Synthesis and Mechanical Behavior of Composite Material Reinforced with *Guadua* Fiber and with a Polyurethane or Polyester Matrix

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A reactive hot-melt resin (polyurethane) was used to manufacture *Guadua* composites with a certain flexibility, high processing speed, good initial rigidity, and high temperature performance. These composites can support a moderate tensile stress, allow for large strains at low stresses, and have a low density and a working temperature range of -40 °C and 110 °C. During the flexural test, bamboo composites with reactive polyurethane matrix do not break or fail during the test. A polyurethane-based reactive hot-melt resin was characterized by tensile tests, Shore hardness tests, differential scanning calorimetry, and thermogravimetry. Besides, a composite material was made with *Guadua* fiber and polyester matrix, which had a greater strength in the test of tension and flexion, although it had a lower percentage of elongation than the composite material with reactive polyurethane. *Guadua* fiber can increase the strength by 266% of polyurethane matrix and 228% of polyester matrix.

Keywords: Hot melt adhesive; Characterization; Composite material; Guadua fiber

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INTRODUCTION

Polymer composite material (PMC) consists of thermoplastic or thermosetting polymer as a matrix with one or more reinforcements, such as carbon, Kevlar, glass, steel, or natural fibers. These composites are used in various industrial applications due to their high specific strength and stiffness, light weight, corrosion resistance, reparability, fatigue resistance, and low cost. PMC can produce good components, as they can be processed conveniently. The mechanical and chemical characteristics of the fibers, matrix, and interface influence in the mechanical properties, besides the way in which the composite deforms and fractures (Andrew *et al.* 2019: Aruchamy *et al.* 2020). The most advanced polymer-matrix structural composites are those that involve continuous fibers, such as carbon fibers, which are attractive for their combination of low density, high strength, and high modulus of elasticity (Marulanda *et al.* 2020). Glass, Kevlar, and carbon fiber reinforced composites do have excellent mechanical properties but are non-renewable, non-ecofriendly, and can cause human health issues. The natural fiber reinforced composites were developed because of their ability to reduce or replace synthetic fibers in many engineering applications (Sharma *et al.* 2020; Lu *et al*. 2020).

The development of biocomposite materials has attracted considerable interest due to environmental problems and depletion of fossil resources. The combination of natural fiber with polymer matrices (both renewable and non-renewable resources) is called a

biocomposite. These composites are very important due to their abundance, light weight, contribution towards reduction of harmful solid-waste deposition and green-house gases emission; in addition, these materials can be recycled and used for different purpose with enhanced properties and even at low price (Tokala and Mulpur 2019; Sormunen and Kärki 2019). Natural fiber-reinforced polymer composite has a huge affinity to interchange the composite made up of synthetic fiber. The mechanical behavior of this biocomposites is influenced by several parameters such as fiber volume fraction, fiber length, fiber-matrix adhesion, fiber orientation, and stress transfer at the interface. Therefore, to improve the mechanical behavior of composite materials, the properties of matrix and fibers must be improved first (Vaghasia and Rachchh 2018; Andrew *et al.* 2019; Kalali *et al.* 2019; Sair *et al.* 2019; Sormunen and Kärki 2019; Tokala and Mulpur 2019; Aruchamy *et al.* 2020; Lokesh *et al.* 2020; Lu *et al.* 2020; Sharma *et al.* 2020) Several composites are utilized nowadays that were developed via innovation together with efficiency at the optimum price. The natural fiber composites have begun to be used in secondary structural applications in the automotive industry, including door panels and package trays, because of their low material and production costs, acceptable mechanical properties, biodegradability, and lower weights (Barari *et al.* 2016).

Guadua is a woody bamboo that belongs to the grass family. Colombia has one of the species that has the best physical-mechanical properties in the world and extraordinary durability: *Guadua* angustifolia. Among commonly used bio-based resources, *Guadua* is a very attractive source for manufacturing high-performance composite materials. Besides, mechanical properties of *Guadua* fibers are comparable to those of glass fibers or hard woods, but *Guadua* fiber are more cost effective than hard woods, due to the much shorter growth cycle. Therefore, *Guadua* fibers are considered promising candidates for the reinforcement of composite materials due to its low-density, high stiffness and high strength, which makes them abundantly available (Lokesh *et al.* 2020; Chin *et al.* 2020). Bamboo grows as a system of roots (technically, rhizomes) that produce culms. Their fast rate of growth can reach full height (up to 20 m) in as little as 3 months. For some species, this is a rate of growth of 1 m a day (Kuehl and Yiping 2012). However, culms will require a period of maturation before they have optimal strength for structural applications. This period ranges from 3 to 6 years. In a similar manner to other materials, in particular timber, the strength of bamboo correlates well with its density. Density values correlate well with compressive, bending, and tensile strength; this is likely to be because denser samples have a higher content of cellulose (Trujillo and Lopez 2016; Gou *et al.* 2019).

The *Guadua* fiber cannot be obtained directly from the *Guadua* culm, and several treatments are needed before the fiber can be obtained. The *Guadua* fiber preparation methods include retting, steam, alkali treatment, degumming, grinding, and crushing. The quality and strength of the fibers is directly affected by the extraction method used. Thermoplastic composites reinforced with chemically treated *Guadua* fiber offer better mechanical and physical properties. For this reason, *Guadua* was treated with 2% NaOH by weight (Lokesh *et al.* 2020). The alkali treatment modifies the fiber surface, which helps to remove the lignin and wax from outer surface resulting in better adhesion between fiber and polymeric matrix (Vaghasia and Rachchh 2018; Chin *et al.* 2020).

The present study focused on the use of *Guadua* fiber and hot melt polyurethane (PUR) as a composite material matrix. The performance of this type of matrix depends on its rapid crystallization during cooling, which favors quick solidification and welding. The interface compatibility between the *Guadua* fiber and the matrix is an important issue in the development of this composite materials since their mechanical properties depend on

the interactions between the reinforcement and the polymer chains. The resin behavior differs with a change of atmosphere because the environmental humidity affects the resin-crosslinking reactions. The glass transition temperature (T_g) depends on the heating and cooling rates used in the DSC run, molecular weight, plasticizer content, sample size, cross-linking, crystallinity, and degree of cure. The hot-melt resin contains ethylene-co-vinyl acetate with wax. The wax is included because its addition increases the crystallization kinetics of the system, which was observed between 50 and 65 °C. The wax is usually seen as a component that can reduce the cost and control the viscosity during processing. To a large extent, the final properties of matrix depend on the degree of crystallization achieved by hot melt polyurethane (Canales *et al.* 2016; Borgaonkar *et al.* 2018; Marulanda *et al.* 2019). The aligned microstructure of *Guadua* fibers is expected to improve the mechanical properties of unidirectional polymer composite materials. The structural and morphological features of the composite materials were investigated by optical microscopy; thermal properties were characterized by thermogravimetric analysis (TGA) and mechanical properties were evaluated by tensile and flexural tests.

EXPERIMENTAL

Guadua was harvested between three and four years, and the central area of the *Guadua* was taken. The *Guadua* culms without nodes were cut from 300 mm x 20 mm x 10 mm and washed with distilled water for 10 min, then chemically treated with 2% wt/v sodium hydroxide (NaOH) for 4 h at room temperature. After reaching the required immersed duration, the *Guadua* were mechanically defibrated using a mill roller machine to obtain the fiber, the chemical and mechanical treatment was repeated four times.

The *Guadua* fibers were washed with distilled water to remove impurities, and a neutral pH was obtained; it was finished with drying at 103 °C for 24 h. Figure 1 shows the *Guadua* fibers and the test pieces for the tension test. Figure 1a shows *Guadua* fibers, which had lengths around 250 mm. In Fig. 1b, the specimens of composite material of *Guadua* and polyurethane as matrix have many visible pores, while the specimens of composite material with polyester resin have very little porosity. All the *Guadua* fibers were not aligned during the manufacturing process since some fibers were misaligned during hand-laying. The manufacturing process of the composite material with a polyurethane matrix is governed by pressure, temperature, and process time. When more time and / or temperature than required is added, the matrix can generate porosity due to the acceleration of curing. In addition, the pressure allows the entry of the polymer into the fiber arrangement and the reduction of pores. Therefore, it is necessary to improve the melting times and the manufacturing pressure of this composite material. The samples were made with percentages of 6, 8, and 10% of *Guadua* fibers, where the best results were obtained with 10% of fiber.

Thermogravimetric differential scanning calorimetry (DSC) and thermogravimetry (TGA) analyses was carried out on an SDT Q600 thermobalance, which allows the simultaneous measurement of DSC and TGA. The thermograms were obtained between 21 °C and 200 °C at a heating rate of 5 °C/min in both air atmosphere. Approximately 7 to 8 mg of each adhesive sample was placed inside an alumina crucible for DSC-TGA thermal analysis.

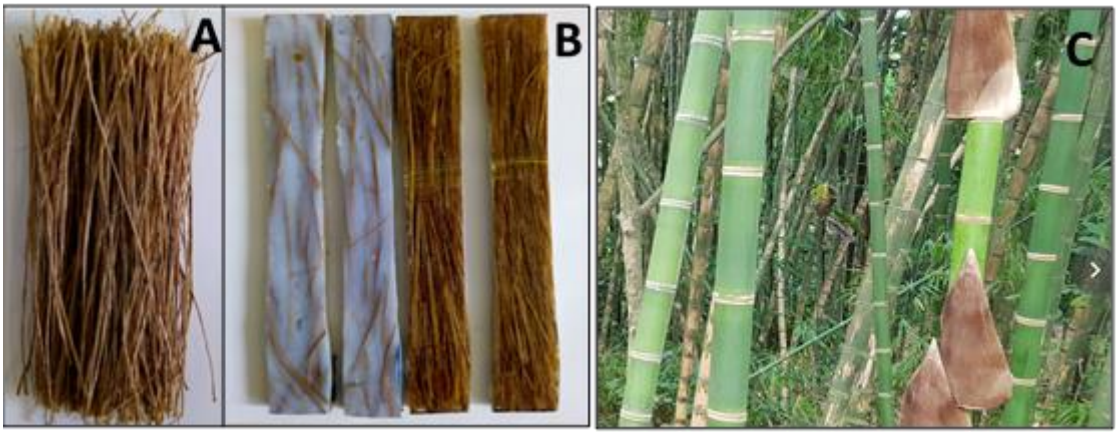


Fig. 1. *Guadua* fibers, specimens to tensile test and *Guadua* plant

For the matrix, 330-mL Jowatherm-Reaktant 642.00 cartridges were used. These cartridges are polyurethane-based reactive hot melt. Because its application properties are a function of temperature, it is necessary to heat and maintain the matrix at a constant temperature to guarantee its viscous liquid state. The growth of hardness hot melt reactive remains high until approximately 48 h of cure, after which the rate of hardening decreases and begins to be much slower. From 188 h, the hardness is stabilized, which was apparent from the observation that at 336 h of curing, the Shore-A hardness was 92. The bond strength increases gradually with time and depends on moisture. In addition, for this type of resin (reactive hot melt) the curing time is an important factor that influences its mechanical properties. The resin hardness did not change when fibers were added (Marulanda *et al.* 2019). To manufacture the composite material test specimens, the matrix was melted at 130 to 140 °C in a muffle furnace with a sensitivity of +/- 3 °C and dimensions of 40 cm × 30 cm × 30 cm. Once the matrix had melted, it was emptied into a Teflon mold at 130 °C, ensuring that the matrix was evenly distributed throughout the mold. The reinforcement (*Guadua* fiber) was introduced into the mold with continued supply of the matrix, once more ensuring that it was evenly distributed. Finally, the mold was closed, and pressure was applied to reduce the porosity and improve the uniformity of the composite material. A sample containing only hot melt polyurethane matrix was also produced and used as the control for comparison. To manufacture the polyester composite material test specimens, the polyester resin was placed in a 200 mL glass beaker and weighed on an electronic balance. Next, catalyst was added with a disposable syringe at the ratio of 100 mL of polyester resin 856 to 1 mL of catalyst (methyl ethyl ketone peroxide-MEKP) and stirred for about two minutes. A thin layer of catalyzed polyester resin was poured into the Teflon mold coated with petroleum jelly as mould release agent. Fiber reinforcement was placed by hand in a mold, and fiber was pressed against the mold with a roller to remove porosity. Then another layer of catalyzed polyester resin was placed into the mold. Finally, the mold was closed, and pressure was applied to reduce the porosity and improve the uniformity of the composite material. The test samples were cured for 24 h, after which they were removed from the molds. Sample containing only a polyester matrix was also produced and used as the control for comparison. After making the composite material, six specimens were cut (250 mm × 25 mm × 2.5 mm) according to stress-test measurements from ASTM D 3039 (2017). Six specimens were cut for each material (110 mm × 13 mm × 3.5 mm) according to flexural tests using standard ASTM D 7264 (2015). The tensile and flexure tests were carried out using a universal testing

machine single-column force tester with capacities of 1,500 lbF (6.7 kN) (MARK-10 ESM1500, Mark-10 Corporation, NY, USA) a constant crosshead speed of 1 mm/min at room temperature (23 °C).

RESULTS AND DISCUSSION

DSC-TGA was performed to observe the matrix behavior in air atmosphere as a function of temperature before and after making the composite material, as can be seen in Fig. 2. The resin behavior differs because the curing time affects the resin-crosslinking reactions. In Fig. 2A it is apparent that the variation of heat capacity led to a small softening of the hot-melt polyurethane at ~38 °C, because the polyolefins in the resin are remarkably flexible. The glass-transition temperature (T_g) of the polyurethane resin before curing time was observed at ~55 °C. This behavior is not observed in Fig. 2B. After curing, the polyurethane only exhibited a small melting of wax and polyolefins, as shown in Fig. 2B. The addition of waxes and polyolefins influenced the glass-transition temperature. The T_g depends on the heating and cooling rates used in the DSC run, molecular weight, plasticizer content, sample size, cross-linking, crystallinity, and degree of cure. Figure 2A shows that the melting of most resin compounds occurred after ~75 °C. The adhesive at 75 °C is not liquid but exists in a gummy state; the Fig. 2B only shows a little fusion of compounds such as waxes and polyolefins. The resin mass gain is different before and after curing. In Fig. 2A, the slope of the mass gain by volatilization and reaction begins at 75 °C to 95 °C, then tries to stabilize to 130 °C, continues with a slope change at 135 °C due to the fluidization of the polyurethane, and 170 °C has a slope change because of the resin degradation. In Fig. 2B, the polyurethane resin has a mass gain and a little mass change at 75 °C, due to melting of some resin compounds.

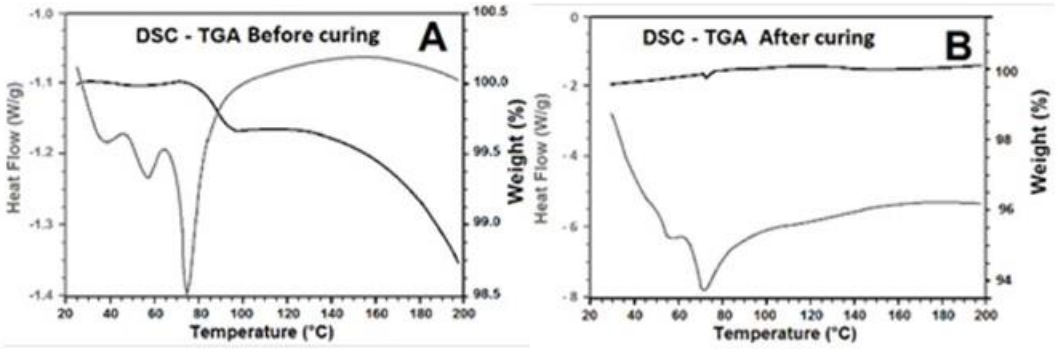


Fig. 2. DSC and TGA analysis for polyurethane resin in air atmospheres before and after of matrix cure

The polyester resin composite material is fragile with respect to the Jowatherm-Reaktant hot melt, which is elastic in nature, but when solidified and after 92 h of curing it becomes a more rigid material. During the stress test, the composite material with polyurethane matrix had an elongation of the matrix before the fibers fracture, contrary to what happens with the fibers in the polyester resin compound where the matrix and the fibers fail at the same time.

The tensile strength of composite materials depends on the properties of their constituents, such as bonding between fiber and matrix, the volumetric fraction, fiber

length, fiber content, and the spatial distribution of the fibers; both the tenacity of the fibers and the interaction between the fibers and the matrix are crucial to the mechanisms involved in breaking stress and energy dissipation (Fernandez *et al.* 2017; Borgaonkar *et al.* 2018).

In Fig. 3, linear/elastic behaviors were observed between the deformation and the stress applied to the reactive polyurethane matrix specimen without reinforcement. This matrix reached deformations of 600%, with applied pressure of 18.5 MPa; the initial measurements of the samples (L_o) were 80 mm and the final measurements before the stress test break (L_f) were around 486 mm. Observing the area under the stress-strain curve; it can be stated that the material had a great toughness, and its data showed a low dispersion (5%). During the tensile test, a long neck phenomenon was observed in the reactive polyurethane specimen. The neck-forming area was propagated along the direction of the tensile test until almost the entire sample had been homogeneously deformed. In some tests, failure was not achieved and after unloading the specimens could not regain their initial shape and maintain a permanent deformation of 150%. Due to the capacity of the tensile test machine, it reached the maximum displacement, and the specimen did not break. The authors continued to reduce the length of the specimens. In this test, no softening of the polyurethane was observed, since during the beginning of the test it showed a linear elastic region and continued with a progressive hardening of the polyurethane until rupture (Fig. 3). This material exhibited viscoelastic behavior in the stress-strain test because it exhibited both viscous and elastic characteristics. The viscoelastic behavior was observed when the deformation of materials caused by stress was applied, linearly with time due to stress.

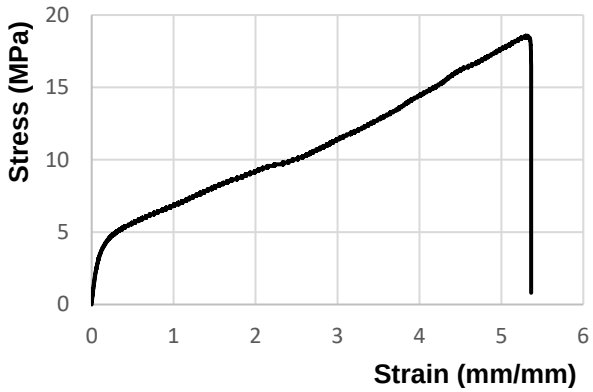


Fig. 3. Results of tensile tests on polyurethane matrix composites

Figure 4 shows the composite material with *Guadua* reinforcement and reactive polyurethane matrix. This material did not have a linear behavior between the matrix and the reinforcement, since at the beginning of the test there were large deformations with small applications of stress. This behavior can be attributed to the arrangement of the matrix and the reinforcement, after which a linear / elastic behavior was observed between the deformation and the stress, leading to an increase with a different slope in the displacement. The composite material of *Guadua* and polyurethane matrix withstood almost 48 MPa (maximum strength), and its elongation displacement was 11.84 mm. This material was more ductile than the composite material with a polyester matrix, since it allowed greater deformation before failing, although it had a high porosity, and it was necessary to continue working to reduce it and improve its mechanical resistance. The

composite material with polyurethane matrix had type-LGM failure mode, according to ASTM D3039 (2017), with high elastic material deformation, but with few yield points before the fracture mechanism.

Figure 4 shows the composite material with *Guadua* reinforcement and a polyester matrix. This material had a higher tensile strength supporting 79.7 MPa (maximum strength) and its elastic limit was 64.1 MPa and elastic modulus was measured between 20 and 80 % of the elastic limit. The maximum elongation displacement was 7.18 mm. This composite material was more fragile and presented greater homogeneity and compaction of the matrix and the *Guadua* fiber reinforcement since the porosity was very low. *Guadua* fiber reinforcements were the elements that improved load transfer, offering the structure greater strength and rigidity. Figure 4b shows the brittle nature that resulted in the catastrophic failure of the material composite, because no yield points existed before the fracture mechanism. The composite material with polyester matrix had type-AGM(2) failure mode, according to ASTM D3039 (2017), which is characterized by a fragile behavior during fracture with low elastic material deformation. The polyester matrix could resist more than 45 MPa, and the composite material with *Guadua* resists almost 80 MPa, increasing a maximum of 177 % the capacity to support the load. The average results for each of the results are detailed in Table 1.

Table 1. Tension and Flexion Tests MOR Results

Test type	Material	Samples	MOR (MPa)	COV (%)	Peak MOR (MPA)
Tensile test	polyurethane	4	34.3	25.5	47.3
	polyester	5	71.26	8.8	79.7
Flexural test	*Guadua* polyurethane	4	22.7	26.4	31.8
	Guadua polyester	7	83.9	14.9	98.7

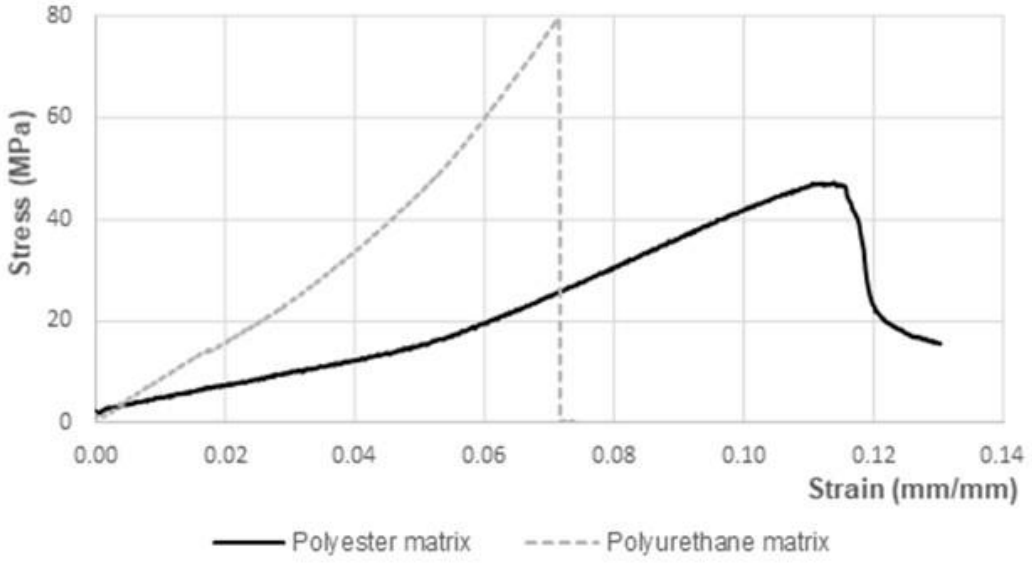


Fig. 4. Results of tensile tests on composites reinforced with *Guadua*

The polyurethane matrix was able to resist 18 MPa, and the composite material with *Guadua* resisted almost 48 MPa, increasing the strength by 266 %. The area under the curve in the stress / deformation diagram of the composite material with *Guadua* fiber with polyurethane was larger than the composite material of *Guadua* with polyester; therefore, it could be said that the composite material with polyurethane will have better impact strength, since composite materials with larger areas under the stress/strain curve are more efficient in absorbing energy. This is a quick and practical way to have a guide to the impact strength of composite materials.

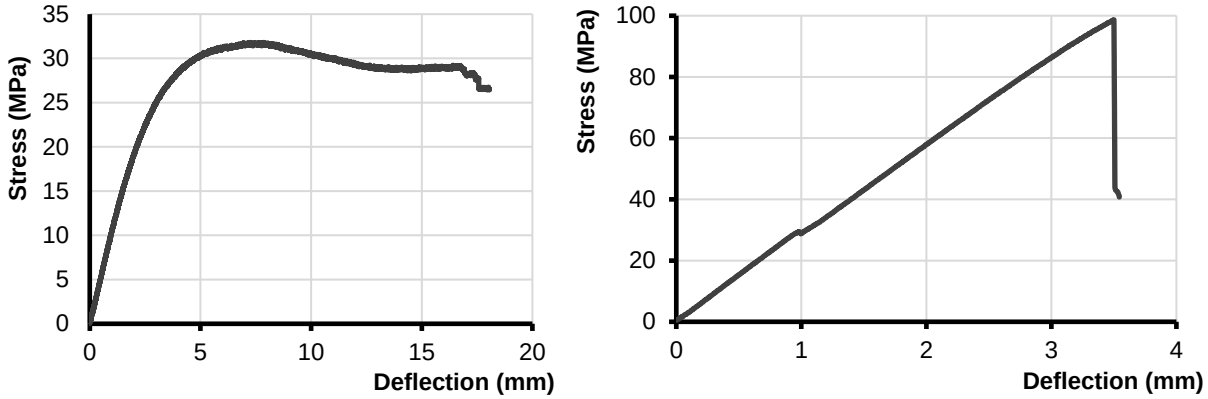
Other studies of natural fibers report different behaviors of natural fibers. Aruchamy *et al.* (2020) reported: The tensile strength of woven cotton composite materials and epoxy matrix reached a maximum of 72.9 MPa at 45% by weight of cotton, so that the strength did not increase after increasing the fiber content more beyond the critical fiber load content. In addition, the composite materials were prepared with mixtures of cotton fibers and bamboo fibers, obtaining a maximum tensile stress of 82.1 MPa with 45% by weight of the fiber mixture (Andrew *et al*. 2019). Venkata Naga and Sarat Babu reported that composite materials with sugarcane fibers, coconut fibers and the hybrid composite based on cashew shell resin exhibited a tensile strength of 11.5, 2.6, and 5.9 MPa, respectively (Tokala *et al*. 2019). Bhavik Vaghasia and Nikunj Rachchh reported that composite materials with mixtures of fiberglass 19% by weight and bamboo fiber from 3 to 15% by weight reached a maximum stress of 108 MPa when they had 9% bamboo fiber (Vagashia *et al.* 2018). Lokesh *et al.* (2020) made a composite material with epoxy-epoxy resin and short bamboo fiber, which reached tensile stresses between 6.74 and 18.07 MPa.

The amount of fiber content that could be added without difficulty in the composite material manufacturing process was 12 % by weight. For higher fiber contents it is difficult to maintain the size and thickness of the plates to make the specimens for the bending and tension test. In addition, the content of air bubbles between the bamboo fiber and the matrix is increased. This leads to discontinuity and reduces the homogeneity of the material, which results in a reduction of the tensile strength with a higher fiber content. This may be due to a lesser amount of resin available to impregnate the fibers, leading to less charge transfer between the fiber and the matrix (Vaghasia *et al.* 2018; Lokesh *et al.* 2020).

Flexural tests were conducted to determine the mechanical properties of the *Guadua* fiber composite and matrix. Figure 5 shows graphs of flexural tests by procedure A for composite materials of *Guadua* fiber with polyester matrix and *Guadua* fiber with reactive polyurethane matrix. The test load was applied at the midpoint of three support points. All the *Guadua* fiber and polyester matrix specimens showed fractures in the middle during the flexural test. *Guadua* composite materials with reactive polyurethane matrix did not break or fail during testing, showing good adhesion between matrix and fiber reinforcements, as bending tests can be used to get a qualitative idea of the strength of the fiber/composite matrix interface. Polyurethane matrix composites underwent deformation during the test, but they returned to their initial state and position after some time of removing the test load. Furthermore, these reactive adhesive composites did not present linear curves, which confirms the ductile behavior at low stresses of the material.

The maximum flexural stress of these materials was 80 MPa for the composite material of *Guadua* fiber and epoxy matrix, 48 MPa for the composite material with polyurethane matrix, and 36 MPa for the reactive adhesive matrix. This makes it possible for composite materials with reactive polyurethane to be employed to manufacture different parts such as suspension springs and bumpers, among others. The benefits offered by hot melt polyurethane over conventional hot melts lie in the cross-linked structure,

which implies excellent strength and adhesion to reinforcement. In addition, hot melt polyurethane provides an advantage compared to polyesters due to its rapid processing, as they do not require drying time.



a. Polyurethane matrix **b.** Polyester matrix

Fig. 5. Results of flexural tests on such composites

CONCLUSIONS

1. *Guadua* fiber is a good alternative as a reinforcement for making polymeric matrix composite materials. *Guadua* fiber can increase the strength by 266% of polyurethane matrix and 177% of polyester matrix. This means a considerable reduction in costs compared to the glass fibers or carbon fibers that are used today; they also have lower density and are friendly to the environment.
2. Tensile specimens made of polyester resin composite had a better performance than specimens with hot melt. This was attributed to high porosity, which reduces the mechanical properties. However, in the bending test the specimens of composite material with matrix of polyurethane did not fail and once the effort was removed, they began to recover the initial position. After a few minutes (5) they returned almost to their initial position.

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