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Morphological and nanomechanical characterization of Guadua *Angustifolia kunth* fiber by means of SEM and AFM

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Abstract

Recent developments in engineering have promoted the use of reinforced composite materials from natural fibers, which provides an opportunity to investigate such materials using state-of-the-art tools. Here we present a morphological and nanomechanical characterization of the parallel section of the axis of guadua *Angustifolia kunth* fibers (GAK) from Colombia, focusing on properties such as hardness (nanoindentation), roughness and topography. Our method was based on the application of scanning electron microscope (SEM) and atomic force microscope (AFM). AFM provided curves of force by displacement, as well as characteristics of dynamic nanoindentation systems and images. Their analysis revealed ridges and valleys on the surface of GAK fibers. The estimated surface roughness of 9.51 nm suggests an adequate value to provide superior adhesion between polymer and fiber. The same conclusion follows from our measurements of hardness, reduced modulus and nanoscale topography. Due to their excellent properties, we conclude that GAK fibers represent an ideal reinforcement material in polymer matrices.

Keywords: fibers of Guadua *Angustifolia kunth*; morphology; nanoindentation; SEM; AFM.

Caracterización morfológica y nanomecánica de la fibra de Guadua *Angustifolia kunth* mediante SEM y AFM

Resumen

El desarrollo reciente en la ingeniería propone el uso de materiales compuestos reforzados a partir de fibras naturales, lo cual genera la iniciativa de estudiarlos mediante herramientas sofisticadas. En esta investigación se muestran los resultados de la caracterización morfológica y nanomecánica como dureza (nanoindentación), rugosidad y topografía de la sección paralela al eje axial de fibras de guadua *Angustifolia kunth*, con el fin de encontrar las propiedades mecánicas a nanoescala de las fibras. Para ello se emplearon los microscopios electrónico de barrido (SEM) y de fuerza atómica (AFM), con este último se obtuvieron curvas de fuerza vs desplazamiento, características de sistemas dinámicos de nanoindentación e imágenes, donde a partir de análisis se encontró que la guadua *Angustifolia kunth* (GAK) presenta crestas y ondulaciones en su superficie, las cuales le brindan una rugosidad adecuada otorgando adherencia entre el polímero y la fibra, además la dureza, el módulo reducido y la topografía a escala nanométrica, lo que permite concluir que las fibras de GAK presentan mejores propiedades para ser utilizadas como material de refuerzo en matrices poliméricas.


Palabras claves: fibras de Guadua *Angustifolia kunth*; morfología; nanoindentación; SEM; AFM.

1. Introduction

Polymer matrix composites consist of a continuous phase (polymer matrix) and a dispersed phase (particles or fibers) [1,2]. Usually the dispersed phase consists of fibers. Most

studies have shown that synthetic fibers such as graphite [3,4] glass [5,6], carbon [7, 8], and thermoplastic resins such as polyesters and polypropylenes [9,10] among others, can be used as reinforcement for composite materials through the incorporation of fibers, thereby improving their properties

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[11]. However, many studies have ignored the high energy requirements and negative impact on the environment associated with the production of those materials. While some fibers are widely used in industry due to their excellent properties, they may on the other hand generate health problems such as asbestosis [12]. In Colombia, a considerable volume of organic waste derived from the production of fique, guadua, coconut, rice husk, bagasse, husk and other husk fibers have contributed to increasing pollution levels [13,14]. Therefore, the incorporation of organic fibers as reinforcement in polymeric matrices would not only avoid the use of synthetic or artificial fibers, but also reduce the environmental impact generated by their disposal.

2. Materials and methods

For our study, fibers of guadua *Angustifolia kunth* were collected in El Peñón, Cundinamarca Department, Colombia. Fiber extraction was mechanically carried out, so that fiber diameter was under 1mm and lengths under 5mm. Surfaces of GAK fibers were then modified with 5% sodium hydroxide (NaOH) solution to remove impurities [36], continuously stirred for half an hour at room temperature, and finally washed with distilled water (neutral pH) to completely eliminate NaOH. Fibers were then left to dry for 12 hours at room temperature, and 24 hours at 60 °C [37].

2.1. Scanning electronic microscopy (SEM)

Fibers were coated with gold to increase conductivity of samples and enable analysis by a Scanning Electron Microscope, JEOL brand, model JSM 6490-LV. ImageJ software was used for processing of digital images and measurement of average diameter and cross-sectional area of fibers.

2.2. Atomic force microscopy (AFM)

To investigate the mechanical behavior of natural fibers at low scale through instrumented indentation tests, we used the AFM brand ASYLUM RESEARCH, model MFP-3D-BI. Fibers were cut down to a length of 5mm, and appropriate points for nanoindentation were identified on their surface. This process consists in applying a force to the fiber with the tip of an indenter or cantilever to create indentation marks. The tip is pressed continuously against the fiber for about 2 seconds, and the resulting displacement is measured. Data on force vs. displacement, and on indenter geometry, were used to calculate hardness (H), reduced modulus (E_r), rupture modulus (H/E_r), resistance to plastic deformation (H^3/E_r^2) and elastic module, after the method proposed in 1992 by Oliver and Pharr. In addition, fibers were swept before and after the indentation at low force using the same indentation tip, thereby generating a topographic record on nanometric scale. The obtained nanoscale topographic images of fiber and indentation traces enabled quantification of the stacking of ductile material around the indenter. For the nanoindentation test, an AC 160 TS Olympus indenter was selected based on the principle that the indenter tip must have an elastic modulus equal to or greater than the sample in order

to generate a deformation. Finally, water left over from the NaOH treatment was slowly neutralization (until neutral pH) by diluted acetic acid (vinegar) and disposed by a hazardous waste management company with a valid environmental license.

3. Results and discussion

3.1. Morphological characterization

Fig. 1 shows the cross section of a GAK fiber from the interior of the cluster and treated with 5% NaOH. The fiber has an irregular and elongated shape, while its transversal area is composed of irregularly shaped microfibrils. Average value was 13301.806 μm^2 .

Fig. 2 shows a comparison between two GAK fibers with and without the 5% sodium hydroxide treatment. The untreated fiber presents irregularities due to the presence of lignin and hemicellulose [38], while the treated fiber exhibits no imperfections, and instead consists of visible microfibrils (observable in the cross-section). Treatment exposes microfibrils aligned in the axial direction, which is relevant for applications to composite materials by providing stronger adherence between the polymer matrix and natural fibers.

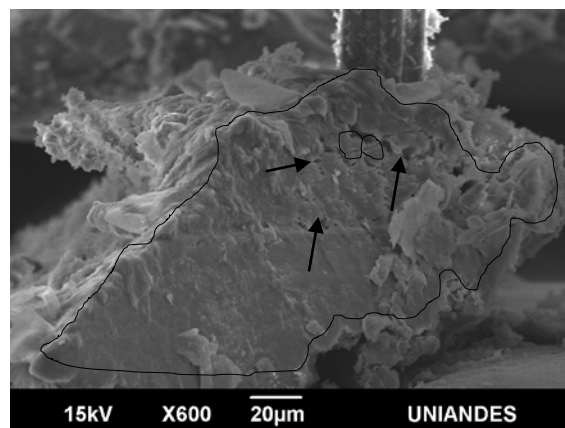


Figure 1. Transversal section of GAK fiber.
Source: Authors.

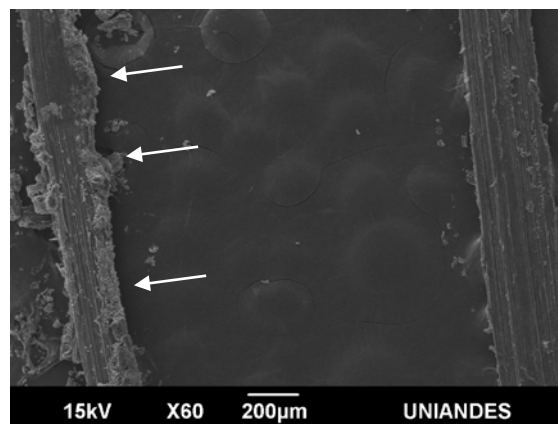


Figure 2. Longitudinal section of GAK fiber, with and without 5% NaOH treatment.
Source: Authors.

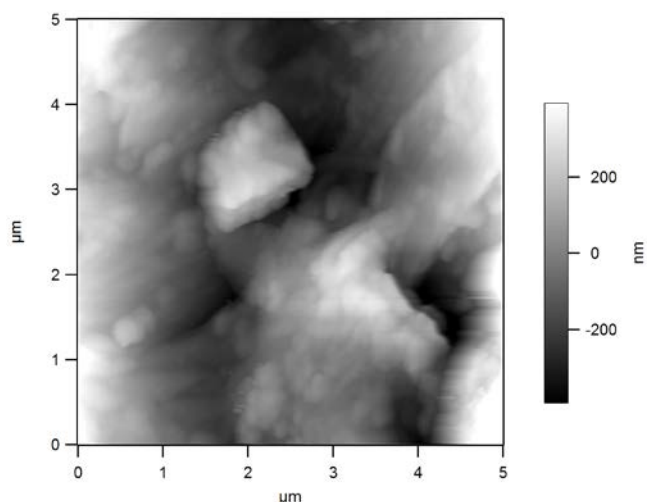


Figure 3. Longitudinal section of GAK fiber before indentation. AFM altitude image.
Source: Authors.

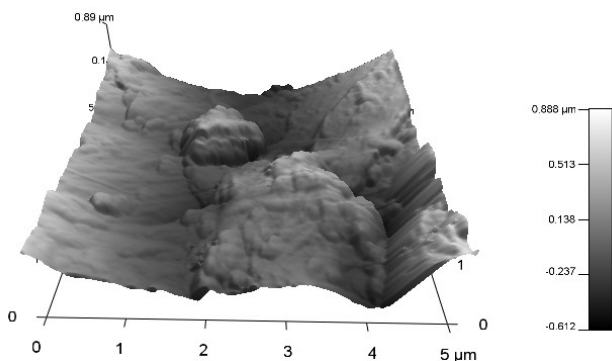


Figure 4. 3D morphology of the longitudinal surface of GAK fiber.
Source: Authors.

3.2. GAK topography

Prior to indentation, we recorded the longitudinal topography of a section of GAK fiber measuring 5 μm x 5 μm . Fig. 3 shows that the GAK fiber displays an irregular structure on most of its surface.

Fig. 4 is a 3D section along the longitudinal axis of the GAK fiber, revealing surfaces without defined shape that protrude between 0.888 μm -0.612 μm relative to the general surface. It exhibits superior roughness (R_a) to the areca palm [39], but inferior to flax [40]. This level of roughness may facilitate adhesion between polymer matrices and GAK fibers, confirming our SEM findings.

3.3. Nanoindentation

Figs. 5 and 6 show a longitudinal section of GAK fibers before and after nanoindentation. In Fig. 5, cellulose microfibrils are observed in lignin within the hemicellulose matrix [34] and aligned with the main axis of the GAK fiber. Due to their orientation, microfibrils confer guadua superior resistance to tension along the main axis of the fiber. In

addition, the distribution of microfibrils confirms that guadua is an orthotropic material [41]. Moreover, Fig. 6 shows 10 nanoimprints with different indentation depths, as shown in Table 2. Nanoimprints 2, 3 and 4 cannot be clearly seen in Fig. 6 since the nanoindentation zone exhibits elastic behavior, recovering by 98.7%, 96.70 and 96.13% respectively after removal of the indentation load, therefore presenting no residual deformation. In contrast, nanoimprints 1, 5-10 presented only minor and non-identifiable recovery.

Fig. 7 shows the loading and unloading curve of nanoimprint 1. The curve is similar for indentations on other nanoimprints, which is most likely explained by the similar elastic properties of GAK fibers in most of their surface.

Following the analysis of curves and the method proposed by Oliver and Pharr, Table 1 presents measures of hardness, reduced modulus, modulus of rupture and resistance to plastic deformation for each of the 10 nanoimprints. Hardness ranges between 10.27 and 13.48 MPa, demonstrating the homogeneity of the longitudinal section.

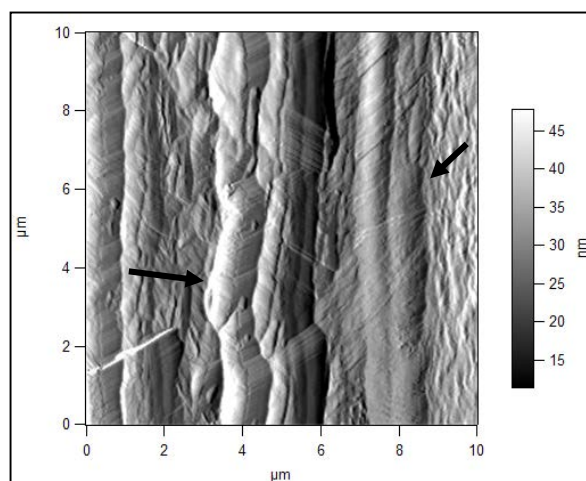


Figure 5. Longitudinal section of GAK fiber prior to indentation. AFM amplitude image.
Source: Authors.

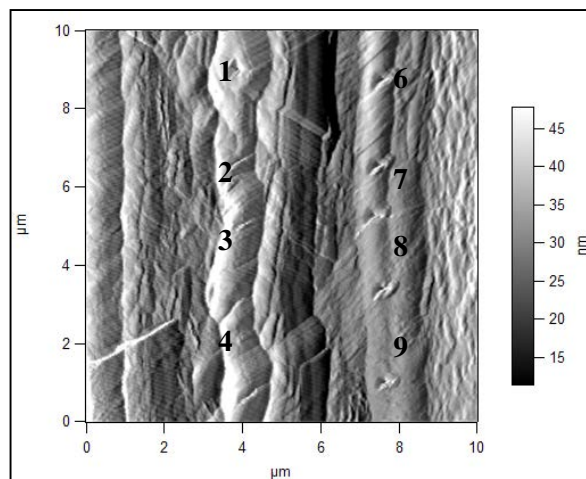


Figure 6. Longitudinal section of GAK fiber after indentation. AFM amplitude image.
Source: Authors.

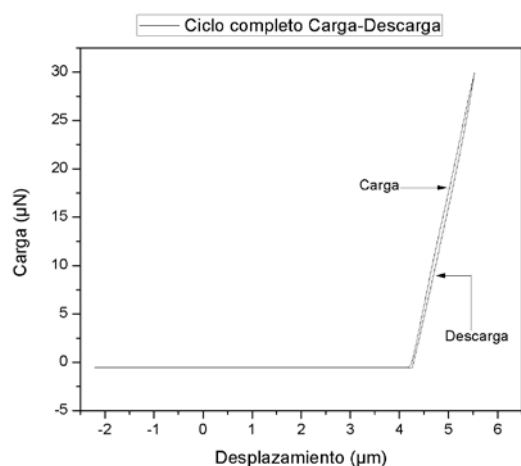


Figure 7. Full loading-unloading cycle of nanoimprint 1.
Source: Authors.

Table 1.

Mechanical properties at the nanoscale of GAK fibers.

777777-curves (Fig. 8), as well as residual depth values (h) for each nanoimprint and the percentage of elastic recovery.

Residual deformation of nanoimprints is variable; however, nanoimprints 2, 3 and 4 show the highest residual

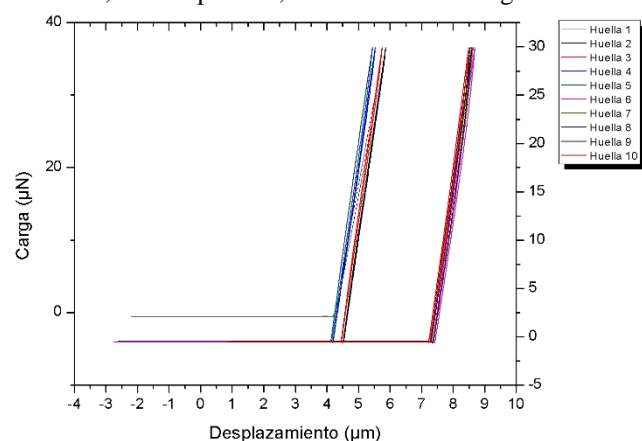


Figure 8. Loading-unloading curves of the 10 nanoimprints on GAK fibers.
Source: Authors.

Table 2.

Elastic properties of GAK fibers.

| Nanoimprint | hmax (nm) | h (nm) | Recovery (nm) | Recovery (%) |
|-------------|-----------|--------|---------------|--------------|
| 1 | 1280,90 | 92,44 | 1188,46 | 92,78 |
| 2 | 1342,21 | 17,49 | 1324,72 | 98,70 |
| 3 | 1298,23 | 42,31 | 1255,92 | 96,74 |
| 4 | 1346,93 | 52,15 | 1294,78 | 96,13 |
| 5 | 1294,50 | 81,30 | 1213,20 | 93,72 |
| 6 | 1273,86 | 80,50 | 1193,36 | 93,68 |
| 7 | 1281,23 | 92,79 | 1188,44 | 92,76 |
| 8 | 1236,41 | 66,06 | 1170,35 | 94,66 |
| 9 | 1242,99 | 85,57 | 1157,42 | 93,12 |
| 10 | 1251,23 | 86,46 | 1164,77 | 93,09 |
| Mean | 1284,85 | 69,71 | 1215,14 | 94,54 |

Source: Authors.

difference, with values of 17.49, 42.31 and 52.15 nm. This confirms that they exhibit the highest values of elastic recovery (Table 2), as shown in Fig. 6 where they do not exhibit an indentation mark. On the other hand, the nanoimprints with the most visible marks in indentation (Fig. 6) show the highest values of residual deformation.

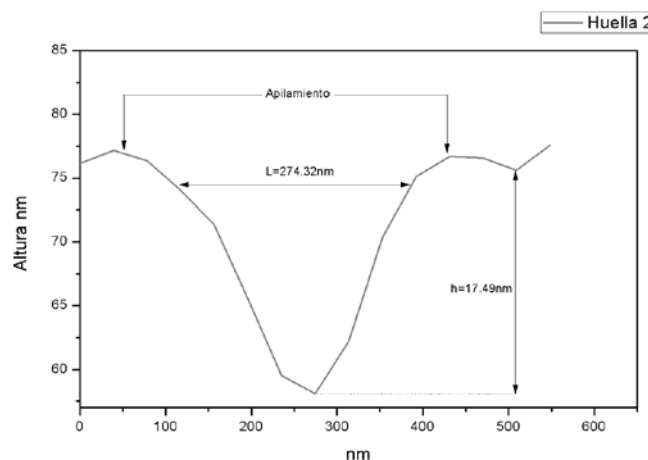


Figure 9. Topographic profile of residual deformation in nanoimprint 2.
Source: Authors.

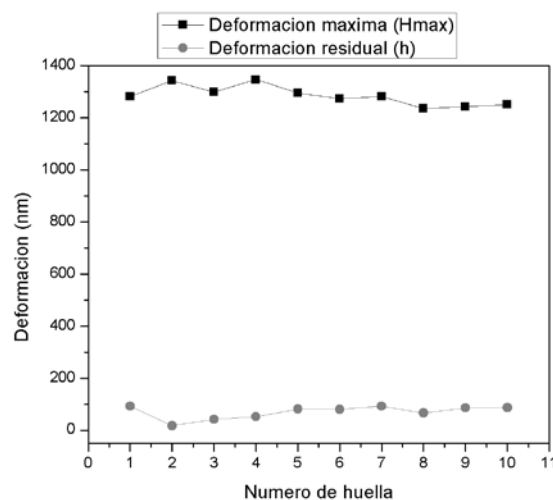


Figure 10. Elastic recovery of GAK fibers after nanoindentation essay.
Source: Authors.

Table 3.

Elastic properties of GAK fibers.

| Mechanical properties | Unit | Value |
|--|------|-------|
| Hardness (H) | MPa | 11,75 |
| Reduced modulus (Er) | MPa | 12,97 |
| Rupture modulus (H/Er) | - | 0,91 |
| Resistance to plastic deformation (H ³ /Er ²) | MPa | 9,68 |
| Elastic recovery | % | 94,54 |

Source: Authors.

Fig. 9 shows the residual deformation on nanoimprint 2 after application of load. Residual depth on all nanoimprints is obtained from the topographic profile, with a value of 17.49nm in nanoimprint 2, which displays a minimum amount of stacking on the sides but no sinking nonetheless. This is due to the characteristics of plasticity and elasticity of the material.

Application of the maximum indentation load (29,91uN) on all nanoimprints resulted in maximum deformations (hmax) of $1290 \pm 60\mu\text{m}$ of the GAK fibers (Table 2). When the load is removed, fibers recover from deformation by an amount ranging between 1160 and 1320 nm (Fig. 10). This demonstrates the elastic recovery of GAK fibers, which averaged 94.54%. Recovery occurs since the atoms in the fiber are not permanently displaced, with the force applied in the indentation being stored as a distortion of fiber interatomic bonds [43].

Table 3 shows the summary of average mechanical properties of GAK fibers resulting from instrumented nanoindentation.

4. Conclusions

Due to their characteristics, fibers of *Angustifolia kunth* guadua are a viable alternative to reinforcement of composite materials. Our sampled fibers had an average cross-sectional area of $13301.806 \mu\text{m}^2$ as measured by SEM. GAK fibers also exhibited ridges and undulations resulting in considerable levels of rugosity, which constitutes a key feature by enabling stronger adhesion to polymer matrices.

Furthermore, AFM characterization demonstrated that the cross section of GAK fibers is formed by microfibrils in the longitudinal direction, providing excellent resistance to tension in that direction. In addition, most of the nanoimprints on the GAK fibers presented minimal stacking, and no sinking. The findings validate the view that GAK fibers are ideally suited to be used as reinforcement in compound materials. Similarly, characterization of the GAK fiber longitudinal section revealed irregularities, which can nonetheless be removed through application of 5% NaOH.

Mechanical characterization at nanometric scale revealed that *Angustifolia kunth* guadua fibers had an average hardness of 11,75MPa, a reduced modulus of 12.97MPa and break of and 0.91 respectively. Characterization also showed that GAK fibers exhibited a plastic deformation resistance of 9.68MPa and an elastic recovery of 94.54%.

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