Effect of benzoylation treatment on tensile properties of sustainable bamboo species: *Pseudoxytenanthera ritcheyi*

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Abstract. This study undertakes a comprehensive exploration into the impact of chemical reagents on the distinctive bamboo species, Pseudoxytenanthera ritcheyi (*P. ritcheyi*). The research involves a meticulous examination of chemical characterization, encompassing subsequent chemical modification processes like alkaline treatment and benzoylation treatment applied to virgin bamboo fibers. The benzoylation treatment, preceded by alkaline pretreatment, is conducted with varying durations—10, 15, and 20 minutes. Various physical and mechanical characterization techniques are employed to assess the hydrophobic nature and strength of the fibers. Tensile properties of both treated and untreated single fibers of *P. ritcheyi* bamboo are measured using a computerized universal testing machine in accordance with ASTM standards. The obtained results are then compared with reference samples and commercially available natural fibers. The primary objective of this investigation is to analyze and juxtapose the axial tensile modulus and ultimate strength of *P. ritcheyi* bamboo fibers, employing different surface modification techniques. The thermal stability of both treated and untreated fibers is scrutinized through thermogravimetric analysis. Additionally, scanning electron microscopy (SEM) is utilized to examine the fracture morphology of the samples. The results obtained are benchmarked against those of other natural fibers, leading to the conclusion that the 15-minute soaked benzoylated *P. ritcheyi* single fibers exhibit exceptional performance in terms of chemical, physical, and mechanical attributes. This research not only contributes valuable insights but also presents a sustainable solution for reinforcement in composite material engineering and product development.

Keywords: Bamboo fibers; Pseudoxytenanthera species; chemical modifications; physical characterization; mechanical characterization; tensile properties; scanning electron microscopy

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1. Introduction

Plant fibers are a promising solution for sustainable development by diminishing the quantity of carbon footprint. As per the reports [1, 2], natural fibers had compatible strength and biodegradability as compared to synthetic fibers. In fiber-based composites, reinforcement fiber plays a critical role in defining the enhancement of mechanical properties [3]. Among the available natural plant wealth bamboo provides excellent mechanical and thermal properties with elegant damage tolerance [4, 5]. Various bamboo-based products were developed using single bamboo fibers, fiber bundles, and bamboo strips [6]. The orderly arranged thick wall structure and higher cellulosic content reveal the superiority of bamboo in high-strength applications [6-8]. Bamboo fibers possess a remarkable strain to failure value which was higher than that of commercially available synthetic fibers such as carbon and glass fibres [9]. Various studies reported [10, 11] that bamboo resulted in higher tensile strength and modulus as compared to wood-based natural fibers. In people's daily lives and cultures, bamboo plays a significant role because of its remarkable specific strength, high specific modulus, rapid growth, and viable flexibility [12-14]. Natural fibers, such as bamboo, exhibit inadequate resistance to moisture, resulting in heightened water absorption and premature fractures during tensile tests. Consequently, their mechanical performance is subpar, limiting their potential applications in industrial settings. Among the myriad bamboo species thriving in the Western Ghats, numerous research studies[15, 16] have highlighted the exceptional physical, chemical, and mechanical attributes of *P. ritcheyi*. Consequently, for this endeavor, individual bamboo fibers from the *P. ritcheyi* species were specifically chosen.

Amidst the diverse array of bamboo species flourishing in the Western Ghats, a multitude of research endeavors, including a notable study[17], have underscored the transformative impact of chemical modification on plant fibers, elevating their material properties significantly. Alkali treatment, often referred to as the Mercerization technique, stands out as a prevalent fiber treatment method renowned for enhancing fiber/matrix adhesion and endowing composites with remarkable mechanical properties [18, 19]. Notably, a study [20] emphasized the impressive outcomes achieved by subjecting Banyan Fiber to a 5% NaOH concentration for 60 minutes, resulting in heightened tensile strength and modulus. Consistently, optimal results were consistently reported at a 5% NaOH concentration with a 60-minute duration across various natural fibers [21], with alkaline treatment being a recurring theme in studies on natural fibers [22]. However, a noticeable gap exists in research regarding the impact of benzyl chloride on natural fiber properties [23]. In this investigation, a meticulous approach was adopted, involving NaOH pretreatment before immersing the fibers in benzoyl chloride [24]. Drawing inspiration from a study [25] showcasing elevated tensile strength in Palmyra palm leaf stalk fibers following benzoyl chloride treatment, our study delves into the detailed exploration of chemical modification processes—specifically, alkaline treatment and benzoylation treatment—and their influence on the tensile behavior of *P. ritcheyi* single bamboo fibers. A thorough comparative analysis was conducted, taking into account varying fiber soaking times (10, 15, and 20 minutes) in benzoylation treatment, and drawing distinctions between untreated (PRF) and NaOH-treated fibers (NPRF) as reported [26]. The findings unearthed in this study not only spotlight the potential of bamboo fibers, particularly from the *P. ritcheyi* species, as reinforcements in natural fiber-reinforced composites but also present a compelling solution for high-performance, sustainable composite material engineering applications.

2. Materials and Methods

2.1 Plant material

Pseudoxytenanthera ritcheyi (*P. ritcheyi*), a unique and underexplored bamboo variety, collected from JNTBGRI, was selected for the present work. The voucher specimen (*Accession No. 64*) was kept in the herbarium for future reference.

2.2 Fibre Extraction and Specimen Preparation

Fibres from the outer portion of *P. ritcheyi* bamboos were isolated mechanically[27]. ASTM D3822-07 standard procedure was followed for the specimen preparation and all the specimens were kept in sealed packets at 23ºC and 65% RH.

2.3 Chemical modification of fibers

The surface hydroxyl groups of cellulose in the bamboo fibres are chemically modified using the benzoylation reaction [28, 29]. The fibres are first soaked with 5% NaOH solution for 2 hours followed by washing with water to remove excess alkali. These alkali-treated fibres are then treated with benzoyl chloride for 10, 15, and 20 min. After that, the benzoylated samples were thoroughly washed with water and kept immersed in the ethyl alcohol for one hour to remove any unreacted benzoyl chloride. Once again, the fibres were washed with distilled water and dried completely at 75ºC overnight in an air oven and the dried samples were kept in vacuum-sealed containers for further analysis. The proposed scheme of reaction is as follows in **Figure 1**.

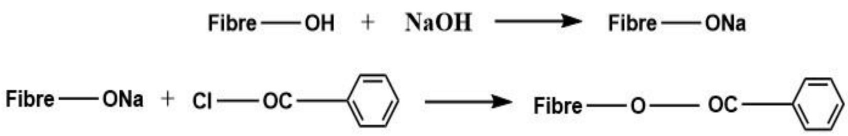


Fig.1. Fibre modification by benzoylation treatment

2.4 Physical characterization of fibers

Measurement of Fibre Diameter. Optical microscopy (LEIKA DM750M model) was used to calculate the diameter of the treated and untreated *P. ritcheyi*fibres and confirmed by Scanning Electron Microscopy (SEM- JEOL JSM-IT510).

Density (ρ). The density of the specimens was measured using the pycnometer setup[30] using toluene as a reference The density of fibres is given by the equation (1),

$$\rho = \frac{(m_2 - m_1)}{(m_3 - m_1) - (m_4 - m_2)} \times \rho_t \qquad (1)$$

Where, $\boldsymbol{\rho}$ and $\boldsymbol{\rho_t}$ defines the density of *P. ritcheyi*bamboo fibers and toluene respectively in g/cm^3. $m_1 m_2$, m_3 and m_4 denotes the empty pycnometer mass, mass of the pycnometer filled with chopped bamboo fibers, mass of the pycnometer filled with toluene, and total mass of the pycnometer filled with both fibers and toluene together in g, respectively

Moisture Content. The moisture content of the samples was determined by oven drying method [31].The precisely weighed samples were placed in a hot air oven at a temperature of 105°C at an interval of 15 minutes. Then weigh the samples at an interval of 15 minutes until reach a weight difference of 0.1% or less. The percentage moisture content [32] in the bamboo fibres was defined by the equation (2),

$$\% \text{ Moisture Absorption} = \frac{(M_0 - M_1)}{M_0} x100 \quad (2)$$

Where, M_0 and M_1 are the fibre weight before and after drying in grams.

Water Absorption. Water absorption capability of both treated and untreated fibres was analysed using the ASTM D570-98 standard. Water absorption tests were conducted for a duration of 1 hour, in alignment with previous studies on various natural fibers [33]. These studies have consistently demonstrated that stable absorption capabilities are achieved within a 60-minute timeframe. This study comprises an estimation of the absorption ability of bamboo fibres with a time interval of 10, 30, and maximum of 60 min. The samples after water immersion were wrapped with soft tissues to remove excess water content and weighed the same using a digital weighing machine with an accuracy of 0.0001 g. The difference in weight before and after immersion was quantified and used for determining the percentage of water absorption. The measurements of water absorption shed insight into the necessity and enhancement of mechanical properties enabled by chemical modification treatments.

2.5 FTIR Analysis

The Infrared spectroscopic analysis of the untreated and treated rhizome powders was carried out using the FTIR-IR Prestige-21 (Shimadzu Corporation, Japan). The powdered bamboo fibres were mixed Potassium bromide powder (ratio 1:10) then formed into a pellet for analysis. The graph was plotted between the percentage of transmitted IR light and the wave number (4000 to 400 cm^{-1}).

2.6 Thermogravimetric Analysis (TGA)

The TGA (Perkin Elmer/TGA4000 instrument) was performed on both treated and untreated samples. In this study, is used to evaluate the thermal degradation of fibres. The test was conducted within a controlled environment of nitrogen flow set at 20 ml/min and at a heating rate of 10°C/min. The temperature ranges from 30°C to 700°C.

2.7 Morphological study by Scanning Electron Microscopy (SEM)

Fracture studies on treated and untreated samples after tensile testing were analyzed by using a SEM (JEOL JSM-IT510). A detailed comparison study was performed regarding the influence of benzoyl chloride on mechanical properties against untreated samples using SEM images.

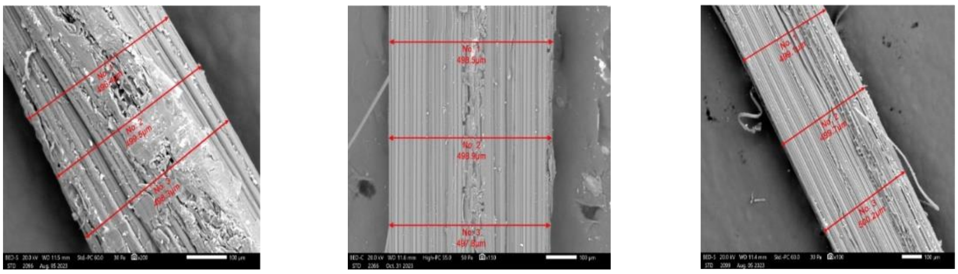
2.8 Tensile Test

Tensile tests were executed to determine the tensile stress and modulus of single bamboo fibres using the ASTM D3822-07 standard. In our study, a Computerized UTM (Make: KALPAK UTM, Model No. KIC-2-1000-C) with a maximum load capacity of 100 kN and a load cell of 0.1 kN is used. Tests were performed on treated and untreated fibers and the results were compared against each other and with some commercially available natural fibers.

3. Results and Discussion

3.1 Physical characterization

The fibres were physically characterized in terms of fibre diameter and density. The variation of diameter along the length of the fibres at three different locations were measured using an optical microscope and depicted the same with SEM (JEOL JSM-IT510) images shown in **Figure 2**. The treated and untreated fibres in the diameter range 0.4mm to 0.5mm were selected for the tensile testing.



PRF ***NPRF*** ***BPRF***

Fig. 2. Measurement of fiber diameter using SEM images.

The density of the fibres is found to improve on the benzoylation treatment (**Table 1**). This can be attributed to the removal of hemicellulose and lignin and the addition of the benzoyl group [28] . BPRF 15 showed highest value for density (1.73 g/cm^3). Earlier reports were available on the effect of benzoylation treatment on natural fibres[34] also confirm that the benzoylation reaction increases the density. The moisture content and percentage water absorption of the samples were also reduced by the benzoylation treatment. The moisture content of the untreated sample was 4.8% which is reduced by 2.5% in BPRF samples. The water absorption pattern of both treated and untreated samples is depicted in **Table 1**.

Table 1. Experimental results of physical characterization

Sl. No.	*P. ritcheyi* bamboo fibers	Density (g/cm^3)	Moisture content (%)	% Water Absorption		
				10 min.	30 min.	60 min.
1	PRF [26]	1.24	4.8	58	78	85
2	NPRF [26]	1.51	3.3	52	62	76
3	BPRF 10*	1.40	3.7	50	65	74
4	BPRF 15*	1.73	2.5	44	57	60
5	BPRF 20*	1.60	2.8	53	69	79

*Designated the soaking time in minutes of *P. ritcheyi* bamboo fibers on *benzoylation treatment*

Tests were carried out for 60 minutes [33], during which we periodically weighed the samples until the weight difference stabilized to within an accuracy of 0.1% or less. Our results were consistent with those reported in previous studies [16, 33]. The differences in density before and after treatments revealed the lesser number of pores in between the fiber components. The influence of benzoyl moiety on -OH groups of cellulose in bamboo fibers makes them hydrophobic. The SEM images shown in **Figure 4(b-c)** predict the morphology of fibers which revealed distinct surface characteristics.

3.2 FTIR Analysis

FTIR analysis conducted on both untreated and treated fibers revealed the presence of diverse functional groups, including phenolic hydroxyl, alkenic, aromatic, and beta glycosidic groups. These findings suggest that the primary constituents of the fibers encompass cellulose, hemicellulose, and lignin [35]. The distinct absorbance frequencies and their corresponding functional groups are detailed in **Table 2**. Assignment of various peaks in the spectrum was based on prior research [16]. All three samples exhibited a comparable spectrum, featuring prominent peaks at approximately 3344 cm^{-1} (indicative of phenolic -OH- stretching), 2923 cm^{-1} (C-H stretching), and 1598 & 897cm^{-1} (characteristic of the β-glycosidic linkage in lignin).

Table 2. FTIR identification chart of absorbance peaks.

Wavenumber (cm^{-1})		**Vibration & Functional Group**	**Corresponding Component**
PRF [16]	**BPRF**		
3344	3556	–O–H stretching	Cellulose, Hemicellulose & lignin
2902	2784	-CH_2& -CH stretching	Cellulose, Hemicellulose & lignin
1737	1735	C=O stretching	Hemicellulose
1597	1524	Aromatic ring vibration	Lignin
1508		Aromatic skeletal vibrations	Lignin
1458	1437	-OH, in plane bending	Cellulose, Hemicellulose & lignin
1388		CH bending	Cellulose & Hemicellulose
	1292	C-O stretch & OH in plane	Polysaccharides
1249	1188	C-O-C Asymmetric stretching	
	1091	C-O-C stretching & Aromatic C-H plane deformation	Cellulose & Lignin
1029		C-O stretching	Holocellulose & Lignin

3.3 Thermogravimetric Analysis

The TGA thermograms of different specimens are shown in **Figure 3**. The untreated fibre shows three stages of degradation corresponding to the loss of moisture (70-110ºC), hemicellulose (240-300ºC), and cellulose (300-350ºC). The maximum decomposition temperature of the PRF, NPRF and BPRF are 333.2ºC, 341.5ºC and 352.6ºC respectively. This clearly indicates that the benzoylated fibre shows exceptionally high thermal stability. Therefore, they can be considered suitable for engineering applications that involve temperatures below this range.

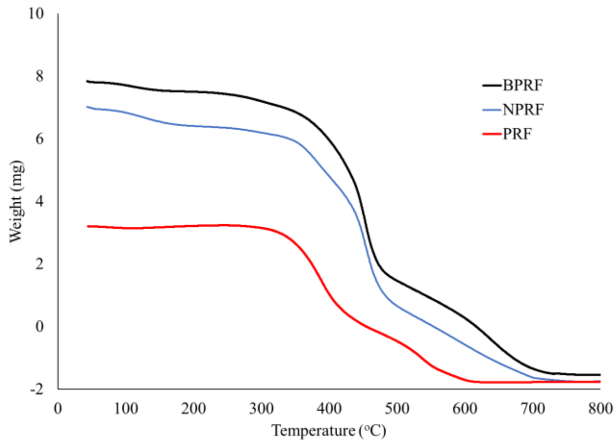


Fig. 3. TGA Curves of treated and untreated *P. ritcheyi* fibres

3.4 Tensile Test

Tensile tests were performed on single fibers of both treated and untreated samples in line with ASTM D3822-07 standard and the results were tabulated in **Table 3**. Tests were conducted on fiber gauge length of 20 mm, which provided the maximum tensile strength reported[15]. The defect-free samples were aligned vertically on the fiber attachment device in UTM using secure grips. Both ends of the fibers were attached with a paper frame tab as per ASTM D3822-07 using proper adhesive. In order to avoid any additional flaws or defects utmost care was taken for handling and fixing the sample specimens on the fiber testing attachment. The results were compared against the reference samples[15] considered and are shown in **Table 3**.

The maximum tensile stress values obtained from the tensile tests at a gauge length of 20mm were 465.56 MPa for untreated samples[15] and 598.54 MPa for benzoylated samples (BPRF 15). This clearly indicates that the at minimum gauge lengths (20 mm), benzoylated fibers of bamboo exhibit higher strength as compared to virgin fibers of *P. ritcheyi*[15].The results were also compared against the available data on the tensile properties of natural fibers reported[34].

Effect of Benzoylation treatment on tensile strength. SEM images of *P. ritcheyi*, show a densely and closely packed arrangement of fibers. The densely packed structure of respective species of bamboo compared to others reported[15] provided remarkable tensile strength. This structural integrity along the tensile direction of fibers distributes loads more efficiently and more resistant to breaking or failure. The morphology of fracture after tensile tests were observed under SEM that shows in **Figure 4** has more uniform and consistent pattern of fiber fracture due to the well-organized fiber orientation.

The effect of chemical modification of *P. ritcheyi* bamboo fiber on tensile stress, tensile modulus and tensile strain are presented in **Table 3**. Experimental data revealed that chemical treatments (Benzoylation treatment) improved the tensile strength of *P. ritcheyi* bamboo fibers. The fibers were treated at an interval of 10 minutes and by comparison, 15 minutes soaking time provided the maximum tensile strength and modulus values. The tensile strength value ranges from 465.56 MPa to 598.54 MPa which was 29% more than the untreated fiber. The values obtained were compatible with several studies reported[36]. The treatment of benzoylation eliminated the strength deduction factors such as hemicellulose and lignin and thus increased the strength dominating component cellulose to higher values. This caused the enhancement of tensile strength by the fibrils to rearrange along the tensile direction of fibres[28].Also, the benzoylation process changed the chemical composition of the fibers which subsequently affected their hydrophilic nature, mechanical properties, surface roughness, crystallinity, and thermal behavior. Interestingly, an increment in treatment duration caused individual bamboo fibers to shift from a brittle to a ductile mode, leading to remarkable tensile properties. This process changed the hydrophilic nature of fibers to a hydrophobic nature as reported in the literature[30].

The tensile modulus of treated single *P. ritcheyi* bamboo fiber shows a similar pattern depicted in the tensile strength of fibers as explained. The improvement in tensile modulus is attributed to the removal of hemicellulose and lignin which increases the cellulose crystallinity index. The tensile strain (elongation at break) of a single *P. ritcheyi* bamboo fiber varied due to benzoylation treatment. Results were tabulated in **Table 3**, observed a decreasing pattern of tensile strain compared against untreated fibers. As expected, the tensile strain value will be lower as there is improvement of the tensile stress properties. During chemical treatment procedures, the wax layer and the impurities vanish which facilitates thinner and stiffer fibres[28]. Reduction in tensile strain is also due to the removal of lignin and hemicellulose provided by the benzoylation treatment.

Table 3. Experimental results of tensile test

Sl. No.	*P. ritcheyi* bamboo fibers	Tensile stress (MPa)	Tensile Modulus (GPa)	Tensile Strain
1	PRF[26]	465.56	48.85	0.57
2	NPRF [26]	501.38	51.77	0.44
3	BPRF 10*	515.78	56.72	0.38
4	BPRF 15*	598.54	65.23	0.11
5	BPRF 20*	508.85	51.66	0.46

*Designated the soaking time in minutes of *P. Ritcheyi* bamboo fibers on***benzoylation treatment***

The tensile strength of treated single *P. ritcheyi* bamboo fiber showed comparable value with available varieties of natural fibers[34] shown in **Table 4**. Moving to tensile modulus some fibers possess the same value as that of chemically modified *P. ritcheyi* bamboofibers. The remarkable improvement in tensile strength and tensile modulus of benzoylated *P. ritcheyi* bamboo fibers compared with untreated ones indicated that the fiber became more ductile due to the removal of lignin and hemicellulose. Even though a decrease in tensile strain of chemically modified *P. ritcheyi* bamboo fibers, they maintain quite a high value compared to other fibers reported shown in **Table 4**. The benzoylation process facilitates the fibers to become stiffer with a decrease in tensile strain.

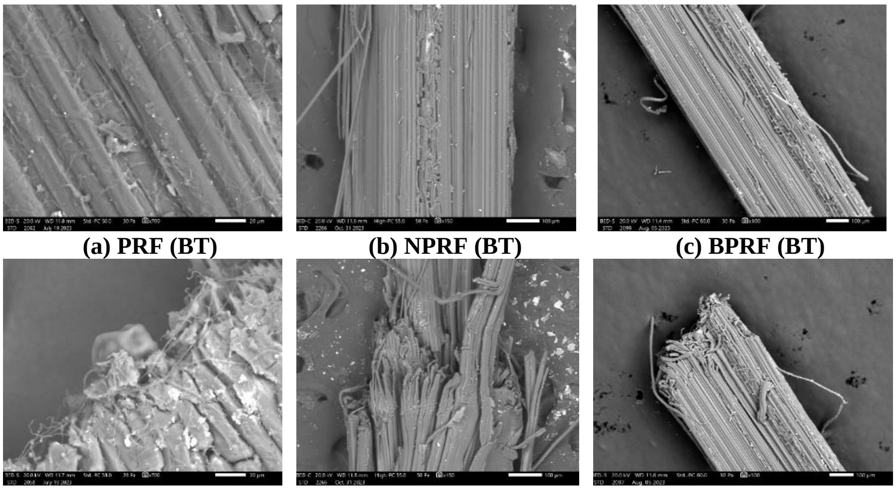
Table 4. Tensile performance study of natural fibers [34]

Natural & Synthetic Fibres	Tensile stress (MPa)	Tensile Modulus (GPa)	Tensile Strain
Jute	393-773	26.5	1.5-1.8
Hemp	690	70	1.6-4
Aramid	3000-3150	63-67	3.3-3.7

Kenaf	215.4	53	1.6
Pineapple	400-627	1.44	14.5
Flax	345-1035	27.6	2.7-3.2
Bagasse	290	17	-
Sisal	511-535	9.4-22	2.0-2.5
Coir	138.7	4-6	30
S-Glass	4570	86	2.8
E-Glass	2000-3500	70	0.5
***P. ritcheyi* Bamboo**	**598.54**	**65.23**	**0.11**

3.5 Fracture Morphology Study

Morphological study after fracture along tensile direction of the treated and untreated fibres were shown in **Figure 4**. Alkaline treatment of fibers changes the fiber surface morphology by removing the hemicellulose and lignin contents which enhanced the surface roughness property of the samples. -OH- groups on cellulosic fibers were transformed to -ONa- groups thereby reducing the hydrophilicity of fibers. By comparing the SEM images of treated with untreated samples, treated one shows better surface roughness followed by the removal of impurities, waxes along the length of fibers. Benzoylated fibers provide a clear image that predicts strong molecular surface microfibrillar structures followed by a thinner and stiffer fiber. During this treatment strength-diminishing factors like hemi cellulose, lignin, layer of wax, impurities etc were removed.



(a) PRF (BT) **(b) NPRF (BT)** **(c) BPRF (BT)**

(d) PRF (AT) **(e) NPRF (AT)** **(f) BPRF (AT)**

Fig. 4. SEM images of chemically treated single P. ritcheyi bamboo fibers: Before (BT) and after (AT) tensile testing.

Chemical treatments increase the cellulose contents of bamboo which enhanced the mechanical properties of single fibers. The addition of benzoyl moiety makes more hydrophobic. Surface modification process enhances the hydrophobic nature and mechanical strength properties, which is evident from the SEM images also.Morphology after fracture revealed a consistent pattern of fiber fracture in benzoylation treatments compared to untreated fibers. The scenario on fiber fracture shown above was in agreement with the increment in tensile strength and modulus properties reported[15].

4. Conclusion

This comprehensive study on the influence of chemical modifications on *Pseudoxytenanthera ritcheyi* (*P. ritcheyi*) bamboo fibers has yielded significant findings with implications for various industries, particularly in composite material engineering. Our research demonstrates that chemical modifications, particularly benzoylation, enhance the tensile strength by 29% and tensile modulus by 34% of *P. ritcheyi* fibers. Furthermore, the addition of benzoyl moieties induces a significant shift from hydrophilic to hydrophobic behavior in the fibers, accompanied by an increase in cellulose content and reduction in hemicellulose and lignin. Physical characterization studies corroborate these findings, with improved hydrophobicity observed in treated fibers, as evidenced by both SEM morphology and mechanical characterization analyses. The comparative study of fracture morphology between untreated and treated fibers provides valuable insights into surface interactions involved in chemical modifications.

Overall, our research underscores the potential of benzoylation as an optimized chemical modification treatment for *P. ritcheyi* fibers, rendering them suitable as sustainable and biodegradable reinforcements in composite materials. These findings hold promise for the development of eco-friendly alternatives to synthetic fibers, contributing to the advancement of green engineering practices and sustainable material technologies. By elucidating the transformative effects of chemical modifications on bamboo fibers, our study paves the way for further exploration and application of natural fiber composites in various industrial sectors, ultimately fostering a more environmentally conscious and sustainable future.

Ethics approval. The authors hereby state that the present work is in compliance with the ethical standards.

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Authors' contribution. NJ led the conceptualization, design, and execution of the analysis, while also overseeing data collection, experiments, and comprehensive data analysis. IS contributed significantly to conceptualization, analysis design, experiment execution, data analysis, and manuscript development. KBK played a crucial role in conceptualization, design, data collection, experimentation, thorough data analysis, manuscript creation, and study supervision. All authors contributed to data interpretation, writing, and revising the manuscript, ensuring its accuracy and integrity.

Declaration of Competing Interest. The authors declare that they have no known competing financial interests or personalrelationships that could have influenced the work reported in this paper.

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