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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.

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

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).

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

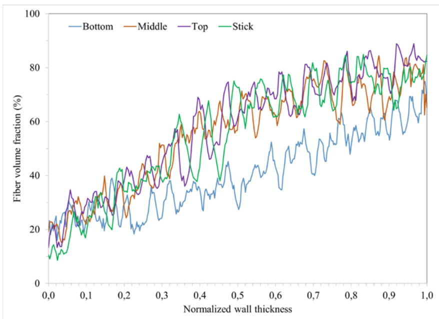

Figure 3. Profiles for fiber volume fraction distribution through the culm wall.

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.

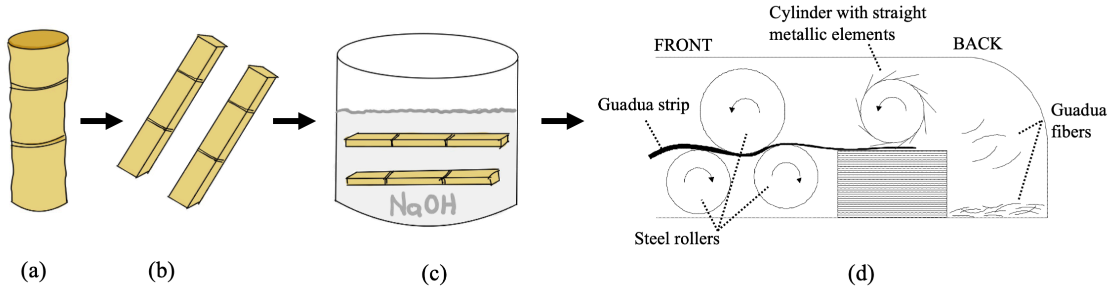

Figure 4. General chemical-mechanical procedure to obtain the bamboo guadua fibers: (a) guadua culm; (b) polished longitudinal strips; (c) strips immersed in NaOH solution concentration using different conditions; (d) scheme of the crushing machine.

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.

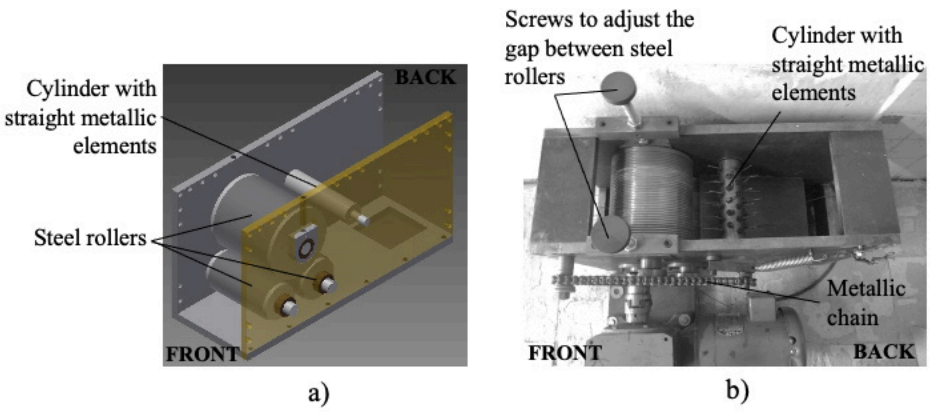

Figure 5. Crushing machine used to obtain bamboo guadua fibers.

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