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Guadua angustifolia bamboo fibers as reinforcement of polymeric matrices: An exploratory study

P. Luna [a,*], J. Lizarazo-Marriaga [a], A. Mariño [b]

[a] Departamento de Ingeniería Civil y Agrícola, Universidad Nacional, Bogotá, Colombia
[b] Departamento de Física, Universidad Nacional, Bogotá, Colombia

HIGHLIGHTS

- Effect on surface appearance of bamboo fibers treated using sodium hydroxide baths.
- Effect on tensile strength of bamboo fibers treated using sodium hydroxide baths.
- Effect on surface appearance of bamboo fibers using a dry plasma etching technique.
- Effect on tensile strength of bamboo fibers using a dry plasma etching technique.

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ABSTRACT

Mechanical behavior of composites depends on the individual properties of each component; however, bonding between fibers and matrix plays a predominant role in the material performance. The interfacial region, which is considered as a zone of property gradients, determines the stress transfer between the bonded fibers and the matrix. A well establish interface linkage assures an adequate mechanical behavior of composite materials.

The main disadvantage for using natural fibers as reinforcement of polymeric matrices is the physico-chemical incompatibility between fibers and matrix. Because of the hydrophilic nature of fibers and the hydrophobic character of resins, it is necessary to treat both materials in order to improve the final properties of composites, avoiding premature and delamination failures.

This paper describes an exploratory research intended to use *Guadua angustifolia* bamboo fibers as reinforcement of polymeric matrices. Two different techniques of compatibilization were employed: first, standard baths of sodium hydroxide as coupling agent were applied to fibers, and second, a novel plasma treatment was implemented for natural fibers. The influence of both compatibilization methods on tensile strength and surface appearance of fibers were evaluated. Results confirm that sodium hydroxide baths decrease the mechanical strength of fibers and clean its surface. In contrast, plasma treatments showed very encouraging results, without changes on mechanical strength and improving the fiber surface.

1. Introduction

Automotive, aerospace and construction industries have shown special interest in developing new polymeric composites due to its versatility of manufacture, and especially to the high cost of production of traditional materials. Developed countries use around 50% of polymers produced on the manufacture of composite materials [1].

Synthetic fibers such as carbon, aramid, glass and nylon are commonly used as reinforcement in composite polymeric materials. However, in the last two decades natural fibers have become an important resource in composite industry [2–6]. The average annual growth rate of global market for polymeric composites reinforced with natural fibers was 38% from 2003 to 2007 [7]. In the future, it is expected that the application of this kind of materials increases.

The use of natural fibers as reinforcement of polymeric matrices gives multiple benefits such as lower abrasion during production, possibility of recycling, higher mechanical properties per weight unit, and lower manufacturing cost per volume unit [8]. Nevertheless, the potential of natural fibers has not been completely profited in the polymeric composite industry due to its

* Corresponding author.

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physicochemical incompatibility with commonly used hydrophobic matrices (polystyrene, polyester, polyethylene, among others). Natural fibers are composed by a high content of cellulose that confers its hydrophilic properties, resulting in a low compatibility between the reinforcement and the matrix, which are the composite phases. Low compatibility may bring poor stress transfer between phases and thus affect the mechanical properties of the final composite material, and in consequence, its use may be limited [2,9–12].

The literature reviewed presents the two commonly used ways to improve the compatibility between polymer matrices and natural fibers: the first is the modification of the physicochemical properties of the matrix, using reactive extrusion processes [13–15]. The second alternative is the modification of the physicochemical properties of natural fibers. This modification can be performed in three different manners: by using coupling agents [2,9,11,16–20]; by a graft polymerization of monomers compatible with the polymer matrix [1]; and subjecting fibers to plasma treatments [21–28].

At industrial level, modification of the physicochemical properties of natural fibers is the most used alternative to increase the compatibility between composite phases. The traditional method to improve the bonding between fiber and matrix employs coupling agents that react with the hydroxyl groups present in the amorphous region of fibers, leaving the cellulose structure exposed in order to bond the polymer matrix. The main disadvantage of using treatments of this type is that they generate a reconfiguration of proportions of the constituents (cellulose, hemicellulose and lignin), reducing the mechanical strength of fibers [29]. However, several studies [2,9,11,16–20], suggest that the mechanical performance of the composite improves when the natural fibers employed have been previously treated with coupling agents.

The modification of surface properties of fiber using plasma is an innovative technique; the aim of this treatment is to increase the mechanical grip on fiber–matrix interface by modifying the surface properties of fibers without major changes in its molecular structure [28]. Some research has been made using natural fibers aiming to evaluate the efficiency of this technique, where different gases have been used, among which the most common were He and Ar. Different exposure times and electrical currents have also been used [21,22,24,23,25,27,26]. In order to establish the effectiveness of the treatment, various polymer matrices have been reinforced using untreated and plasma treated natural fibers [21,22,25,27,26]. The main conclusion of these researches is that the composite tends to be more resistant as a result of the increased bonding between fibers and the polymer matrix. Such resistance was attributed to the increasing surface roughness of the reinforcing material.

This paper presents the results obtained during an exploratory study of some techniques employed to modify the surface properties of *Guadua angustifolia* bamboo fibers. In order to do this, a common coupling agent such as sodium hydroxide (NaOH) and a novel plasma technique were used. The surface appearance was assessed using a scanning electron microscope, while the influence of each treatment on the mechanical properties was evaluated using tensile tests. These treatments were made in order to, in second stage, develop a polymeric composite material using as reinforcement *Guadua angustifolia* bamboo fibers, that could be used as non-structural material in buildings.

2. Material and methods

2.1. Fiber extraction

Guadua angustifolia bamboo fibers were extracted from the bottom part of culms, using a compression load similar to that described by [30]. Initially, bamboo raw samples were cut into segments of rectangular cross section with 10 cm of length, which were immersed in a solution containing sodium hydroxide (NaOH) at 10% during 96 h. After immersion, segments were carefully washed up using tap water. The rectangular segments were crushed, applying compression loads of about 100 kN. Finally, the fibers were separated using a brush. Using this procedure, fibers with around 10 cm length were obtained.

2.2. Coupling agent treatment

After extraction, fibers were divided into two groups corresponding to the concentration of the solutions (2 and 10%) of NaOH employed in this treatment as coupling agent. The immersion times for both solutions were 10 and 60 min. All the immersions were performed at atmospheric pressure and room temperature.

After this, fibers were carefully washed using tap water and dried at room temperature for two days before the mechanical tests.

2.3. Plasma treatment

In this study, a dry etching plasma treatment (physical sputtering) was carried out. It works at low pressure and high energy. Using this treatment, it is expected that the fiber surface roughness could be increased due to ions bombardment. The treatment was carried out by using a D.C *etching* system with Ar gas. The fibers were exposed to ion bombardment for 300 s with a current of 50 mA at a working pressure of 50×10^{-2} mbar (0.37 Torr). The objective of this treatment is to increase the fiber surface roughness by an ion bombardment from the plasma. Before being treated, fibers were placed on a carbon conductive film in order to ensure the efficiency of the treatment and achieve fixation of the samples during the processing.

2.4. Fiber mechanical testing

In order to study the influence of treatments on mechanical properties of fibers, tensile tests were carried out on specimens without treatment and treated using the coupling agent proposed or plasma technique.

Tensile tests were performed following the guidelines established on ASTM C1557-14: Standard Test Method for Tensile Strength and Young's Modulus of Fibers. The rate of load application was 2 mm/min, in which the failure of fiber was achieved in 30 s, according to the indications of ASTM C1557-14. Before the tests, single bamboo fibers were glued to paper frames as shown in Fig. 1. This setup was implemented in order to avoid deviations of axial load due to incorrect positioning of the specimen in the testing grips. After adjusting the fiber specimen, this frame was carefully cut.

Due to the fact that *Guadua angustifolia* bamboo fibers have an irregular cross section [31], the average cross section (A) was calculated using Eq. 1, where m and L are the fiber mass and length respectively, and ρ is the apparent density, which according to [32] is 1.44 g/cm^3.

$$A = \frac{m}{\rho L} \tag{1}$$

The average tensile strength was calculated using Eq. 2, where P_{max} is the maximum load resisted on each mechanical tests, and A is the average cross section.

$$\sigma = \frac{P_{max}}{A} \tag{2}$$

2.5. Morphological analysis

A scanning electron microscope (SEM) was used to take images of *Guadua angustifolia* bamboo fibers surface, in order to evaluate the changes in its surface by sodium hydroxide baths or plasma treatment.

Fig. 2 shows the experimental procedure used on this research schematically.

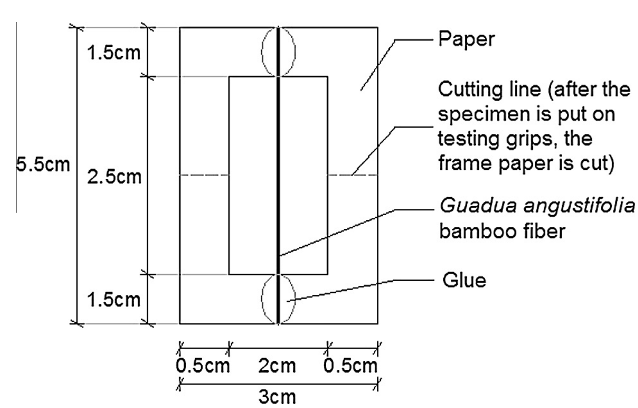

Fig. 1. Paper frame used on tensile test of fibers.

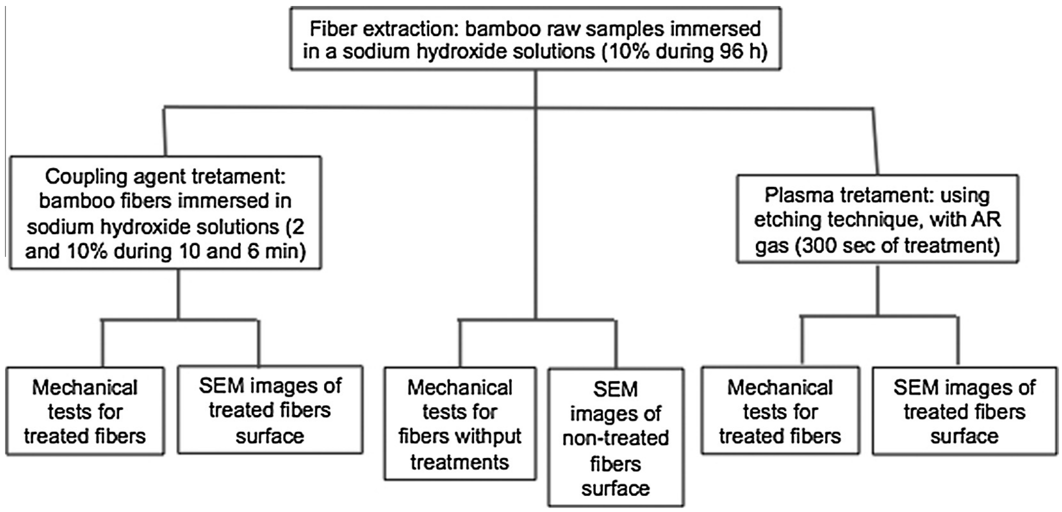

Fig. 2. Diagram of experimental procedure.

Table 1
Tensile stress for *Guadua angustifolia* bamboo fibers.

Treatment	σ_t (MPa)	S.D. (MPa)	C.V. (%)
N	179.37	78.36	44
P	173.65	52.87	30
H210	167.37	65.73	39
H260	148.30	82.57	56
H1010	127.08	67.12	53
H1060	60.01	28.10	47

σ_t: tensile strength; S.D: standard deviation; C.V.: coefficient of variation.

3. Results and discussion

3.1. Fiber mechanical testing

Table 1 presents the results of the average tensile strength for *Guadua angustifolia* bamboo fibers. N values correspond to fibers without treatment; P to fibers with plasma treatment; H210 and H260 to fibers immersed in sodium hydroxide with a concentration of 2% for 10 and 60 min respectively; and H1010 and H1060 to the same treatment with a concentration of 10% for 10 and 60 min respectively.

Results showed that plasma treatment does not reduce the tensile resistance; similar results were found by [27] for jute fibers treated by oxygen plasma. On the other hand, treatments using sodium hydroxide reduce the resistance, and the decrease was more significant when the immersion was done using a higher concentration and during a long time. According to [29], coupling agent treatments can result in a reconfiguration of hemicellulose and lignin constituents, which has a direct effect on fiber strength.

3.2. Morphological analysis

Fig. 3 shows SEM micrographs of the fiber surface for untreated and plasma treated fibers. Before plasma treatment (Fig. 3a), the fiber surface presented some irregularities generated by the presence of non-cellulosic materials (hemicellulose and lignin constituents). After plasma treatment (Fig. 3b), the roughness of fiber surface increases, as a result of the ion bombardment.

It is believed that in immersions using sodium hydroxide it works as a cleaner, removing the non-cellulosic materials from the cracks and pits on the fiber surface (Fig. 4). Fig. 4a) shows the fiber surface before the treatment. The irregularities observed are produced by hemicellulose and lignin constituents. Fig. 4b) and c) shows the surface of fibers treated with a solution of 2% during 10 and 60 min respectively. In Fig. 4b) the cleaning of the surface is not completely efficient and some irregularities are observed; and in 4c) it is evident that the surface is regular, which suggests that the content of non-cellulosic materials is lower. Fig. 4d) and e) shows the surface of fibers treated with a solution

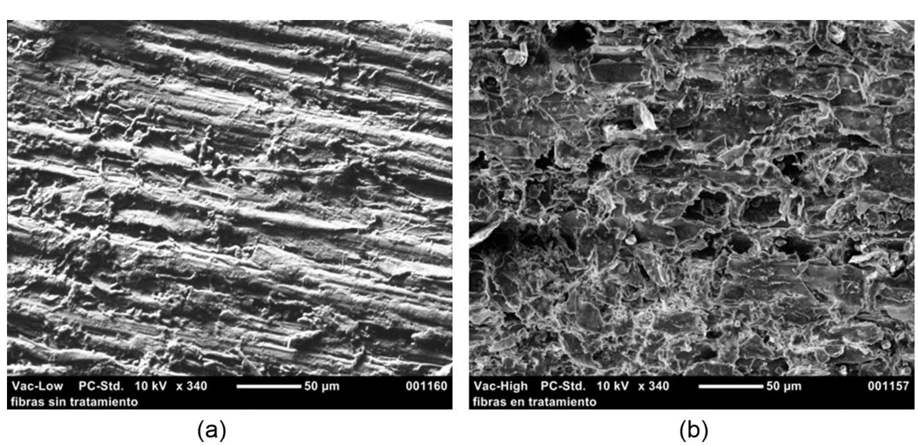

Fig. 3. SEM micrographies for: (a) untreated, and (b) plasma treated *Guadua angustifolia* bamboo fibers.

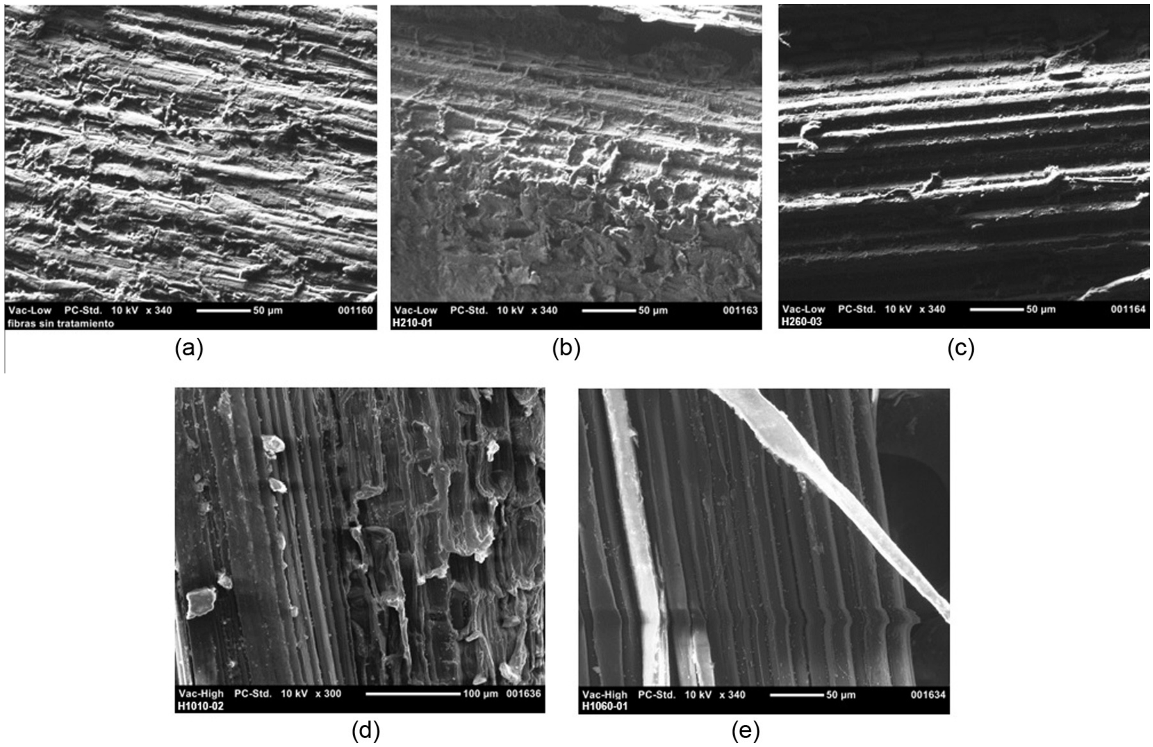

Fig. 4. SEM micrographies for *Guadua angustifolia* bamboo fibers: (a) untreated, and different baths of sodium hydroxide (b) H210, (c) H260, (d) H1010 and (e) H1060.

of 10% during 10 and 60 min; in Fig. 4d) some non-cellulosic materials are visible; in Fig. 4e) the fiber surface is cleaner and it can be observed that the bamboo fiber was divided into microfibers.

Bamboo fibers are composed by microfibers, which from the mechanical point of view work together to resist loads. Baths using NaOH remove hemicellulose and lignin constituents which work as glue-like materials, decreasing mechanical strength (Table 1). Plasma treatment affects the fiber surface, and the results in terms of tensile resistance suggest that the ion bombardment could has no implications on fibers mechanical behavior.

All analysis carried out using SEM micrographs are qualitative observations, which are preliminar results. These should be complemented through other experimental measurements, which are out of the scope of this paper.

4. Conclusions

It can be concluded that the treatment with the coupling agent (sodium hydroxide solutions) used in this research notably affects the tensile strength of *Guadua angustifolia* bamboo fibers. Although the coupling agent works as surface cleaner, it also affects the tensile strength because it removes the hemicellulose and lignin constituents. SEM images suggest that this treatment works as a surface cleaner, but for higher concentrations and prolonged times it results on the division of macrofiber.

Results showed that the use of plasma for treatment of *Guadua angustifolia* bamboo fibers, could increase the adherence between the reinforcement and different polymer matrices. Fibers that were treated with plasma did not show changes in its mechanical strength.

The treatment applied as well as the results obtained stimulate us to propose further exploration on these techniques. Thus, a working second stage of this research seeks to employ this treatments in the development of a polymeric composite material for non-structural elements in buildings.

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