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ARTICLE

Alkali and Plasma-Treated *Guadua angustifolia* Bamboo Fibers: A Study on Reinforcement Potential for Polymeric Matrices

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ABSTRACT

This study focuses on treating *Guadua angustifolia* bamboo fibers to enhance their properties for reinforcement applications in composite materials. Chemical (alkali) and physical (dry etching plasma) treatments were used separately to augment compatibility of *Guadua angustifolia* fibers with various composite matrices. The influence of these treatments on the fibers' performance, chemical composition, and surface morphology were analyzed. Statistical analysis indicated that alkali treatments reduced the tensile modulus of elasticity and strength of fibers by up to 40% and 20%, respectively, whereas plasma treatments maintain the fibers' mechanical performance. FTIR spectroscopy revealed significant alterations in chemical composition due to alkali treatments, while plasma-treated fibers showed minimal changes. Surface examination through Scanning Electron Microscopy (SEM) revealed post-treatment modifications in both cases; alkali treatments served as a cleanser, eliminating lignin and hemicellulose from the fiber surface, whereas plasma treatments also produce rough surfaces. These results validate the impact of the treatments on the fiber mechanical performance, which opens up possibilities for using *Guadua angustifolia* fibers as an alternative reinforcement in composite manufacturing.

KEYWORDS

Bamboo fibers; *Guadua angustifolia*; alkali treatment; dry etching plasma treatment; composite reinforcement

1 Introduction

Currently, natural fibers are extensively employed as reinforcement within the composite industry, mainly alongside polymer matrices [1–4] in uses that do not demand extensive load-bearing capabilities. Examples such as pineapple, cotton, fique, flax, and bamboo illustrate the widespread adoption of natural fibers for reinforcement purposes. Research by Peças et al. [5] indicates that cotton, pineapple, and bamboo are among the most frequently discussed natural fibers within the Science Direct database. This trend may be linked to the global distribution and accessibility of these plant sources, their immediate suitability for composite reinforcement applications and their inherent mechanical properties. With an annual growth rate projected at 9.59%, the market value of natural fiber composites is estimated to up to USD 41 billion by 2025 [6].

Bamboo is a no timber product belonging to the Gramineae (Poaceae) family, Bambusoideae subfamily, comprising 1575 species from 116 genera worldwide [7]. These plants thrive in both tropical and temperate climates globally, spanning a range of habitats from sea levels to elevated mountainous regions [8]. A particularly remarkable attribute of these forest resources is their rapid growth rate. Chin et al. [9] noted that due to their structural variation as to thermal and mechanical properties, bamboo has been used recently in the composite industry, showing great potential. However, the literature suggests that the most compelling characteristic, as reinforcement in composites, is their high strength-to-weight ratio [5,10].

Guadua angustifolia stands out as the leading bamboo species in Latin America, originating from Colombia, Venezuela, and Ecuador. In Colombia, approximately 51,000 ha of forest are covered with this bamboo type [11], which is integral to the folklore, ecosystem, social customs, and cultural development of some regions. In Colombia, *Guadua angustifolia*, in its raw (tubular) form, has been a construction material since ancient times, and its mechanical properties have been extensively investigated [12–14]. Laminated *Guadua angustifolia* has also been the subject of numerous studies [15,16]. In contrast to the extensive research on raw and laminated *Guadua angustifolia*, there is a lack of research on *Guadua angustifolia* fibers [17]. In this research, *Guadua angustifolia* was chosen as the study material, considering that in Colombia it is an abundant resource frequently used as a construction material.

The bamboo plant could be divided into two parts as shown in Fig. 1. The above-ground section includes the culm, branches, and leaves, whereas the underground section compromises the rhizome (Fig. 1a). The culm is comprised of alternating nodes and internodes (Fig. 1b). The wall thickness, external diameter, and internodal length generally vary in the culm. In *Guadua angustifolia* species, the wall thickness and external diameter decrease linearly from base to top. Regarding wall thickness, the variation ranges from 20 to 5 mm, while the external diameter varies from 100 to 30 mm. On the other hand, the length of the internodes at both the top of the culm and base, which is of shorter length than in the middle, spans from 150 to 350 mm [14]. From an anatomical perspective, the culm has vascular bundles within parenchyma tissue. As depicted in Fig. 1e, there is variation in the shape, size, and density of vascular bundles across the wall, with the outer section being smaller and denser than the middle and inner sections. Vascular bundles are composed of two large metaxylem vessels, one or two protoxylem elements, fiber bundles and phloem (Fig. 1f). As depicted in Fig. 1g, fiber bundles (fibers) consist of microfibrils or elementary fibers.

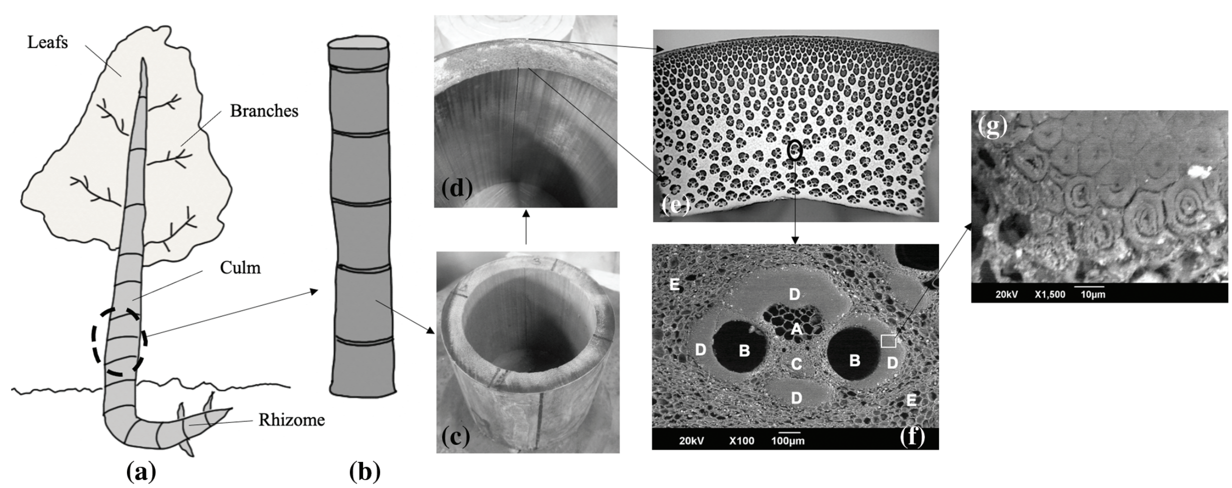

Figure 1: Parts of a bamboo plant: (a) plant; (b) culm; (c) and (d) wall; (e) distribution of the culm wall; (f) vascular bundle structure: (A) metaxylem vessel component, (B) protoxilem, (C) phloem, (D) fiber bundle (fiber), (E) parenchymal tissue; (g) microfibrils

Generally, in a bamboo culm, fibers have approximately 40% of the volume and 70% of the weight [18]. Torres et al. [19] carried out research to measure the volume of fibers in the Guadua *angustofolia* species using advanced field microscopy and high-resolution imaging techniques. They found that the fiber volume in the lower, middle and upper parts of the culm are 34%, 41% and 43%, respectively. Bamboo exhibits a tensile strength that generally exceeds that of wood by more than a factor of two, and its specific tensile strength exceeds that of steel by approximately three times. Furthermore, the elongation at break of bamboo fibers typically falls between 4%–10%, exceeding the values observed for both glass and carbon fibers, while being on par with those of PVA (polyvinyl alcohol) fibers [20].

From an environmental perspective, the principal advantages of using natural fibers as reinforcement include that they are renewable resources, their production consumes very little energy, and they contribute to CO_2 absorption [21]. The technical advantages of natural fibers, including their high specific stiffness and strength, low density, and cost-efficiency, are particularly notable. Nonetheless, there are disadvantages, such as high variability in properties: mechanical strength, moisture absorption, and durability, and limited bonding in the fiber-matrix interphase [22,23]. Many researchers argue that the chief drawback of employing natural fibers in composite materials is the poor adhesion between phases [24–26], stemming from the fibers' hydrophilic properties and the polymers' hydrophobic characteristics [5,22]. This could be solved by modifying the physicochemical properties of natural fibers through chemical or physical methods [27].

The primary goal of chemical treatments is to modify the hydrophilic characteristics of fibers, which are a consequence of the significant amount of hydroxyl groups (-OH) within their chemical structure [28], predominantly found in hemicellulose and lignin [24]. Consequently, these treatments aim to remove these components [25,29,30], enhancing the chemical compatibility of fibers and polymers. Several studies employed alkali (NaOH) solutions for this purpose, achieving significant improvements in the adhesion between polymers and fibers [25,26,31–33]. However, the effect of such treatments on the mechanical performance of fibers is inconclusive. For instance, Sanchez et al. [17] reported a decrease of approximately 20% in tensile strength when bamboo fibers were treated with a 10% NaOH solution. Rajeshkumar et al. [34] treated *Phoenix Sp*. fibers using different concentrations (5%, 10% and 15%) for 1 h, and found that even though the tensile strength increased for all treatment conditions, a decrease in the modulus of elasticity was obtained for fibers using 5% and 15% NaOH. In contrast, Zidi et al. [32] examined multiple concentrations of NaOH (1%, 2%, 3%, 4%, and 5%) and various durations (from 1 to 5 h) on sisal fibers, noting improvements in tensile strength from each treatment. Furthermore, Zhang et al. [35] noted that a 6% NaOH treatment on bamboo fibers raised both their modulus of elasticity and tensile strength by 38% and 14%.

Physical methods are designed to modify the structural and surface properties of fibers to enhance bonding within composite phases [22,36], through processes such as plasma treatments, vacuum ultraviolet irradiation, ozone, and corona treatments, among others [37]. Plasma formation occurs via highly energetic procedures that involve subjecting a gas to electric discharges or heating it to elevated temperatures. If a potential difference is applied and there is a gas flowing, the gas breaks down into positive ions and electrons that can conduct electricity and initiate a gas discharge. This discharge takes place if an electric field strength in the electrodes suffices to ionize the gas, a phenomenon termed breakdown. The resulting ionized gas, or plasma, consists of positive ions and electrons [38]. Cold plasmas are characterized by electrons with temperatures higher than those of positive ions [39], are frequently used in materials science. Cold plasma is usually produced under low (10^{-4} to 10^{-2} kPa) or atmospheric pressures with power sources such as radio frequency (RF), direct current (DC), or microwave (MW), and is frequently boosted by supplementary electric or magnetic fields [40]. DC discharges are commonly utilized because of their simplicity, while MW and RF discharges offer better atom ionization [38].

Cold plasmas are mainly used for surface functionalization, thin film deposition, and etching [41]. These plasmas primarily modify surface properties with a minor impact on bulk properties [36,40,42–44].

Research has shown that plasma treatments (etching) can improve the bonding of natural fibers to polymeric matrices, although these studies are fewer than those for alkali treatments. Plasma gases such as oxygen, helium, argon, or nitrogen have been employed to treat various natural fibers [23,45], effectively enhancing their interfacial adhesion to different matrices [46–50]. These treatments have also been shown to improve the fibers' mechanical performance, including increases in the tensile strength and the elastic modulus [51–53].

The principal objective of this research is to furnish technical data to advance the utilization of *Guadua angustifolia* fibers in composite materials. As previously discussed, it is imperative to alter the fibers' physicochemical properties to enhance the interfacial bonding within the composite phases. To this end, both chemical and physical treatments have been implemented in this study. Subsequently, the modified fibers were subjected to through physical and chemical characterization to evaluate their suitability as an alternative reinforcement within the composite industry.

2 Methods

2.1 Guadua angustifolia Fibers

In this research, *Guadua angustifolia* culms aged between 3 and 6 years were selected, whose fibers were obtained from the lower part of the plant. The fibers were obtained using an initial chemical-mechanical process, schematically depicted in Fig. 2. The extraction process began by splitting the *Guadua angustifolia* culms into longitudinal strips (Fig. 2b). The purpose of the chemical process was to soften the material, which was accomplished by immersing the strips for 3 h in a 2.5% sodium hydroxide solution, previously heated to 80°C; the temperature was maintained during the immersion period (Fig. 2c). Following immersion, the strips were thoroughly rinsed with tap water. The fibers were then isolated through a mechanical process using the crushing machine described in [54], and illustrated in Fig. 2d. After extraction, all fibers were meticulously cleansed with drinking water and they were left to dry at laboratory conditions. The average length of extracted fibers was 91.64 mm.

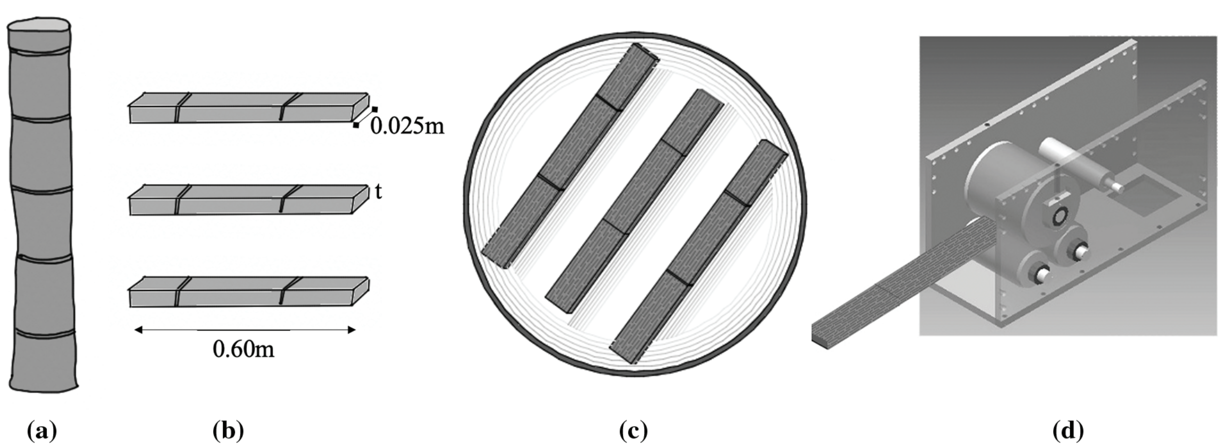

Figure 2: General chemical-mechanical process to obtain *Guadua angustifolia* bamboo fibers: (a) *Guadua angustifolia* culm; (b) longitudinal strips; (c) strips immersed in 2.5% NaOH solution heated to 80°C; (d) crushing machine

2.2 Fiber Treatments

Chemical and physical treatments were applied to *Guadua angustifolia* fibers. Given the potential use of these fibers in the composite industry, these treatments aimed at altering the fibers' physicochemical properties and improving the bonding between composite phases.

2.2.1 Alkali Treatments (Chemical Approach)

In this study, alkali treatments were employed to decrease the hydrophilicity of the extracted fibers. Accordingly, the fibers were bathed in NaOH solutions using 1%, 2.5%, 5%, and 10% concentrations for 1 h. Following this period, the fibers were intensely bathed using clean water.

2.2.2 Dry Etching Plasma Treatments (Physical Approach)

A dry etching plasma treatment was used to change the surface roughness of fibers. This was accomplished by physically bombarding the surface of the fibers to remove material [38]. The treatments were conducted using a DC etching device (Bal-tec SCD 050), which operates with argon gas to generate plasma. Plasma treatments were carried out applying a current of 30 ± 3 mA, a pressure of 10^{-2} kPa and a voltage of 500 ± 10 V with exposure times of 400, 1000, 1500 and 2000 s.

2.3 Tensile Tests on Treated Fibers

Tensile tests were performed to assess how the treatment conditions impacted the mechanical response of *Guadua angustifolia* fibers. To accurately capture the mechanical properties, 16 replicas were investigated. The sample size accounted for an expected error of 20 MPa in fiber strength, with a 90% confidence level. Tensile tests were carried out following the ASTM C1557-14, employing a Shimadzu universal testing machine using a 50 N load cell. A loading rate of 1.5 mm/min was used, achieving fiber failure within the standard-recommended 30 s. Samples were placed on paper frames to prevent axial load misalignment during testing, as depicted in Fig. 3. The gauge length was set to 40 mm, and the frame was carefully cut after the sample was secured in the testing grips.

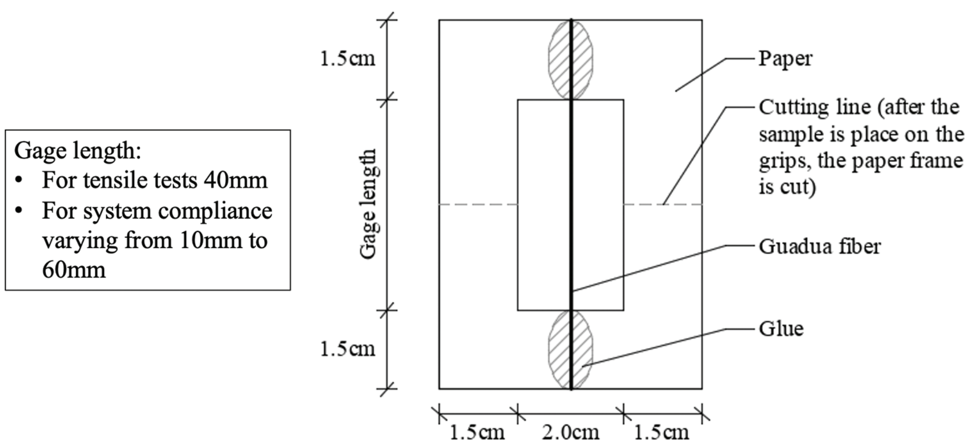

Figure 3: Fiber setup for mechanical characterization of treated *Guadua angustifolia* fibers

Tensile strength (σ) of each sample was determined using Eq. (1), where P denotes the failure load, and A represents the fiber's cross-sectional area. Previous research [55] has detailed the methodology for measuring fiber cross-sectional areas, ensuring accurate measurements by ensuring that the fracture plane is consistently perpendicular to the observer's view.