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# Preliminary Study on the Compatibilization Techniques of Natural Fibers as Reinforcement of Polymeric Matrices

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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-established interface linkage ensures an adequate mechanical behavior of composite materials.

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

This paper describes an exploratory research aimed to use *Guadua angustifolia* (guadua) bamboo fibers as reinforcement of polymeric matrices. Two different compatibilization techniques were employed: first, sodium hydroxide baths were applied as a coupling agents, and second, plasma was used as a novel treatment. The influence of both compatibilization methods on the mechanical properties of fibers were investigated. Results confirmed that the use of sodium hydroxide decreases the tensile strength of fibers. In contrast, plasma treatments showed encouraging results, without changes on the mechanical strength.

## INTRODUCTION

Composite materials are formed by a dispersed phase (reinforcement material) embedded into a continuous phase (matrix). The assessment of the material performance depends on their mechanical properties; in fact, the properties, distribution, and interaction among their constituent elements are essential for describing the material behavior. Distinctively, the physicochemical interactions between components are liable to the process of transferring stresses from the matrix to the reinforcement material (Daniel & Ishai 1994; Valadez 1999; Hodgkinson 2000; Agarwal et al. 2006).

According to different authors (Bledzki et al. 1996; Bledzki & Gassan 1999; Rijswijk et al. 2001; Mohanty et al. 2002; Faruk et al. 2013), the interest of using fibers from natural and renewable resources has risen during the last two decades. In consequence, their market has increased steadily; as a matter

of fact, from 2003 to 2007 the growth rate was 38% worldwide and it is expected to double by 2020 (Faruk et al. 2012).

There are multiple benefits in using natural fibers as reinforcement of polymeric matrices. First, they have high mechanical properties per unit of weight and the cost of manufacturing per unit of volume is fairly low (Valadez 1999; Tapia et al. 2006; Taj et al. 2007; Spear 2009). Nonetheless, the high content of cellulose of the natural fibers gives to the material some hydrophilic properties (Barkoula et al. 2008; P. K. Kushwaha & Kumar 2009; Song et al. 2015), which make them chemically incompatible with most of the polymers used in the composite materials industry. As a result, the market and industrial applications of composites using natural fibers are limited to products that demand low mechanical characteristics (Barkoula et al. 2008; Han et al. 2008; Song et al. 2015; Islam et al. 2013).

Therefore, it is essential to ensure a suitable bonding between fibers and polymeric matrices to take the higher advantages of the mechanical capacity offered by natural fibers. In this regard, some research have proposed two procedures in order to overcome the limitations of natural fibers. One suggest modifying the chemical properties of the matrix by using reactive extrusion processes (Raquez et al. 2008; Garnier et al. 2010; Haque & Pracella 2010) or with plasma treatments (Švorčík et al. 2006). The second one indicates a change on the physicochemical properties of the fibers by using coupling agents (Bledzki et al. 1996; George et al. 2001; Kalia et al. 2009; Elvy et al. 1995; Araújo et al. 2008; P. Kushwaha & Kumar 2009; Lee et al. 2009; Kim & Netravali 2013), making a graft polymerization of monomers compatible with the polymer matrix (Valadez 1999; Herrera-Franco & Valadez-González 2005), or using plasma treatments (Gassan & Gutowski 2000; Marais et al. 2005; Oraji 2008; Xu et al. 2006; Morshed et al. 2010; Ragoubi et al. 2010; Seki et al. 2010; Amirou et al. 2013)

The most common procedure used in the industry is the modification of the fiber properties, in particular by using coupling agents. These coupling agents react with hydroxyl groups that are present at the fiber surface amorphous region, exposing the cellulose structure to react with the binding materials (Kabir et al. 2013; Li et al. 2007b; Guimaraes et al. 2013). The main disadvantage of using this kind of treatment is the reconfiguration of the fiber's structure that diminish its mechanical properties (Kabir et al. 2013; Bledzki & Gassan 1999; Faruk et al. 2012).

Another recently investigated alternative for the modification of the fibers' physicochemical properties is treat them using plasma (Gassan & Gutowski 2000; Marais et al. 2005; Oraji 2008; Xu et al. 2006; Morshed et al. 2010; Ragoubi et al. 2010; Seki et al. 2010; Amirou et al. 2013). The objective of this is increasing their superficial roughness, hence the mechanical adherence grows between the composite material phases. Applying this method, the macroscopic characteristics of the fiber remains at the same level because it is a superficial treatment. Another advantage of this method corresponds to the environmental benefits because it does not generate hazard or toxic waste.

This paper summarizes the results obtained in the early stages of a research aiming the development of a composite material using a polymeric matrix reinforced with *Guadua Angustifolia* (guadua) bamboo fibers. Two compatibilization methods have been explored to modify the fibers' physicochemical properties: employing sodium hydroxide solutions as a coupling agent and using a plasma technique.

## MATERIALS AND EXPERIMENTAL PROCEDURE

**Fiber extraction**. Guadua bamboo fibers were extracted from the lower part of culms, aged between three to five years. Bamboo stems came from the department of Quindío, Colombia. Similar to the procedure described by Deshpande et al. (Deshpande et al. 2000), the fibers extraction started by cutting 10 cm length segments of rectangular cross section and immersed them into a sodium hydroxide solution (NaOH) at 10%. After 96 hours of immersion, the samples were carefully washed up using tap water, crushed by loads nearly to 100 kN and the fibers were separated using a metal brush. Finally, the

fibers were again watched up with tap water and air dried during a week before applying any compatibilization treatment.

## Coupling agent treatment

Sodium hydroxide was chosen as coupling agent into two solution concentrations: 2% and 10%. The fibers treated with this technique were separated in two groups accordingly with the concentration employed. Immersion times used correspond to 10 and 60 minutes for both concentrations. These treatments were performed at atmospheric pressure and room temperature.

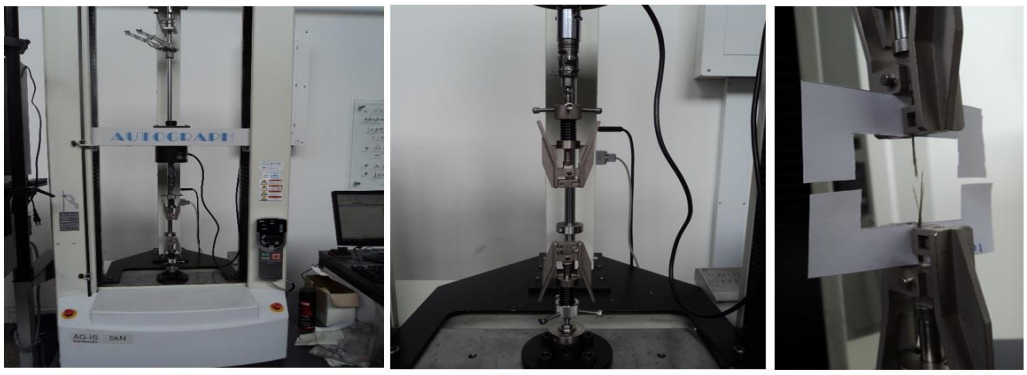
**Plasma treatment**. The plasma treatment was carried out by using a D.C. sputtering system with Argon gas. The fibers were exposed to ion bombardment for 300 seconds with a current of 50 mA at a working pressure of $5x10^{-2}$ mbar. In order to ensure the efficiency of the treatment and achieve fixation of the samples during the procedure, the fibers were placed on a conductive carbon film before being treated.

**Tensile tests.** The influence of each compatibilization treatment on the fibers' mechanical behavior was measured through a tensile test on samples with no treatment and specimens treated with the techniques explained above. Every test was performed following the standard ASTM1557-14 (ASTM 2014) using a Shimadzu universal testing machine, a loading speed of 1.5mm/min and a load cell of 50 N. Figure 1 shows the testing machine and the restraint system used in this research. All the samples were set up on paper frames, as shown in figure 2, using 40 mm of gage length; the frame was carefully cut after adjusting the specimen on the testing grips.

It was established the equipment's system compliance according to the ASTM1557-14 standard and using the fibers extracted through the methodology mentioned above without any compatibilization treatment. There were tested samples using different gage lengths: 10, 20, 30, 40, 50 and 60 mm, and using five samples per each length. The total cross-head displacement during the fiber tests, $\Delta L$, was determined using equation 1, where $F$ was the applied force, $l_o$ was the gage length, $E$ was the young's modulus, $A$ was the cross-sectional area of the fiber, and $C_s$ was the system compliance. Hence, by plotting ΔL/F versus lo/A, it was obtained a straight line with a constant slope (1/E) and the intercept with the vertical axis corresponds to the system compliance $C_s$. Thereby, the fiber elongation, $\Delta l$, can be determined with equation 2.

$$\frac{\Delta L}{F} = \frac{l_o}{EA} + C_S \quad (1)$$

$$\Delta l = \Delta L - C_s F \quad (2)$$




a) b) c)

**Figure 1. Tensile test: a) universal testing machine, b) restraint system, c) restraint system detail.**

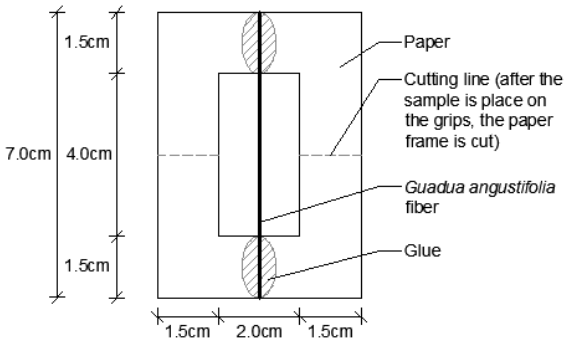



**Figure 2. Paper frame used for fiber tensile tests**

The tensile strength of each fiber, $\sigma_t$, was determined using equation 3, where $P_{max}$ was the sample's failure load and $A$ was the cross-sectional area of the fiber at fracture plane.

$$\sigma_t = \frac{P_{max}}{A} \tag{3}$$

The fibers' cross-sectional area at the fracture plane was measured with high accuracy taking micrographs through a metallographic microscope Leco500 and a 100X magnification. Prior the micrographs were taken, the fibers were put in a transparent resin and afterward, the software ImageJ used to calculate the actual measurement. This procedure ensured that the perpendicular axis of the image cross-section matched the observer's eyes axis, which avoided optical distortions.

A statistical analysis led to identifying significant differences among the data of tensile strength values and maximum elongation measures from the tests. Initially, through the Shapiro-Wilk tests, it was verified whether the distribution of each data set were normal. The homoscedasticity was verified among the data collected through the Levene's test. An ANOVA analysis was applied in the cases to fulfill both criteria of normality and homoscedasticity; in the opposing cases, it was used the Kruskal-Wallis test (Montgomery & Runger 2002). The significance level for every case was 0.05.

## RESULTS AND DISCUSSION

**System compliance and gage length effect.** Figure 3 shows the result of the system compliance determination for the universal testing machine and the restraint system used; the atypical data were excluded from this graph. The point cloud was analyzed through a linear least-squares regression that was extrapolated to the vertical axis; the cut-off point obtained is the $C_s$ value. The correlation coefficient $R^2$ in the linear regression was 0.63.

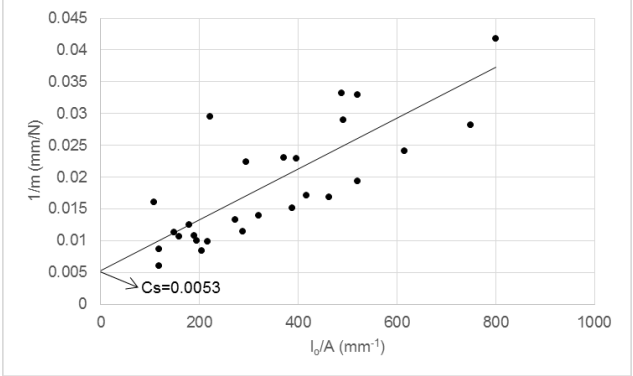



**Figure 3. Determination of system compliance**

Table 1 shows the results of tensile strength and maximum elongation for fibers with different gage lengths, these results are also shown in figures 4 and 5. The maximum elongation values were fixed according to the system compliance. The ANOVA analysis on tensile strength data did not reveal substantial differences among the values for each gage length. Similar outcomes were reported by Osorio et al (Osorio et al. 2010; Osorio et al. 2011) for guadua fibers, and for Alves et al. (Alves et al. 2013) for jute fibers. In contrast, there were differences among the maximum elongation values according to the Kruskal-Wallis test. Samples of 10, 20 and 60 mm gage length showed significant variations, in fact, as higher was the gage length, higher was the maximum elongation. In table 1 different letters for the same mechanical property indicate that there are significant differences between those values.

**Table 1. Tensile strength and maximum elongation for fibers tested with different gage length**

| GL (mm) | $\sigma_t$ (MPa) | $\Delta l$ (mm) |
|---|---|---|
| 10 | 219.98 ± 39.41[a] | 0.2874 ± 0.0628[ac] |
| 20 | 210.97 ± 60.26[a] | 0.2642 ± 0.0651[ac] |
| 30 | 183.58 ± 42.65[a] | 0.3763 ± 0.1205[ab] |
| 40 | 175.34 ± 48.47[a] | 0.6512 ± 0.1697[ab] |
| 50 | 199.46 ± 55.47[a] | 0.5286 ± 0.2468[ab] |
| 60 | 188.49 ± 58.65[a] | 0.7367 ± 0.2376[b] |

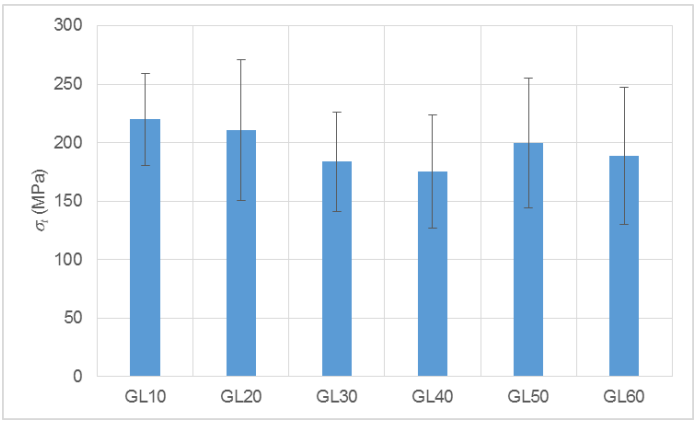



**Figure 4. Average tensile strength for different gage length**

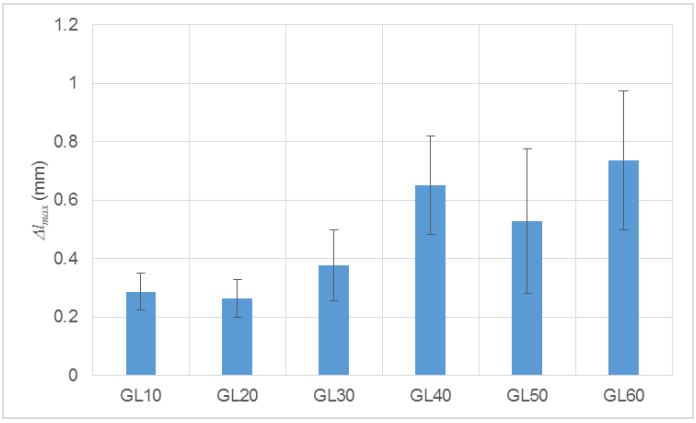



**Figure 5. Maximum elongation for different gage length**

**Tensile behavior.** This section shows the results obtained after the treatment of the fibers. Table 2 shows the results obtained from each treatment used, where the maximum elongation values were corrected with the system compliance found. Accordingly, letter N denotes fibers without any treatment after the extraction procedure; letter P indicates fibers with plasma treatment; H210 and H260 correspond to fibers treated with the coupling agent using solutions at 2% for 10 and 60 minutes respectively. Finally, H1010 y H1060 are fibers handled with the same treatment but using solutions at 10% for 10 and 60 minutes respectively. These results are also show in figures 6 and 7.

**Table 2. Tensile strength and maximum elongation for different fiber treatments**

| Treatment | $\sigma_t$ (MPa) | $\Delta l$ (mm) |
|---|---|---|
| N | $223.70 \pm 26.85^{c}$ | $0.8503 \pm 0.2198^{a}$ |
| P | $234.38 \pm 32.80^{c}$ | $0.5864 \pm 0.2635^{a}$ |
| H210 | $203.46 \pm 35.92^{c}$ | $0.7991 \pm 0.2214^{a}$ |
| H260 | $144.63 \pm 35.23^{b}$ | $0.6638 \pm 0.2336^{a}$ |
| H1010 | $100.42 \pm 25.38^{ab}$ | $0.7334 \pm 0.2358^{a}$ |
| H1060 | $75.18 \pm 21.73^{a}$ | $0.5550 \pm 0.2251^{a}$ |

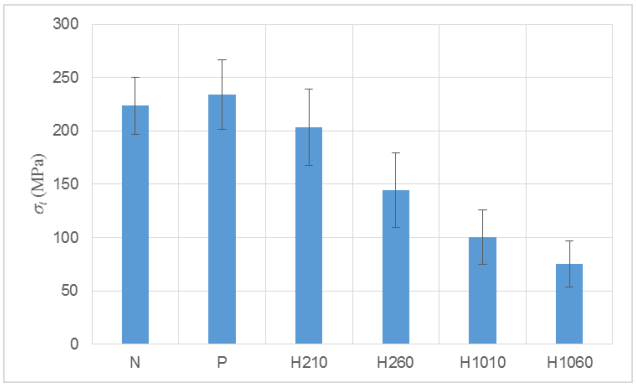



**Figure 6. Average tensile strength for different fiber treatments**

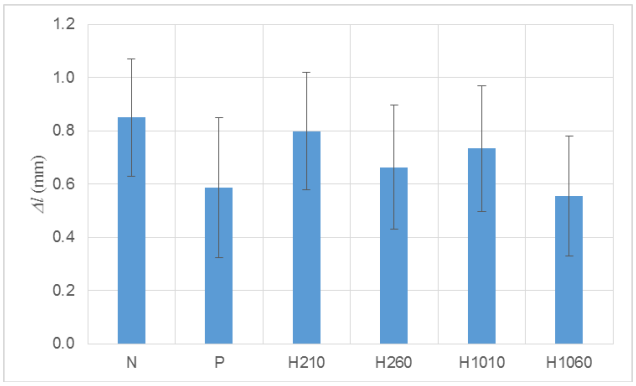



**Figure 7. Average maximum elongation for different fiber treatments**

ANOVA data analysis on tensile strength values pointed out substantial variations among some of the values obtained. However, there are no differences between the average fiber strength for N, P and H210 cases. The trend of tensile strength shows a decreasing with the increase on the solution concentration and treatment time. Seki et al. (Seki et al. 2010) report no change on the mechanical strength of jute fibers treated with oxygen plasma, in addition, it was reported that the treatment increases the fiber superficial roughness. According to Kabir et al. (Kabir et al. 2013), treatments with

NaOH produce a reconfiguration of hemicellulose and lignin, which are important components of natural fibers. In this way, the chemical treatment causes a downward in the mechanical capacity. On the other hand, ANOVA analysis on the maximum elongation data suggests that there are no substantial differences of this mechanical property among the different treatments applied.

## CONCLUSION

The coupling agent treatment used in this research decrease the mechanical strength of *Guadua Angustifolia* bamboo fibers, which can be a consequence of the components reconfiguration of the fiber. On the other hand, the treatment with argon plasma does not change the material strength. It was assumed that this treatment acts only superficially, avoiding any microscopic or compositional alteration.

The outcomes of this research will be examined with depth detail during the second stage of the research, which aims the development of a composite material with a polyester matrix reinforced by *Guadua Angustifolia* bamboo fibers.

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