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Transforming bamboo-guadua: Fiber extraction and characterization for biocomposite materials

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ABSTRACT: Guadua *angustifolia*, a South American bamboo species, has a long-standing tradition of use as a construction material. Despite numerous studies on its mechanical and structural properties, research on its fibers and extraction processes is scarce. This paper aims to enrich the technical-scientific understanding necessary for enhancing the utilization of Guadua *angustifolia* bamboo fibers. It outlines an experimental program that developed and assessed an extraction methodology. The fibers produced were characterized by their length, density, water absorption, mechanical properties, morphological features, and FTIR spectroscopy. The extraction method yielded fibers up to 20cm long, with an average density of 1.4g/cm^3 and a tensile strength of approximately 350MPa, among other properties.

KEY WORDS: Natural fibers; Bamboo-guadua fibers; Extraction methodology; Physical characterization; Mechanical characterization.

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RESUMEN: *Transformando el bambú-Guadua: extracción y caracterización de las fibras para materiales biocompuestos.* La Guadua *angustifolia*, una especie de bambú sudamericana, tiene una larga tradición de uso como material de construcción. A pesar de existir numerosos estudios sobre sus propiedades mecánicas y estructurales, las investigaciones sobre sus fibras y procesos de extracción son escasas. Este artículo tiene como objetivo enriquecer el conocimiento técnico-científico necesario para aumentar la utilización de las fibras de bambú Guadua *angustifolia*. Propone un programa experimental que desarrolló y evaluó una metodología de extracción. Las fibras obtenidas se caracterizaron por su longitud, densidad, absorción de agua, propiedades mecánicas, características morfológicas y espectroscopia FTIR. El método de extracción arrojó fibras de hasta 20 cm de largo, con una densidad promedio de 1.4 g/cm^3 y una resistencia a la tracción alrededor de 350 MPa, entre otras propiedades.

PALABRAS CLAVE: Fibras naturales; Fibras de bambú-guadua; Metodología de extracción; Caracterización física; Caracterización mecánica.

1. INTRODUCTION

Due to the increasing environmental awareness, researchers and industries have shown interest in substituting polluting materials with sustainable and renewable resources. Consequently, natural fibers, obtained from animals, plants or agricultural wastes (1, 2), have become attractive to many industries, including the composite sector (3). The global market for natural fiber composites is expected to grow by 5.2% from 2021 to 2026, reaching an estimated 0.6 billion USD (4).

Guadua *angustifolia* (guadua), a bamboo species that is highly prevalent in South America (5), is native to Colombia, Ecuador and Venezuela (6). This plant has been integral to the folklore, environment, and socio-cultural development of the western region of Colombia. Although the mechanical and physical properties of guadua have been extensively investigated in both raw (7-12) and laminated forms (13-19), research on guadua fibers is lacking (20-22), especially concerning the methodologies used for extracting its fibers (23-25). This may be due to the high difficulty and cost of the extraction process (26, 27).

Methods currently used for obtaining natural fibers include mechanical extraction, chemical methods, and combination of both (28). Each extraction method has specific characteristics that make it suitable depending on the requirements of the extraction process and, more importantly, the desired characteristics of the obtained fiber, which depends on the intended application. Mechanical extraction methods are highlighted by their high-performance efficiency and low environmental impact; however, they have a very high energy consumption. Conversely, chemical methodologies are less energy-intensive but are not-ecofriendly. Chemical-mechanical methodologies allow for high yield with lower energy consumption; however, these, like chemical methodologies, may not be environmentally sustainable due to the use of chemical agents (29).

Mechanical methods can involve heat steaming, steam explosion, retting, crushing, rolling in a mill and griding (30). These methods typically produce coarser bamboo fibers (27), which may contain large amounts of non-cellulosic components, negatively affecting the mechanical performance of fibers. In case of crushing processes, Sreenivasan et al. (31) design a mechanical decorticator that appears to be efficient for obtaining fibers from the soft leaves of *Sansevieria cylindrica* plants, producing fibers suitable for use as reinforcement in composites. For bamboo, the crushing process involves breaking the culm into small pieces using a crushing roller and extracting the coarse fibers using a pin roller (32). According to Popat & Patil (33), the main challenge in using this procedure to obtain bamboo fibers is that is produces short fibers, which tend to become powdered after mechanical over-processing.

Common procedures in chemical methodologies include alkali or acid retting, chemical assisted natural retting (CAN) and degumming (34). Compared to mechanical methods, chemical procedures are more efficient at removing non-cellulosic components (35). Alkali retting is effective at obtaining clean fibers with significantly less damage (32). One of the most widely investigated alkaline-retting solutions is sodium hydroxide (NaOH), which is more affordable and commercially available than other alkalis (27). NaOH solution concentration range from 0.5% to 15.0%, with immersion times ranging from 15 minutes and 48 hours and temperatures from room temperature to 100°C. [29, 33, 49–53]. From a mechanical perspective, the main disadvantage of alkali retting is that the excessive removal of binding components can affect the stress transfer between elementary fibers, reducing the mechanical performance of the fibers (36).

Despite the various methods available for extracting natural fibers, there is a significant gap in the literature regarding the extraction of Guadua *angustifolia* fibers, particularly in balancing the preservation of the fiber's structural integrity with the controlled removal of lignin and hemicellulose. This balance is crucial, as it directly impacts the fibers' performance in composite materials. Excessive removal of non-cellulosic components, such as lignin and hemicellulose, can weaken the stress transfer between fibers, thereby reducing their mechanical properties. On the other hand, insufficient removal of these components can result in poor bonding to polymer matrices, diminishing the effectiveness of the fibers as reinforcements. Furthermore, the specific chemical and structural composition of Guadua *angustifolia* fibers differs from other bamboo species and natural fibers, suggesting that the strategies developed for other plants may not be directly applicable to Guadua *angustifolia*. Therefore, tailored extraction methodologies are needed to optimize the use of guadua fibers in composite applications, ensuring both effective removal of non-cellulosic materials and the retention of mechanical strength, which justifies a focused approach for this unique species.

This research contributes to overcome this gap by presenting a novel extraction methodology specifically designed for Guadua *angustifolia*, one that balances the removal of lignin and hemicellulose while preserving the lignocellulosic structure necessary for reinforcing composites. Previous studies on bamboo fibers have largely focused on other species (27, 37-39), but the distinct properties of Guadua *angustifolia* fibers require a specialized approach, which this study introduces. The proposed methodology enhances the potential of guadua fibers for use in a variety of composite matrices, contributing to sustainable material development in various industrial applications.

This paper describes an experimental program aimed at studying a proposed extraction methodology for Guadua *angustifolia* fibers, with the main purpose of promoting the use of these fibers as reinforcement of composite materials. The characterization of the fibers obtained through this methodology was essential to evaluate their suitability for such applications. Length and bulk density measurements were carried out to assess the basic physical properties of fibers, which are critical for ensuring uniformity in composite reinforcement. Water absorption behavior was analyzed to understand the fibers' interaction with moisture, as excessive water retention can negatively affect the long-term performance of composites. The mechanical properties of the fibers were evaluated to determine their potential to enhance the strength and durability of the composite matrices. Morphological analysis through scanning electron microscopy (SEM) was used to examine the surface characteristics and fiber structure, providing insights into the degree of delignification and the overall quality of the extraction process. Fourier-transform infrared (FTIR) spectroscopy was employed to identify the functional groups present in the fibers, allowing for a detailed chemical analysis to verify the removal of non-cellulosic components like lignin and hemicellulose. Additionally, before initiating the extraction process, the number of fibers available within the guadua plant was assessed to determine the fiber distribution profiles and the percentage of fibers present in the culm walls at different sections along the plant's height. This comprehensive characterization not only ensures that the proposed extraction method yields high-quality fibers but also highlights their potential for a broad range of composite applications.

2. MATERIALS AND METHODS

2.1. Fiber distribution along the wall thickness

The fiber distribution within the guadua culm was investigated using digital image analysis. Four different heights of the plant were analyzed: stick, top, middle and bottom. Annular sections approximately 5cm in length were cut from the midpoint of each culm, as shown in Figure 1.a. Rectangular segments with a width of 1cm were obtained from the middle point of each culm, matching the thickness of the culm wall (Figures 1.b and 1.c). Samples for microscopic observations were prepared from each circular segment as illustrated in Figure 1.d. Using sandpaper of various granulometries, one of the cross-sections of the samples was polished, finishing with a clean cloth.

To ensure accurate identification of fiber bundles during the digital image analysis, white toothpaste was used to fill the hollow spaces of the metaxylem, protoxylem, and phloem. These vascular structures appear as dark areas under the microscope and can be easily mistaken for fiber bundles if left unfilled (Figure 2.a). The white toothpaste helps to clearly distinguish these features from the fiber bundles by providing contrast in the cross-sectional images (Figure 2.b). This step ensures that the analysis focuses only on the actual fiber distribution within the culm wall.

Cross-sectional images of the samples were captured using a Motic SMZ168 stereo microscope with 3X magnification.

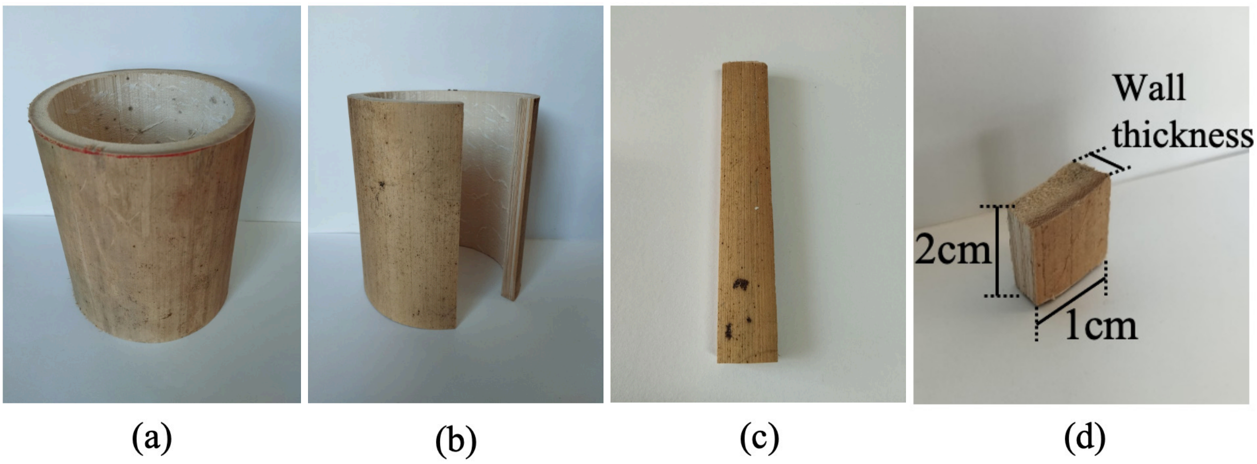


Figure 1. Sample preparation for fiber distribution analysis: (a) annular section; (b) and (c) rectangular segment; (d) sample.

The process of image analysis is depicted in Figure 2, which was conducted using ImageJ software. Initially, images of curved segments (Figure 2.b) were transformed into images of segments with a rectangular shape (Figure 2.c). To optimize the contrast between the fibers and the matrix, considering the uneven exposure to light, the images were filtered. Then, a binary image was produced as shown in Figure 2.d, where fiber bundles are black, and matrix and veins are white. This image, with m×n pixels, was transformed into a matrix of the same dimensions.

(a) (b) (c) (d)

Figure 2. Image analysis for fiber distribution measurement: (a) prepared cross-section without filled veins using toothpaste; (b) prepared cross-section with veins filled using toothpaste; (c) initial curved segment transformed into a rectangular shape; (d) binary representation.

Fiber density was calculated using Equation [1]; each pixel value at row i and column j of the matrix is considered as v_ij. The fiber volume fraction, expressed as a percentage, was then plotted as a function of the normalized wall thickness. The results for each analyzed part of the culm are presented in Figure 3. The inner and outer limits are delineated between 0 and 1, with the inner layer consistently having a lower fiber density. This characteristic is explained by the plant's evolutionary development, which has adopted this strategy to enhance its mechanical behavior naturally (40).

$$A_i = \frac{\sum_{j=1}^{n} v_{ij}}{n} \quad [1]$$

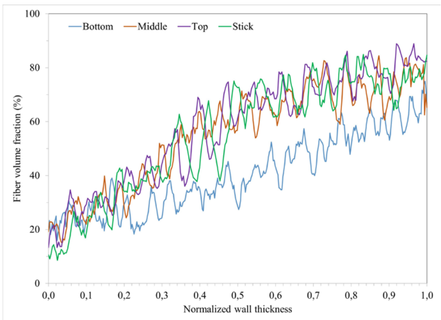


Figure 3. Profiles for fiber volume fraction distribution through the culm wall.

Table 1. Fiber content at different parts of the culm.

Location	Fiber percentage V_f (%)
Bottom	40.52
Middle	55.54
Top	57.82
Stick	58.09

The fiber content (V_f, as a percentage) for different culm locations is presented in Table 1, obtained using the formula $V_f = \Sigma A_i$. A variation in the fiber content is evident along the height of the culm, being lower at the base and higher at the top (stick). These observations indicate that as the wall thickness decreases with height, there is also a reduction in the parenchymal tissue of the plant, more so than in the vascular bundles, aligning with findings reported by (41).

2.2. Bamboo-guadua fiber extraction methodology

Guadua culms, ranging from three- to six-year-old, were selected for fiber extraction. The extraction methodology adopted was a combined chemical-mechanical process. The general steps for obtaining the fibers depicted in Figure 4. The method starts by splitting the guadua culms into longitudinal strips measuring approximately 0.025m in width, t in wall thickness, and 0.60m in length. To remove the outer coating layer and the nodal protuberances, these strips were mechanically polished. Guadua is recognized for its higher hardness (42) compared to other bamboo species; therefore, to soften the material, a chemical procedure using sodium hydroxide solutions (NaOH) of different concentrations and exposure times were employed, with temperature control detailed subsequently. After the chemical procedure, the strips were thoroughly rinsed with tap water to remove any remaining NaOH. A specialized crusher with four steel rolls was then designed and used in this research to crush the softened strips, allowing for the mechanical separation of fibers. Thicker strips required multiple passes through the crusher, gradually reducing the distance between the rollers.

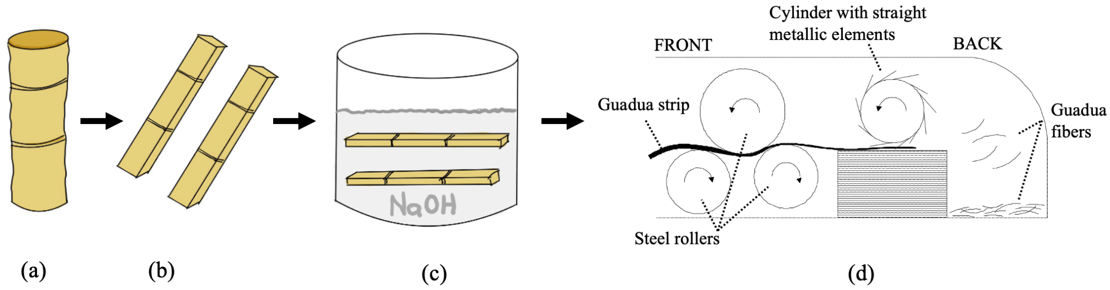


Figure 4. General chemical-mechanical procedure to obtain the bamboo guadua fibers: (a) guadua culm; (b) polished longitudinal strips; (c) strips immersed in NaOH solution concentration using different conditions; (d) scheme of the crushing machine.

The extracted fibers were washed extensively with fresh tap water until all traces of NaOH were eliminated. Subsequently, fibers were soaked in clean running water for a full day. To confirm the complete removal of NaOH from the fibers after the washing process, a random sample (approximately 2g) was submerged in 100 ml of distilled water for three minutes. After this period, the pH level of the distilled water was measured, and if it was close to 7.0 ± 0.5, it indicated that the NaOH had been successfully removed from the extracted fibers (43-45). In cases where the pH was not within this range, the washing step was repeated. A digital pH tester (Hanna Instruments HI98130) was used to measure the pH value. Once the NaOH removal was confirmed, the fibers were left to dry at ambient laboratory conditions.

Figure 5 illustrates the crushing machine developed specifically for this study, which mechanically separates guadua fibers. The machine features three steel rollers at the front to compress the softened strip. At the rear, a cylinder with straight metal elements brushes the crushed strip, facilitating fiber separation. The gap of the upper front roller can be adjusted for efficiency. The cylinders are powered by an electric motor connected to a geared motor on the lower roller. The remaining rollers (two at the front and one at the rear) are driven by a chain and pinion system. To enhance brushing efficiency, the rear cylinder rotates at a higher speed than front ones.

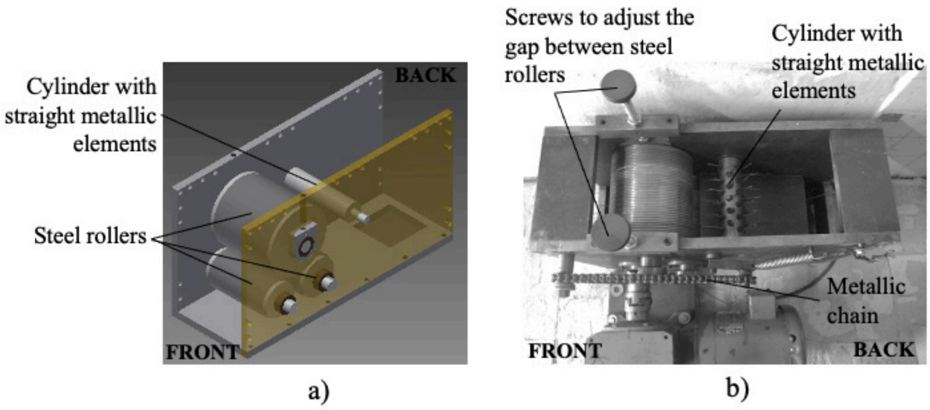


Figure 5. Crushing machine used to obtain bamboo guadua fibers.

The variables investigated in this study included the NaOH solution concentration, temperature, and immersion time of the strips. These variables were selected based on a comprehensive literature review and represent the frequently used conditions for natural fiber extraction, employing chemical, mechanical, or combined methods. The NaOH solution concentration ranges from 0.5% to 15.0%, with immersion times varying from 15 minutes to 48 hours (shorter times for heated solutions), and temperatures ranging from room temperature to 100°C (34, 37, 46-50). A previous study (51) showed that immersing the strips for 96h increase the ease of the extraction process.

Thus, two temperature conditions were evaluated: ambient laboratory temperature and a heated solution at 80°C. For the ambient temperature procedures, solution concentrations were 2.5%, 5.0%, 7.5%, and 10.0%, with immersion times of 24, 48, 72, 120, and 192h. Due to the acceleration of fiber separation by heated solutions (43, 52), the concentration was set at 2.5% with immersion times of 2, 3, and 4h for the 80°C procedures. As a control, a set of fibers was also extracted without any NaOH immersion by soaking polished strips in tap water at 0% NaOH concentration for 90 days (2160h) before mechanical separation. Figure 6 outlines the experimental program undertaken for the extraction process.

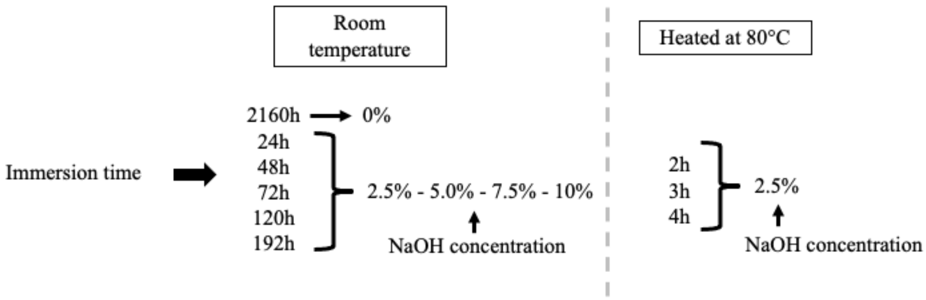


Figure 6. Fiber extraction variables and nomenclature used to identify each group.

2.3. Guadua fiber extraction study

The most important variables in establishing a methodology for guadua fiber extraction are the quantitative measurement of tensile strength and a heuristic approach to assessing the easiness of extraction. Tensile strength is crucial for the mechanical properties of composites materials, while the easiness of extraction significantly impacts the feasibility of industrial production processes. Accordingly, a Final Score index (*FS*) was introduced to evaluate both variables, incorporating tensile strength data and a score representing the easiness of extraction for each condition. This approach provided a comprehensive understanding of how the extraction variables influenced the mechanical properties of fibers. To ensure robust mechanical behavior characterization, 16 replicates were tested for each variable. The sample size was determined with a confidence level of 90% and an allowable margin of error around 20MPa in fiber strength. Mechanical testing was conducted following the ASTM C1557-14 standard, on a universal testing machine equipped with a 50N load cell. A test loading rate of 1.5mm/min was chosen, leading to fiber failure in approximately 30s, conforming to the standard's recommendations. Figure 7 illustrates the fibers mounted on paper frames; a technique used to prevent axial load deviations during tests. A gage length of 4.0cm was employed, and the frame was meticulously cut after securing the sample in the testing grips.

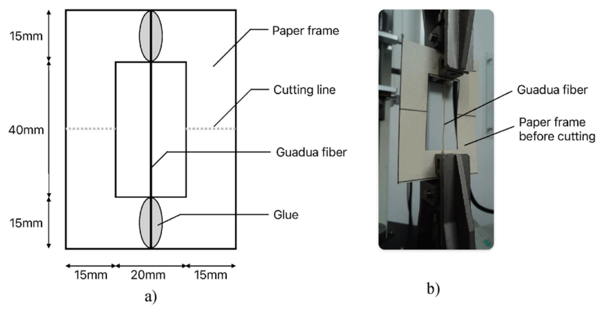


Figure 7. Fiber assembly for testing: (a) paper frame; (b) specimen on testing grips.

The tensile strength (σ) was calculated using Equation [2], where *P* is the maximum load sustained by the fiber and *A* represents the fiber's cross-sectional area. Previous study (53) has detailed a precise method for measuring the fiber area, ensuring accurate results as the plane of failure is orthogonal to the observer's view.

$$\sigma = \frac{P}{A} \qquad [2]$$

The ease of extraction score is a metric developed to quantify the efficiency of the extraction process. It considers the difficulty of obtaining fibers and the yield of material extracted. Scores ranged from 0 to 1, where 0 corresponded to extremely difficult extraction conditions and 1 to conditions of minimal difficulty. Intermediate scores of 0.25, 0.50, and 0.75 denoted difficult, moderately difficult, and easy extraction processes, respectively.

Equation [3] defines the *FS* for each extraction condition, combining the normalized average tensile strength (σ_N)

and the ease of extraction score (*ES*). The σ_N was computed as the ratio of the average strength to the maximum average strength observed across all conditions. As indicated in Equation [3], the *ES*, being a qualitative measure, was assigned a lower weight due to its inherently greater uncertainty.

$$FS = 0.8\sigma_N + 0.2ES \qquad [3]$$

2.4. Characterization of extracted fibers

The guadua fibers obtained through the proposed extraction process were subjected to a comprehensive physical and chemical characterization to assess their properties for potential use in composite materials. The physical characterization involved measuring the fibers' length, density, water absorption capacity, and examining their surface morphology using Scanning Electron Microscopy (SEM). Additionally, the chemical characterization was performed through Fourier Transform Infrared Spectroscopy (FTIR) to analyze the functional groups present in the fibers. These characterization techniques provide essential information on the structural and chemical changes induced by the extraction process, which are critical for evaluating the fibers' suitability as reinforcement in composites

The length of approximately 200 randomly selected fibers was measured using a digital caliper with a precision of 0.01cm. Fiber density was determined following ASTM D3800- 99 (procedure A), which specifies the use of immersion methods for determining density. Approximately 80 randomly selected fiber samples, each weighing about 1g, were used to calculate the average density. Fibers were submerged in 1500 ml of tap water at an average temperature of 22°C to displace the water volume, and their weights were recorded using an analytical scale with 0.001g precision. Measurements were taken in a controlled environment at 23°C and 50% relative humidity.

Following the procedure used by (54), water absorption of the fibers was evaluated using 1.5g samples immersed for various durations (0 to 24 hours). After immersions, the samples were centrifuged for 1 minute to remove excess water and then oven-dried at 50°C for 24 hours to avoid thermal degradation. This temperature was similar to the one used by (55). Although this drying temperature may not have been sufficient to guarantee complete water removal, the consistent use of this condition across all measurements allows for the observation of water absorption trends. In future studies, higher drying temperatures could be employed to ensure complete water removal for a more accurate determination of moisture content. Weights measurements were taken at 2-hour intervals until successive measurements showed a difference of less than 0.010g. The water absorption capacity of fibers was calculated as the moisture content (*MC*) using Equation [4], where w_w and are w_d the weights of wet and dry samples, respectively.

$$MC = \frac{w_w - w_d}{w_d} \times 100 \qquad [4]$$

Functional groups in guadua fibers were identified through Fourier transform infrared spectroscopy (FTIR) analysis. Samples were equilibrated at room temperature before testing with a Nicolet iS 10 FTIR spectrometer (Thermo Fisher Scientific), executing 40 scans per spectrum across a range of 4000 and 800 cm^{-1}. This range was selected based on the literature review, where this technique was used for different natural fibers (27, 48, 49, 55)

The surface morphology analysis was performed through SEM imaging with a Tescan Vega 3 SB scanning electron microscope. Before observations, fibers samples were coated with a thin layer of gold using a plasma sputtering technique. All the observations were performed in high vacuum and operating in secondary electron mode. The accelerating voltage was 10kV.

3. RESULTS AND DISCUSSION

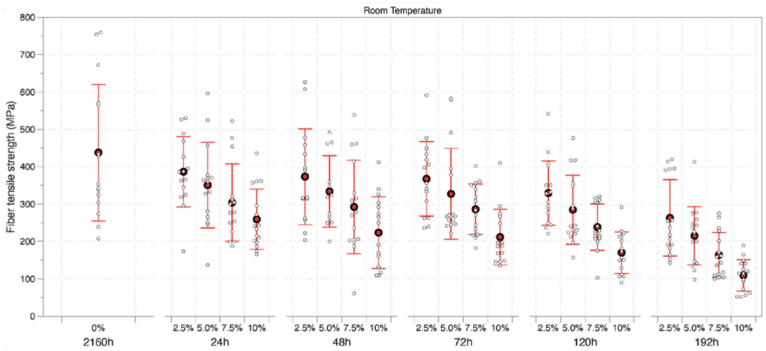
3.1. Guadua fiber extraction results

Figure 8 presents the average tensile strength for each extraction variable involved in the extraction process. The results are categorized by immersion times and temperature conditions. Hence, Figure 8.a illustrates outcomes at room temperature, while Figure 8.b displays those for immersions heated to 80°C. The figures represent experimental data with empty circles, standard deviation with error bars, and the mean values with red circles. Outliers identified using boxplots were excluded from statistical analysis.

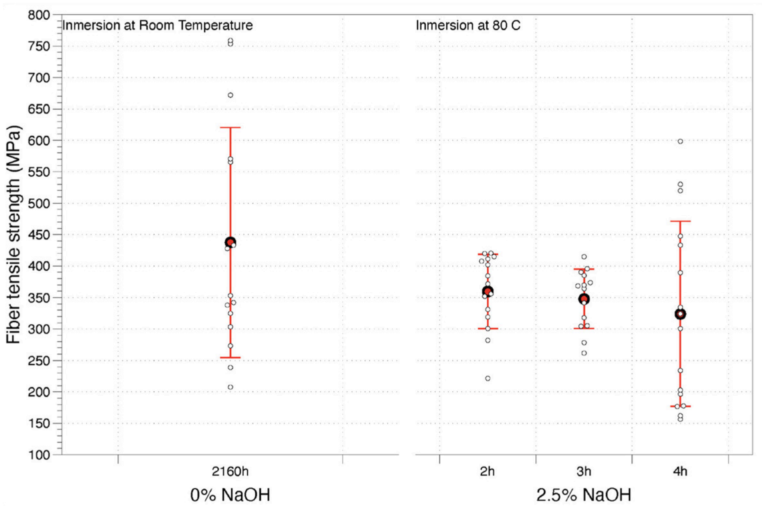
As indicated in Figure 8, the tensile strength of fibers is highly variable, consistent with findings from previous studies (52, 56-60). The coefficient of variation (COV), defined as the ratio of average tensile strength to standard deviation, ranged between 11 and 60%.

Heating the alkaline solution is known to accelerate the separation of hemicellulose and lignin. Specimens immersed in a 2.5% NaOH solution and heated to 80°C reached sufficient softness after 2h to begin the mechanical separation process. Conversely, samples immersed at room temperature in the same concentration required 24h to soften adequately. A notable decrease in tensile strength was observed with increasing NaOH concentration and immersion time. This is attributable to: 1) significant loss of hemicellulose and lignin, impairing stress transfer between fibers (36) and, 2) the alkaline solution degrading the crystalline structure of the cellulose in the fibers (52, 57, 58).

For identical solution concentrations at varying immersion times, a higher alkali concentration led to a further decrease in fiber strength. With 2.5% and 5% concentrations, there was about a 35% strength reduction for fibers extracted from 24-hour and 192-hour immersions. Similarly, the maximum tensile strength reduction for 7.5% and 10% concentrations approached 50% between the 24-hour and 192-hour immersion comparisons. The heated solution's fiber strength decline was approximately 10%, comparing 2-hour and 4-hour immersions.



a)



b)

Figure 8. Tensile strength for extracted fibers: (a) at room temperature; (b) heated to 80ºC.

To evaluate the effect of each independent variable (NaOH solution concentration, temperature, and immersion time) on the tensile strength of extracted fibers, a two-way ANOVA with replications was carried out. This analysis was made to determine the statistical significance of each factor, as well as the interactions between them. The factor considered were: NaOH concentration (levels of 0%, 2.5%, 5%, 7.5% and 10%), immersion time (levels of 24, 48, 72, 120, 192 and 2160h), and temperature during immersion (two levels "laboratory" for room temperature and 80ºC). The ANOVA was made with a confidence level of 95% ($p<0.05$), using the fibers' tensile strength as dependent variable. The results are shown in Table 2.

Table 2. Results two-way ANOVA with replications.

Source of variation	Sun of Squares	Degrees of Freedom	Mean Square	F-value	p-value
Samples	19157878.97	23	832951.25	364.04	0
Factors	20213670.69	3	6737890.23	2944.77	0
Interaction	51142170.79	69	741190.88	323.94	0
Residual	3294849.80	1440	2288.09		

ANOVA results indicate that the NaOH solution concentration, temperature and immersion time significantly influence the tensile strength of extracted fibers. As previously stated in the methodology, 16 replicates were tested for each variable to ensure the repeatability and reproducibility of the results. This sample size was selected to provide a robust characterization of the mechanical behavior with a confidence level of 90% and a margin of error within 20 MPa in fiber strength. Findings are consistent with previous studies, which highlight that NaOH modify the structure of natural fibers, enhancing of reducing their mechanical properties depending on the used conditions (20, 61-63). Immerse the guadua strips using elevated temperature seems to be less effect on decreasing fibers' tensile strength but can also degrade the fiber if the immersed time is excessive. The significant interactions between factors suggest that combinations of long immersion times with higher concentrations and temperature, may produce detrimental effects depending on the specific conditions.

Figure 9 presents the assigned ease of extraction score (*ES*) for both room temperature and 80ºC immersions. Extraction following 2160h of immersion proved difficult, yielding a minimal volume of fibers, thus warranting a lower *ES*. As depicted, increased NaOH concentration, immersion time, and temperature facilitated the procurement of individual fiber due to sodium hydroxide's capacity to dissolve lignin and hemicellulose, which naturally bond the fibers (30). Higher concentrations and extended immersion times resulted in more substantial natural binder removal.

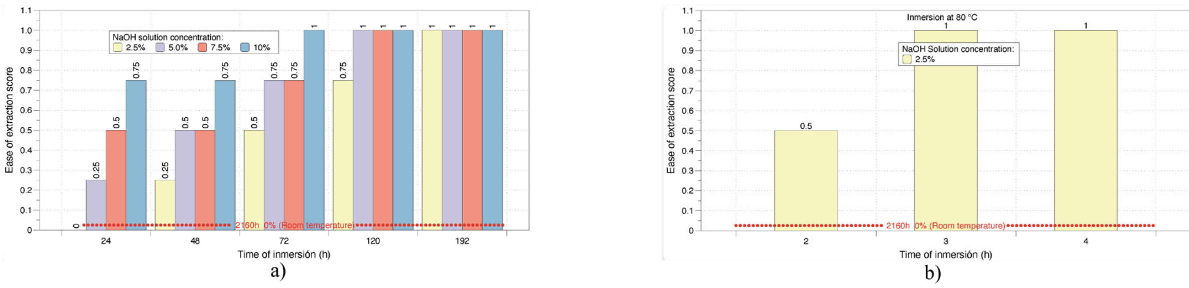


Figure 9. Easiness of extraction score assigned to fibers extracted using immersions: (a) at room temperature; (b) heated to 80ºC.

Figure 10 shows the Final Score (*FS*) calculated for various extraction conditions. At room temperature (Figure 10.a), the score diminished with heightened solution concentration and extended immersion duration due to the pronounced fiber strength degradation. For fibers extracted using heated solutions (Figure 10.b), there was and is an *FS* increase when immersion time extended from 2 to 3 hours, attributed to the improved extraction ease. However, the *FS* remained relatively constant between 3-hour and 4-hour conditions. Despite fibers from 0% NaOH solutions at room temperature over 2160h demonstrating the highest tensile strength, the optimum *FS* was achieved with fibers from segments immersed in a 25% NaOH solution heated for 3 hours.

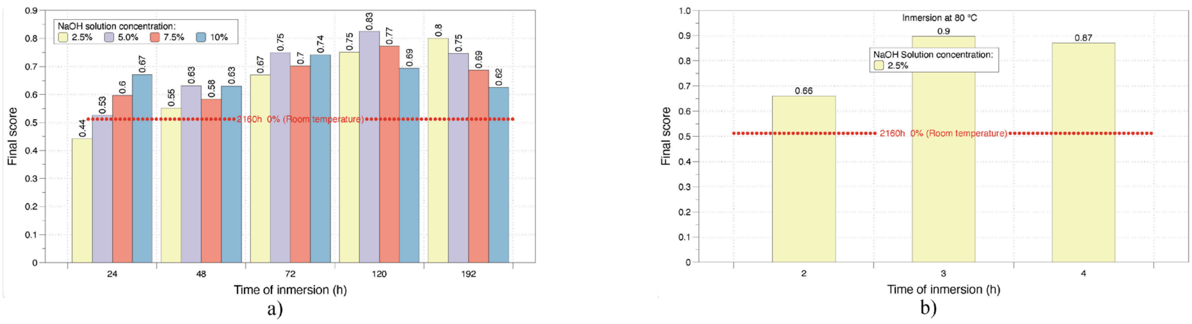


Figure 10. Final score for fibers extracted using immersions: (a) at room temperature, (b) heated to 80ºC.

The adopted guadua fiber extraction methodology corresponds with the highest *FS*. Guadua softening, achieved by a 3-hour immersion in a 2.5% sodium hydroxide solution at 80ºC, precedes fiber separation through the crusher. Such extraction conditions yield fibers with the tensile strength necessary for reinforcing composite materials while also considering ease of extraction, a vital factor for industrial application. Figure 11 showcase a sample of the extracted fibers.

3.2. Results on characterization of extracted fibers

Table 3 shows the lengths and densities of guadua fibers. The fiber's average density corresponds closely to those reported by other researchers for different natural fibers (64, 65). The length is consistent with the internodal length of the culm base, from which the fibers were extracted. It has been noted that bamboo fiber length correlates with internodal distance (66), which, for guadua culms, ranges from 100mm to 350mm (40). With a COV > 60%, the length measurements exhibit high variability, likely due to the vigorous action of the crushing machine.

Figure 11. Guadua fibers obtained using the proposed extraction methodology.

Table 3. Density and length of guadua fibers.

Property	Average	Maximum	Minimum	COV (%)
Density (g/cm^3)	1.36	1.57	1.18	5.40
Length (mm)	91.64	205.00	11.00	62.27

Figure 12 presents the average *MC* values. The initial *MC* corresponds to the fibers' atmospheric moisture absorption, a typical property of natural materials (67). Higher immersion times resulted in *MC* values exceeding 100%, indicative of fiber swelling and infiltration of water into spaces between microfibrils. Natural fibers tend to swell in moist conditions due to the formation of new hydrogen bonds with water molecules, disrupting those within the fiber cell wall (68), allowing water to occupy inter-microfibrillar spaces (69).

The FTIR spectrum displayed in Figure 13 features vibrational modes characteristic of lignocellulosic fibers (37, 70, 71). The broad, strong band from 3600 to 3200 cm^{-1} is attributed to hydroxyl group stretching (72). The peak at 2850 cm^{-1} signifies C-H stretching vibrations in hemicellulose and cellulose (73). Water adsorbed in crystalline cellulose corresponds to peaks between 1660 and 1600 cm–^{1} (73). Aromatic C-H bending in lignin is associated with the peaks from 1510cm^{-1} to 1450cm^{-1} (74, 75), and cellulose bending is indicated near 1421cm^{-1} (76). The prominent peak between 1100 and 1000 cm^{-1} corresponds to C-H vibrations in cellulose and C-O stretching, in alignment with findings from other studies on bamboo fibers (76, 77).

To further understand the chemical modifications introduced by the extraction process, the obtained FTIR spectrum were compared with those reported in the literature for (i) bamboo in its raw state, (ii) bamboo fibers extracted using different techniques, and (iii) natural fibers extracted using procedures similar to those employed in this research. These comparisons aim to evaluate three key aspects: (i) how the extraction process alters the chemical structure of fibers compared to bamboo in its natural state, (ii) how the fibers obtained using the established procedure compare to those obtained using different methods, and (iii) whether the established extraction procedure yields similar results to those obtained under similar extraction conditions.

The comparison with FTIR spectra, of raw bamboo species peaks (78, 79) showed that while the broad band associated with hydroxyl groups (3600 to 3200 cm^{-1}) and C-H stretching vibrations (2850 cm^{-1}) remains present, the intensity of these peaks is slightly reduced, indicating partial removal of hemicellulose and lignin. This aligns with typical results from alkaline extraction, where non-cellulosic components are partially dissolved. Despite these changes, the essential structure of the fibers remains intact, as seen in the preservation of key cellulose and hemicellulose.

Comparing the FTIR results with those of bamboo fibers extracted using other techniques, such as mechanical extraction (80) and more aggressive chemical treatments like acid hydrolysis (81), the proposed method demonstrates less intense delignification. The FTIR spectra of mechanically extracted fibers show similar functional groups, particularly hydroxyl and aromatic C-H bending, but with a more preserved lignin structure. In contrast, fibers subjected to acid hydrolysis show a more pronounced reduction in lignin and hemicellulose peaks, indicating a greater removal of non-cellulosic components.

Finally, a comparison with *Juncus* fibers extracted under similar conditions [45] highlights a notable similarity in the preservation of cellulose and hemicellulose, as indicated by comparable hydroxyl and C-H stretching bands. However, the guadua fibers in this study showed slightly higher intensities in the C=O carbonyl stretching region (~1700 cm^{-1}), suggesting variations in lignin structure or retention between different fiber types. This may be attributed to the specific composition of guadua or the minor differences in extraction conditions.

In summary, the FTIR analysis suggests that the established extraction method strikes an effective balance between removing non-cellulosic components and preserving the lignocellulosic structure. The comparisons with raw bamboo, mechanically and chemically extracted fibers, and *Juncus* fibers demonstrate that the proposed method maintains a significant portion of the fibers' natural structure, making them suitable for further use in composite applications where fiber integrity is crucial.

As observed in Figure 14, the guadua fibers comprise many individual fibers aligned in parallel clusters (Figures 14.a, 14.b, 14.d and 14.f), which is a typical feature of lignocellulosic fibers (50, 82). Surface irregularities observed are likely due to lignin, hemicellulose, and oils coating the fibers, which is consistent with incomplete removal of non-cellulosic components, a phenomenon often observed in alkaline treatment of natural fibers (21, 51). The pits in Figures 14.b, 14.c, and 14.g are characteristic of parenchyma structures (83, 84). At the 5µm scale (Figures 14.f, 14.g and 14.h), fibers exhibit a generally smooth surface with minor, continuous ridges, suggesting that the extraction process effectively preserved the integrity of the fiber's outer surface while allowing for some delignification and removal of hemicellulose.

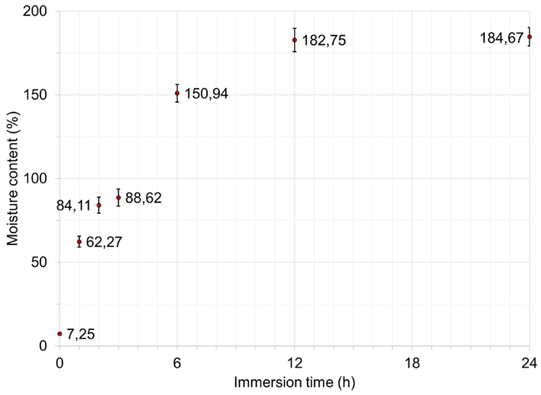


Figure 12. Moisture content of extracted fibers for different immersion times: 0, 1, 2, 4, 6, 12, and 24h.

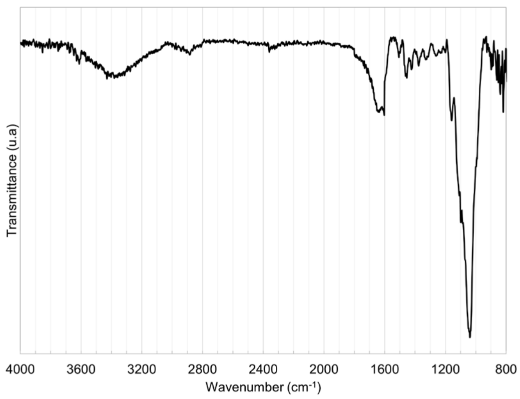


Figure 13. FTIR speWcatrvoensucmobpery (c mfo-1)r extracted fibers.

When comparing these observations to those obtained by Naili et al. (46) for *Juncus* fibers, using similar conditions that those used in this study, is noted that moderate concentrations of NaOH effectively preserve fiber integrity while significantly reducing non-cellulosic materials, which aligns to the findings of this research. In contrast, the Guadua angustifolia nanofibers obtained by (81) using more aggressive delignification process showed a near-complete removal of lignin and cellulose. This comparison emphasizes the importance of selecting an extraction method based on the desired fiber properties, depending on the specific application.

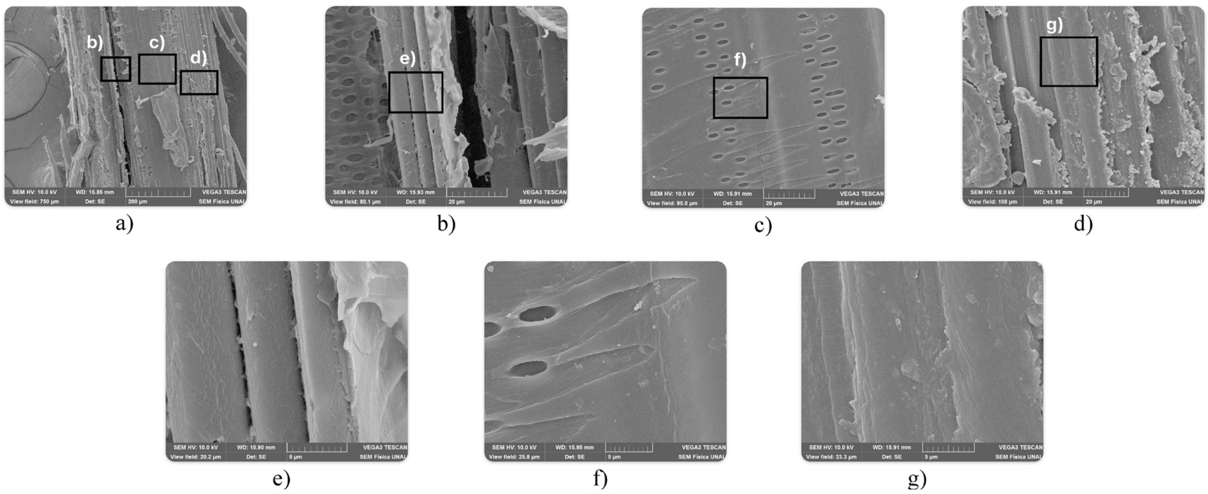


Figure 14. Surface morphology of fibers: (a) 200µm; (b), (c) and (d) 20µm; (e), (f) and (g) 5µm.

4. CONCLUSIONS

The extraction process described in this study encompasses both mechanical and chemical procedures. Utilizing this approach, fibers from Guadua *angustifolia* bamboo were obtained with considerable mechanical strength and relative ease of extraction, factors of significant importance in the composites industry.

It is concluded from the results that using sodium hydroxide solutions to isolate fibers adversely impacts their mechanical properties. Nevertheless, NaOH notably expedites the separation process, an effect further enhanced by heating during the immersion of the raw material. The reduction in mechanical properties of guadua fibers could be attributed to two phenomena: 1) compromised stress distribution among microfibrils, following the loss of binding compounds such as lignin and hemicellulose; and 2) degradation of the fibers' crystalline cellulose structure.

This study focused on establishing an extraction methodology for Guadua *angustifolia* fibers and their characterization. Results regarding the performance of these fibers as reinforcement in composites are not included here. However, it is important to point out that fibers obtained using the procedure described in this work have been evaluated as reinforcement in polyester matrices, showing satisfactory results in terms of compatibility and mechanical performance. For more details on the impact of these fibers as reinforcement in composites, readers can refer to a previous publication [59]. Future research will further explore the interaction between Guadua *angustifolia* fibers obtained with the proposed methodology and various matrices, as well as optimize the extraction process to enhance fiber performance in structural applications.

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Authorship contribution statement

Patricia Luna: Conceptualization, Data curation, Formal analysis, Funding acquisition, Investigation, Methodology, Writing – original draft.

Juan Lizarazo-Marriaga: Conceptualization, Formal analysis, Funding acquisition, Investigation, Methodology, Supervision, Validation Writing - review & editing.

Declaration of competing interest

The authors of this paper declare that they have no financial, professional or personal conflicts of interest that could have inappropriately influenced this work

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