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Article

Caracterización física y mecánica de compuestos de *Guazuma crinita* Mart. a base de polipropileno virgen

Physical and mechanical characterization of *Guazuma crinita* Mart. composites based on virgin polypropylene

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Resumen

Se elaboraron materiales compuestos plástico madera con base en partículas de madera de la especie *Guazuma crinita* (bolaina blanca), proveniente del raleo de 4, 5 y 6 años, con polipropileno virgen (PP), además se utilizó como agente acoplante anhídrido maleíco de polipropileno (MAPP). La producción de probetas se hizo por el método de extrusión, compresión térmica y corte por láser. Las partículas de bolaina blanca se tamizaron con tamaños de malla ASTM: -40/+60, -60/+80 y -80/+100. Las proporciones de mezcla polipropileno/bolaina fueron: 70/30, 80/20 y 90/10. Todas las formulaciones incluyeron 2 % de MAPP como agente acoplante. Se evaluaron las propiedades físicas de contenido de humedad, densidad, absorción e hinchamiento; así como, las propiedades mecánicas de flexión estática, tensión y resistencia al impacto. Para la discusión de resultados se realizó una caracterización anatómica de la fibra (longitud, diámetro, espesor de pared, diámetro del lumen y coeficiente de esbeltez), además de un análisis químico de los componentes de la misma (extractivos, holocelulosa, lignina y cenizas). Los resultados permiten apreciar influencia de la variable proporción de mezcla sobre las principales propiedades físicas, así como de la variable tamaño de partícula respecto a la tensión. La edad de la madera no representó una fuente de variabilidad significativa.

Palabras claves: *Guazuma crinita* Mart., materiales compuestos plástico madera, módulo de elasticidad, polipropileno, propiedades físicas, propiedades mecánicas.

Abstract

Wood plastic composite materials based on *Guazuma crinita* wood particles (White *Bolaina*) from forestry thinning of 4, 5 and 6 years old and virgin polypropylene (PP) were prepared, using polypropylene maleic anhydride (MAPP) as a coupling agent. Specimens were made by extrusion, thermal compression and laser cutting. White bolaine particles were sieved with ASTM mesh sizes: -40/+60, -60/+80 and -80/+100. Proportions of polypropylene/*Bolaina* mixture were: 70/30, 80/20 and 90/10. All formulations included 2 % MAPP as a coupling agent. Physical properties as moisture content, density, absorption and swelling were assessed, as well as the mechanical properties of static bending, tension and impact resistance. Additionally, for discussing the results an anatomical characterization of the White *Bolaina* wood fiber (length, diameter, wall thickness, lumen diameter and slenderness coefficient) and a chemical analysis of its components (extractives, holocellulose, lignin and ash) was carried out. Results allow to appreciate a direct relationship between the variable mixing ratio and the mainly physical properties, as well as between the variable particle size with respect to tension. Wood age did not represent a significant source of variability.

Key words: *Guazuma crinita* Mart., wood plastic composites, modulus of elasticity, polypropylene, physical properties, mechanical properties.

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Introduction

Many lignocellulosic fibers have been proposed as reinforcement in composite materials throughout human history. Of special interest is the use of fast-growing wood fibers, as it is a natural, low-cost, renewable and highly available resource for its industrial purposes; as well as for his contributions in the physical and mechanical characteristics in related final products (Satyanarayana *et al.,* 2009).

In addition, society's demand for the use of products originated by waste from industrial and single-use processes, such as sawdust and plastic, respectively, must be considered as well. All this has allowed sectors of the construction and automobile industry to develop a variety of products, including railings, window frames, door panels, moldings, floors, seat upholstery, etcetera (Clemons, 2002).

In the composite materials the optimum particle size is sought, as well as its adequate proportion, according to its final use. Composite materials reinforced with vegetable fibers have been positioning themselves strongly in the market, favored by their low cost, high durability and the use of waste in their elaboration (Wolcott and Englund, 1999; Klyosov, 2007).

There is a poor research background related to plastic and wood composites in Peru (Lázaro *et al*., 2016a; Lázaro *et al*., 2016b; Gonzáles *et al*., 2018), despite the efforts made by universities, organizations of the State and private entities for its promotion; there is only one national company that produces plastic and wood and markets them. Its main market is the construction companies that use them for multi-family projects, due to their low cost of installation and maintenance, as well as their long service time, a valuable feature for this purpose.

The definition of the raw material to be used is important, since it must come from a promising species industrially and with existing plantations. Such is the case of the *Guazuma crinita* Mart. (White *Bolaina*), a widespread forest species as an alternative for forest plantations in the Peruvian Amazon. The main product of White *Bolaina* is sawn wood, with which tongue and groove are manufactured for interiors and

exteriors, glued boards, moldings, furniture interior covers and other types of carpentry (Guerra *et al*., 2008).

Based on the above, in the present study the aim was to achieve a characterization of virgin polypropylene-based composite materials, reinforced with White *Bolaina* particles, through the evaluation of physical and mechanical properties.

Materials and Methods

Wood plastic composite materials were made, for which, as a reinforcement material, thinning wood of 4, 5 and 6 years of *Guazuma crinita* (White *Bolaina*) was used from a forest plantation in the province of *Puerto Inca*, department of *Huánuco,* Peru.

As a thermoplastic matrix, a polypropylene homopolymer with a flow rate of 12.5 g / 10 min^{-1} (2.16 kg / 230 $°C^{-1}$) was used. As a coupling agent, maleic polypropylene anhydride (MAPP) was used - which already has several efficiency studies (Correa *et al.,* 2007) - with a melting temperature of 167 °C; in 2 % concentration for all formulations.

The wood was allowed to peel, bark, grind and sift until obtaining particles of three mesh sizes (ASTM) -40/+60, -60/+80 and -80/+100. The sieved particles were dried in an oven at 103 °C ± 2 °C for 48 h to obtain a moisture content <5 %. The different proposed formulations were prepared (Table 1). The extrusion of the materials was carried out in a single screw extruder machine, at a temperature between 160-175 °C and 30 rpm, then the extruded material was milled to continue pressing.

Table 1. Formulations with different ages, particle sizes, proportions and treatment.

Age (years)	Mesh sizes ASTM	Particle size (µm)	Composition		Treatment code
			Bolaina **(%)**	**Polypropylene (%)**	
4	+40/-60	400-250	30	70	T-4/40/30
			20	80	T-4/40/20
			10	90	T-4/40/10
	+60/-80	250-177	30	70	T-4/60/30
			20	80	T-4/60/20
			10	90	T-4/60/10
	+80/-100	177-149	30	70	T-4/80/30
			20	80	T-4/80/20
			10	90	T-4/80/10
5	+40/-60	400-250	30	70	T-5/40/30
			20	80	T-5/40/20
			10	90	T-5/40/10
	+60/-80	250-177	30	70	T-5/60/30
			20	80	T-5/60/20
			10	90	T-5/60/10
	+80/-100	177-149	30	70	T-5/80/30
			20	80	T-5/80/20
			10	90	T-5/80/10
6	+40/-60	400-250	30	70	T-6/40/30
			20	80	T-6/40/20
			10	90	T-6/40/10
	+60/-80	250-177	30	70	T-6/60/30
			20	80	T-6/60/20
			10	90	T-6/60/10
	+80/-100	177-149	30	70	T-6/80/30
			20	80	T-6/80/20
			10	90	T-6/80/10

The composite boards were made in a hydraulic press, at a speed of 0.9 cm s^{-1} and a pressure of 40 bar; curing of the material took between 4-5 minutes at a temperature between 177-195 °C. A total of 1 080 specimens were made in an 80 W power laser machine;40 were tested for each treatment, according to the following standards: ASTM D1037-99 for moisture and density (ASTM, 1999), ASTM D570-98 for absorption and swelling (ASTM, 1998), ASTM D638-03 for tension (ASTM, 2003b), ASTM D790-03 for static bending (ASTM, 2003a) and ASTM D5420-04 for impact resistance (ASTM, 2004).

The statistical model of the factorial design used was:

$$X_{ijk} = \mu + E_i + T_j + P_k + (ET)_{ij} + (EP)_{ik} + (TP)_{jk} + (ETP)_{ijk} + \varepsilon$$

Where:

X_{ijk} = Corresponding observation to the i^{th} replication

μ = Mean of all observations of the treatment

E_i = Parameter that measures the effect of the main age variable

T_j = Parameter that measures the effect of the main particle size variable

P_k = Parameter that measures the effect of the main mixture proportion variable

$(ET)_{ij}$ = Effect of the interaction between age and particle size variables

$(EP)_{ik}$ = Effect of the double interaction between the age and mixture proportion variables

$(TP)_{jk}$ = Effect of the double interaction between the particle size and mixture proportion variables

$(ETP)_{ijk}$ = Effect of the triple interaction among the age, particle size and mixture proportion variables

ε = Experimental error

An analysis of variance was applied with the Statistical Analysis System version 9.1 (SAS) program, with a 95 % confidence interval.

The anatomical characterization of the White *Bolaina* fibers was carried out in accordance with the procedure standard for studies of wood anatomy Ibama (1991). The values of length, width and wall thickness of at least 25 fibrous bundles obtained after the defibration process were taken, with a LEICA ICC50 HD camera coupled to a LEICA DM500 microscope with magnifications of 4X, 10X and 40X.

The chemical description of *G. crinita* fibers was carried out following the standards: TAPPI T 264 CM-97 (TAPPI; 1997a) for wood preparation in chemical analysis, TAPPI T 204 CM-97 (TAPPI; 1997b) for extractives, Jayme method -Wise for holocellulose, TAPPI T 222 OM-98 (TAPPI; 1998) for insoluble lignin and TAPPI T 211 OM-93 for ashes (TAPPI, 1993).

Results and Discussion

An evaluation of the physical and mechanical properties of the PP- White *Bolaina* composite material (Table 2) was performed, as well as a characterization (Table 3) and chemistry of the *G. crinita* fiber in its three ages.

Table 2. Average values and standard deviation of the physical and mechanical properties of the 4, 5 and 6 year old PP-White *Bolaina* composite material.

Treatment	Moisture content (%)	Density (g cm-^{3})	Absorption (%)	Swelling (%)	Tension		Static bending		Impact (J)
					Maximum resistance (MPa)	Module of elasticity (Gpa)	Maximum resistance (MPa)	Module of elasticity (Gpa)	
T-4/40/30	2.2 [0.08]	0.90 [0.02]	14.0 [2.27]	5.3 [0.42]	14.0 [0.67]	1.0 [0.10]	30.2 [1.88]	1.3 [0.05]	0.56 [0.06]
T-4/40/20	1.5 [0.13]	0.87 [0.02]	7.7 [1.63]	4.0 [1.63]	16.7 [0.43]	0.9 [0.21]	30.4 [2.04]	1.1 [0.14]	0.62 [0.05]
T-4/40/10	1.0 [0.06]	0.76 [0.06]	17.4 [6.60]	3.2 [1.45]	16.7 [2.12]	0.9 [0.15]	28.8 [2.34]	0.9 [0.08]	0.50 [0.05]
T-4/60/30	2.2 [0.07]	0.91 [0.01]	11.9 [0.83]	4.2 [0.56]	11.6 [1.12]	0.7 [0.08]	26.2 [4.28]	1.2 [0.11]	0.58 [0.07]
T-4/60/20	1.5 [0.04]	0.87 [0.01]	8.2 [1.32]	3.3 [0.98]	12.9 [1.12]	0.6 [0.06]	28.8 [1.71]	1.1 [0.09]	0.48 [0.09]
T-4/60/10	0.9 [0.10]	0.81 [0.03]	9.3 [5.98]	3.7 [0.23]	15.5 [1.05]	0.5 [0.04]	33.7 [1.00]	1.1 [0.04]	0.46 [0.01]
T-4/80/30	2.2 [0.09]	0.91 [0.02]	12.2 [1.79]	5.8 [1.09]	13.9 [1.47]	0.7 [0.17]	27.4 [0.77]	1.2 [0.05]	0.56 [0.04]
T-4/80/20	1.6 [0.11]	0.87 [0.01]	6.9 [0.92]	3.1 [0.71]	13.7 [0.77]	0.6 [0.03]	30.6 [1.97]	1.2 [0.08]	0.54 [0.05]
T-4/80/10	2.0 [0.05]	0.83 [0.04]	5.7 [1.75]	3.7 [0.50]	15.7 [1.30]	0.6 [0.14]	31.4 [2.24]	1.1 [0.10]	0.49 [0.03]
T-5/40/30	2.7 [0.32]	0.91 [0.01]	16.3 [3.66]	5.8 [0.85]	13.2 [0.98]	0.8 [0.05]	29.8 [2.34]	1.1 [0.13]	0.45 [0.03]
T-5/40/20	1.9 [0.48]	0.89 [0.02]	18.8 [4.27]	4.2 [0.62]	13.3 [0.63]	0.8 [0.13]	28.3 [1.27]	1.0 [0.07]	0.45 [0.05]
T-5/40/10	1.7 [0.33]	0.85 [0.02]	6.7 [2.81]	4.6 [1.04]	16.2 [1.50]	0.8 [0.17]	33.1 [3.66]	1.1 [0.14]	0.44 [0.06]

T-5/60/30	2.4 [0.25]	0.90 [0.02]	11.7 [0.46]	5.2 [0.57]	11.9 [0.92]	0.7 [0.08]	28.8 [1.31]	1.2 [0.15]	0.46 [0.02]
T-5/60/20	1.4 [0.26]	0.90 [0.02]	9.1 [1.70]	4.1 [0.29]	13.4 [0.86]	0.8 [0.09]	29.3 [1.31]	1.2 [0.17]	0.44 [0.03]
T-5/60/10	1.8 [0.38]	0.83 [0.04]	9.1 [3.26]	4.0 [0.38]	13.2 [1.25]	0.7 [0.05]	29.8 [2.52]	1.1 [0.19]	0.44 [0.02]
T-5/80/30	2.0 [0.22]	0.93 [0.01]	10.6 [0.55]	4.9 [1.04]	11.2 [0.89]	0.6 [0.03]	25.6 [1.81]	1.0 [0.08]	0.45 [0.04]
T-5/80/20	1.3 [0.14]	0.90 [0.01]	8.4 [1.45]	4.2 [0.55]	13.7 [0.64]	0.7 [0.06]	27.4 [1.65]	1.0 [0.07]	0.45 [0.01]
T-5/80/10	2.1 [0.41]	0.86 [0.01]	6.0 [2.13]	3.3 [0.62]	12.9 [0.71]	0.9 [0.11]	27.3 [3.20]	0.9 [0.15]	0.47 [0.04]
T-6/40/30	2.0 [0.16]	0.90 [0.01]	11.6 [0.64]	5.2 [0.46]	12.3 [0.95]	0.8 [0.10]	29.3 [5.07]	1.2 [0.21]	0.48 [0.04]
T-6/40/20	1.6 [0.17]	0.88 [0.01]	6.4 [1.22]	3.7 [0.63]	16.8 [0.65]	0.8 [0.06]	27.8 [1.50]	1.1 0.11]	0.42 [0.03]
T-6/40/10	1.4 [0.11]	0.82 [0.01]	5.1 [1.54]	4.0 [1.14]	18.8 [0.84]	0.8 [0.09]	31.4 [3.07]	1.0 [0.11]	0.52 [0.02]
T-6/60/30	2.1 [0.11]	0.90 [0.01]	10.8 [0.96]	5.2 [0.71]	12.7 [0.79]	0.7 [0.11]	25.5 [0.47]	1.0 [0.09]	0.46 [0.02]
T-6/60/20	1.7 [0.19]	0.86 [0.02]	6.0 [0.39]	3.6 [0.63]	14.4 [1.37]	0.7 [0.09]	29.0 [1.92]	1.2 [0.07]	0.48 [0.03]
T-6/60/10	1.6 [0.39]	0.84 [0.02]	6.6 [2.41]	3.6 [1.74]	15.0 [1.77]	0.8 [0.07]	33.6 [1.12]	1.1 [0.03]	0.46 [0.03]
T-6/80/30	2.7 [0.45]	0.90 [0.03]	9.9 [1.30]	5.0 [1.25]	10.7 [0.25]	0.7 [0.10]	26.9 [0.98]	1.1 [0.10]	0.44 [0.02]
T-6/80/20	1.8 [0.28]	0.90 [0.01]	6.8 [1.13]	4.3 [0.97]	13.2 [0.91]	0.7 [0.06]	27.2 [3.19]	1.1 [0.19]	0.47 [0.01]
T-6/80/10	1.2 [0.19]	0.84 [0.01]	7.8 [2.12]	4.7 [0.77]	14.2 [1.92]	0.9 [0.04]	31.0 [1.74]	1.1 [0.06]	0.51 [0.04]

Table 3. Dimensions of anatomical elements of interest in 4, 5 and 6 years old *G. crinita* fibers.

Feature/ Age (years)	4	5	6
Fiber length (µm)	1 399 [496.3]	1 554 [294.7]	1 100 [141.6]
Fiber total diameter (µm)	26.7 [6.4]	27.9 [5.49]	26.3 [6.54]
Fiber wall thickness (µm)	2.1 [0.3]	2.1 [0.36]	2.1 [0.3]
Fiber lumen diameter (µm)	22.4 [6.4]	23.7 [5.3]	22.0 [6.58]
Slenderness coefficient	53.4 [19.2]	57.4 [14.76]	44.1 [10.4]

Anatomy Description

The exclusive analysis of the fibers was made because of its possible influence on the physical and mechanical properties of the composite material.

The average fiber length reached its highest value for the age of 5 years (1 554 µm), followed by 4 years (1 399 µm) and 6 years (1 100 µm), respectively. According to IAWA (1989), these dimensions are classified as medium length fibers (900-1 600 µm); therefore, all of them were considered that way. The average fiber diameter was 28, 27 and 26 µm, for ages 5, 4 and 6 years without confirming a significant difference.

According to Ibama (1991), the fiber diameter is described as medium. The average wall thickness for the three ages (2 µm) is defined as very thin (IAWA, 1989). The slenderness coefficient (also known as long/wide ratio) reached its highest value for age 5 years (57), followed by age 4 years (53) and age 6 years (44), respectively. These results are shown in Table 3.

Chemical caracterization

The contents of extractives, holocellulose, lignin and ashes were very similar for the three ages evaluated. The content of extractants and ashes were numerically lower than the values recorded by Oluwadare and Asagbara (2008), who carried out a study of the chemical composition of *Sterculia setigera* Delile, a species of the same family as *Guazuma crinita* (Table 4). The results of lignin content kept numerical similarity with the values found by Oluwadare and Asagbara (2008). Holocellulose content values were numerically higher than those recorded by Pettersen (1984) for *Guazuma tomentosa* Kunth, a species of the same genus as *Guazuma crinita*.

Table 4. Chemical characterization of White *Bolaina* wood 4, 5 and 6 years old.

Feature / Age (years)	4	5	6
Extractives (%)	3.08 [1.01]	1.98 [0.60]	2.11 [0.21]
Holocellulose (%)	72.01 [3.40]	75.43 [6.27]	72.36 [0.36]
Lignine (%)	22.75 [0.59]	21.81 [1.57]	24.27 [1.16]
Ahes (%)	1.21 [0.18]	1.22 [0.04]	1.11 [0.14]

Physical properties

Moisture content

Figure 1 shows the average final humidity values for all samples of the PP- White *Bolaina* composite material, which range from 2.2 to 0.9 % for year 4, from 2.7 to 1.3 % for year 5 and from 2.1 to 1.2 % for year 6. Moisture content values maintain numerical similarity in the three ages studied, although statistically age is an influential variable. The chemical composition of the *G. crinita* fibers showed almost no differences in their three ages.

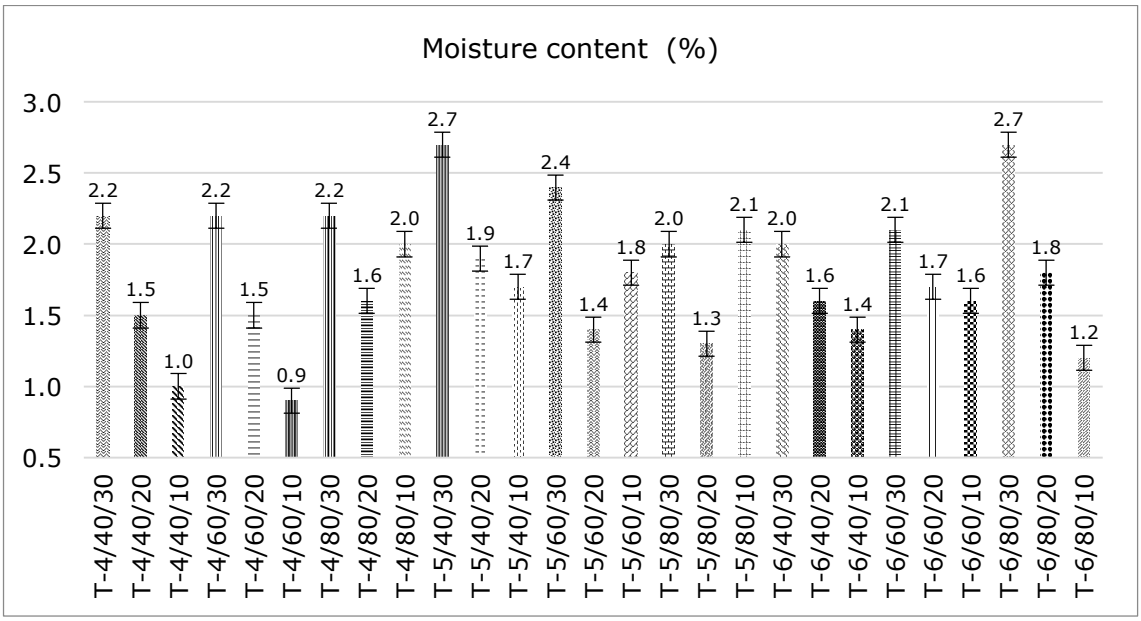

Figure 1. Moisture content of PP- White *Bolaina* composite materials.

The proportion of particles in the PP- White *Bolaina* composite material and the final humidity show a proportional relationship, which is due to the inherent hygroscopic nature of the wood. The carbohydrates that make up the cell wall in plant fibers, such as cellulose and hemicelluloses, have hydroxyl groups (OH), which are quite similar to water (Caulfield *et al*., 2005; Bouafif *et al*., 2009). Likewise, the average holocellulose content (cellulose and hemicelluloses) in the *G. crinita* fibers for their three ages under study is above 70 %, confirming a strong affinity between the fibers and the surrounding humidity. In plastic-wood composite materials, lignocellulosic components are responsible for moisture gain; matrixes usually have a hydrophobic character (Klyosov, 2007; Caicedo *et al.,* 2015).

Cárdenas (2012) refers to a maximum range of acceptability for moisture content in composite materials of 2 %. The same author recorded moisture content values of 0.27 to 0.31 % for composite materials made by the injection method.

Statistical analysis indicated that the variables age and mixing ratio had a highly significant influence ($p \leq 0.0041$). The double age * particle size interaction and the triple interaction behaved in a similar way on the moisture values ($p \leq 0.0040$).

Bulk density

Figure 2 shows the average values of apparent density for all samples of the PP- White *Bolaina* composite material. Values range from 0.91 to 0.76 g.cm^{-3} for year 4, from 0.93 to 0.83 g.cm^{-3} for year 5 and from 0.90 to 0.82 g.cm^{-3} for year 6. Age 5 years records the highest values in apparent density; however, both the anatomical and chemical characterization performed on the *G. crinita* fibers had little difference among the three ages under study.

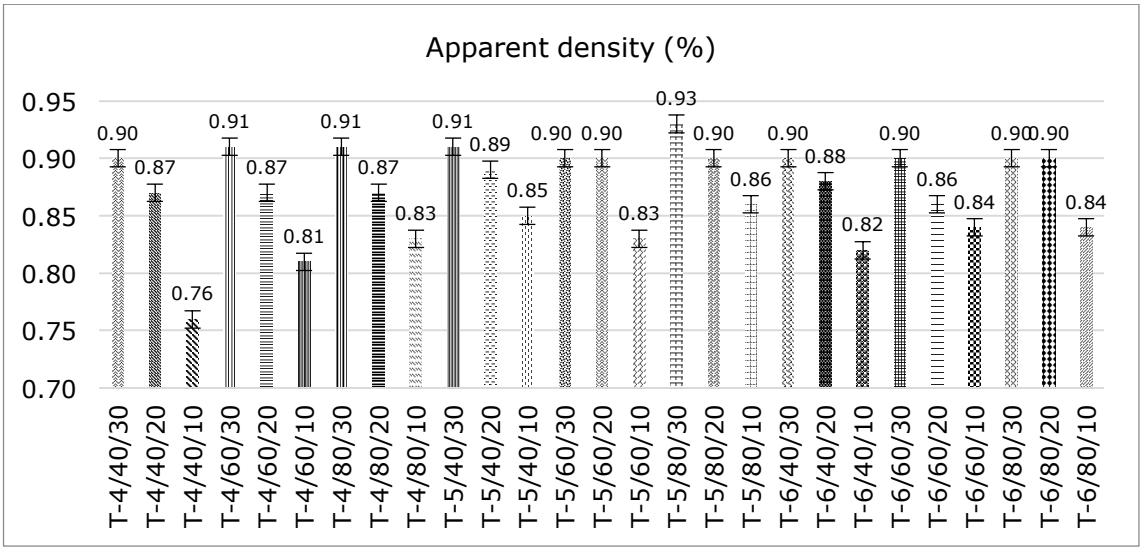

Figure 2. Bulk density of PP-White *Bolaina* composite materials.

Statistical analysis indicated that the variables age, particle size and mixing ratio had a highly significant influence ($p \leq 0.0015$); however, double interactions and multiple interaction did not act that way upon density ($p \geq 0.0405$).

In regard to particle size, the treatments that include the smallest particles had the highest density values, given the greater ease of encapsulation of the material (Fabiyi, 2007; Klyosov, 2007; Cárdenas, 2012). Likewise, there is a slight increase in density values when the proportion of particles in the composite increases. Although the *G. crinita* wood has a low density, its increase favors the density of the composite material.

Moya *et al.* (2012) calculated density values between 0.98 and 1.04 g.cm^{-3} for composite materials reinforced with pine sawdust. Cárdenas (2012) determined density values between 1.06 and 1.11 g.cm^{-3} for polypropylene and pinewood composites made by injection method; Lázaro *et al*. (2016a) accomplished similar results using the combined extrusion and compression method. In turn, Soatthiyanon (2010) determined density values between 1.01 and 1.14 g.cm^{-3} for different types of composite materials. The bulk density values obtained in the present study were lower compared to those of the cited literature.

Absorption and swelling

Figure 3 shows the average absorption and swelling values for the PP- White *Bolaina* composite material during two months of immersion in water.

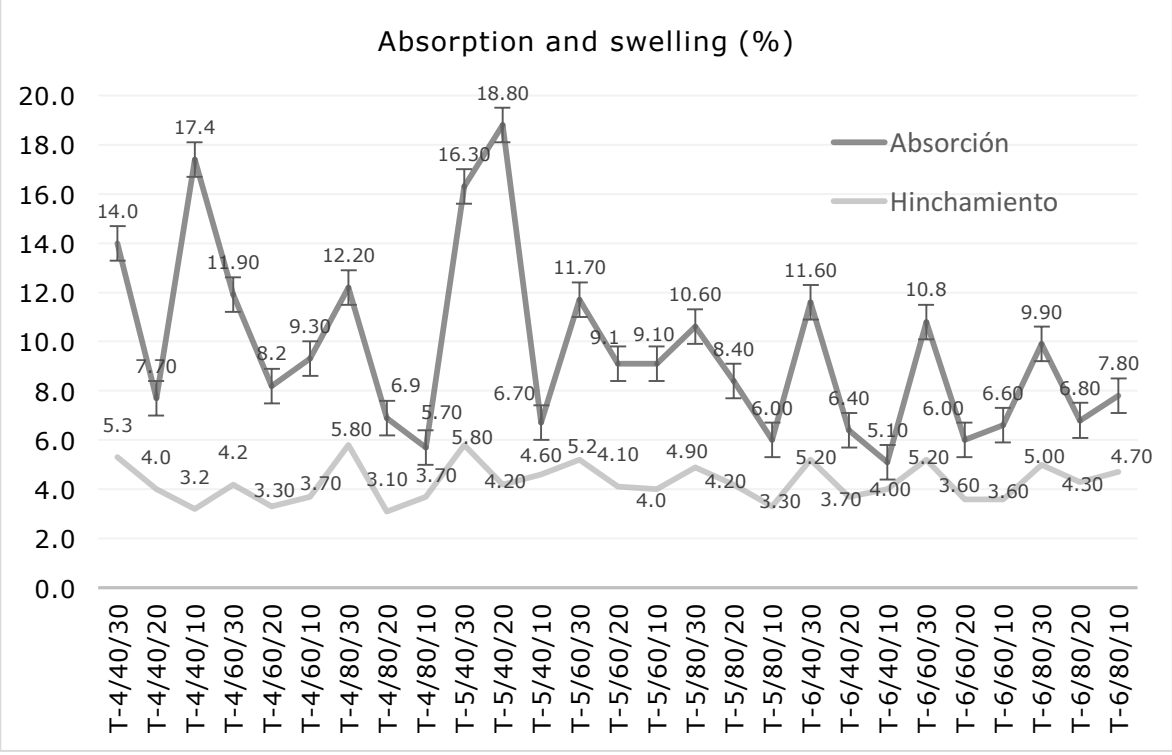

Figure 3. Average value of absorption and swelling in PP- White *Bolaina* composite materials.

The average absorption values vary from 17.4 to 5.7 % for year 4, from 18.8 to 6.0 % for year 5 and from 11.6 to 5.1 % for year 6. Although the age variable presented a highly significant difference ($p \leq 0.0001$), the chemical composition of *G. crinita* fibers for their three ages is very similar, so it is evident that it does not influence the results from absorption.

A slight decrease in absorption values is noticed when using smaller particles, which is consistent with Fabiyi (2007) and Fuentes-Talavera *et al.* (2014). When the interface in the composite material is homogeneous and compact, the fibrous elements are embedded within the matrix unable to absorb moisture from the outside. Large particles are difficult to embed through the matrix, leaving exposed regions where they absorb moisture (Simonsen and Rials, 1996; Caulfield *et al.,* 2005). Likewise, a direct relationship between absorption values and the proportion of particles is observed. Klyosov (2007) points out that most plastics used in composite materials practically do not absorb water; therefore, the incorporation of cellulosic particles is responsible for significantly increasing water absorption.

Soattiyanon (2010) reported absorption values between 8 and 9 % for different types of composite materials during periods of immersion greater than 6 months in materials processed by injection, a process that ensures a better coating of the fiber and therefore a greater resistance to absorption. In turn, Lázaro *et al.* (2016a) reported absorption values of 14 to 15 % in polypropylene and bamboo composites, results similar to the present investigation.

Statistical analysis indicated a highly significant influence ($p \leq 0.0001$) for the three variables; in a similar way, the double interactions age * particle size and age * mixing ratio, as well as the triple interaction, had significant influence on the absorption values ($p \geq 0.0007$).

The average swelling values varied between 5.8 and 3.1% for year 4, from 5.8 to 3.3 % for year 5 and from 5.2 to 3.6 % for year 6. Ages 5 and 6 years had the greatest increase in swelling for the first two hours of immersion in water. In general, treatments with larger particles reached the highest swelling values, since the fiber is not fully encapsulated, a trend reported in different studies (Okubo *et al*., 2004; Mattos *et al*.,

2014). A direct relationship between the swelling values and the proportion of particles, as in absorption. This is due to the hydrophilic nature of wood particles, especially the presence of hydroxyl groups (OH) in cellulose and hemicelluloses, major components in wood (Caulfield *et al*., 2005; Bouafif *et al*., 2009).

Cárdenas (2012) reported increases in swelling close to 10 % in composite materials reinforced with pinewood (50 % of the total weight), for periods of immersion greater than five months. In another investigation, Gonzáles *et al.* (2018) recorded swelling values of 2.6 % in composite materials with bamboo (30 % of the total weight), for the first 24 hours of immersion and in materials made by extrusion and compression. The swelling values obtained in the present study exceeded those indicated by Gonzáles *et al.* (2018), but were lower than those from Cárdenas (2012), who experimented with longer immersion periods and composite materials made by injection.

Statistical analysis indicated that the mixing ratio variable had a highly significant influence ($p = 0.0001$) on the swelling values.

Mechanical properties

Rupture Module (MOR)

Figure 4 shows the average values of rupture modulus (MOR) in static bending and tension for all samples of the PP- White *Bolaina* composite material. The average values of MOR in bending range from 33.7 to 26.2 MPa for year 4, from 33.1 to 25.6 MPa for year 5 and from 33.6 to 25.5 MPa for year 6.

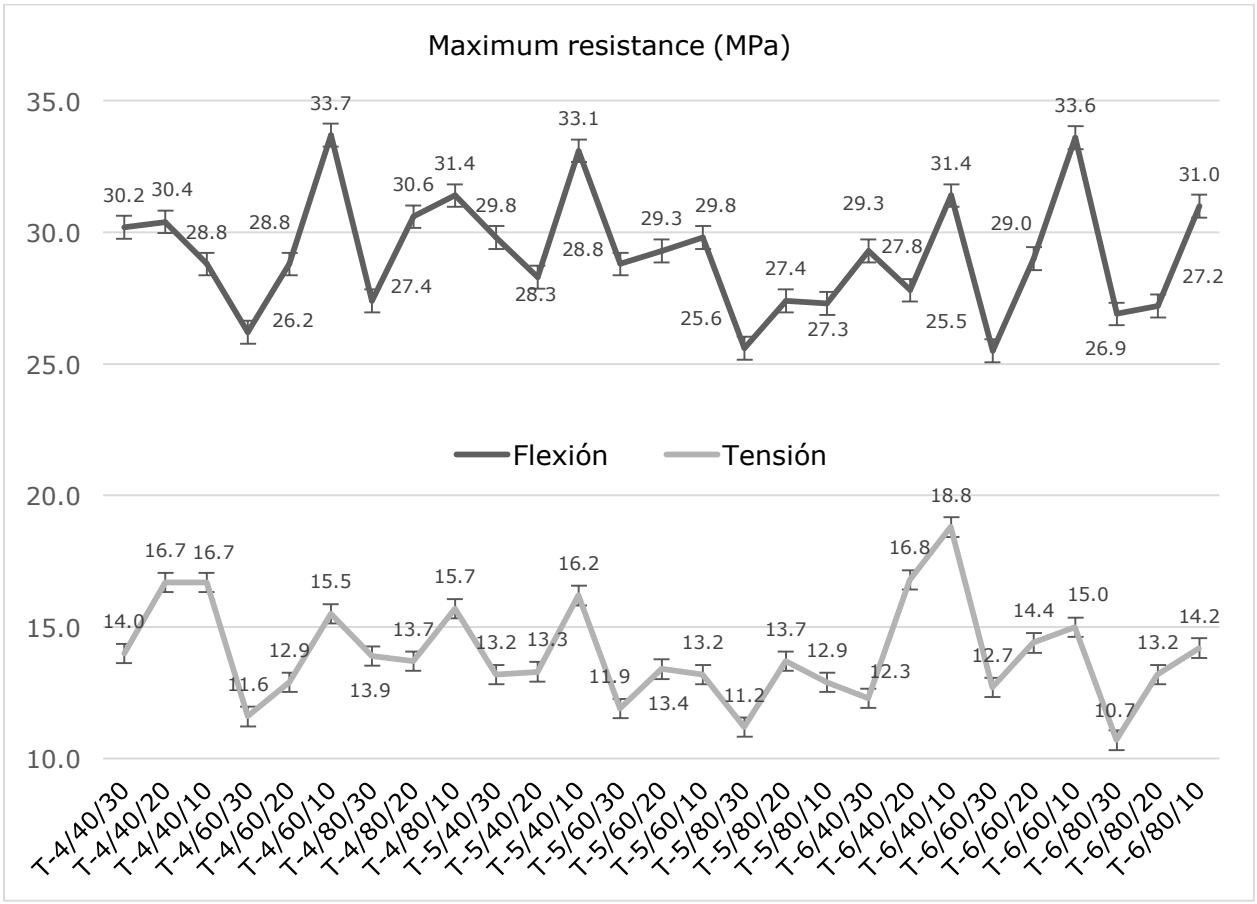

Figure 4. Rupture module (MOR) in static bending and parallel tension of PP- White *Bolaina* composite materials.

An inverse relationship between the proportion of particles and the values of MOR in bending is observed, where treatments with less proportion of particles obtained high values of MOR. According to Klyosov (2007), as the ratio of lignocellulosic fibers in composite material increases, the MOR begins to decrease.

The MOR values in bending obtained in the present study are similar to those referred by Cárdenas (2012) and Klyosov (2007) (31-34 MPa and 21-26 MPa respectively). Other researchers such as Idrus *et al.* (2011) and Ravi *et al.* (2014) agree to state otherwise, more fibers in the composite material improve the values of MOR in bending. An explanation for this behavior is the poor reinforcement / matrix interaction in the composite material due to the manufacturing method, as well as to the anatomical characteristics (wall length and thickness) of White *Bolaina* fibers. It should be noted

that the responsibility for conferring mechanical resistance to the composite rests with the fibers. Statistical analysis indicated a highly significant influence (p = 0.0001) for the mixing ratio variable on the bending MOR values.

The average values of MOR in tension vary between 16.7 and 11.6 MPa for year 4, from 16.2 to 11.2 MPa for year 5 and from 18.8 to 10.7 MPa for year 6. The values of MOR in tension keep numerical similarity in the three ages studied, although statistically age is an influential variable. The slenderness coefficient of the *G. crinita* fiber showed almost no differences in its three ages.

Regarding the size of the particles, a moderate increase in the values of the MOR in tension is detected when larger particles are incorporated. In an investigation, Stark and Berger (1997) argued that this characteristic increases when the particle size increases to reach 250 µm (60 ASTM), at which point the tension MOR begins to decrease. However, Nourbakhsh *et al*. (2010) and Bledzki *et al.* (2005) consider that the tensile strength in PP-wood composite materials increases when particle size decreases, and they attribute this behavior to an improvement in interfacial adhesion between the wood particles and the matrix.

A significant increase in the MOR values in tension is observed when the proportion of wood particles in the composite material is reduced (p = 0.0001). This phenomenon has been pointed out by other researchers (Klyosov, 2007; Ravi *et al*., 2014), who agree that high concentrations of wood particles reduce the MOR of the composite material.

Caulfield *et al.* (2005) recorded tension MOR values of 44.9 MPa for polypropylene and poplar fiber composites (30 % of total weight). In another investigation, Stark and Rowlands (2003) reported MOR values of 29.4 and 37 MPa for wood flour composites at 40 and 20 % of the total weight, respectively. Cárdenas (2012) reported MOR values between 19 and 25 MPa for polypropylene and pinewood composites made by injection method.

Statistical analysis indicated for the variables age, particle size and proportion of particles, a highly significant influence (p = 0.0001); in the same way, the double interactions age * particle size and age * mixing ratio, as well as the triple interaction, had significant influence on the values of MOR in tension (p≤0.0093).

Modulus of elasticity (MOE)

Figure 5 shows the average elastic modulus (MOE) values in static bending and tension for all samples of the PP- White *Bolaina* composite material. The average values of MOE in static bending range between 1.3 and 0.9 GPa for year 4, 1.2 to 0.9 GPa for year 5 and 1.2 to 1.0 GPa for year 6.

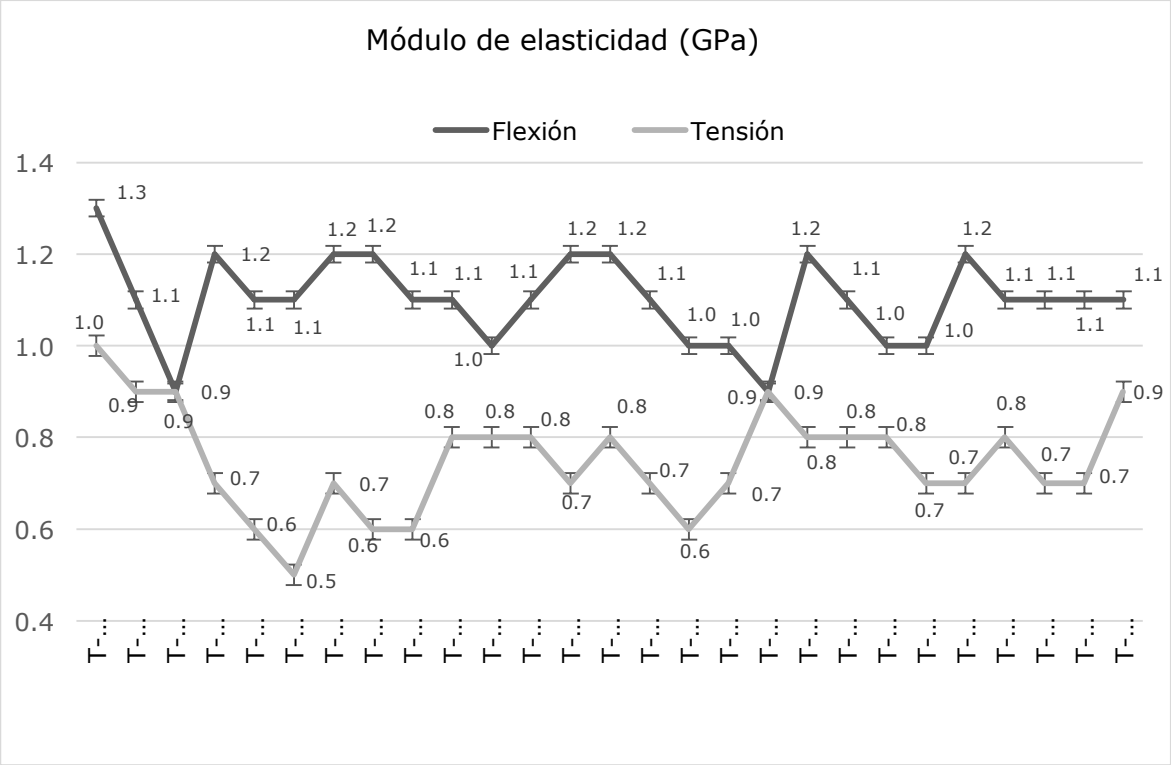

Figure 5. Modulus of elasticity (MOE) in static bending and tension of PP- White *Bolaina* composite materials.

The treatments with the highest proportion of particles obtained higher values of MOE in bending. Wood fibers generally exhibit good bending behavior, which is why composite materials with more fiber demand more effort to achieve deformation (Caulfield *et al*., 2005; Idrus *et al*., 2011). However, reinforcing with more particles the composite material does not necessarily produce improvements in the MOE in bending. Ravi *et al*. (2014) indicate that the empty spaces, the low interaction between

fibers and a poor dispersion of them in the matrix, negatively influence the mechanical properties of the composite material.

For the mixing ratio variable, the statistical analysis indicated a highly significant influence ($p = 0.0017$) on the values of MOE in static bending.

The average values of MOE in tension vary from 1.0 to 0.5 GPa for year 4, from 0.9 to 0.6 GPa for year 5 and from 0.9 to 0.7 GPa for year 6.

A slight increase in the values of the MOE in tension is verified when larger Bolaina particles are used, as it occurred with the rupture module. The highest values of MOE in tension correspond to treatments with larger particles, and they were lower than those mentioned by Caulfield *et al*. (2005), Lisperguer *et al*. (2013) and Cárdenas (2012).

The low inter-phase adhesion between the *G. crinita* particles and the polypropylene matrix has possibly generated areas of high heterogeneity inside the composite material, reducing its resistance to deformation (Essabir *et al*., 2015). Likewise, the anatomical characteristics of the *Bolaina* fibers in their three ages registered low values, with medium-sized fiber lengths and very thin wall thicknesses, undesirable characteristics for stress tests (García *et al.,* 2003).

For the particle size variable, the statistical analysis indicated a highly significant influence ($p = 0.0001$); in a similar way, the double interactions age * particle size and age * mixing ratio had a highly significant influence on the MOE values in tension ($p \leq 0.0005$).

Impact resistence

Figure 6 presents the average values of impact resistance for all samples of the PP-White *Bolaina* composite material. Values range from 0.62 to 0.46 J for year 4, from 0.47 to 0.44 J for year 5 and from 0.52 to 0.42 J for year 6.

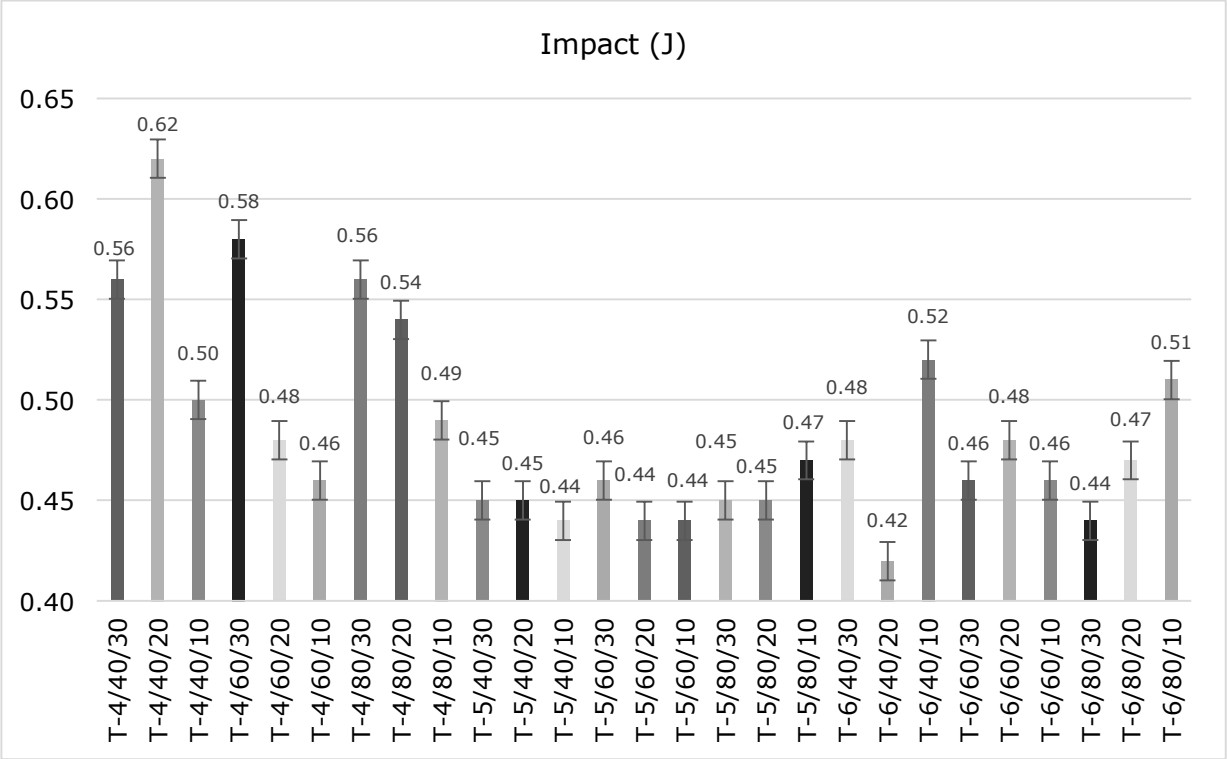

Figure 6. Impact resistance of polypropylene composite materials - White *Bolaina*.

The values for 4 years sample were slightly higher, although the slenderness coefficient of the *G. crinita* fiber had almost no differences in its three ages.

The treatments with larger and smaller particles, respectively, achieved the highest impact resistance values. This irregular behavior can be explained by the poor reinforcement/matrix interaction in the composite material due to the manufacturing method. The presence of wood as reinforcement in the PP matrix generates areas where the effort is concentrated, which leads to the beginning of cracks and the potential failure of the composite material (Nourbakhsh *et al*., 2010).

Stark and Berger (1997) observed that as the particle size increased, the impact resistance for different composite materials also did. However, this does not fit the results of the present study in which the matrix is primarily responsible for absorbing the energy produced by the impact. Durowaye *et al.* (2014) indicated that increases in the amount of wood particles reduce the ability to absorb energy from the matrix,

which decreases the impact resistance on the composite material, influence also reported by Yuan *et al.* (2008).

Statistical analysis indicated that the variables age and particle size exerted a highly significant influence ($p \leq 0.0002$); in a similar way, double interactions and triple interaction affected impact resistance values ($p \leq 0.0004$).

Conclusions

Wood age did not have a numerically significant influence, except for the physical property of absorption and the mechanical property of impact resistance.

The proportion of particles in the PP- White *Bolaina* composite material showed a direct relationship with the physical properties of moisture content, density, swelling and absorption, as well as with the mechanical property of MOE in static bending; while the relationship was inverse with respect to the values of MOR in tension and static bending, in addition to impact.

The particle size in the PP- White *Bolaina* composite described a direct relationship with respect to the values of MOR and MOE in tension.

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Conflict of interests

The authors declare no conflict of interests.

Contribution by author

Alexei Rodolfo Córdova Contreras: raw material processing, composite materials processing, evaluation of physical and mechanical properties, data analysis; Aldo Joao Cárdenas Oscanoa: research advice, writing of the manuscript; Héctor Enrique Gonzáles Mora: research planning and advice.

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