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Physical and Nanomechanical Characterization of Fique Fiber (*Furcraea gigantea*) by Atomic Force Microscopy-AFM

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Abstract: The use of natural fibers as reinforcement of composite materials (i.e., plastic and wood) has increased in recent decades because of its great availability and the fact that it is an option as renewable resources source. For these reasons, it is important to understand the mechanical behavior of these natural's fibers and optimize the design of composite materials. To this purpose, characterization techniques have been used to determine the properties of different materials. This study presents the results of measurements and calculations of mechanical properties, namely, Hardness (H) and modulus of Elasticity (E) of fique fibers (*Furcraea gigantea*) from Popayán, Colombia as optional alternative to incorporate natural fibers and to collaborate for a sustainability environment. Methodologically, Atomic Force Microscopy (AFM) and nanoindentation tests were used to characterize the topography, roughness and mechanical behavior of fique fibers. The characterization of images in situ confirmed stacking but not subsidence in most of the traces, demonstrating a homogeneous behavior on the fiber surfaces. The stress-displacement curves showed an average E = 841 GPa and H = 15 MPa indicating that the AFM mode nanoindentation technique was able to effectively characterize the mechanical properties of the fique fibers. The fibers have great potential for use as reinforcement in composite materials.

INTRODUCTION

Natural fibers have had a wide variety of applications, usually artisanal and industrial and have been implemented in composite materials because of the perception of their sustainable development and efficiency associated with their mechanical properties[1-5]. In comparison, synthetic fibers have been widely used as reinforcement material in polymeric compounds but they cause environmental problems because of the energy consumption required in their manufacture. Additionally, the growing production of plastic containers has a negative impact on the environment and so, it is important to reduce, reuse and recycle those materials.

Given the above, new composite materials have been developed to reduce the environmental impact[6, 7]. In the study and manufacture of these materials it has been shown that natural fibers have great potential for reinforcement because they improve the behavior of and have advantages over traditional composite materials reinforced with synthetic fibers[8, 9]. The advantages are that natural fibers are an easily available, recoverable and renewable resource[5] and that they are biodegradable, low-density, non-toxic and of limited economic value[3, 10, 11, 12, 13]. This has accelerated the mechanical, chemical and thermal characterization of natural fibers, in order to learn more about their characteristics and behavior and use them properly in composite materials, thereby achieving optimal and efficient designs that facilitate longer service times. Some studies have focused on determining the behavior of natural fibers when incorporated into composite materials such as banana[2, 4], coconut[5, 14], bamboo[15, 16] and sisal[17, 18]. It is possible to use other natural fibers such as fique which is native to Colombia[10] where about 30,000 tons are produced annually[19]. It has been shown that the incorporation of fique fibers in polymeric matrices has a substantial influence on the thermal stability of thermoplastic compounds[19] and on mechanical properties[20] and they do not emit any toxic or harmful components.

To ascertain the small-scale mechanical behavior of natural fibers, Atomic Force Microscopy (AFM)[21] has been implemented through instrumented indentation tests. These tests consist of deforming a material to determine its mechanical properties such as Hardness (H) and modulus of Elasticity (E). This technique uses force spectroscopy to construct a Force curve (F) as a function of Displacement (D) and from this determine the mechanical behavior of an analyzed material.

In this research, we present a mechanical and topographic characterization of fique fibers (*Furcraea gigantea*) superficially treated with sodium hydroxide, with the aim of learning the behavior and viability of it as reinforcement in composite materials.

MATERIALS AND METHODS

In our study, we selected fique fibers (*Furcraea gigantea*) from the city of Popayán, Colombia. Mechanical processes in such a way that fiber diameters were <1 mm and lengths >20 cm extracted the fibers. Subsequently, the fibers were modified superficially using sodium hydroxide (NaOH) at 5% to remove impurities[22]. The fibers were continuously stirred for half an hour at room temperature in the solution and were then washed with distilled water of neutral pH to completely remove the NaOH. Finally, the fibers were subjected to drying, which took 12 h at room temperature and 24 h at 60°C[23].

Through a cutting process the fibers attained a length of 5 mm in order to conduct the AFM analysis using an Asylum Research model MFP-3D-BIO. Ultimately, strategic points of the fiber surface were identified where nanoindentation was performed and the topography recorded. For the nanoindentation test, an Olympus AC160TS indenter was used. Our analysis was that proposed by Oliver and Pharr in 1992[24].

RESULTS AND DISCUSSION

When analyzing the fique fiber using AFM before the indentation measurements, the longitudinal topography of a fiber section (10×10 μm) was obtained (Fig. 1). It is seen that the fique fiber has a corrugated structure.

Figure 2 is the section (5×5 μm) marked in Fig. 1, in which it can be seen that the fique fiber is composed of microfibers with surface irregularities reaching depths between -0.717 and 0.783 μm. These

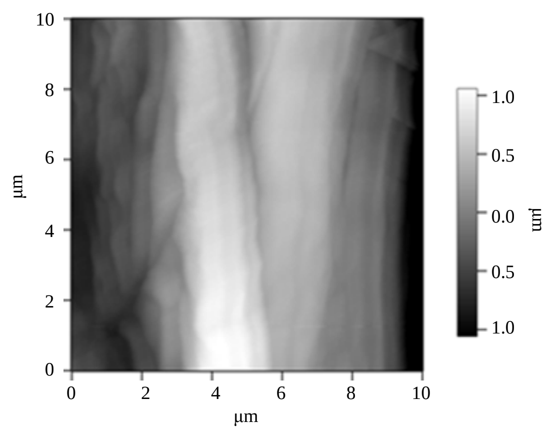

Fig. 1: AFM height image of fique fiber *Furcraea gigantea* before indentation

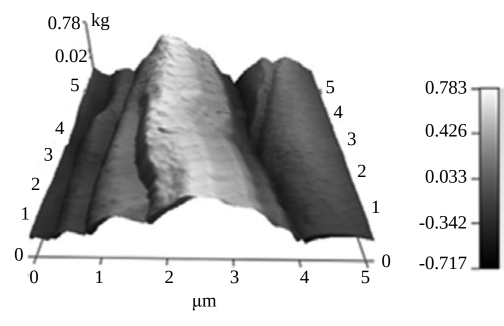

Fig. 2: 3D fique fiber morphology of *Furcraea gigantea*

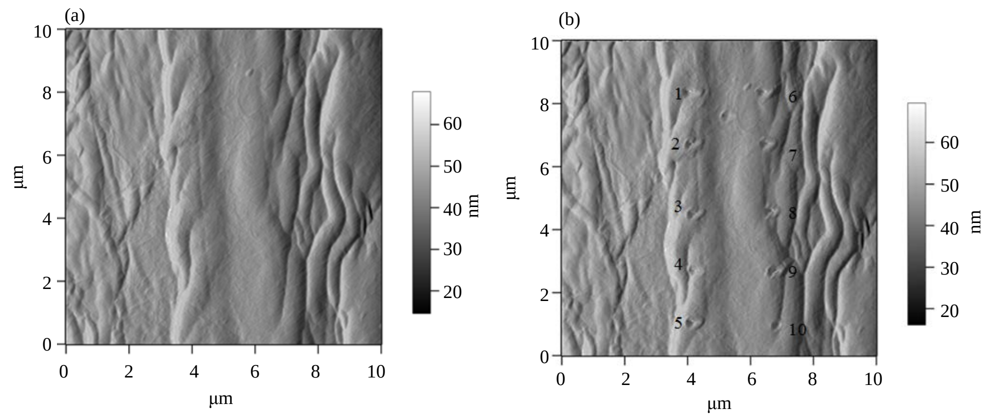

Fig. 3(a, b): AFM amplitude image of longitudinal section of fique fiber, (a) Before nanoindentation and (b) After nanoindentation

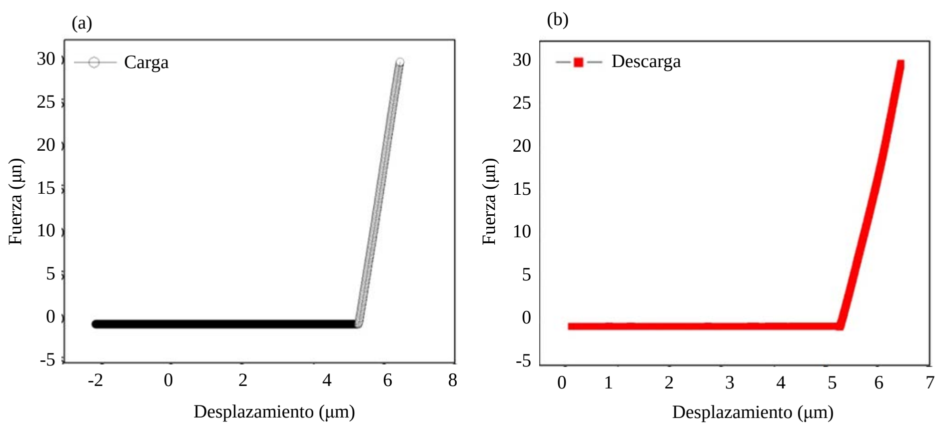

Fig. 4(a, b): Force-displacement, (a) Load curve obtained for fique fiber and (b) Discharge curve obtained for fiber

characteristics yield a roughness of 146.85 nm which is favorable because it allows greater adhesion between polymeric matrices and the fibers.

Figure 3 shows a longitudinal section of the fique fiber before and after nanoindentation. Panel b reveals that the shape and size of the 10 footprints produced by the load were similar.

Figure 4 shows the curve of loading and unloading of footprint 6. Such behavior was evidenced for each of the indentations performed, probably because the fique fibers had similar characteristics throughout the surface. From analysis of the curves and the method proposed by Oliver-Pharr and poisson ratio values are presented for each of the fingerprints in Table 1. It is seen that most of the H values are between 14.81 and 16.60 MPa indicating that the fiber shows homogeneity in the longitudinal section. However, the H variation in footprint 1 relative to the others could be given by the form of load distribution. The average H value of fique fiber is greater than that of bamboo fiber or hemp stalk[21, 25].

E, a parameter obtained from indentation, ranged between 751 and 930 GPa which are large values compared to those of sisal fibers[26] and are produced by the arrangement or crystallinity of cellulose microfibers[27].

Figure 5 shows the residual deformation of footprint 6 after application of the load. The other footprints had deformations similar to those shown in Table 1. The residual depth result of footprint 6 obtained from the topographic profile is 80 nm. This also shows substantial stacking on the sides but no subsidence. In general, upon applying the maximum indentation load, the fique fiber showed deformations (h) of 1.15±0.1 µm. When the load was removed, the fiber recovered between 55 and 120 nm of deformation. The above indicates that *Furcraea gigantea* has an average elastic recovery of 93.25%. This is because the atoms of the fiber are not permanently displaced because the force of the indentation is stored as a distortion of the interatomic bonds of the fiber.

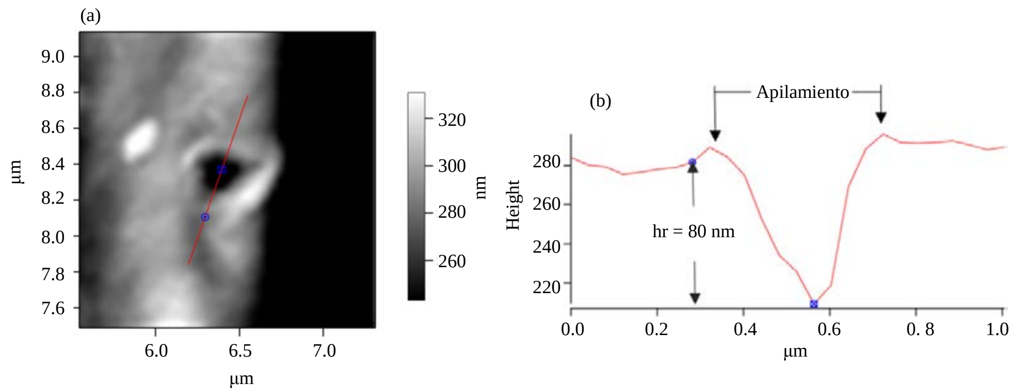

Fig. 5(a, b): Nanoindentation on fique fiber, (a) AFM image of footprint 6 and (b) Topographic profile of footprint 6

Table 1: Physical and nanomechanical properties of fique fiber *Furcraea gigantea*

Footprint	h maximum deformation (nm)	hr Residual deformation (µm)	H Hardness (MPa)	E modulus of elasticity (GPa)
1	1260	120	9.58	751
2	1190	88	15.30	815
3	1160	55	15.68	856
4	1050	79	16.60	852
5	1140	69	15.61	920
6	1140	80	15.62	755
7	1150	62	16.58	808
8	1190	83	15.15	828
9	1210	86	14.81	897
10	1210	64	15.45	930
Average	1170	79	15.04	841

CONCLUSION

The topography of the fique fibers (*Furcraea gigantea*) has ridges and undulations that give them a roughness of 14 nm. This is an important characteristic because it facilitates greater adhesion with polymeric matrices, confirming that the fibers have great potential for use as reinforcement in composite materials. However, the average H and E found for the fique fiber were 15 MPa and 841 GPa, respectively. These values are larger than those reported for other fibers such as sisal, jute and bamboo.

Most of the traces of the fique fiber indicated stacking but not sinking. Thus, the fiber had an average elastic recovery behavior of 93.25%.

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