Effect of Thermo-hydro-mechanical Densification on the Wood Properties of Three Short-rotation Forest Species in Costa Rica

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Alnus acuminata, *Vochysia ferruginea*, and *Vochysia guatemalensis* are three low-density wood species used for reforestation in Costa Rica. The goal of this work was to study a thermo-hydro-mechanical densification process and test the characteristics of densified wood of these species. Twelve densifying treatments based on temperature, compression time, and use/no use of steam were tested. The variables of the densification process and the properties of the densified wood were determined. The results showed that the densification percentage was over 80% for wood of *A. acuminata* and over 70% for wood of *V. ferruginea* and *V. guatemalensis*. In the three species, the densification process was influenced by initial density. The influence of temperature during the densification process affected the heating rate and color change. An increase in the modulus of elasticity and modulus of rupture in static bending and in the hardness of the densified wood relative to the normal wood was observed, as well as a clear positive correlation of the properties with final density and maximum load, the latter being highly correlated with initial density. This showed that initial density was significant in the densification process and affects wood properties.

Keywords: Tropical species; Hardwoods; Low density; Thermo-hydro-mechanical process

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INTRODUCTION

Wood supply for the industrial and construction sectors at the world level has decreased during the last few years (Toppinen *et al*. 2018). For this reason, the interest in fast-growing wood species has grown (Cossalter and Pye-Smith 2003). However, low density and limited mechanical properties are the main characteristics of those species because the trees are harvested while still young and the proportion of juvenile wood is dominant (Zobel and Van Buijtenen 1989; Kamke 2006; Cahyono *et al*. 2015). The high price of wood in Costa Rica led to the search for new opportunities to allow the replacement of high value timber species with less valuable or marginal species (Nölte *et al*. 2018).

Alnus acuminata, *Vochysia ferruginea*, and *Vochysia guatemalensis* are fast-growing hardwood species used in commercial reforestation programs in Costa Rica (Moya 2018). These are low-density, soft, and easy to work with wood species (Tenorio *et al*. 2016). However, the mechanical properties of these species are limited, which makes them unsuitable for structural uses, being limited to products of low structural demand and low commercial value markets (Serrano and Moya 2011). Due to these reasons, these species have been rarely studied and ignored by industries. Currently, studies can be found on the properties and behaviour of these and other reforestation species in industrial processes in Costa Rica (Moya *et al*. 2019). However, studies aiming at improving their mechanical properties are not available.

Many wood densification processes have been developed to improve the wood's mechanical and physical properties (Fang *et al*. 2012). Such processes can increase the wood density in three ways: (i) mechanical compression by reducing the empty spaces; (ii) impregnation of the empty spaces with some substance, and (iii) combination of the previous processes (Fang *et al*. 2012). However, unlike densification by mechanical compression, chemical impregnation affects the natural and sustainable character of the wood, and is usually more expensive (Navi and Heger 2004). Mechanical compression combined with steam and heat, called thermo-hydro-mechanical (THM) densification, has been studied as an environmentally friendly alternative to increase the wood density and improve its mechanical properties, achieving enhanced dimensional stability without using chemical products (Bekhta *et al*. 2009; Büyüksari *et al*. 2012; Arruda and Del Menezzi 2013; Candan *et al*. 2013; Moya *et al*. 2013; Tu *et al*. 2014).

There are different THM densification processes, generally consisting of: (i) softening the wood structure, which can be attained at certain temperatures and moisture contents; (ii) compressing the wood, usually between two metal plates, and (iii) keeping the wood deformation obtained, by thermal modification in many cases (Sandberg *et al*. 2013). The THM densification process improves the natural properties of the wood and produces stable materials (Navi and Heger 2004; Sandberg *et al*. 2013). The heat treatment can improve resistance to decay (Huang *et al*. 2012), decrease hygroscopicity (Metsä-Kortelainen *et al*. 2006), and improve the dimensional stability (Esteves *et al*. 2007). The moisture induces a mechano-sorptive effect and further softens the wood, which enables mechanical compression of wood without cell wall fracture (Bao *et al*. 2017).

Most research on THM densification has focused on softwood species and closed systems, where the densification process takes place inside reactors (Navi and Heger 2004). Therefore, studies on hardwood tropical species, whose anatomical structures affect to a further extent the result of the process, are scarce (Navi and Heger 2004). This is because the compression properties of the wood depend on the frequency, size, and distribution of its anatomical structures (Darwis *et al*. 2017). For hardwoods, these structures are dominated by vessels, fibers, and radial parenchyma arranged in more complex matrixes (Gibson 2012) than the fiber tracheids of softwood species (Fratzl and Weinkamer 2007).

The aim of this study was to investigate the THM densification process and evaluate its effect on the characteristics of the densified wood of three fast-growing hardwood species used in commercial reforestation in Costa Rica: *A. acuminata*, *V. ferruginea*, and *V. guatemalensis*. The variables in the densifying process along with the physical and mechanical properties of the densified wood were determined.

EXPERIMENTAL

Origin and Characteristics of the Wood before Densification

The study tested the wood of *Alnus acuminata*, *Vochysia ferruginea*, and *Vochysia guatemalensis* from fast-growth plantations located in Cartago and Alajuela in Costa Rica.

The trees used were approximately 8 years old, which normally implies a low heartwood content (Tenorio *et al*. 2016). Therefore, the wood used was mostly sapwood. Wood samples of 300 mm long × 70 mm wide × 20 mm thick of each species were prepared. Before densification, thickness, width, length, density, moisture content, and color were measured for each of the samples, in total were 240 samples per species (Table 1).

Table 1. General Characteristics of the Wood Before Densification

Species	Initial Thickness (mm)	Initial Wood Density (g/cm^3)	Initial Moisture Content (%)	Initial Wood Color Parameter		
				*L**	*a**	*b**
Alnus acuminata	19.7 (0.02)	0.43 (0.03)	9.97 (0.83)	71.65 (6.36)	14.18 (3.01)	22.45 (1.85)
Vochysia ferruginea	20.0 (0.03)	0.45 (0.05)	10.68 (1.13)	69.05 (3.00)	13.36 (1.77)	19.81 (1.48)
Vochysia guatemalensis	20.4 (0.02)	0.39 (0.05)	12.56 (0.57)	69.55 (1.89)	9.84 (1.92)	20.89 (2.16)

The values in parenthesis are standard deviations.

The density was calculated as the ratio of weight and volume determined by measuring the initial thickness, width, and length. The moisture content was calculated as the ratio between the initial weight and the oven-dry weight, expressed as a percentage according to ASTM D4422-16 (2016). For color measurements, a Mini Scan XE Plus spectrophotometer (HunterLab, Reston, VA, USA) was used along with the CIE *L*a*b** system to measure the reflectance spectrum. The range of this measurement was from 400 to 700 nm with an opening at the point of measurement of 11 mm. For the observation of reflection, the specular component (SCI mode) was included at a 10º angle, which is normal for the specimen surface (D65/10), a field of vision of 2º (Standard observer, CIE 1931), and the standard illuminant D65 (corresponding to daylight in 6500 K). The Mini Scan XE Plus generated three parameters for each measurement, namely: *L** (luminosity), *a** (color trend from red to green), and *b** (color trend from yellow to blue).

Densification Process

Three temperatures, 140 °C, 160 °C, and 180 °C for *A. acuminata* and *V. guatemalensis* and 140 °C, 150 °C, and 160 °C for *V. ferruginea*, two compression times of 10 and 15 min, and the application of water steam or just heat (as the initial stage before wood compression) were used in the densification process. In total, 12 treatments were tested with 20 specimens per densification treatment, resulting in 240 specimens per species (Table 2). The differences in temperature for *V. ferruginea* in comparison with the other two species are due to the fact that when carrying out the first tests with 180 °C the surface of the wood samples burned and, in some cases, parts adhered to the metal plates, making it impossible to evaluate the process, probably due to low thermostability of this species (Moya *et al*. 2017). Therefore, it was decided to work with different temperatures for this species.

The densification process was the same described in Tenorio and Moya (2019). The process consisted of three stages. Stage 1 was the steaming or heating stage, where steam was applied to half of the wood samples for 10 min, while the other half was only heat-treated. Stage 2 was the compression stage, where the wood samples were compressed perpendicular to the grain until reaching a target thickness of 9 mm (degree of compression of 55%) during 10 or 15 min. Stage 3 was the stabilization stage, where the samples were kept compressed and heated while unloaded for another 10 min. Throughout the process, the metal plates kept one of the three temperatures constantly.

Table 2. Treatments Used in the Densification Process of the Wood of Three Species

Species	Temperature (°C)	Densification Time (min)	Steam	Code of Treatment
Alnus acuminata and *Vochysia guatemalensis*	140	10	Yes	140-10-ST*
			No	140-10-WT
		15	Yes	140-15-ST
			No	140-15-WT
	160	10	Yes	160-10-ST
			No	160-10-WT
		15	Yes	160-15-ST
			No	160-15-WT
	180	10	Yes	180-10-ST
			No	180-10-WT
		15	Yes	180-15-ST
			No	180-15-WT
Vochysia ferruginea	140	10	Yes	140-10-ST
			No	140-10-WT
		15	Yes	140-15-ST
			No	140-15-WT
	150	10	Yes	150-10-ST
			No	150-10-WT
		15	Yes	150-15-ST
			No	150-15-WT
	160	10	Yes	160-10-ST
			No	160-10-WT
		15	Yes	160-15-ST
			No	160-15-WT

*This treatment code means 140 °C of heat treatment, 10 min of compression time, and with steam application

Evaluation of the Densification Process

To determine the maximum load and the internal heating rate of the wood, a temperature control probe that was placed at the centre of the sample thickness was used to monitor and record the load and temperature during the densification process. Additionally, each sample's thickness was determined at the end of stages 2 and 3. After the densification process, the width, length, weight, color, density, and densification percentage were determined.

The same procedure used before the densification process was used for color measurements. The density was calculated as the ratio between the weight and volume of the wood sample after stage 3. For the volume calculation, the dimensions of stabilization thickness, length, and width were determined. The densification percentage was calculated as the ratio of the sample initial density and the density after densification.

The thickness determined in stages 2 and 3 helped determine the compression ratio and the spring back of wood thickness. The compression ratio was calculated as the ratio of the initial thickness and the compression thickness, expressed as a percentage. The spring back of the thickness of the densified wood was calculated as the absolute value of the ratio of compression thickness and stabilization thickness, expressed as a percentage.

Properties of the Densified Wood

In the evaluation of the wood properties, the thickness swelling was determined according to ASTM D4933-16 (2016). The wood samples were conditioned to 18% equilibrium moisture content, at 20 °C, and 85% relative humidity. Next, the thickness swelling was calculated as the ratio of the thickness after conditioning and the thickness before conditioning, expressed as a percentage. The mechanical properties, static bending, modulus of rupture, modulus of elasticity, and Janka hardness were determined following the ASTM D143-14 (2016) standard. A total amount of 20 specimens per species, per treatment, were prepared for each test. In total, 13 treatments were performed, 12 densification treatments and one un-densified wood treatment.

Microscopic Examination

For the microscopic examination, sections 10 mm × 50 mm × the densified thickness were taken from the densified specimens, and sections 10 mm × 50 mm × 20 mm were taken from the un-densified wood specimens. All samples were polished in their transversal sections using a sander (Struers, Tegramim 30 Model, Cleveland, OH, USA). The procedure is detailed in Tenorio *et al.* (2021).

After polishing, a microscope (Zeiss, Axioscope Model, Jena, Germany) was used to take the pictures of the anatomical structures of each specimen, using an Axicam 503 Color camera (Zeiss, Jena, Germany) in reflexion mode using a 10x and 50x lens. The un-densified specimens were photographed in reflexion mode using a 10x lens. The image editing ZEN program (Zen Pro, Zeiss, Version 2.3, Jena, Germany) was used.

Statistical Analysis

Compliance of the measured variables with the assumptions of normal distribution, homogeneity of variance, and outliers was verified. An analysis of variance was applied to verify the effect of the densification treatments in each one of the variables obtained during the densification process, in the physical properties, and mechanical properties per species (Table 2).

The Tukey test was used to determine the statistical differences between the means of the variables measured. A correlation analysis between the variables obtained during densification and the initial characteristics of the wood was performed. Correlation analysis was additionally performed between the physical and mechanical properties of the densified wood, and the variables obtained during the process of densification, independently for each species. The analysis of variance, the Tukey tests, and the correlation analysis were performed with the SAS software (SAS Institute Inc., v.4.11, Cary, NC, USA).

RESULTS

Evaluation of the Densification Process

Table 3 presents the results obtained as part of the evaluation of the densification process. A compression ratio of approximately 55% can be observed for the three species. No statistical differences were found between the treatments applied for any of the species.

Table 3. Characteristics of the Wood of Three Forest Species After Densification

Species	Treatment	Compression Ratio (%)	Final Density (g/cm^3)	Densification Percentage (%)	Spring Back (%)	Color Change (ΔE^*)
Alnus acuminata	140-10-ST	54.95^{A}	0.79^{A}	85.91^{A}	8.53^{A}	5.79^{D}
	140-10-WT	54.80^{A}	0.80^{A}	84.07^{A}	9.24^{A}	6.20^{D}
	140-15-ST	54.84^{A}	0.78^{A}	83.50^{A}	8.85^{A}	5.65^{D}
	140-15-WT	54.81^{A}	0.80^{A}	85.33^{A}	7.01^{A}	5.92^{D}
	160-10-ST	55.02^{A}	0.78^{A}	86.42^{A}	7.97^{A}	7.96^{BCD}
	160-10-WT	54.78^{A}	0.76^{A}	83.04^{A}	8.38^{A}	7.49^{CD}
	160-15-ST	54.92^{A}	0.81^{A}	85.52^{A}	6.61^{A}	11.07^{ABC}
	160-15-WT	54.61^{A}	0.80^{A}	82.93^{A}	8.36^{A}	8.49^{ABCD}
	180-10-ST	54.52^{A}	0.75^{A}	82.61^{A}	8.54^{A}	11.73^{A}
	180-10-WT	54.79^{A}	0.79^{A}	87.95^{A}	5.47^{A}	11.10^{ABC}
	180-15-ST	54.85^{A}	0.77^{A}	81.59^{A}	8.45^{A}	11.14^{ABC}
	180-15-WT	54.68^{A}	0.77^{A}	84.96^{A}	5.62^{A}	11.52^{AB}
Vochysia ferruginea	140-10-ST	55.52^{A}	0.78^{A}	71.84^{A}	18.39^{AB}	2.55^{C}
	140-10-WT	55.27^{A}	0.80^{A}	73.23^{A}	14.83^{AB}	3.09^{C}
	140-15-ST	55.59^{A}	0.81^{A}	74.67^{A}	14.87^{AB}	4.01^{C}
	140-15-WT	55.49^{A}	0.81^{A}	72.78^{A}	15.31^{AB}	2.85^{C}
	150-10-ST	55.55^{A}	0.82^{A}	79.97^{A}	14.02^{AB}	5.36^{BC}
	150-10-WT	55.06^{A}	0.78^{A}	72.08^{A}	18.88^{A}	4.75^{BC}
	150-15-ST	54.86^{A}	0.80^{A}	81.41^{A}	12.05^{AB}	5.32^{BC}
	150-15-WT	55.20^{A}	0.82^{A}	80.00^{A}	12.81^{AB}	3.89^{C}
	160-10-ST	55.66^{A}	0.81^{A}	80.72^{A}	13.20^{AB}	11.21^{A}
	160-10-WT	55.49^{A}	0.79^{A}	74.45^{A}	12.85^{AB}	8.43^{AB}
	160-15-ST	54.95^{A}	0.74^{A}	72.90^{A}	12.85^{AB}	11.59^{A}
	160-15-WT	55.14^{A}	0.79^{A}	78.93^{A}	11.04^{B}	11.19^{A}
Vochysia guatemalensis	140-10-ST	56.09^{A}	0.64^{B}	72.31^{BC}	19.97^{A}	1.74^{DE}
	140-10-WT	55.87^{A}	0.66^{AB}	71.54^{BC}	16.96^{AB}	1.08^{E}
	140-15-ST	56.11^{A}	0.66^{AB}	81.41^{A}	12.14^{B}	2.69^{CDE}
	140-15-WT	55.04^{A}	0.68^{AB}	76.69^{ABC}	12.02^{B}	0.95^{E}
	160-10-ST	55.78^{A}	0.67^{AB}	75.59^{BC}	13.96^{AB}	3.07^{CD}
	160-10-WT	56.64^{A}	0.64^{B}	76.70^{ABC}	16.67^{AB}	2.15^{DE}
	160-15-ST	54.02^{A}	0.69^{AB}	67.83^{C}	15.28^{AB}	3.25^{CD}
	160-15-WT	56.38^{A}	0.68^{AB}	77.14^{ABC}	13.94^{AB}	1.97^{DE}
	180-10-ST	57.72^{A}	0.73^{A}	87.76^{A}	11.69^{B}	7.67^{A}
	180-10-WT	55.47^{A}	0.70^{AB}	76.06^{ABC}	12.09^{B}	4.44^{BC}
	180-15-ST	56.10^{A}	0.70^{AB}	77.50^{ABC}	12.08^{B}	5.26^{B}
	180-15-WT	55.02^{A}	0.69^{AB}	77.63^{ABC}	12.61^{B}	5.06^{B}

Note: Different letters for each parameter represent statistical differences between different treatments (significance level 0.05)

The final density, the densification percentage, and the spring back for *A. acuminata* were 0.78 g/cm^3, 84.49%, and 7.75% respectively, while no differences were observed between the densification treatments (Table 3).

For *V. ferruginea* the final density was 0.80 g/cm^3 on average and the densification percentage was 76.08%, and no differences between the densification treatments were observed. The spring back was 14.26% with minimal differences between the treatments. Treatment 150-10-WT presented the highest value and 160-15-WT the lowest (Table 3). For *V. guatemalensis*, the final density was 0.68 g/cm^3 on average; 180-10-ST presented the highest value, whereas 140-10-ST and 160-10-WT presented the lowest values. The densification percentage was 76.55%. Treatments 140-15-ST and 180-10-ST showed the highest values, while 160-15-ST had the lowest value. The spring back was 14.12%. Treatment 140-10-ST showed the highest percentage of spring back, while treatments 140-15-ST, 140-15-WT, and all treatments with temperature of 180 °C presented the lowest averages (Table 3).

Color change in wood after densification was more pronounced in *V. ferruginea* with 5.23, followed by *A. acuminata* with 4.18, and *V. guatemalensis* with 2.48 on average (Table 3). Treatments with the temperature of 180 °C for *A. acuminata* and *V. guatemalensis*, and 160 °C for *V. ferruginea* presented higher color changes, while treatments with the temperature of 140 °C for the three species presented the lowest color changes (Table 3).

Evaluation of the maximum load applied to wood in stage 2 of the densification process showed that greater load was applied to *A. acuminata* (201439 N) followed by *V. ferruginea* (175666 N) and next by *V. guatemalensis* (138323 N). *A. acuminata* and *V. ferruginea* showed no differences between the treatments, while *V. guatemalensis* treatment 140-15-WT presented the highest value and 140-10-WT and 160-10-WT the lowest values (Fig. 1 a through c).

Concerning the heating rate, in general, similar averages were observed for the three species: 3.69 °C/min for *A. acuminata*, 3.40 °C/min for *V. ferruginea*, and 3.92 °C/min for *V. guatemalensis*. In addition, densified wood with the highest temperatures, 180 °C for *A. acuminata* and *V. guatemalensis* and 160 °C in *V. ferruginea*, presented the highest heating rates. Moreover, the wood with 10 min compression time showed a higher heating rate than the wood under 15 min compression, while the wood densified without steam showed the highest values of heating rate compared to wood densified with steam (Fig. 1 d through f). As for *A. acuminata* wood, treatments 180-10-ST and 180-10-WT presented higher heating rate values, while 140-15-ST showed the lowest value (Fig. 1d). For *V. guatemalensis* and *V. ferruginea*, 180-10-WT and 160-10-WT showed the highest heating rate and 140-15-ST the lowest (Fig. 1 e and f).

Properties of Densified Wood

Statistical differences were observed between the treatments applied regarding the averages obtained for the properties evaluated in the densified wood (Table 4). The thickness swelling for *A. acuminata* showed an average of 41.71%. Treatment 160-15-ST presented the highest value, while treatment 140-10-ST was the lowest. For *V. ferruginea*, the average thickness swelling was 23.42% for all the densification treatments. Treatment 160-15-WT had the highest value and 140-10-WT the lowest. As for *V. guatemalensis*, treatment 140-15-ST had the highest thickness swelling average, and treatments 160-10-

WT and 180-15-ST the lowest, for an average between the treatments of 20.73% (Table 4). For the three species the thickness swelling in the un-densified wood was less than 1%.

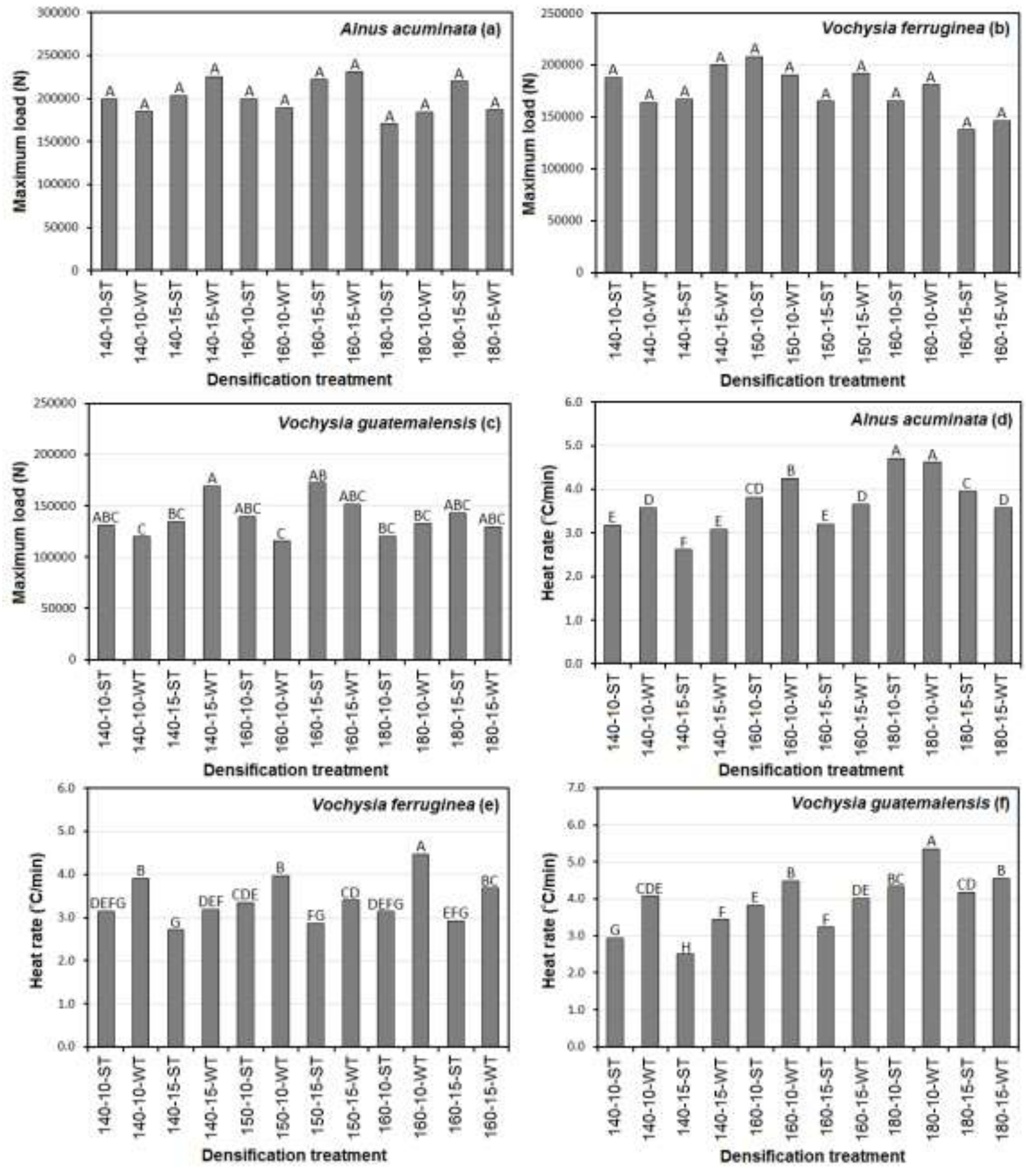

Fig. 1. Maximum load and heating rate during the THM densification process of the wood for *Alnus acuminata* (a and d), *Vochysia ferruginea* (b and e), and *Vochysia guatemalensis* (c and f). Different letters for each parameter represent statistical differences between different treatments (significance level 0.05)

With respect to mechanical properties, no differences in the modulus of elasticity (MOE) and modulus of rupture (MOR) in static bending were observed between treatments for the densified wood of *A. acuminata* (13.18 GPa and 134.22 MPa on average, respectively) (Table 4). Average MOE and MOR for densified wood of *V. ferruginea* was

14.11 GPa and 84.78 MPa, respectively. For MOE, treatment 160-15-WT presented the highest value and treatment 150-10-WT the lowest.

Table 4. Physical and Mechanical Properties of Densified Wood of Three Forest Species

Species	Treatment	Thickness Swelling (%)	MOE in Static Bending (GPa)	MOR in Static Bending (MPa)	Hardness (N)
Alnus acuminata	140-10-ST	37.89^{C}	12.56^{A}	126.17^{A}	7772.26^{A}
	140-10-WT	39.06^{BC}	13.78^{A}	136.91^{A}	8017.61^{A}
	140-15-ST	44.34^{AB}	14.20^{A}	147.60^{A}	6924.15^{A}
	140-15-WT	43.42^{AB}	12.52^{A}	140.57^{A}	7275.96^{A}
	160-10-ST	41.94^{ABC}	12.65^{A}	128.99^{A}	7495.72^{A}
	160-10-WT	44.01^{AB}	13.42^{A}	140.89^{A}	7432.95^{A}
	160-15-ST	44.54^{A}	14.00^{A}	142.15^{A}	7427.59^{A}
	160-15-WT	41.86^{ABC}	12.15^{A}	120.98^{A}	7661.85^{A}
	180-10-ST	39.23^{BC}	12.73^{A}	128.05^{A}	7384.06^{A}
	180-10-WT	40.72^{ABC}	13.95^{A}	145.16^{A}	7046.68^{A}
	180-15-ST	41.26^{ABC}	13.48^{A}	130.42^{A}	8181.47^{A}
	180-15-WT	42.26^{ABC}	12.81^{A}	125.06^{A}	7731.08^{A}
	Un-densified	0.89^{D}	7.94^{B}	62.18^{B}	3042.75^{B}
Vochysia ferruginea	140-10-ST	18.81^{CD}	15.52^{AB}	97.56^{A}	6405.17^{AB}
	140-10-WT	17.04^{D}	13.34^{BC}	96.28^{A}	6406.60^{AB}
	140-15-ST	19.50^{CD}	14.30^{ABC}	89.19^{ABC}	6446.07^{AB}
	140-15-WT	25.79^{BC}	14.30^{ABC}	91.02^{AB}	7392.78^{A}
	150-10-ST	21.96^{BCD}	13.03^{BC}	82.89^{ABCD}	6780.70^{AB}
	150-10-WT	19.51^{CD}	12.49^{C}	89.07^{ABCD}	5610.31^{B}
	150-15-ST	26.28^{BC}	14.90^{ABC}	71.31^{BCD}	6348.66^{AB}
	150-15-WT	28.58^{B}	13.10^{BC}	98.03^{A}	5816.61^{AB}
	160-10-ST	21.45^{CD}	13.86^{ABC}	65.47^{CD}	6098.83^{AB}
	160-10-WT	19.99^{CD}	14.25^{ABC}	86.24^{ABCD}	5824.01^{AB}
	160-15-ST	26.24^{BC}	14.24^{ABC}	78.74^{ABCD}	6053.80^{AB}
	160-15-WT	35.88^{A}	16.25^{A}	64.91^{D}	6145.32^{AB}
	Un-densified	0.83^{E}	9.63^{D}	56.79^{E}	2348.22^{C}
Vochysia guatemalensis	140-10-ST	28.49^{AB}	9.52^{BCD}	99.69^{A}	7186.55^{ABC}
	140-10-WT	20.98^{CD}	9.03^{CDE}	94.47^{AB}	5731.70^{DE}
	140-15-ST	30.25^{A}	9.99^{ABC}	100.22^{A}	8548.87^{A}
	140-15-WT	26.45^{AC}	10.21^{ABC}	107.25^{A}	8317.37^{AB}
	160-10-ST	23.29^{BC}	11.32^{A}	110.81^{A}	7053.08^{BCD}
	160-10-WT	13.71^{E}	7.52^{EF}	70.99^{DE}	7052.22^{BCD}
	160-15-ST	29.05^{AB}	10.88^{AB}	108.81^{A}	6646.33^{CDE}
	160-15-WT	16.60^{DE}	8.97^{CDE}	93.46^{AB}	7419.09^{ABC}
	180-10-ST	15.57^{DE}	8.82^{CDE}	76.65^{BD}	5610.94^{E}
	180-10-WT	15.21^{DE}	7.62^{EF}	75.05^{BD}	5727.97^{DE}
	180-15-ST	12.75^{E}	7.00^{F}	66.77^{DE}	5667.80^{DE}
	180-15-WT	16.42^{DE}	8.96^{CDE}	78.83^{BD}	5344.20^{E}
	Un-densified	0.67^{F}	8.14^{DEF}	52.15^{E}	2008.17^{F}

Different letters for each parameter represent statistical differences between different treatments per specie (significance level 0.05)

All treatments increased MOE and MOR in flexion and hardness values in *A. acuminate* densified wood (Table 4). For densified wood of *V. guatemalensis,* treatment 160-10-ST showed the highest average MOE and treatments with low temperature (140-160 °C) obtained the highest MOR in flexion (Table 4). In *V. guatemalensis*, the averages for un-densified wood were below the averages obtained for densified wood in the 12 treatments used, except to180-10-WT and 180-15-ST for MOE (Table 4).

For hardness test, the un-densified wood had the statistically lower value in *V. ferruginea* (Table 4), and there were few differences between the densification treatments. In the wood of *V. guatemalensis* there were many differences between the treatments. Treatment 140-15-ST had the highest value, and the un-densified wood had the statistically lowest average compared to densification treatments (Table 4).

Relationship Between Variables

Regarding the relationships between the initial characteristics of the wood and the variables obtained in the densification process, the initial density had the highest number of correlations with the variables obtained in the wood densification process for all three wood types (Table 5). In *A. acuminata,* the initial thickness of the wood presented a positive correlation with the degree of compression, and a negative correlation with the heating rate (Table 5). The initial density was related to almost all the variables of the densified process, except for color change. A positive correlation was observed between the initial density, final density, the maximum load, and the spring back. A negative correlation was observed with the compression ratio, the densification percentage, and the heating rate (Table 5). An aspect to highlight in *A. acuminata* is that the initial moisture content did not show any relationship with the variables obtained in the densification process (Table 5).

For the *V. ferruginea* densified wood, the initial thickness showed a positive correlation with the compression ratio. Initial density correlated to almost all the variables in the densification process, except for the heating rate. A positive correlation was observed with the final density, color change, maximum load, and spring back. A negative correlation was shown with the compression ratio and the densification percentage. The initial moisture content correlated negatively with color change and positively with the heating rate (Table 5).

For the *V. guatemalensis* wood, the initial thickness showed positive correlation with the final density and negative correlation with the heating rate. The initial density correlated to almost all the variables in the process of densification except color change and correlated negatively with all the remaining variables. Moisture content correlated positively with the densification percentage and with the heating rate (Table 5).

As for the relationship between the variables obtained in the densification process and the wood properties, the final density presented the greatest number of correlations with the wood properties for the three species (Table 6). In *A. acuminata* the thickness swelling and the MOE in static bending, showed a positive correlation with the compression ratio, the final density, and the densification percentage, and a negative correlation with the spring back. As for the MOR in static bending and hardness, both correlated positively with the final density and maximum load (Table 6).

Table 5. Correlation Coefficients Between the Initial Characteristics of the Wood and the Variables Obtained in the THM Densification Process

Species	Variable	Compression Ratio	Final Density	Densification Percentage	Spring Back	Color Change	Maximum Load	Heating Rate
Alnus acuminata	Initial Thickness	0.56**	0.08^{ns}	0.07^{ns}	-0.04^{ns}	0.06^{ns}	0.11^{ns}	-0.19**
	Initial Wood Density	-0.16*	0.73**	-0.39**	0.15*	-0.03	0.77**	-0.14*
	Initial Moisture Content	0.08^{ns}	-0.07^{ns}	-0.02^{ns}	0.06^{ns}	0.06^{ns}	-0.06^{ns}	-0.05^{ns}
Vochysia ferruginea	Initial Thickness	0.73**	0.09^{ns}	0.10^{ns}	-0.02^{ns}	0.02^{ns}	0.09^{ns}	0.01^{ns}
	Initial Wood Density	-0.15*	0.81**	-0.35**	0.27**	0.14*	0.73**	0.10^{ns}
	Initial Moisture Content	0.11^{ns}	-0.02^{ns}	-0.02^{ns}	0.04^{ns}	-0.17**	-0.08^{ns}	0.17**
Vochysia guatemalensis	Initial Thickness	0.07^{ns}	0.16**	0.04^{ns}	0.10^{ns}	0.04	0.07^{ns}	-0.15*
	Initial Wood Density	-0.33**	0.73**	-0.46**	0.26**	0.10	0.73**	0.08^{ns}
	Initial Moisture Content	-0.03^{ns}	0.06^{ns}	0.13*	-0.13^{ns}	-0.09^{ns}	0.03^{ns}	0.22**

**: Statistically significant level 0.1; *: statistically significant level 0.05; ^{ns}: not significant

Table 6. Correlation Coefficients Between the Variables Obtained in the THM Densification Process and the Densified Wood Properties

Species	Variable	Compression Ratio	Final Density	Densification percentage	Maximum Load	Heating Rate	Spring Back
Alnus acuminata	Thickness swelling	0.13*	0.19**	0.56**	0.10^{ns}	-0.09^{ns}	-0.62**
	MOE in static bending	0.16*	0.33*	0.15*	0.14*	0.03^{ns}	-0.14*
	MOR in static bending	0.03^{ns}	0.37**	-0.01^{ns}	0.22**	0.01^{ns}	-0.05^{ns}
	Hardness	0.02^{ns}	0.50**	-0.08^{ns}	0.47**	0.01^{ns}	-0.05
Vochysia ferruginea	Thickness swelling	-0.15*	-0.23**	0.11^{ns}	-0.18*	-0.07^{ns}	-0.21**
	MOE in static bending	0.01^{ns}	0.16*	0.35**	-0.03^{ns}	-0.12^{ns}	-0.44**
	MOR in static bending	0.01^{ns}	0.18**	-0.29**	0.35**	0.08^{ns}	0.33**
	Hardness	0.02^{ns}	0.44**	-0.00	0.32**	-0.12^{ns}	-0.13^{ns}
Vochysia guatemalensis	Thickness swelling	0.05^{ns}	-0.08^{ns}	0.12^{ns}	0.10^{ns}	-0.63**	-0.06^{ns}
	MOE in static bending	-0.05^{ns}	0.37**	-0.08^{ns}	0.35**	-0.42**	-0.01^{ns}
	MOR in static bending	-0.20**	0.40**	-0.30**	0.49**	-0.38**	0.11^{ns}
	Hardness	-0.05^{ns}	0.39**	-0.05	0.55**	-0.40**	0.01^{ns}

**: Statistically significant level 0.1; *: statistically significant 0.05; ^{ns}: not significant

For *V. ferruginea*, the thickness swelling had a negative correlation with the compression ratio, final density, maximum load, and spring back. The MOE in static bending had a positive correlation with the final density and the densification percentage, and a negative correlation with the spring back.

The MOR in static bending had a positive correlation with the final density, maximum load, and spring back, and a negative correlation with the densification percentage. The hardness had a positive correlation with the densification percentage and maximum load (Table 6).

In the densified wood of *V. guatemalensis* the thickness swelling only showed a negative correlation with the heating rate. The MOE in static bending had a positive correlation with the final density and the maximum load, and a negative correlation with the heating rate. The MOR in static bending showed a positive correlation with the final density and maximum load, and a negative correlation with the compression ratio, the densification percentage, and the heating rate. Hardness had a positive correlation with the final density and the maximum load, and there was not any correlation found with other variables (Table 6).

Microscopic Evaluation

Cross-sections of the un-densified and densified specimens' anatomy features of the three species are presented in Fig. 2. The three species presented solitary and multiple diffuse vessels (Fig. 2a, e, and i). According to previous anatomical description in the same species (Moya *et al*. 2019), *A. acuminata* presents a pore frequency of 16 pores/mm^2 with small diameters (75 µm) and short lengths (150 µm). *V. ferruginea* presents 2.84 pores/mm^2 of medium diameter (145 µm) and length (346 µm). *V. guatemalensis* has 2.88 pores/mm^2, with regular size diameters (169 µm) and length (339 µm). Fibers in the three species are irregularly arranged in rows perpendicular to the growth rings or parallel to ray parenchyma, which is typical of non-stored fibers. The rows are crooked due to the presence of large vessels. *A. acuminata* formed more uniform fiber rows than the other two species. *A. acuminata* features finer rays than those of the other two species, which are multi-serial, showing 44 to 8 series per ray (Moya *et al*. 2019).

In the cross-sections of densified wood of the three species, the effect of the densification process can be perceived in the size of their anatomical structures and the differences in the vessels. The vessels tended to collapse and flatten completely in the case of *A. acuminata* (Fig. 2d). For the other two species, the vessels tended to collapse but not flatten completely (Fig. 2h and i). For the three species, the vessels formed a row horizontal or perpendicular to the application of the compression strength (Fig. 2b, f, and j). The deformation occurring in the vessels caused the close rays to collapse and lose their original shape in the three species. Most fibers tended to make an "S" shape, especially those that were close to the collapsed vessels (Fig. 2c, d, g, h, k, and l). In the case of the rays of *A. acuminata*, the frequency of the waves was greater than those of *V. guatemalensis* and *V. ferruginea*.

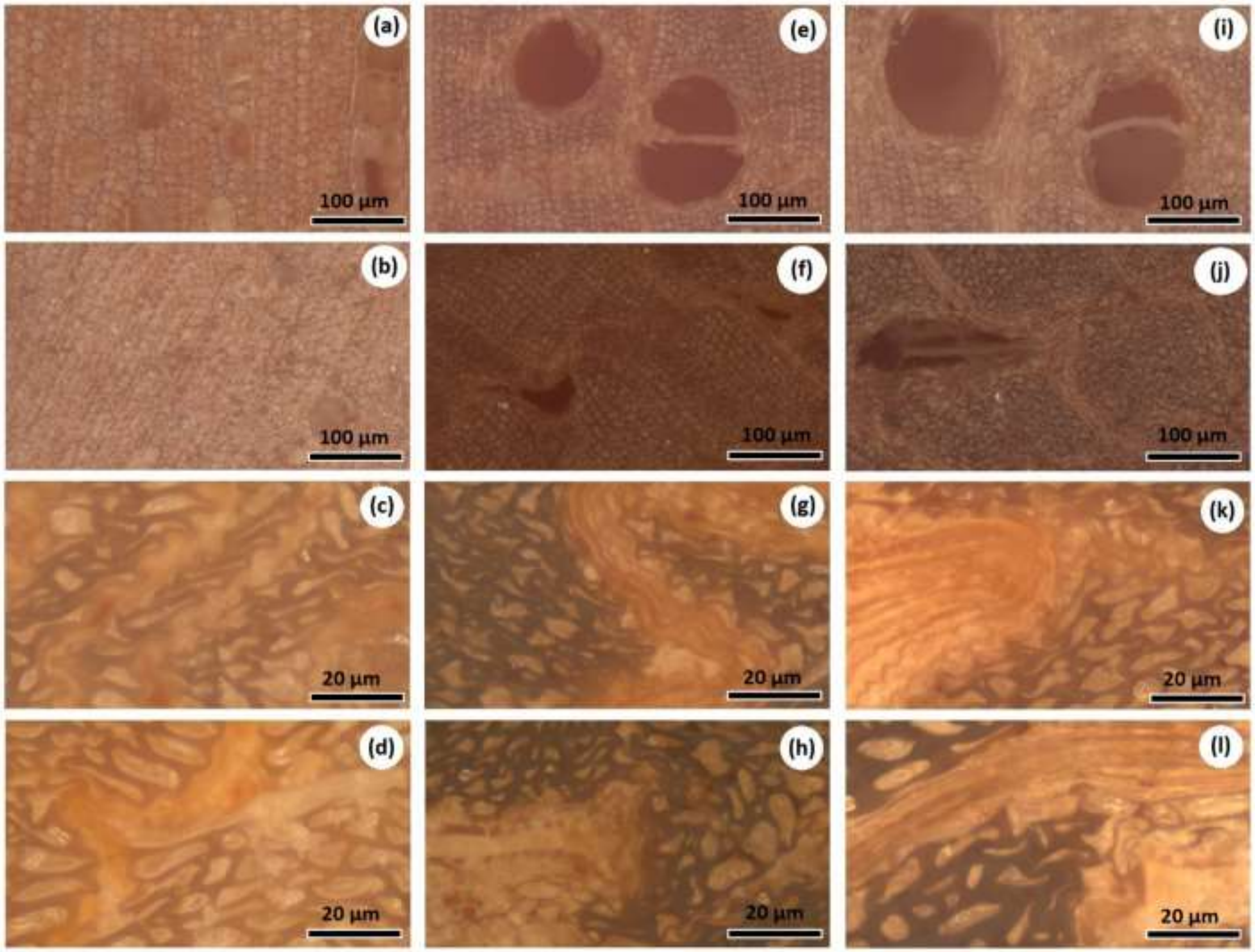

Fig. 2. Anatomical features of un-densified and densified wood of *Alnus acuminata* (a, b, c, and d), *Vochysia ferruginea* (e, f, g, and h), and *Vochysia guatemalensis* (i, j, k, and l)

DISCUSSION

THM Densification Process Evaluation

For *A. acuminata* and *V. ferruginea*, the treatments (Table 2) used in the densification process had no influence on compression ratio, final density, densification percentage, and maximum load applied during stage 2 (Table 3 and Fig. 1). In the case of *V. guatemalensis*, some differences were noticed between treatments in the parameters evaluated in the densification process (Table 3). However, it was not possible to identify any behavioural patterns related to the treatments (Table 3, Fig. 1). Only in variables such as color change and heating rate was there an influence of densification temperature noticed for the three species (Table 3 and Fig. 1).

This demonstrated that other variables influence the densification process of these species, as is the case of the initial density of wood and the anatomical features (Table 5 and Fig. 2). The effect of the initial density was evidenced by the correlation analysis performed between the initial characteristics of the wood of the three species and the variables evaluated in the densification process. Where the initial density had a high positive correlation with the final density and the maximum applied load ($R^2 > 0.73$), and a negative correlation with the compression ratio and densification percentage (Table 5).

The results above show that the initial density is one of the most important factors in the densification process of the three species. This was to be expected because the moisture content and the initial thickness of the wood were controlled before the process. In contrast, the wood density before densifying is a harder feature to control because of its variability within the tree (Zobel and Van Buijtenen 1989), especially when dealing with wood from tropical climate species, which feature high variability radially and throughout the trunk (Moya and Muñoz 2010; Tenorio *et al*. 2016).

The distribution and size of the vessels, fibers, and the characteristics of the rays of each of the species additionally affect the densification process, especially in stage 2 of the process. Wang and Cooper (2005) pointed out that transversal compression of the wood is highly dependent on its anatomical structure. During radial compression, the weakest part of the wood deforms much faster and more dramatically. For hardwoods, the weakest part is the vessels and the compression of the wood depends on the frequency and size of them. *A. acuminata* has a higher frequency of smaller vessels (Fig. 2a) in relation to the other two species (Fig. 2e and i), which during the densification process tend to collapse and flatten more (Fig. 2c and d) than the vessels of the other two species, causing this species to present a higher densification percentage (Table 3). The vessels of the other two species, that are larger in diameter, tend to be less flattened and have a lower densification percentage (Table 3).

As expected, color change of the wood of the three species was higher in treatments with higher temperatures (Table 3). Temperature has a direct influence on wood color. It affects its chemical composition (Pohleven *et al*. 2019) and causes wood darkening (Salca *et al*. 2016). The color changes obtained after wood densification were caused by the hydrolysis of the hemicelluloses (Candelier *et al*. 2016), mainly on the wood surfaces in contact with the metal plates responsible for transmitting heat. The heat of the metal plates causes a decrease in luminosity (L^*), which can be attributed to the degradation or modification of components through reactions, such as oxidation, dehydration, decarboxylation, and hydrolysis (Kocaefe *et al*. 2008), as well as lignin darkening, which is associated with the generation of chromophoric groups (Salca *et al*. 2016), which cause darker color at higher temperatures (Table 3, Fig. 1).

Like the color change, the heating rate was higher in treatments with higher temperatures, in treatments when 10 min compression was used in stage 2 of the process, and when no steam was applied (Fig. 1). This suggested that better conditions could be achieved to reach uniformity in the densification of the cross-section of the wood with these treatments. Likewise, the correlation analysis performed (Table 5) indicated that the heating rate was influenced by the initial thickness of the wood, the moisture content, and the initial density (Table 5). Therefore, the propagation of heat is faster in wood with lower density, lower thickness, and higher moisture content. The fast propagation of heat in the wood is an important factor in the densification process (Wu *et al*. 2019). The internal parts quickly reach the appropriate temperatures so that the hydrogen bonds in the hemicelluloses, in the amorphous areas of the cellulose, and the lignin bonds achieve the appropriate stick-slap to reach visco-elastic deformation of the anatomical elements of the wood and thus adequate densification (Bao *et al*. 2017).

The spring back was much lower in *A. acuminata* than in *V. ferruginea* and *V. guatemalensis* (Table 3). Some studies reported that the elastic-strain energy stored in the semi crystalline micro fibrils and lignin of wood is the main cause of the recovery in the THM process (Navi and Heger 2004; Inoue *et al*. 2008). During this process, the bond between the microfibers and lignin is weakened under the action of temperature and moisture presented in the THM process, and then there is a freeing of the internal stress (Liu *et al.* 2014). In this case, it is possible that the wood of *A. acuminata* has a lower hydrolysis of hemicellulose so that the release of the elastic-strain is lower and therefore its stabilization recovery.

Other studies indicated that there was a significant reduction in the spring back in the THM process when a high compression ratio occurs, as a result of the rupture of cross-links responsible for the memory effect in wood, and lignin softening (Inoue *et al*. 2008; Darwis *et al*. 2017). However, in this case there were no differences in the compression ratios between treatments, while between species the compression ratios were similar. However, the densification percentage of *A. acuminata* was higher (84.49%) compared to *V. ferruginea* (76.08%) and *V. guatemalensis* (76.55%). This approximately 8% higher densification in *A. acuminata* may be the cause of a lower spring back (Table 3).

Properties of the Densified Wood

With respect to evaluation of the properties of the densified wood, thickness swelling in the three species presented many differences between the treatments (Table 4). Importantly, none of the treatments reached 100% thickness recovery. The values of thickness swelling of the densified wood of *A. acuminata* doubled those of the other two species (Table 4), which was not to be expected given that *A. acuminata* presented greater densification percentage and lower spring back than those of *V. ferruginea* and *V. guatemalensis* (Table 3).

The high values of thickness swelling obtained for *A. acuminata*, despite its high densification percentage, may be the result of the compression of its anatomical structures in the densification process (Fig. 2). This species has a higher frequency of vessels with diameter and length smaller (Fig. 2a) than those of *V. ferruginea* (Fig. 2e) and *V. guatemalensis* (Fig. 2i). Thus, when the densified wood of *A. acuminata* was subjected to changes in temperature and humidity, this greater number of vessels tended to absorb more moisture and were able to recover their thickness, contrary to the vessels of *V. ferruginea* and *V. guatemalensis*, which although they are larger in diameter are less frequent in relation to those of *A. acuminata*, so the moisture they absorb could be less (Table 3).

Regarding the mechanical properties, an improvement was observed in most of the properties analysed in relation to the un-densified wood for the three species, with the exception of the MOE in static bending in some treatments of *V. guatemalensis* (Table 4). This showed that the process successfully improved the mechanical properties of the wood and that the results can be attributed to the densification of the cells during the THM densification process, which translates into an increase in density and therefore an improvement in the hardness property (Tu *et al*. 2014). Keckes *et al*. (2003) pointed out, in relation to the change in the mechanical properties of densified wood, that deformation during densification does not deteriorate cell stiffness, because during the compression process the amorphous regions redistribute, and in the absence of deterioration, densified wood tends to improve its properties in relation to un-densified wood.

The correlation analysis between the properties of the densified wood and the process variables (Table 6) showed that the final density of the wood presented more correlations with the properties of the wood. The final density was positively correlated with the MOE and MOR in static bending and with the hardness of the three species (Table 6). Some authors pointed out that there is a relationship between the increase in MOE and MOR and the increasing compression ratio (Bao *et al*. 2017). However, it should not be affirmed that the increase in the mechanical properties of densified wood was a product of the compression ratio obtained, because correlation analyses only relate the compression ratio with the MOE in static bending of *A. acuminata* (Table 6). In addition, the compression ratio was similar between species as it was derived from the target thickness of the densification process. The above statements indicate that in a densification process that started with wood of a uniform thickness and a certain target thickness, the mechanical properties were governed by the final density obtained from the process and not by the process parameters, such as compression ratio, densification percentage, heating rate, or spring back.

CONCLUSIONS

1. According to the results obtained, the densification process carried out allowed densifying the wood of *A. acuminata* with a densification percentage greater than 80% and the wood of *V. ferruginea* and *V. guatemalensis* with a densification percentage greater than 70%. The densification process of the three species was influenced by the initial density of the wood. The treatments used only influenced variables, such as heating rate and color change, where the treatments with higher temperatures had the highest values.
2. Although the *A. acuminata* wood had a higher densification percentage and a lower spring back, it obtained the highest percentage of thickness swelling. This was a possible consequence of the behaviour of its anatomical structure during the compression stage. Regarding the mechanical properties, there was an increase in the MOE and MOR in static bending and in the hardness of the densified wood in relation

to the un-densified wood of the three species. The treatments used showed positive correlation of these mechanical properties with the final density and the maximum load.

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