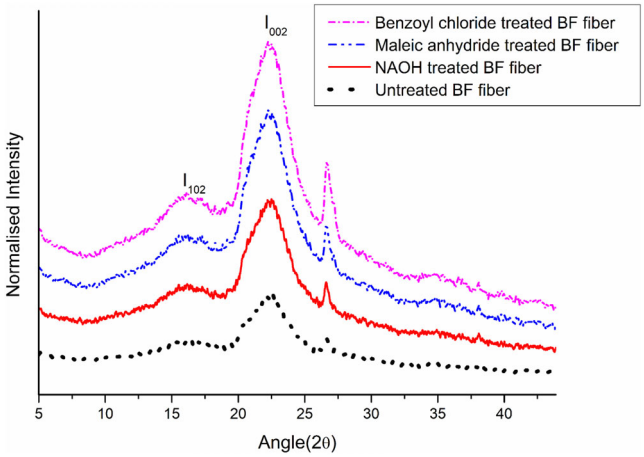


FIGURE 4 X-ray diffraction of *Borassus flabellifer* (BF) fibers.

treated as compared to other treated and raw fibers.[17] A high peak is observed at 1022 cm^{-1} in the band range between 920 and 1140 cm^{-1} is caused by the C—O stretching vibrations in cellulose which found to more intense in case of all chemically treated fibers as compared to the raw fiber.[18] A second peak, demonstrating the glucose linkage in cellulose, is visible at 897 cm^{-1}.[17]

4.3 | X-ray diffraction

All the BF fibers with and without surface modifications are shown in Figure 4 as XRD images. Around 2θ of 16° and 22°, two sharp peaks were detected, which represent the crystallographic planes of the cellulose (I_{101} and I_{002}). The peaks for the untreated BF fibers were found at 16.020 and at 22.10 respectively. The shifting of the higher peak, that is, I_{002} towards the right, is indicated for the alkaline-treated and maleic anhydride-treated fibers, which can be found at 22.200 and 22.260 respectively. The lower peak, which shows I_{101} for surface-modified fibers, in particular for benzoyl chloride-treated fiber, seems slightly skewed to the right and was acquired close around 16.12°, and the higher peak, that is, I_{002} was found nearby at 22.32°. According to the figure, all BF fibers, whether raw or surface-modified, exhibit the same signal properties. All treated fibers' crystallinity index rise relative to untreated fibers, showing an increase in the proportion of crystalline cellulose, with benzoyl chloride-treated fiber exhibiting the highest index. The good crystalline structure of the fiber surface is the result of the chemical treatment's removing amorphous materials.[10] All the various surface-modified BF fibers' crystallinity indices were measured and displayed in Table 1. The following Equation (5) has been used to compute the crystallinity index (CI).[20]

$$\mathrm{CI}\% = [(I_{002} - I_{101})/I_{002}] * 100 \quad (5)$$

Table 1 displays crystallinity measurements in chemically treated fibers compared to natural fibers. It was observed that BF fiber treated with benzoyl chloride has a greater crystallinity index of 44.58% than that of other chemical treatments.

TABLE 1 Measures of crystallinity in chemically treated versus natural fibers.

Material	I_{002}	I_{101}	I_c (%)
Untreated BF fiber	33.94	20.42	39.83
Alkali treated BF fiber	47	27.72	41.02
Maleic anhydride treated BF fiber	44	24.48	44.34
Benzoyl chloride treated BF fiber	47.73	26.45	44.58

Abbreviation: BF, *Borassus flabellifer*.

4.4 | Energy-dispersive x-ray analysis

The proportions of carbon and oxygen in untreated and various chemically treated BF fibers are shown in Table 2. An energy-dispersive x-ray (EDX) studies was performed to assess the oxygen-to-carbon ratio present in the BF fibers. Lignin serves as an adhesive and gives plant cells their structural integrity. A higher carbon content means that there is more lignin in the fiber.[21] It was found that for raw BF fiber the O/C ratio is 0.494. The natural fiber has more lignin than other types of

TABLE 2 Energy-dispersive x-ray analysis of *Borassus flabellifer* (BF) fibers.

Taxonomy of sample	% of carbon	% of oxygen	Ratio of O/C
Untreated BF fiber	66.9	33.1	0.494
NAOH treated BF fiber	64.1	35.9	0.560
maleic anhydride treated BF fiber	57.4	41.4	0.721
Benzoyl chloride treated BF fiber	55.4	43.2	0.779

chemically treated fiber due to its higher carbon content. It has been noticed that the O/C ratio increases (0.494–0.779) as a result of chemically modifying the surface of fibers. As indicated in the table, the BF fiber treated with benzoyl chloride had the highest oxygen-to-carbon ratio of 0.779. The amount of cellulose in the BF fiber increases as the extraneous materials including wax, hemicellulose and some parts of lignin is removed from the fiber surface by benzoyl chloride treatment. The o/c ratio increases by 0.560 and 0.721, respectively, in the alkaline and maleic anhydride-treated fibers.

4.5 | Thermogravimetric analysis

Figure 5 displays the TGA curves for both raw and treated BF fibers. Findings indicate that there are two primary processes by which the BF fibers thermally degrade. From 50 to 120°C, the untreated BF fiber loses weight more quickly than surface-modified BF fibers. This is owing to the evaporation of moisture from the fiber. For raw BF fiber, weight reduction was about 15%, while for surface-modified fibers, it was roughly 9%. Hemicellulose along with lignin depolymerize at the temperature range of 240–350°C.[18] Weight loss occurs in this temperature range; untreated BF fiber loses about 45% of its weight. However, all of the modified fibers exhibit a predicted reduction in weight of 36%–39% in this range of temperatures, suggesting better thermal stability of the fiber following surface modification. It is found that the residual ash takes up almost 24% of the total weight of the treated BF fiber when it is heated to 450°C. Chemically treated fibers can contain up to 32% more ash than untreated fibers because there is more cellulose-lignin bonding.[17,22]

4.6 | Tensile test

The results of the tensile testing are shown in Figures 6 and 7 and Table 3. The removal of materials like hemicellulose and lignin from the raw fiber leads to a more

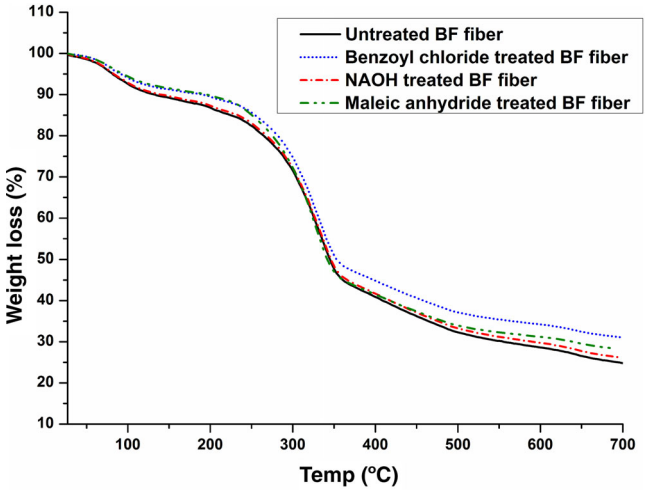


FIGURE 5 Thermogravimetric analysis of treated and untreated *Borassus flabellifer* (BF) fibers.

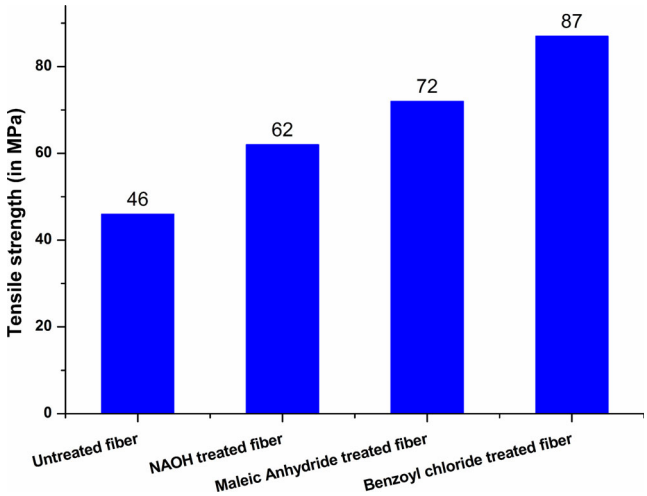


FIGURE 6 Tensile strength treated and untreated fibers.

densely packed, crystalline cellulosic structure, which is what strengthens the fiber.[6] It has been revealed that surface-modified BF fibers treated with Benzoyl chloride exhibit a significant increase in tensile strength and Young's modulus as compared to fibers any other samples such as NAOH treated BF fiber, Maleic anhydride treated BF fiber and raw BF fiber, which is also supported by FTIR, XRD, SEM and EDX analysis.

4.7 | Water absorptivity

Figure 8 demonstrates the findings of a 105 days water absorption test on all BF fibers, including raw and surface-modified fibers. It was found that the BF fibers' water absorption pattern exhibits non-Fickian behavior[15] The presence or absence of boundaries is the primary

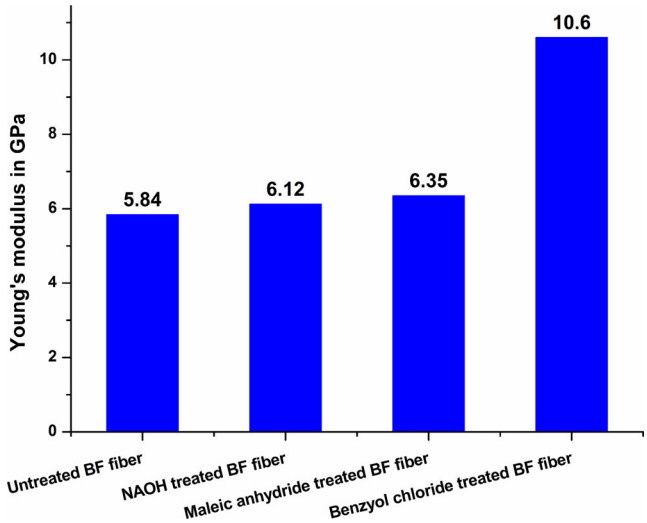


FIGURE 7 Young's modulus of *Borassus flabellifer* (BF) fibers.

TABLE 3 Tensile strength of the treated and untreated samples.

Treated samples	Tensile strength (in MPa)	Young's modulus (in GPa)
Untreated fiber	46	5.84
NaOH-treated fiber	62	6.12
Maleic anhydride-treated fiber	72	6.35
Benzoyl chloride-treated fiber	87	10.6

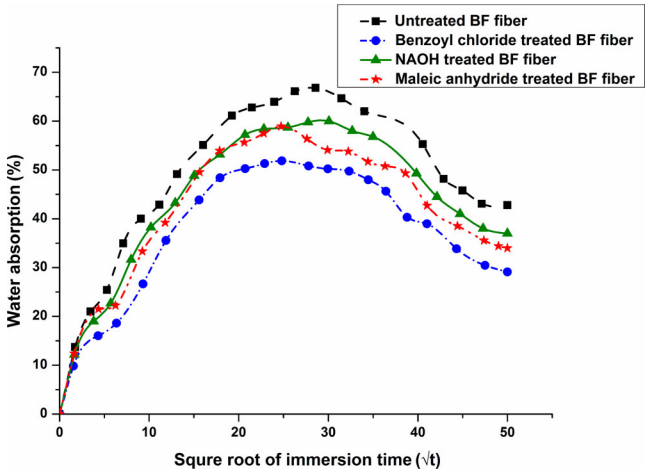


FIGURE 8 Water absorptivity of *Borassus flabellifer* (BF) fibers.

distinction between Fickian and Non-Fickian Diffusion; Fickian diffusion lacks boundaries, but Non-Fickian diffusion has a distinct boundary Water molecules enter the amorphous regions of the cellulose and hemicellulose during the test, causing hydrogen bonds to be broken and the intermolecular distances of the cellulose chain to increase. This causes the cellulose chain to stretch, thus result in fiber stretch out the rate of water absorption first rises linearly with time, but after a considerable amount of time, the rate starts to decline and ultimately approaches the steady state. A decreasing tendency was seen in both treated and untreated BF fiber about 55 days later. This could be the result of hydro-degradation or mechanism of material hydrolysis.[20] It was found that compared to all surface-modified BF fibers, raw fiber absorbs more water. Benzoyl chloride-treated fibers show the best results, by absorbing least amount of water.

5 | CONCLUSION

The surfaces of BF fibers were subjected to a number of modifications. The BF fibers were treated with a variety of chemicals, including alkaline, maleic anhydride, peroxide, and benzoyl chloride. The highest tensile strength is shown by the benzoyl chloride-treated fiber, although surface-modified fibers exceed natural BF fiber in this regard. According to the TGA curves, the treated fibers are thermally stable up to 240°C. According to XRD, the treated fibers had a greater crystallinity index than the untreated fiber. The lignin-cellulose complex is proven to have a better crystalline structure by the BF fiber treated with benzoyl chloride, supported by FTIR and SEM, and EDX analysis. When compared to natural and other treated fibers, the chemically benzoyl chloride-treated BF fiber has a very low water absorption property. According to the experiments stated above, chemically treated BF fibers offer better properties. The results of benzoyl chloride treatment on BF fiber are the most remarkable.

The water absorption property for untreated BF fiber is at its maximum since it contains all elements including hemicellulose, wax, and lignin. This highlights the fiber's hydrophilic nature. Hemicellulose, lignin, and wax removal after surface treatment promotes the crystalline structure of fiber. Surface treatment with benzoyl chloride and maleic anhydride reduce the treated fibers' capacity to absorb water. This is due to the chemical functional groups attached to the fiber surface reduce the hydrophilicity of BF fiber. Furthermore, when BF fiber surfaces were treated with alkaline, the water absorption rates are lower than the untreated fibers. This may be due to exclusion of hemicellulose which is more hydrophilic by nature.

CONFLICT OF INTEREST STATEMENT

There are no competing interests that the authors can identify that are pertinent to the subject matter of this

research. No organization provided the authors with funding for the work they submitted.

DATA AVAILABILITY STATEMENT

The corresponding author is willing to provide the datasets created during and/or analyzed during the current work upon reasonable request.

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