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Incidence of mercerization treatment in the mechanical properties of bamboo fibre bundles "Guadua Angustifolia Kunth" from colombian origin

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Abstract
In this article, bamboo fibre bundles of "Guadua Angustifolia Kunth" specie were isolated from different locations of the basa zone: upper, middle and lower, through mechanical extraction method. The elastic modulus and the tensile strength were obtained with preliminary tensile tests. Applying the statistical analysis known as ANOVA, it was determined that the mechanical properties are similar in all the extension of the basa zone. From there, fibre bundles were extracted randomly, and a part of the fibre bundles was treated with NaOH (mercerization). Later, tensile tests with different calibration lengths were made for calculate the elastic modulus and the tensile strength of the treated and untreated fibre bundles. Best results belonged to the treated fibre bundles. A good correspondence between the results obtained in this work and the results reported in the literature was concluded.

Keywords: natural fibres; bamboo; alkaline treatment; extraction of fibres; mechanical properties.

Incidencia del tratamiento de mercerización en las propiedades mecánicas de haces de fibras de bambú “Guadua Angustifolia Kunth” de origen colombiano

Resumen
En este artículo se empleó el método de extracción mecánica para aislar haces de fibra de bambú de la especie GAK desde las zonas superior, central e inferior de la parte de la planta llamada basa. Se hicieron ensayos preliminares de tensión, obteniéndose el módulo elástico y la resistencia a la tensión. Se determinó por medio de un análisis estadístico ANOVA que las propiedades mecánicas eran similares en toda la zona de la basa. De ahí, se extrajeron haces de fibras de forma aleatoria y una parte fue tratada con NaOH (mercerización). Posteriormente, se realizaron ensayos de tensión variando la longitud de calibración y, se calculó la resistencia a la tensión y el módulo elástico de los haces de fibras no tratados y tratados. Los mejores resultados pertenecieron a los haces de fibras tratados. Se concluye una buena correspondencia entre los resultados obtenidos y los reportados en la literatura.

Palabras clave: fibras naturales; bambú; tratamiento alcalino; extracción de fibras; propiedades mecánicas.

1. Introduction

Since the start of our civilization, natural fibers have been used in basic elements such as baskets, ropes, fabrics, floors, and roofs [1]. However, as the global energy crisis and ecological risks increase, interest has arisen in the research and development of biodegradable and high-performance products [2]. In recent years, natural fibers such as hemp, linen, jute, kenaf, oil palm, and

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bamboo have been considered in several applications such as automobiles, furniture, packaging, and construction. This is a result of the superior advantages they possess over synthetic fibers in terms of their relative low cost, low weight, less damage to processing equipment, better surface finish in molded parts compounds, and adequate specific mechanical properties.

The aforesaid fosters the consideration of using natural fibers for various applications and under different loading conditions [3].

In industrial applications, synthetic fibers such as fibers of glass, carbon, and kevlar are highly used [4]. However, this type of material generates high environmental pollution and its disposal after the finalization of its lifecycle is difficult. The use of these fibers, such as fiberglass, in the case of being incorporated into automotive spare parts, due to their density, increases the use of petroleum-derived fuels, which contribute to the increase in environmental problems generated by the large amount of CO_2 emissions, which for the year 2013, was around 4,996 metric tons per capita worldwide [5].

A natural fiber is understood as a fibrous material that can be extracted from animals, minerals, and vegetables. The vegetal fibers that we address in this article are composed mainly of cellulose, hemicellulose, and lignin. Further, they possess additional components in less proportion, as shown in Table 1. Cellulose imposes mechanical strength to the fibers (resistance and rigidity), lignin is the hydrophobic part, and hemicellulose, the most amorphous component, improves water absorption and mechanically facilitates elasticity. These vegetable fibers present highly dispersed mechanical properties and are dependent on their chemical composition and the conditions in which they are found, mainly of their degree of humidity, their cultivation processes, and extraction methods [10] However, Table 2 shows that the specific mechanical properties of these natural fibers are similar or even superior to those presented by synthetic fibers such as fiberglass [7,8,12].

The bundles of natural bamboo fibers from the "Guadua Angustifolia Kunth (GAK)" species can be a good alternative for industrial applications as they possess adequate mechanical properties, as well as additional advantages such as low density and low cost. Moreover, they exhibit rapid growth and are classified as a renewable resource [12,13].

The bamboo is divided into six zones: rhizome, bottom, middle, top, stick, and leader, as shown in Fig. 1 [14], and according to literature, the fiber bundles with superior mechanical properties are found in the middle zone. [15].

It is important to point out the importance of choosing a good extraction method, as it is difficult to separate the fibrous bundles from the plants. When lignin and other components are removed, the cellulose microfibrils are also affected, which may increase the fragility of the bundles and decrease their mechanical properties [15]. Therefore, different extraction methods and chemical, physical, and biological surface treatments have been applied, with which significant differences have been obtained in terms of mechanical properties [14,16]. One of the most widely used surface treatments is mercerization or basic hydrolysis. As part of this treatment, the natural fiber is first impregnated with an alkali solution (caustic soda). Then, tension is applied while it is rinsed to eliminate caustic soda, thus improving the luster and facilitating the handling of natural fiber in later processes and the removal of lignin and hemicellulose.

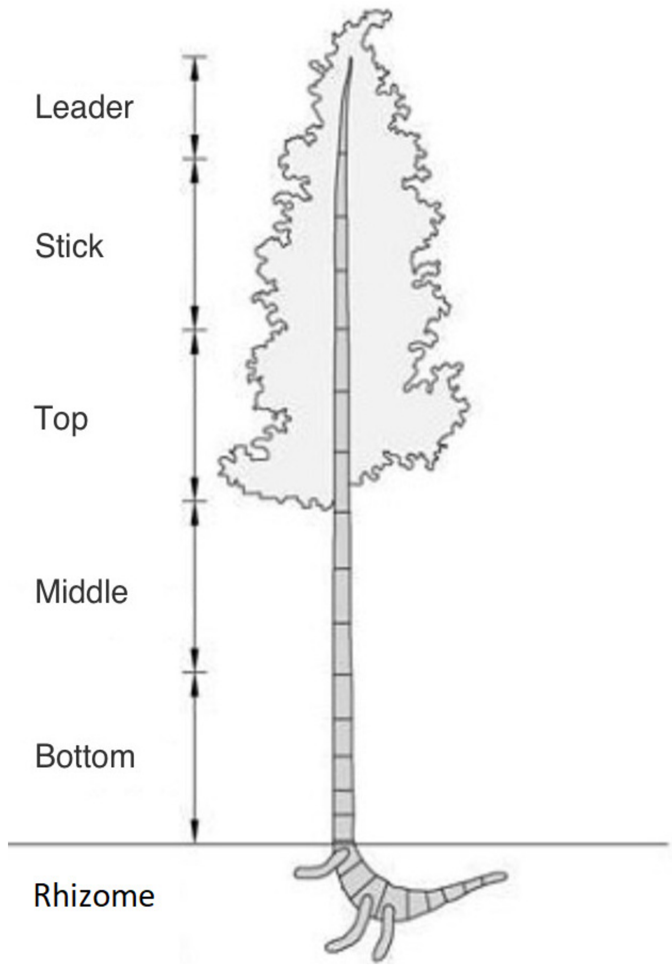

Figure 1. Parts of a bamboo plant.
Source: [14]

Given the above, the objective of this work was to determine the mechanical properties, specifically the elastic modulus and tensile strength at different calibration lengths, of Guadua *Angustifolia Kunth* bamboo fiber bundles obtained from the middle zone, after being isolated by mechanical extraction and having applied a mercerization treatment.

2. Materials and methods

2.1. Materials

Untreated GAK bamboo stalks or culms, with an average age of 4 years, from the municipality of Santa Fe de Antioquia (Colombia) provided by the National University of Colombia at Medellin were used as raw materials. This region is located at an altitude of 625 meters above sea level and its average temperature is 27°C [17]. For the mercerization treatment, analytical grade sodium hydroxide was used at a concentration of 5% w/v.

2.2. Mechanical characterization of GAK

2.2.1. Obtaining GAK fiber bundles through mechanical extraction

The GAK fiber bundles were isolated using an IMOCOM milling machine.

In this first stage, GAK culms were randomly selected from the middle region, which was divided into three zones: upper, middle, and lower. Subsequently, the fiber bundles of the different zones were extracted through the mechanical extraction method.

The GAK culms were cut at an approximate height of 15 cm (internode) and placed on a milling machine. Next, a tungsten beading mill with a working angle of 90°, a speed of 1050 rpm, and a cutting depth of 0.3 mm was used. This assembly is depicted in Fig. 2.

After the initial experimentation, through a statistical analysis from an ANOVA table, it was determined that the mechanical properties in all the middle zones (upper, middle, and lower) were similar. Therefore, in the second stage, the fiber bundles were mechanically removed in random manner before a chemical surface treatment was applied on their surfaces to remove non-cellulosic components.

2.2.2. Chemical surface treatment

The fiber bundles were treated with an alkaline 5% w/v sodium hydroxide solution at a constant temperature of 30°C for a period of 30 min. A ratio of 20 g of fiber bundles per liter of alkaline solution was used. Subsequently, the treated fiber bundles were washed with abundant distilled water until obtaining a neutral pH and were dried in a DIES oven at a temperature of 100.0 ± 0.1° C for 12 h.

Table 1.
Chemical composition of different natural vegetable fibers.

Fiber	Cellulose (%)	Hemicellulose (%)	Lignin (%)	Pectin (%)	Waxes (%)	Ashes (%)
Cotton	82.7	5.7	28.2	5.7	0.6	ND
Jute	64.4	12.0	0.2	11.8	0.5	0.5-2.1
Linen	64.1	16.7	2.0	1.8	1.5	13.1
Ramie	68.6	13.1	0.6	1.9	0.3	ND
Sisal	65.8	12.0	9.9	0.8	0.3	4.2
Bamboo	48.2-73.8	12.5-73.3	10.2-21.4	0.37	ND	2.3
Hemp	55-80.2	12-22.4	2.6-13	0.9-3.0	0.2	0.5-0.8
Kenaf	37-49	18-24	15-21	8.9	0.5	2.4-5.1
Abaca	56-63	15-17	7-9	0.3	0.1	3.2
Henequen	77.6	4-8	13.1	ND	ND	ND
Oil palm	42.7-65	17.1-33.5	13.2-25.3	ND	0.6	1.3-6.0
Areca	35-64.8	29-33.1	13-26	9.2-15.4	0.5-0.7	1.1-2.1
Sugarcane Bagasse	28.3-55	20-36.3	21.2-24	ND	0.9	1-4
Coconut	19.9-36.7	11.9-15.4	32.7-53.3	4.7-7.0	ND	ND
Pineapple	57.5-74.3	80.7	4.4-10.1	1.1	3.3	0.9-4.7

Source: Adapted from [6]

Table 2.
Mechanical properties of different vegetable and synthetic fibers.

	Density (g/cm^3)	Tensile strength (MPa)	Modulus of elasticity (GPa)	Specific tensile strength ($MPa/g/cm^3$)	Specific modulus of elasticity ($GPa/g/cm^3$)	Elongation (%)
Linen	1.4	800-1500	60-80	571.43-1071.43	42.86-57.14	1.2-1.6
Hemp	1.48	550-900	70	371.62-608.11	47.30	1.6
Kenaf	1.45	930	53	641.38	36.55	1.6
Jute	1.46	400-800	10-30	273.97-547.94	6.85-20.55	1.8
Ramie	1.5	500	44	333.33	29.33	2
Coconut	1.25	220	6	176	4.8	15-25
Sisal	1.33	600-700	38	451.13-526.32	28.57	2-3
Cotton	1.55	400	12	258.06	7.74	3-10
Sugarcane Bagasse	1.2	20-290	19.7-27.1	16.67-241.67	16.42-22.58	1.1
Henequen	1.4	430-580	-	307.14-414.29	-	3-4.7
Pineapple	1.5	170-1627	82	113.33-1084.67	54.67	1-3
Banana	1.35	355	33.8	262.96	25.04	5.3
Bamboo	1.1	391-713	18-55	355.45-648.18	16.36-50	-
E-glass	2.5	2000-3500	70	800-1400	28	2.8
Aramid (Std)	1.4	3000-3150	63-67	2142.86-2250	45-47.86	3.3-3.7
Carbon (Std PAN)	1.4	4000	230-240	2857.14	164.29-171.43	1.4-1.8

Source: Adapted from [7-9]

2.2.3. Test Tube preparation for tensile tests

The treated and untreated bundles of extracted GAK fibers were separated manually. Subsequently, the test tubes were prepared for mechanical tests under the ASTM D3822-14 standard, as shown in Fig. 3 [18]. The calibration lengths used in the first and second experimentation stages were 10 mm and 15 mm, respectively. The apparent diameter of each fiber bundle was calculated by the projected profile technique [1] using a LEICA microscope at a magnification of 50×, supported by the IQMATERIALS software.

Afterwards, these samples were conditioned for 12 h before testing at a relative ambient humidity of 69% ± 1% and a temperature of 21.0 ± 0.1° C, as per the ASTM D1776-15 standard [19].

Fig. 4 denotes a flowchart of the extraction process and chemical surface treatment of the fiber bundles.

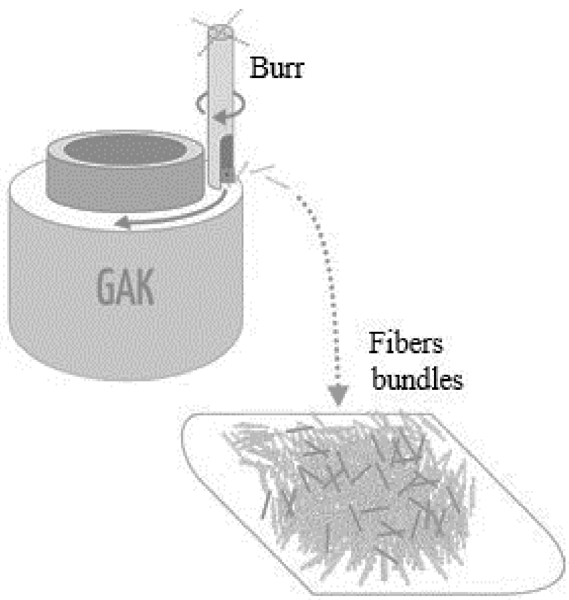

Figure 2. Assembly of the GAK culm in the milling machine.
Source: The Authors.

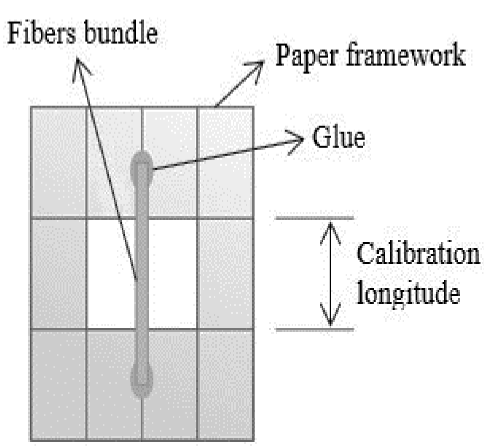

Figure 3. Test tube for tensile tests.
Source: The Authors.

Table 3.
Parameters used in tensile tests

Parameter	Value	Units
Velocity before the test	3	mm/min
Test velocity	1.2	mm/min
Displacement scale	1	mm

Source: The Authors.

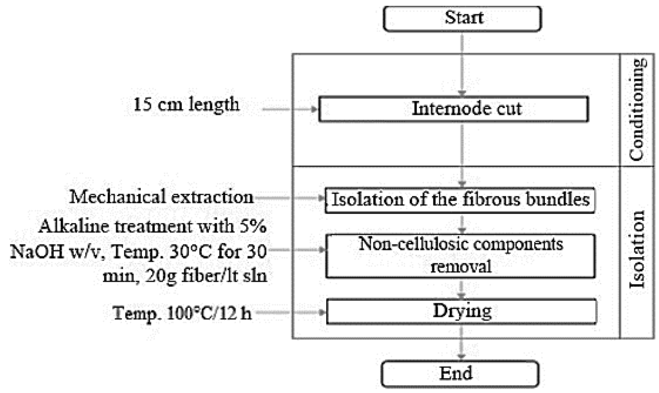

Figure 4. Flow chart of the extraction process and chemical surface treatment of the GAK fiber bundles.
Source: The Authors.

2.2.4. Mechanical characterization of GAK fibers bundles

About 320 tensile tests were performed using a TAXT Plus texturometer from the Agroindustrial Research Group-GRAIN laboratory at the Universidad Pontificia Bolivariana at Medellin.

A 50 Kgf load cell was used and the data were analyzed by the Exponent software. The parameters used in these tensile tests are shown in Table 3.

Based on the ASTM D3822-14 standard and using the calibration lengths mentioned in the first and second stages, some of the mechanical properties were calculated, specifically the tensile strength and the elastic modulus [18].

3. Results and discussion

The results obtained in the different mechanical tests were analyzed using the Statgraphics software [20].

3.1. First stage: mechanical characterization of GAK fibers from the middle area

According to the statistical analysis made from the ANOVA table, it can be determined that the "middle zone" factor, divided into upper, middle, and lower, does not present a statistically significant difference in its mechanical properties (elastic modulus and tensile strength), since the F-ratio is very small (inter-group and intra-group quotient estimates) and the P-Value is greater than 0.05, as may be observed in Tables 4 and 5.

Additionally, from the multiple range tests, it was concluded that there is only one homogeneous group, which means that the fiber bundles from the different middle zones have similar mechanical properties, as shown in Tables 6 and 7. This is confirmed in the Mean charts, where the results overlap, as evidenced in Figs. 5 and 6.

Table 4.
ANOVA Statistical analysis for the elastic modulus

Middle zone modulus of elasticity ANOVA table					
Source	**Sum of Squares**	**DF**	**Mean Squares**	**F-Ratio**	**P-Value**
Between groups	0.64	2	0.32	0.35	0.71
Within groups	8.33	9	0.92		
Total (Corr.)	8.97	11			

Source: The Authors.

Table 5.
ANOVA Statistical analysis for tensile strength.

Middle zone tensile strength ANOVA test					
Source	**Sum of Squares**	**DF**	**Mean Squares**	**F-Ratio**	**P-Value**
Between groups	4672.15	2	2336.07	1.46	0.28
Within groups	14429.60	9	1603.29		
Total (Corr.)	19101.70	11			

Source: The Authors.

Table 6.
Multiple range test for the elastic modulus.

Middle zone modulus of elasticity multiple range tests Method: 95.0 LSD percentage			
Middle zone	**Cases**	**Mean (GPa)**	**Homogeneous Groups**
Middle	4	4.73	X
Upper	4	5.26	X
Lower	4	5.16	X

Source: The Authors.

Table 7.
Multiple Range Test for Tensile Strength.

Middle zone tensile strength multiple range tests Method: 95.0 LSD percentage			
Middle zone	**Cases**	**Mean (GPa)**	**Homogeneous Groups**
Middle	4	163.25	X
Upper	4	210.83	X
Lower	4	194.39	X

Source: The Authors.

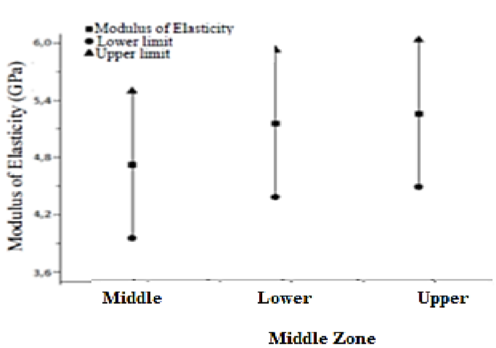

Figure 5. Mean chart for the elastic modulus.
Source: The Authors.

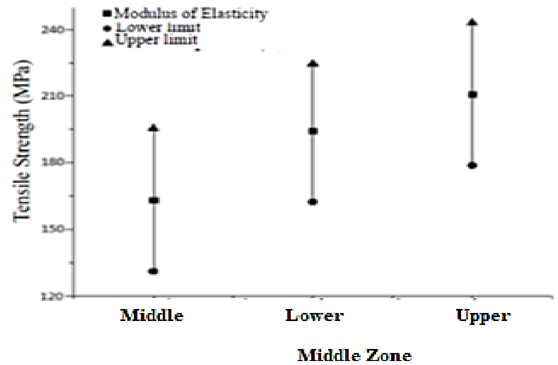

Figure 6. Mean chart for tensile strength.
Source: The Authors.

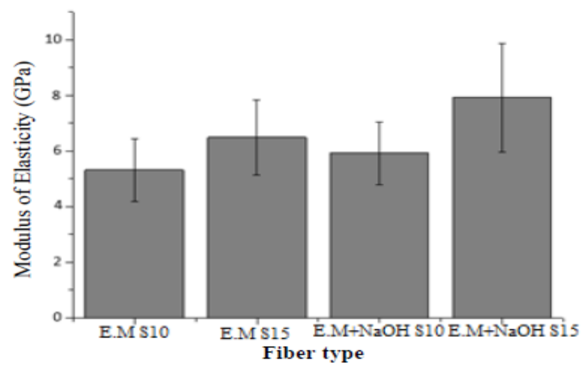

Figure 7. Elastic modulus obtained before and after the chemical surface treatment.
Source: The Authors.

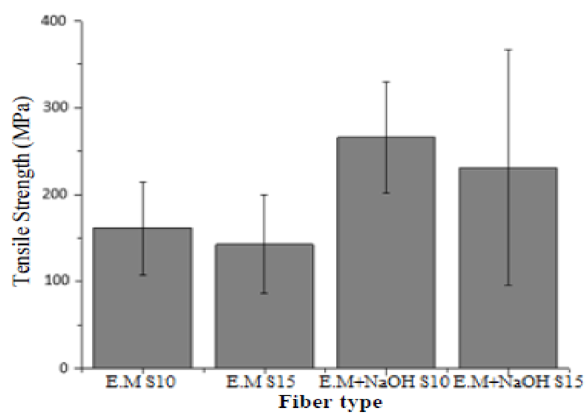

Figure 8. Tensile strength obtained before and after the chemical surface treatment.
Source: The Authors.

3.2. *Second stage: mechanical extraction and chemical surface treatment*

In Figs. 7 and 8, we can observe the high standard deviations from elastic modulus and tensile strength properties, originated by the high heterogeneity in shape and size of the fiber bundles. In addition, these bundles evidence

Table 8.
Natural fibers with inferior mechanical properties compared to what is found in this research.

Natural fiber	Tensile strength (MPa)	Modulus of elasticity (GPa)
Coconut	175	4-6
Oil palm	80-248	0.5-3.2
Piassava	134-143	1.07-4.59
GAK S10 (For this experiment)	161.36-265.98	5.31-5.92
GAK S15 (For this experiment)	142.55-230.97	6.49-7.92

Source: Adapted from [11,27]

high diameter variability, which is a fundamental tensile strength parameter. In addition, the results are very sensitive to variables, such as the processing method, cultivation conditions, type of surface treatment, and percentage of humidity, which determine the final characteristics of this type of natural material [21-24]. Moreover, the best results for both the elastic modulus and the tensile strength are presented after the surface treatment with NaOH for both calibration lengths (span 10 and 15). The aforementioned is due to the fact that the mechanical extraction by itself does not remove non-cellulose components such as lignin and hemicellulose, which have an amorphous structure that significantly affects the mechanical strength of the fiber bundles, thus contributing a greater quantity of microdefects within them and counteracting the crystalline structure of the cellulose component [25]. Given the above, when applying a surface treatment with NaOH to these fiber bundles, the abovementioned components are removed, which improves the mechanical properties of these materials [26]. Furthermore, the best elastic modulus values were obtained at a 15 mm span, since this property improves as the number of bonds aligned in the direction of the applied force increases, which is consistent with the results reported in the literature [24].

However, the results obtained for the tensile strength are quite different. Here, the best results are achieved with 10 mm span since at greater lengths, there is a higher probability of finding defects and/or irregularities. These defects become stress concentrators, and therefore, facilitate the fracture of the fiber bundle [14,24]. Finally, the results obtained in this research study, with respect to other natural fibers, present better mechanical properties, as shown in Table 8.

4. Conclusions

The preliminary mechanical characterization tests and the statistical analysis corroborate the similarity of the mechanical properties existing along the middle zone in the GAK bamboo culm.

Regarding the extraction of GAK fiber bundles, a better yield could be observed when the surfaces are treated with NaOH, since this treatment removes components such as lignin and hemicellulose, thus improving the mechanical properties of the fiber bundles.

In term of calibration lengths, a better tensile strength is obtained when using a span of 10 mm, since there is a smaller number of defects and/or irregularities that can generate fractures in the fiber bundle. Otherwise, for the elastic modulus, the best result is reported when using a 15 mm span because there is a greater number of bonds aligned in the direction of the applied force, which plays an important role in this property.

Although the mechanical properties of untreated GAK fiber bundles are inferior compared with those of treated GAK fiber bundles, they can be used in applications where fiber bundles do not require a high mechanical performance.

The abovementioned results demonstrate the potential of using natural fibers in industrial applications where synthetic fibers have been traditionally implemented.

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