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DENSIFICACIÓN SUPERFICIAL DE MADERAS DE PLANTACIONES FORESTALES PARA USOS EN PISOS

(DOCUMENTO I)

INVESTIGADORES:

Roger Moya R, Ph.D.
Carolina Tenorio M, M. Eng.
Angel Navarro Mora, M.Sc.

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ÍNDICE GENERAL

Densificación superficial de maderas de plantaciones forestales para usos en pisos

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1. **Autores:**
   1.1. Ing. Roger Moya R, Ph.D. (Coordinador).
   1.2. Ing. Carolina Tenorio M, M. Eng.
   1.3. Ing. Angel Navarro, M.Sc.

2. RESUMEN

El factor más importante al escoger una madera para piso es su densidad. En Costa Rica las maderas comúnmente utilizadas para piso se encuentran vedadas, en peligro de extinción o con restricción de corta, lo anterior ha conducido a la búsqueda de nuevas oportunidades que permitan su reemplazo por especies de menor densidad cuyas propiedades mecánicas tienden a ser bajas. El proceso de densificación hace posible que maderas de baja o moderada densidad sean modificadas para alcanzar los requerimientos de dureza necesarios para su uso. Debido a lo anterior el objetivo de este proyecto fue aumentar la densidad de la madera de tres especies de plantaciones forestales (*Alnus acuminata*, *Vochysia guatemalensis* y *Vochysia ferruginea*) mediante un tratamiento termo-hidro-mecánico y conocer los cambios en las características y propiedades de la madera ya densificada y evaluar su posible uso en pisos. Se desarrolló un sistema de densificado con la capacidad de controlar la aplicación de temperatura, carga (compresión) y vapor, mediante la adaptación de un aditamento a una máquina universal de ensayos. Se analizaron variables propias del proceso de densificado y en la madera densificada se determinaron propiedades físicas, mecánicas y cambios en su anatomía y en su perfil de densidad y se evaluó su comportamiento en aplicaciones de piso. El proceso de densificación realizado permite densificar la madera de *A. acuminata* con porcentajes de densificación mayores al 80 % y para la madera de *V. ferruginea* y *V. guatemalensis* mayores al 70 %. Se observó un aumento en los módulos de elasticidad y ruptura en flexión estática y en la dureza de la madera densificada en relación a la madera sin densificar para las tres especies. La madera densificada de las tres especies presentó cuatro perfiles de densidad como consecuencia de la temperatura de densificación y de la anatomía inicial que presentaba la madera. En cuanto a la evaluación en aplicaciones de piso, se observó que la madera densificada de *V. ferruginea* tiende a ser más frágil que la de las otras dos especies, provocando que presente un comportamiento más pobre en la mayoría de los ensayos. Los resultados sugieren que cada especie posee un comportamiento único en el proceso de densificación debido a sus características intrínsecas, por lo que para desarrollar este tipo de procesos es necesario un amplio conocimiento de las propiedades iniciales de la madera y su anatomía.

Abstract

Density is the most important factor when choosing a wood for flooring. In Costa Rica, the wood commonly used for flooring are in danger of extinction or with cutting restrictions, these has led to the search for new opportunities that allow its replacement by species of lower density whose mechanical properties tend to be low. The densification process makes it possible for wood of low or moderate density to be modified to reach the hardness requirements necessary for its use. The objective of this project was to increase the density of the wood of three species of forest plantations (*Alnus acuminata*, *Vochysia guatemalensis* and *Vochysia ferruginea*) by a thermo-hydro-mechanical treatment and to know the changes in the characteristics and properties of wood already densified and evaluate its possible use in floors. A densified system was developed with the ability to control the application of temperature, load (compression) and steam, by adapting an attachment to a universal testing machine. Variables specific to the densification process were analyzed and in the densified wood physical, mechanical properties, changes in their anatomy and density profile were determined and their behavior in floor applications was evaluated. The densification process carried out allows densification of *A. acuminata* wood with densification percentages greater than 80% and for *V. ferruginea* and *V. guatemalensis* wood greater than 70%. An increase was observed in the modulus of elasticity and rupture in static flexion and in the hardness of densified wood in relation to un-densified wood for the three species. The densified wood of the three species presented four density profiles as a result of the densification temperature and the initial anatomy of the wood. Regarding the evaluation in floor applications, it was observed that the densified wood of *V. ferruginea* tends to be more fragile than that of the other two species, causing it to exhibit a poorer behavior in most trials. The results suggest that each species has a unique behavior in the densification process due to its intrinsic characteristics, so in order to develop this type of processes it is necessary to have a broad knowledge of the initial properties of the wood and its anatomy.

3. PALABRAS CLAVE

Especies tropicales, latifoliadas, baja densidad, proceso termo-hidro-mecanico.

Key words: tropical species, hardwoods, low density, thermo-hydro-mechanical process

4. INTRODUCCIÓN

La madera como un elemento constructivo proporciona una estética agradable, aislamiento térmico, resistencia mecánica, es fácil de mantener y con los cuidados adecuados presenta una larga duración (Blanco-Flórez et al. 2015). No obstante, al ser utilizada en pisos, comparándola con otros materiales, la madera está sujeta a daños por objetos, degaste de elementos abrasivos y por tráfico de personas, entre otros. El factor más importante al escoger una madera para piso es su densidad. Existe una alta correlación entre la densidad de la madera y sus propiedades mecánicas, una alta densidad significa una mayor resistencia mecánica y por lo tanto una mayor dureza (Sadatnezhad et al. 2017). Es por esto que la madera solida proveniente de especies de alta densidad ha sido ampliamente utilizada para pisos.

En Costa Rica en la actualidad las maderas comúnmente utilizadas para piso se encuentran vedadas, en peligro de extinción o con restricción de corta (Moya et al. 2013). Esto ha provocado que los precios de los pisos de madera sean elevados, promoviendo la importación de maderas o productos a base de madera para satisfacer las demandas de este sector (Serrano y Moya, 2011). Lo anterior ha conducido a la búsqueda de nuevas oportunidades que permitan el reemplazo de maderas de especies de alto valor por especies de menor valor o marginales (Nölte et al. 2018). Sin embargo, su densidad y sus propiedades mecánicas tienden a ser bajas debido a que los árboles son cosechados mientras aún son jóvenes y la proporción de madera juvenil tiende a ser dominante (Zobel and van Buijtenen 1989; Kamke 2006; Cahyono et al. 2015).

Alnus acuminata, *Vochysia ferruginea* y *Vochysia guatemalensis* se caracterizan por ser maderas latifoliadas de rápido crecimiento utilizadas en los programas de reforestación comercial de Costa Rica (Moya 2018). Poseen madera de baja densidad, son suaves y fáciles de trabajar (Tenorio et al. 2016). Sin embargo, sus propiedades mecánicas son bajas y por ende no son consideradas para usos estructurales por lo que sus usos se limitan a productos de baja demanda estructural y para mercados de bajo valor comercial (Serrano and Moya 2011). Es debido a las bajas propiedades mecánicas de la madera de estas especies que han sido poco estudiadas y que de cierta forma han sido ignoradas por las industrias. En la actualidad, es posible encontrar estudios sobre las propiedades y el comportamiento en procesos industriales de estas y otras especies de reforestación en Costa Rica (Moya et al. 2019). No obstante, hasta el momento se carece de estudios que busquen mejorar de sus propiedades mecánicas.

Muchos procesos de densificación de madera han sido desarrollados para mejorar sus propiedades mecánicas y físicas (Fang et al. 2012). Estos procesos pueden aumentar la densidad de la madera mediante tres formas: (i) por compresión mecánica reduciendo los espacios vacíos, (ii) mediante la impregnación de los espacios vacíos con alguna sustancia, (iii) o utilizando una combinación de compresión mecánica e impregnación (Fang et al. 2012). Sin embargo, a diferencia de la densificación por compresión mecánica, la impregnación química afecta el carácter natural y sustentable de la madera y suele ser más caro (Navi and Heger 2004). La compresión mecánica combinada con vapor y calor llamada densificación termo-hidro-mecánica ha sido estudiada como una alternativa ambientalmente amigable para aumentar la densidad de la madera y mejorar sus propiedades mecánicas logrando una mejor estabilidad dimensional sin necesidad del uso de productos químicos (Bekhta et al. 2009; Büyüksari et al. 2012; Candan et al. 2013; Arruda and Del Menezzi 2013; Tu et al. 2014).

Existen diferentes procesos de densificación termo-hidro-mecánica, pero en general consisten de: (i) suavizar la estructura de la madera, lo cual se puede lograr a ciertas temperaturas y contenidos de humedad, (ii) comprimir la madera, generalmente se realiza entre dos platos de metal y (iii) mantener la deformación obtenida en la madera, que en muchos casos se obtiene mediante modificación térmica (Sandberg et al. 2013). El proceso de densificación termo-hidro-mecánica mejora las propiedades naturales de la madera y produce materiales más estables (Navi and Heger 2004; Sandberg et al. 2013). El tratamiento térmico puede mejorar la resistencia a la descomposición (Huang et al. 2012), disminuye la higroscopicidad (Metsä-Kortelainen et al. 2006) y mejora la estabilidad dimensional (Esteves et al. 2007). La humedad induce un efecto de mechano-sorption y suaviza la madera para permitir la compresión mecánica sin que se produzcan fracturas de las paredes celulares (Bao et al. 2017).

La mayoría de investigaciones sobre densificación termo-hidro-mecánica han sido realizadas en maderas de especies coníferas y en sistemas cerrados (Navi and Heger 2004). Por lo que este tipo de investigaciones son escasas en maderas tropicales latifoliadas, cuyas estructuras anatómicas influyen en mayor medida en el resultado del proceso (Navi and Heger 2004), ya que las propiedades de compresión de la madera dependen de la frecuencia, tamaño y distribución de sus estructuras anatómicas (Darwis et al. 2017), las cuales en el caso de las maderas latifoliadas se encuentran dominadas por vasos, fibras y radios dispuestos en matrices más complejas (Gibson 2012) que las fibro-traqueidas de las coníferas (Fratzl and Weinkamer 2007).

Debido a lo anterior el objetivo de este proyecto aumentar la densidad de la madera de tres especies de plantaciones forestales (*Alnus acuminata*, *Vochysia guatemalensis* y *Vochysia ferruginea*) mediante un tratamiento termo-hidro-mecánico y conocer los cambios en las características y propiedades de la madera ya densificada y evaluar su posible uso en pisos. Se desarrolló y evaluó un sistema de densificado con la capacidad de controlar la aplicación de temperatura, carga (compresión) y vapor, mediante la adaptación de un aditamento a una máquina universal de ensayos de 60 ton (Artículo 1). Se analizaron variables propias del proceso de densificado y en la madera densificada se determinaron propiedades físicas, mecánicas (Artículo 2) y cambios en la micromorfología (anatomía) de la madera y en el perfil de densidad de la madera densificada (Artículo 3). Finalmente, se evaluó el comportamiento que presentaba la madera densificada en aplicaciones de piso (Artículo 4). Los resultados obtenidos permitirán que especies con propiedades mecánicas pobres puedan ser utilizadas en productos de alto valor como por ejemplo en piso, si son modificadas mediante procesos de densificación termo-hidro-mecánica.

5. ARTÍCULOS CIENTÍFICOS:

Artículo 1: Development of a thermo-hydro-mechanical wood densification system evaluated in Vochysia guatemalensis from forest plantations in Costa Rica

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Carolina Tenorio[1] and Roger Moya[2]

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Development of a Thermo-Hydro-Mechanical Device for Wood Densification Adaptable to Universal Testing Machines and Its Evaluation in a Tropical Species

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Carolina Tenorio[1] and Roger Moya[2]

Development of a Thermo-Hydro-Mechanical Device for Wood Densification Adaptable to Universal Testing Machines and Its Evaluation in a Tropical Species

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[1] Escuela de Ingeniería Forestal, Instituto Tecnológico de Costa Rica, PO Box 159-7050, Cartago, Costa Rica, https://orcid.org/0000-0003-2901-7079

[2] Escuela de Ingeniería Forestal, Instituto Tecnológico de Costa Rica, PO Box 159-7050, Cartago, Costa Rica (Corresponding author) e-mail: rmoya@itcr.ac.cr, https://orcid.org/0000-0002-6201-8383

ABSTRACT

There is considerable interest in the development of thermo-hydro-mechanical (THM) treatments to increase the density of low-density woods. However, the development of processes for improving the properties of low-density woods in regions with lower investments in investigations and development, such as tropical areas, is often limited by the equipment and infrastructure constraints. The aim of this work is to outline the development and construction of an inexpensive THM densification device (THM device) that controls the application of heat, load, and steam, by adapting an accessory to a 60-Mg universal testing machine (UTM). The THM device was evaluated using the *Vochysia guatemalensis* wood (tropical species). The inexpensive densification THM device uses steaming, temperature, and compression and can be adaptable to a 60-Mg-capacity UTM. It also allowed us to successfully conduct the densification process on 20 by 70 by 300 mm wood samples of a low-density tropical species. However, there were some drawbacks discovered: for example, the steam application, pressure, and temperatures were controlled independently, which was not convenient. It would be more convenient to concentrate them in a single control unit. Additionally, a high variation was observed in the compression and stabilization thickness.

Keywords

density evaluation, testing machine, fast-growth plantation, tropical species

Introduction

Wood properties are correlated with density.[1,2] For example, a high density is essential for structural applications in which mechanical resistance is an important factor.[3] Because an increase in the density improves the mechanical and hardness properties of the wood,[1] several processes have been developed to improve them.[4,5] Densification allows for the replacement of high-density woods by low-density, commercially unattractive woods[6] that are modified and used in high-value-added products.[7,8]

Many densification processes have been developed to improve the mechanical properties of wood. The first process has been used since the beginning of the last century.[9] However, after a century of investigations, there has been a consensus on the thermo-hydro-mechanical (THM) densification method—which utilizes compression combined with heat and steam application—as one of the most appropriate methods for wood densification when considering the dimensional stability and economic aspects.[10–12] This method increases the wood density by reducing the empty spaces within the anatomical structure of the wood.[9] The interest in wood densification has grown recently because of competition from other materials and the reduced structural quality of wood.[13]

On the other hand, there is limited investment in research and development of the wood densification processes in some universities and research centers, especially in the tropical areas, which limits the availability of economic resources and equipment needed to improve the properties of wood.[14] For example, Fang et al.[9] describe the densification process for veneer in Quebec, Canada. They use expensive and specialized press, which is difficult to access in tropical area countries. Recently, Kamke and Rathi[15] and Liu, Kamke, and Guo[16] developed an expensive and modern press for the densification of samples with viscoelastic thermal compression. Using this approach, the densification process was performed adequately, and it gave excellent results in the densification of wood.

In addition, because of the limited infrastructure of many of these centers, many of the tests have to be sent to universities located in developed countries; hence, the numbers and dimensions of the samples must be reduced. The lack of infrastructure, therefore, becomes a problem in the study of wood species in regions with limited economic resources. In order to increase the potential for studying the species of these regions, the development of inexpensive equipment that can be built with appropriate resources and coupled to the existing infrastructure at university campuses in tropical regions is fundamental.

In order to promote the use of less frequently utilized low-density wood species in regions with lower investment in investigation and development, it is necessary to develop processes to improve their wood properties, such as densification. However, there is a lack of equipment and infrastructure supporting the improvement of wood properties in research centers. The objective of this work is to develop and build an inexpensive THM densification device that controls the application of heat, load, and steam by adapting an accessory to a 60-Mg universal testing machine (UTM). This THM device presents the basic concepts necessary to build a THM device and evaluates its performance on a tropical species (*Vochysia guatemalensis*).

Materials and Methods

DENSIFICATION SYSTEM DESIGN

A 60-Mg Tinius Olsen UTM was used, model Super L60 (Tinius Olsen, Horsham, PA, USA). A THM device was coupled to the UTM to densify 70-mm-wide and 300-mm-long wood samples. The THM device allowed load application by compressing the samples at set times and speeds for each test. In addition, the THM device supplied low-pressure steam to both faces of the wood for a specified time, as well as a rapid temperature control, quickly reaching 100°C to 200°C and remaining stable throughout each test. Lastly, the THM device recorded the temperature and load values applied to the wood samples relative to time. It is important to mention that the THM device is removable, so the UTM can still serve its original function.

The proposed densification THM device was divided into two parts: (1) the densification unit, which includes the aforementioned compression, steaming, and temperature applications, and (2) the control unit, which controls all the variables involved in the densification unit. In addition, the variation of the internal temperature of the wood and its thickness with respect to time were also recorded.

PROVENANCE AND CHARACTERISTICS OF THE WOOD

Once the densification system was designed and manufactured, it was tested on *V. guatemalensis*, a tropical low-density broadleaf species. *V. guatemalensis* features anatomical characteristics and chemical compositions different from those of conifers, which are the species commonly utilized in wood densification. The wood employed in this study came from a forest plantation located in the northern region of Alajuela in Costa Rica belonging to Maderas Cultivadas de Costa Rica S.A. (https://www.maderascultivadas.com/). The trees utilized were approximately eight years old. According to Tenorio et al.,[17] this species presents a specific gravity of 0.32, which is considered low. The wood is whitish, with diffused porosity, large and mostly simple vessels, and multiple 2- to 3-cell pores. Relative to other structures, the rays are medium sized (2–4 series) and moderately abundant. The parenchyma is scarce vasicentric, short-winged and aliform, lozenge and confluent. In addition to its low density, *V. guatemalensis* was chosen because it is widely used in commercial reforestation in Costa Rica.[17]

EVALUATION OF THE DENSIFICATION SYSTEM

Test Conditions

In the initial stage, previous to the compression of the wood, three temperatures (140°C, 160°C, and 180°C) and two densification times (10 and 15 min) were used. A water steam application was also occasionally used. Thus, the work was conducted with 12 treatments, and there were 20 samples per treatment, resulting in 240 samples in total. The wood samples were 300 by 70 by 20 mm, with a tangential pattern and equilibrium moisture content (EMC) of 12 %.

Wood Characteristics Before Densification

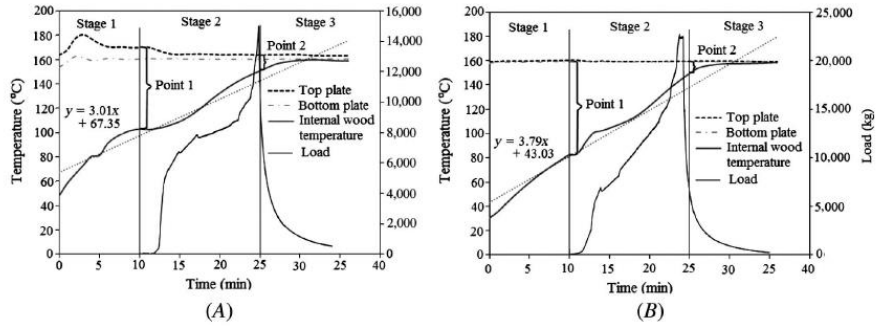
The thickness, density, moisture content (MC), and color were measured for each sample. The density was calculated as a ratio between the weight and volume, determined by measuring the initial thickness and the initial width and length. The MC was calculated as the difference between the initial weight and the oven-dry weight, expressed as a percentage according to ASTM D4442, *Standard Test Methods for Direct Moisture Content Measurement of Wood and Wood-Based Materials*.[18] A HunterLab MiniScan XE Plus spectrophotometer (Hunter Associates Laboratory, Inc, Reston, Virginia, USA) was used for color measurements, and the CIEL*a*b* system was used for measuring the reflectance spectrum. The range of the reflectance spectrum was from 400–700 nm with an opening of 11 mm at the point of measurement. For observing the reflection, the specular component (SCI mode) was included at a 10° angle, which is normal for the sample surface (D65/10), with a field of vision of 2° (Standard observer, CIE 1931) and an illumination standard of D65 (corresponding to daylight in 6,500 K). The MiniScan XE Plus generated three parameters for each measurement, namely L* (luminosity), a* (tendency of color from red to green), and b* (tendency of color from yellow to blue).

Evaluation of the Conditions of the Process to Validate the Densification System Operation

The performance of the densification THM device was evaluated in each one of its stages (fig. 1) as follows: (1) steaming or heating stage, in which the steam was applied to half of the wood samples for 10 min, while the other half were only heated; (2) the compression stage that was evaluated by compressing the wood samples perpendicularly to the grain for over 10 to 15 min until a target thickness of 9 mm was reached; and (3) the stabilization stage, in which the samples were kept compressed and heated but unloaded for 10 more minutes.

The THM device operation was verified by monitoring the load and temperature of the faces and interior of the wood sample. In each stage of the densification process, the following parameters with respect to time were recorded: (i) the temperature of the upper and lower faces of the wood sample (fig. 1); (ii) the internal temperature of the wood sample, which was modeled with respect to time by means of a simple linear regression to obtain the

FIG. 1 Variation of temperature in the top and bottom plates and wood samples and compression force plotted with respect to time during the densification process of *V. guatemalensis* over 25 minutes: (*A*) with steam application and (*B*) without steam application.



slope of the straight line corresponding to the internal heating rate of the wood sample (**fig. 1**). The linear model was utilized because this model presented the best adjusted, evaluated by coefficient of determination (r^2); and the last parameter recorded was (iii) the load applied to the wood sample until reaching the target thickness (maximum load) (**fig. 1**). The temperature of the faces of the sample and the internal temperature of the wood were plotted with respect to time, and the difference between these temperatures at the start and end of Stage 2 of the compression was calculated (**fig. 1**).

Evaluation of the Characteristics of the Densified Wood to Validate the Densification THM Device

As part of the verification of the densification THM device, various parameters were measured in each one of the samples of densified wood, namely, compression thickness, stabilization thickness, color, density, and percentage of densification. The density was calculated as a ratio between weight and volume of the wood sample after Stage 3 (stabilization). To calculate the volume, the dimensions (stabilization thickness, length, and width) of the samples were measured. The percentage of densification was calculated as a ratio between the initial density of the sample and the density of the densified sample. For color measurements, the same procedure used before the process of densification was utilized.

Results

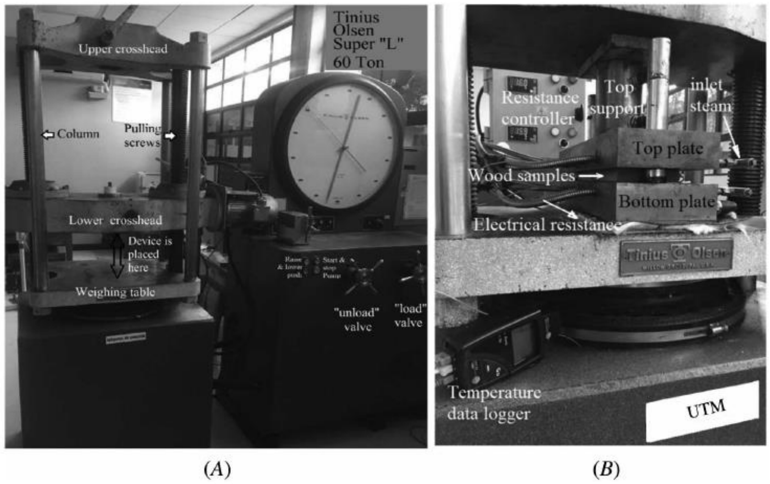
DENSIFICATION THM DEVICE

Densification THM DEVICE

The densification THM device designed consists of an accessory attached to the UTM Tinius Olsen, model Super L60, and has a capacity of 60 Mg (Tinius Olsen, Horsham, PA, USA). When the THM device was designed, the author thought that the force of a 60-Mg (maximum compression test of 28 MPa) UTM capacity would be enough to perform the compression on a piece of wood with the dimensions 70 by 300 mm. For THM processing, the minimum compression stress required is at least 4 MPa.

The UTM has a hydraulic loading system and rugged four-column construction for exceptional load frame rigidity and crossheads that are adjustable to meet specific requirements (**fig. 2*A***). In addition, the THM device is capable of transmitting heat, steam, and load to the wood samples, however, it must receive water to produce the steam (**fig. 2*B***). The UTM has a compression and tension area, but the THM device is placed in the compression area where a lower crosshead is adjusted in relation to the weighing table with a raise/lower

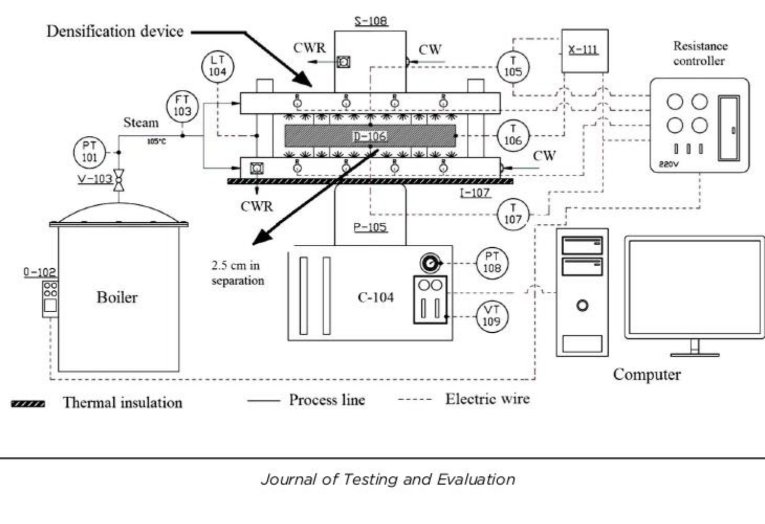
FIG. 2 UTM used and its main parts (*A*) and densification THM device attached to the UTM (*B*).



push, which is run by a crosshead motor and pulling screws (fig. 2*A*). The lower adjustable crosshead remains fixed during testing. The adjustment is determined by the THM device height and is not to be used for applying load.

All test loads are applied by the upward movement of the hydraulically driven piston in the UTM (C-104, fig. 3). The load and unload valves control the rate of loading and unloading by regulating the flow of oil from the pump on the hydraulic power unit to the hydraulic cylinder under the weighing table. Maintaining a uniform

FIG. 3 Control and data capture diagram of the densification THM device. O-102 = evaporator control; CWR = cold water return; T 105 = temperature control 1; CW = cold water; S 108 = top support; P-105 = piston; C-104 = compressor; V-103 = globe valve; FT = flow transmitter; T-106 = wood temperature control; T-107 = temperature control 2; X-111 = temperature data logger; R = electrical resistance; VT = velocity transmitter; LT = lever transmitter; PT = pressure transmitter.



loading rate is aided by an automatic valve that strives to keep the cylinder and pump pressures in equilibrium. As the oil moves through the load valve and into the hydraulic cylinder, the precision ground and lapped piston move upward, thus applying load to the specimen. Compression testing is performed in the area between the weighing table and the lower adjustable crosshead. Then, in the THM device design, the compression load of the UTM is transmitted via the weighing table (fig. 2A) to the THM device (fig. 2B).

This THM device applied a load to the wood by compressing two metal plates that were 5-cm thick, 45-cm long, and 15-cm wide (fig. 3, T-106). As indicated, the UTM transmitted the load application via the bottom plate, while the top plate served as a support (fig. 2B). Heat was applied to the metal plates using eight 500-W cartridge heaters (four in each plate), each with a diameter of 12.5 mm and length of 15 cm, distributed in the center of the plate length with a gap of 25 cm between them (fig. 3, I-107).

For steam production, a pilot small boiler with a 20–25-L capacity and a single steam outlet was used (fig. 3, B-101). The heat for the steam production was supplied by a flame produced by liquefied gas (fig. 3). The steam was generated at a temperature of 105°C (controlled by a thermocouple) and pressure of 478 Pa and was supplied at 5m liters/hours. One important observation when steam was supplied was that there was a small increase in the temperate (approximately 10°C) of the plate (fig. 1A), which was not present when the steam was not supplied (fig. 1B). The steam was supplied to the device's lateral side in both plates. The steam was distributed into the plates, and afterwards, the steam went out through the small holes (2 mm in diameter) on the surface of the plate. The holes were distributed in a 2.5-cm spacing grid on the surface of the plate. In the steam stage, the wood sample was placed at a height of 1.0 cm on the surface of the bottom plate using two supports with a spacing of 25 cm. In addition, the top plate was also placed with a gap of 1.0 cm from the top surface of wood samples during the steam stages.

Control Unit

The densification condition was controlled by the UTM and the THM device. The UTM provides the motion control of loading via a hydraulically driven piston in the UTM (fig. 3, C-103), so the controller for the THM accessory needs to only control the plate temperature and a solenoid valve for the steam line (fig. 3, V-103, T 105, and T 107). The control or monitoring in the compression stage was carried out by an electrovalve that could record the load applied to the wood samples at intervals of 50 µs in the UTM Tinius Olsen (fig. 3, PT 108 and VT 109). The UTM software and a computer were utilized for the recording. To control the temperature, two Type J thermocouples were placed close to the surface of the upper and lower plates and were monitored by the THM device (fig. 3). These two thermocouples (one per plate) were connected to a temperature controller that determined the temperature the plates should reach and helped to keep this temperature constant during the test (fig. 3, T 105 and T 107). In addition, two other Type J thermocouples were attached to each plate (upper and lower) in order to record the plate's temperature (fig. 3, T 105 and T 107). The thermocouples were monitored by a TESTO data capture device (fig. 2C), model T175-177 (Testo North America, West Chester, Pasadena, USA) that was connected to a computer to record the temperature during test development. Steam application supplied to the plates was controlled by a single manual stopcock (fig. 3, PT 101). A valve to record the pressure was placed inside the airtight container (fig. 3, V-103). In this case, the steam application was controlled rather than recorded during the test development because its value was kept constant.

The internal temperature of the wood samples was controlled by means of a Type J thermocouple placed in the center (according to length and thickness) of each piece of wood, and it was monitored in the same way as the plate thermocouples, with the same data capture equipment connected to a computer (fig. 3, T 106).

As for the thickness of the wood sample, it was measured at the start (initial thickness) and then at the end of the compression (compression thickness) and stabilization (stabilization thickness) stages. These thickness values were recorded manually using a caliper.

The specimen thickness was controlled in the following way: the difference between the initial thickness and the target thickness was calculated. This difference was divided by the densification time (10 or 15 min), and then this value was used as the speed of application of the load. During the whole densification period, the sample

TABLE 1

Average values for the wood samples of *Vochysia guatemalensis* from fast-growth plantation before the densification process

Temperature, °C	Time, min	Steam	Initial Thickness, mm	Initial Wood Density, g/cm³	Initial MC, %	Wood Initial Color Parameter L*	a*	b*
140	10	+	20.37 (0.33)	0.39 (0.06)	12.78 (0.36)	69.27 (2.68)	10.88 (1.28)	21.90 (1.64)
		−	20.42 (0.10)	0.39 (0.03)	12.91 (0.39)	69.32 (1.54)	9.78 (2.22)	20.81 (2.27)
	15	+	20.33 (0.22)	0.37 (0.03)	13.03 (0.54)	68.99 (1.56)	9.34 (1.94)	20.65 (1.94)
		−	20.36 (0.17)	0.39 (0.06)	12.98 (0.57)	69.87 (1.84)	10.00 (1.90)	20.89 (2.32)
160	10	+	20.44 (0.14)	0.38 (0.04)	12.62 (0.65)	69.02 (2.07)	9.78 (2.23)	20.45 (2.57)
		−	20.43 (0.17)	0.36 (0.04)	11.93 (0.59)	69.31 (1.69)	10.51 (2.10)	21.59 (2.43)
	15	+	20.39 (0.13)	0.41 (0.06)	12.08 (0.23)	69.66 (2.59)	9.79 (1.64)	20.37 (1.71)
		−	20.39 (0.16)	0.38 (0.02)	12.25 (0.52)	69.69 (1.73)	9.70 (1.67)	21.10 (1.50)
180	10	+	20.33 (0.18)	0.39 (0.03)	12.50 (0.39)	69.89 (1.89)	10.10 (1.75)	21.03 (2.33)
		−	20.43 (0.12)	0.40 (0.05)	12.47 (0.40)	70.03 (1.30)	8.84 (1.47)	20.15 (1.70)
	15	+	20.45 (0.16)	0.40 (0.05)	12.79 (0.35)	70.04 (1.67)	10.01 (2.18)	21.00 (2.66)
		−	20.42 (0.12)	0.39 (0.04)	12.49 (0.40)	69.48 (1.72)	9.34 (2.02)	20.71 (2.44)

Note: + and − mean with steam and without steam, respectively.

thickness was measured constantly between the two plates with a caliper (fig. 2*B*) with the objective of controlling the constant decrease in the thickness.

CHARACTERISTICS OF THE WOOD BEFORE THE DENSIFICATION PROCESS

V. guatemalensis samples utilized in the different treatments of the densification process presented average values of initial thickness that varied from 20.33 mm to 20.44 mm, an initial density from 0.36 to 0.41 g/cm³, an MC from 11.93 to 13.03 %, and the following color parameters: L* from 68.99 to 70.04, a* from 8.84 to 10.88, and b* from 20.15 to 21.90 (Table 1).

EVALUATION OF THE DENSIFICATION THM DEVICE

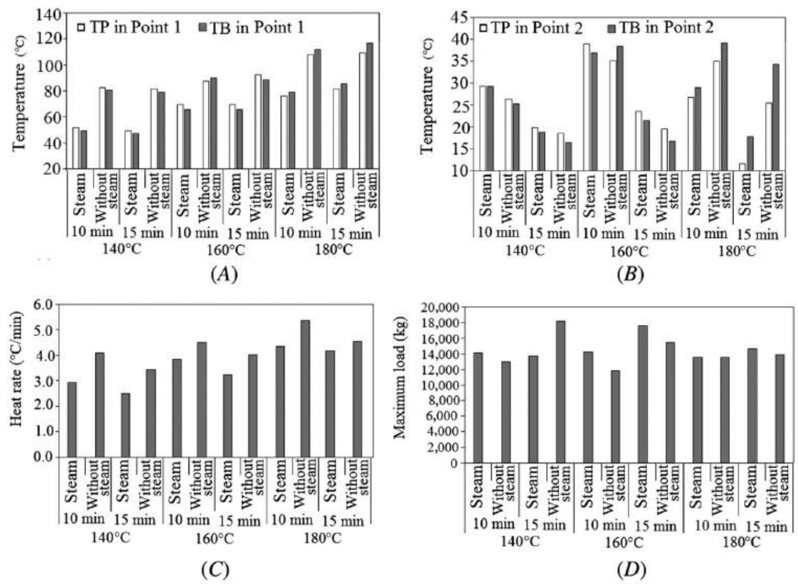
Temperature monitoring of the densification THM device allowed the observation of temperature differences between the two metal plates and the internal temperature of the wood samples. At Point 1 (initial compression stage, fig. 1), steam application showed that the temperature differences between the plates and the wood samples were lower than those for the wood samples without steam (fig. 4*A*). At Point 2 (final compression stage, fig. 1*A* and 1*B*), differences were observed with the compression times: the wood that was subjected to 10 min of compression tended to present higher differences in temperature between the metal plates and the wood than the wood that was subjected to 15 min of compression (fig. 4*B*).

As for the densification temperatures at Point 2, constant monitoring of this variable showed that the wood samples subjected to temperatures of 140°C and 160°C in addition to steam application showed higher differences in temperature than those without steam application. In contrast, the wood samples subjected to 180°C without steam showed the highest temperatures (fig. 4*B*).

The constant monitoring of the internal wood sample temperature, set in the densification THM device, allowed the derivation of the heating rate. The steamed wood samples showed lower values than those of the wood without steam application, regardless of the compression time and the temperatures of the metal plates (fig. 4*C*). Likewise, the wood compressed for 15 min showed a heating rate lower than that of the wood samples compressed for 10 min (fig. 4*C*).

The electrovalve (PT 108, fig. 3) used for monitoring the load applied to the sample indicated that the maximum load applied ranged from 12,928 kg to 18,167 kg (fig. 4*D*). It was not possible to observe a tendency in the behavior of the maximum load with the various treatments applied to the wood samples (fig. 4*D*).

FIG. 4 Temperature differences between the top (TP) and the bottom (BP) plates, with the wood at the beginning (*A*) and end of the compression process (*B*); heating rate of the wood during the densification process (*C*); and maximum load applied in the compression stage (*D*).



CHARACTERISTICS OF THE WOOD AFTER BEING TREATED WITH THE DENSIFICATION THM DEVICE

Figure 5 shows the average values obtained for initial thickness, compression thickness, and stabilization thickness of the densified wood of *V. guatemalensis* densified using the THM device. Before densification, the wood samples presented an initial thickness between 20.33 mm and 20.44 mm (Table 1), whereas after densification, the

FIG. 5

Average compression thickness, stabilization thickness, and initial thickness of *Vochysia guatemalensis* wood from a fast-growth plantation in the densification process.

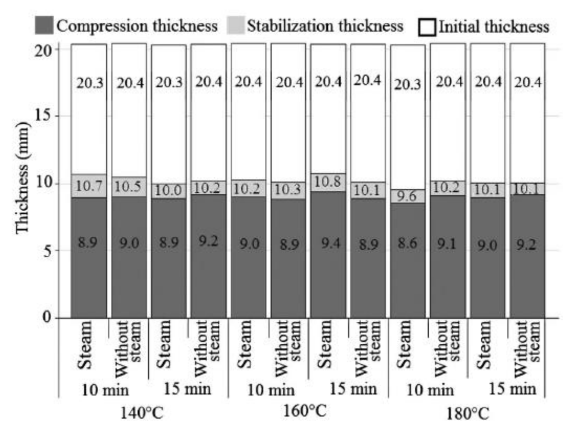


TABLE 2

Average values for maximum load and characteristics of the wood samples of *Vochysia guatemalensis* wood from a fast-growth plantation after the densification process

Temperature, °C	Time, min	Steam	Degree of Compression, %	Degree of Stabilization, %	Final Wood Density (g/cm^3)	Densification, %	Color Change, ΔE*
140	10	+	56.1 (1.5)	47.3 (5.8)	0.65 (0.07)	72.20 (7.35)	1.74 (1.12)
		−	55.9 (0.7)	48.5 (2.7)	0.67 (0.05)	73.76 (9.77)	1.08 (0.51)
	15	+	56.1 (1.2)	50.8 (2.3)	0.66 (0.06)	81.41 (9.18)	2.69 (1.34)
		−	55.0 (3.6)	49.7 (4.1)	0.68 (0.09)	76.55 (12.54)	0.95 (0.32)
160	10	+	55.8 (2.1)	49.7 (2.5)	0.67 (0.06)	75.59 (8.68)	3.07 (1.83)
		−	56.6 (1.8)	49.4 (4.1)	0.62 (0.06)	73.13 (13.99)	2.15 (1.16)
	15	+	54.0 (4.9)	47.1 (5.0)	0.69 (0.06)	67.83 (15.47)	3.25 (1.27)
		−	56.4 (0.9)	50.3 (1.6)	0.67 (0.04)	74.87 (6.35)	1.97 (0.45)
180	10	+	57.7 (3.0)	52.9 (3.0)	0.73 (0.04)	87.76 (10.31)	7.67 (3.41)
		−	55.5 (3.6)	50.1 (4.3)	0.70 (0.05)	74.21 (14.20)	4.44 (1.84)
	15	+	56.1 (1.9)	50.8 (3.2)	0.70 (0.08)	77.50 (12.86)	5.26 (1.98)
		−	55.0 (4.3)	50.7 (2.6)	0.68 (0.08)	74.99 (8.90)	5.06 (1.93)

Note: + and − mean with steam and without steam, respectively.

compression thickness ranged from 8.6 mm and 9.4 mm (fig. 5), which means that the THM device achieved a degree of compression from 55.0 % to 57.7 % (Table 2). However, in the stabilization stage, the thickness increased from 9.6 mm to 10.8 mm (fig. 5), which yielded a degree of compression after stabilization from 47.3 % to 52.9 % (Table 2). There was an increase in the thickness of the densified wood after the stabilization stage, which varied from 1.0 mm to 1.5 mm (fig. 5). Importantly, the parameters evaluated showed no tendency or variation resulting from the type of treatment applied to the wood.

Table 2 shows the average values obtained for the final density, the densification percentage and the ΔE* of the wood after the stabilization stage, using the THM device for densification. The densified wood achieved an average final density ranging from 0.65 to 0.73 g/cm^3. The highest values were obtained at 180°C (>0.68 g/cm^3). The densification THM device allowed *V. guatemalensis* wood to achieve a densification percentage between 72.20 % and 87.76 % (Table 2). The color change of the wood samples due to the applied heat, measured by the ΔE* of the L*a*b*, ranged between 1.08 and 7.67, with the highest color changes observed in wood densified at 180°C (>4.44) (Table 2).

Discussion

DENSIFICATION THM DEVICE

The THM device allowed us to conduct the densification process in 20 by 70 by 300 mm samples of a low-density tropical wood species, *V. guatemalensis*, by means of a UTM with a 60-ton capacity. Importantly, although the steam, pressure and heat application could be controlled, all of these parameters worked as independent elements, which is why it would be convenient to concentrate them in a single control unit by means of a program such as LabVIEW. However, this would increase the cost of control.

Testing machines for small samples have a maximum capacity of 60 tons; therefore, the THM device can be easily adapted in order to allow processes such as densification. This is not the case when a greater capacity is needed because the existing testing machines do not have the necessary structure for attaching devices to them. Large tonnage machines require tests to be carried out on strong floors, making their use for processes like the one described complex.

The steaming unit used in this study (B-101, fig. 3) worked properly and showed no problems with the steam pressure or steam release through the holes of the metal plates. In addition, the unit was inexpensive and was also easy to manufacture. However, the 10°C increment in the temperature of the plate with steam supplied must be considered (fig. 1*A*). It is recommended that the target temperature be 10°C lower.

It should be noted that the densification THM device allows the user to change the conditions of the densification process easily. This characteristic allows the determination of the best combination of treatments (temperature, compression time, and steam application) that a wood species needs for densification. This is vital in areas with a tropical climate where there are a great variety of species with characteristics that allow for the application of techniques that improve their properties.

EVALUATION OF THE DENSIFICATION THM DEVICE

The temperature differences found between the plates and the interior of the wood seem to be due to the treatments applied (temperature, compression time. and steam application). Use of steam at the beginning of the densification process (Stage 1, fig. 1) increased the internal wood temperature at this stage, resulting in a lower difference in temperatures between the plates and the wood at Point 1 of the compression stage (fig. 4*A*).

At the end of the compression stage (Point 2), the temperature differences between the plates and the interior of the wood were caused because of the different compression times applied. For example, the wood subjected to 10 min of compression showed higher differences than the wood that was compressed for 15 min (fig. 4*B*). These differences in temperature could be related to a higher heating rate (fig. 4*C*). Thermal conductivity is a measure of the rate of heat flow through the thickness of a material subjected to a temperature gradient, and it is affected by various factors, including density.[15,16] In the case of wood, as its density increases, so does its conductivity.[15,16] Hence, the wood compressed for 10 min showed a higher heating rate compared with the wood compressed for 15 min.

The treatments applied (temperature, compression time, and steam application) showed no variation with respect to the maximum load (fig. 4*D*). Nevertheless, improved control of the densification THM device regarding the target thickness might establish a relationship between the load and the treatments applied.

CHARACTERISTICS OF THE WOOD DENSIFIED WITH THE THM DEVICE PROPOSED

It was impossible to observe a trend in the measured thicknesses (compression and stabilization thickness) due to the treatments applied (temperature, compression time, and steaming) (fig. 5). However, a high variation was noticed between the treatments (temperature, compression time, and steam application) in the compression and stabilization thickness (fig. 5), which can be attributed to the fact that the proposed densification THM device prevents appropriate control of the target thickness (9 mm in 10 or 15 min). As previously indicated in the proposal of the densification THM device, the final thickness of the wood sample is controlled by means of a caliper and a manual timer. These types of controls hinder precision in achieving the target thickness of the wood, causing the variations observed.

The thickness increased after the stabilization stage (Stage 3) (fig. 5), a phenomenon known as elastic recovery. The springback is the dimensional recovery suffered by wood after deformation as a consequence of its bioelastic nature and the release of the forces stored in the wood during its densification.[15,19] When the load on the compressed wood was eliminated, the elastic deformation recovered instantaneously, producing an increase in the thickness (called stabilization of thickness), which varied from 1.0 mm to 1.5 mm in relation to the compression thickness.

Although differences were observed in the final thickness of the wood caused by the densification THM device, the system allowed for an increase in the density of the *V. guatemalensis* wood from 0.36–0.41 g/cm^3 to 0.65–0.73 g/cm^3, which corresponds to a densification percentage between 72.2 % and 87.7 % (Tables 1 and 2). Those values are common in densified species from temperate climates and other latitudes.

MC is an important parameter for the densification process because MC has an influence in the glass transition temperatures of the polymers of wood[6]; however, the variations of MC found in the samples (Table 1) probably will not have influenced the densified wood. These wood parameters will be affected when there are large variations in the MC value.[6] The color changes found in the wood samples after the densification process were low (ΔE^* 1.08 to 7.67). According to Gonnet,[20] this range of values is imperceptible or hardly perceptible by the human eye. Thus, although high temperatures (140°C, 160°C, and 180°C) were used, color changes were low. Color changes in the wood normally appear after long periods of exposure to high temperatures,[21] in contrast with the densification process proposed here, where changes were observed after 30 to 35 min in total.

Conclusions

The inexpensive densification THM device that was developed using steaming, temperature, and compression and that can be adaptable to a 60-Mg-capacity UTM allowed us to successfully conduct a densification process on 20 by 70 by 300 mm wood samples of a low-density tropical species. However, some drawbacks were observed: although the steam, pressure, and temperature applied could be controlled, all of these parameters worked as independent elements, which is why it would be convenient to concentrate them in a single control unit. High variation was observed in the compression and stabilization thickness because of the lack of convenient control of the target thickness.

The evaluation of the densified wood (*V. guatemalensis*) revealed that the density increased from 0.36–0.41 g/cm^3 to 0.65–0.73 g/cm^3, i.e., a percentage increase in the densification from 72.2 % to 87.7 %. In addition, the temperatures used caused color changes imperceptible or hardly perceptible to the human eye (ΔE^* 1.08 to 7.67) in the densified wood.

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Artículo 2: Effect of thermo-hydro-mechanical densification in the wood properties of three short-rotation forest species in Costa Rica

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Effect of thermo-hydro-mechanical densification in the wood properties of three short-rotation forest species in Costa Rica

Carolina Tenorio[a], Roger Moya[b]*

[a] Escuela de Ingeniería Forestal, Instituto Tecnológico de Costa Rica, Cartago, Costa Rica. orcid.org/0000-0003-2901-7079. ctenorio@itcr.ac.cr

[b] Escuela de Ingeniería Forestal, Instituto Tecnológico de Costa Rica, Cartago, Costa Rica, orcid.org/ 0000-0002-6201-8383,

*rmoya@itcr.ac.cr.

Abstract

Alnus acuminata, *Vochysia ferruginea* and *Vochysia guatemalensis* are fast-growing hardwood species used in commercial reforestation programs in Costa Rica. The wood of these species has low density and low mechanical properties. The objective of this work was to study a thermo-hydro-mechanical densification process and the characteristics of densified wood of these species. Twelve densifying treatments based on temperature (three conditions), compression time (two conditions) and used/not used of steam were tested. The variables of the densification process (degree of compression, densification percentage, stabilization recovery, maximum load, heat rate and color change) and the properties of the densified wood (final density, thickness swelling, MOE and MOR in static bending and hardness) 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 the initial density of the wood. The influence of temperature during the densification process was observed on the variables of heat rate and color change. An increase in the MOE and MOR in static bending and in the hardness of the densified wood relative to the un-densified wood for the three species was observed, as well as a clear positive correlation of the properties with the final density and the maximum load, the latter being highly correlated with the initial density. This means that the initial density was significant in the densification process and affects the wood properties.

Keywords: tropical species, hardwoods, low density, thermo-hydro-mechanical process.

1. Introduction

Wood supply for the industrial and construction sectors at world level has decreased during the last years [1]. For this reason, the interest on fast growing wood species has grown [2]. However, low density and limited mechanical properties are main characteristics of those species because the trees are harvested while still young and the proportion of juvenile wood is dominant [3–5]. 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 [6].

Alnus acuminata, *Vochysia ferruginea* and *Vochysia guatemalensis* are fast-growing hardwood species used in commercial reforestation programs in Costa Rica [7]. These are low-density, soft and easy to work-with wood species [8]. 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 [9]. It is due to these reasons that have been barely studied and to some extent ignored by the industries. Currently, studies can be found on the properties and behaviour of these and other reforestation species in industrial processes in Costa Rica [10]. Yet, studies aiming at improving their mechanical properties are not available so far.

Many wood densification processes have been developed to improve the wood's mechanical and physical properties [11]. 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 [11]. However, unlike densification by mechanical compression, chemical impregnation affects the natural and sustainable character of the wood and is usually more expensive [12]. Mechanical compression combined with steam and heat, called thermo-hydro-mechanical densification (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 [13–18].

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 [12, 19]. The heat treatment can improve resistance to decay (Huang et al. 2012), decrease hygroscopicity [20] and improve the dimensional stability [21]. The moisture induces a mechano-sorptive effect and further softens the wood and this enables mechanical compression of wood without cell wall fracture [22].

Most research on THM densification has focused on softwood species and on closed systems [12]. Therefore, studies on hardwood tropical species whose anatomical structures affect to a further extent the result of the process, are scarce [12]. This is because the compression properties of the wood depend on the frequency, size and distribution of its anatomical structures [23] which, in the case of hardwoods, are dominated by vessels, fibres and radial parenchyma arranged in more complex matrixes [24] than the fibre-tracheids of softwood species [25].

On this account, the aim of this study is 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 (degree of compression, % densification, stabilization recovery, maximum load, heat rate and color change) and the physical (final density and thickness swelling) and mechanical (MOE and MOR in static bending and hardness) properties of the densified wood, were determined. The results obtained will make it possible to utilize species with poor mechanical properties to make new high-performance wood products, if modified by means of THM densification.

2. Materials and methods

2.1. Origin and characteristics of the wood before densification

The study tested the wood of *Alnus acuminata*, *Vochysia ferruginea* and *Vochysia guatemalensis* from forest plantations located in Cartago and Alajuela in Costa Rica. The trees used were around 8 years old, which normally present low heartwood content [8]. Therefore, the wood used was mostly sapwood. Wood samples of 300 mm long x 70 mm wide x 20 mm thick of each species were prepared. Before densification, thickness, width, length, density, moisture content and color were measured for each one of the samples (Table 1).

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 D-4422 [26]. For color measurements, a HunterLab Mini Scan XE Plus spectrophotometer was used and the CIEL*a*b* system to measure the reflectance spectrum. The range of this measurement is 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 Skan XE Plus generated three parameters for each measurement, namely: L* (luminosity), a* (color trend from red to green), and finally b* (color trend from yellow to blue).

Table 1 General characteristics of the wood of three forest species 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)

Legend: the values in parenthesis mean standard deviation.

2.2. 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 (10 and 15 minutes) 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 (detailed in Table 2) were tested and 20 samples per densification treatment, resulting in 240 samples per species (Table 2).

Table 2 Treatments used in the THM densification process of the wood of three forest species.

Species	Temperature (°C)	Densification time (min)	Steam	Number of treatment
Alnus acuminata y Vochysia guatemalensis	140	10	Yes	T1
			No	T2
		15	Yes	T3
			No	T4
	160	10	Yes	T5
			No	T6
		15	Yes	T7
			No	T8

	180	10	Yes	T9
			No	T10
		15	Yes	T11
			No	T12
Vochysia ferruginea	140	10	Yes	T1
			No	T2
		15	Yes	T3
			No	T4
	150	10	Yes	T5
			No	T6
		15	Yes	T7
			No	T8
	160	10	Yes	T9
			No	T10
		15	Yes	T11
			No	T12

The densification process was the same described in Tenorio and Moya (2019). Consisted of three stages: 1) steaming or heating stage, where steam was applied to half of the wood samples during 10 minutes, while the other half was only heat-treated; 2) 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 minutes; and 3) stabilization stage, where the samples were kept compressed and heated while unloaded during 10 more minutes. Throughout the process the metal plates kept one of the three temperatures constantly.

2.3. Evaluation of the densification process

In order to determine the maximum load and the internal heat rate of the wood, a temperature control probe 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 (compression and stabilization thickness). After the densification process, the width, length, weight, colour, density and densification percentage were determined.

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

The thickness determined in stages 2 and 3 (compression and stabilization thickness) helped determine the degree of compression and the stabilization recovery of wood thickness. The degree of compression was calculated as the ratio of the initial thickness to the compression thickness, expressed as a percentage. The stabilization recovery of the thickness of the densified wood was calculated as the absolute value of the ratio of the compression thickness to the stabilization thickness, also expressed as a percentage.

2.4. Properties of the densified wood

In the evaluation of the wood properties, the thickness swelling was determined according to ASTM D4933-16 [27]. The wood samples were conditioned to 18% equilibrium moisture content, at 20 °C and 80% relative humidity. Next, the thickness swelling was calculated as the ratio of the thickness after conditioning to the thickness before conditioning. The result was presented as a percentage. The mechanical properties static bending (modulus of rupture and modulus of elasticity) and Janka hardness were determinated following ASTM D143-14 standards [28]. A total of 20 samples per species, per treatment, were prepared for each test. In total, 13 treatments were performed (12 densification treatments and un-densified wood as control).

2.5. 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 (Table 2) in each one of the variables obtained during the densification process (degree of compression, % of densification, stabilization recovery, maximum load, heat rate and colour change) and in the physical (final density and thickness swelling) and mechanical properties (MOE y MOR in static bending and hardness) per species. 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 (initial thickness, initial density and initial moisture content) was performed. Also, 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., Cary, NC).

3. Results

3.1. Evaluation of the densification process

Table 3 presents the results obtained as part of the evaluation of the densification process. A degree of compression of approximately 55% can be observed for the three species. No statistical differences were found between the treatments applied for any of the species. The final density, the % of densification and the stabilization recovery 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 and the % of densification was 76.08%, and again no differences between the densification treatments were observed. The stabilization recovery was 14.26 % with minimal differences between the treatments. Treatment T6 presented the highest value and T12 the lowest (Table 3). As for *V. guatemalensis*, the final density was 0.68 g/cm^3 on average; T9 presented the highest value, and T1 and T6 the lowest values; the % of densification was 76.55%; treatments T3 and T9 showed the highest values, while T7 the lowest value. The stabilization recovery was 14.12%; treatment T1 showed the highest percentage of stabilization recovery, while treatments T3, T4 and T9 a T12 presented the lowest averages (Table 3).

Colour 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). Differences between the treatments were observed in the three species; treatments T9 and T12 presented higher colour changes, while treatments T1 and T4 presents the lowest colour changes (Table 3).

Table 3 Characteristics of the wood of three forest species after the THM densification process.

Species	Treatment	Degree of compression (%)	Final density (g/cm^3)	% of densification	Stabilization recovery (%)	Color change (ΔE^*)
Alnus acuminata	T1	54.95^{A} (0.61)	0.79^{A} (0.06)	85.91^{A} (10.32)	8.53^{A} (5.89)	5.79^{D} (1.06)
	T2	54.80^{A} (0.77)	0.80^{A} (0.06)	84.07^{A} (8.47)	9.24^{A} (4.42)	6.20^{D} (1.53)
	T3	54.84^{A} (0.62)	0.78^{A} (0.06)	83.50^{A} (9.32)	8.85^{A} (4.56)	5.65^{D} (1.22)
	T4	54.81^{A} (0.63)	0.80^{A} (0.05)	85.33^{A} (8.38)	7.01^{A} (3.54)	5.92^{D} (1.04)
	T5	55.02^{A} (1.06)	0.78^{A} (0.06)	86.42^{A} (12.56)	7.97^{A} (5.00)	7.96^{BCD} (3.68)
	T6	54.78^{A} (0.60)	0.76^{A} (0.05)	83.04^{A} (9.58)	8.38^{A} (4.52)	7.49^{CD} (1.32)
	T7	54.92^{A} (0.64)	0.81^{A} (0.06)	85.52^{A} (8.74)	6.61^{A} (4.70)	11.07^{ABC} (9.96)
	T8	54.61^{A} (0.68)	0.80^{A} (0.07)	82.93^{A} (14.39)	8.36^{A} (6.55)	8.49^{ABCD} (1.80)
	T9	54.52^{A} (0.47)	0.75^{A} (0.06)	82.61^{A} (10.90)	8.54^{A} (4.91)	11.73^{A} (2.83)
	T10	54.79^{A} (0.58)	0.79^{A} (0.05)	87.95^{A} (10.61)	5.47^{A} (4.63)	11.10^{ABC} (2.06)
	T11	54.85^{A} (0.56)	0.77^{A} (0.06)	81.59^{A} (9.66)	8.45^{A} (5.29)	11.14^{ABC} (1.77)
	T12	54.68^{A} (0.75)	0.77^{A} (0.06)	84.96^{A} (9.94)	5.62^{A} (3.95)	11.52^{AB} (2.90)
Vochysia ferruginea	T1	55.52^{A} (0.90)	0.78^{A} (0.08)	71.84^{A} (9.33)	18.39^{AB} (7.03)	2.55^{C} (1.55)
	T2	55.27^{A} (0.55)	0.80^{A} (0.08)	73.23^{A} (10.82)	14.83^{AB} (7.15)	3.09^{C} (2.95)
	T3	55.59^{A} (0.64)	0.81^{A} (0.06)	74.67^{A} (11.34)	14.87^{AB} (7.29)	4.01^{C} (2.15)
	T4	55.49^{A} (0.45)	0.81^{A} (0.10)	72.78^{A} (9.31)	15.31^{AB} (6.19)	2.85^{C} (1.90)
	T5	55.55^{A} (0.90)	0.82^{A} (0.07)	79.97^{A} (14.38)	14.02^{AB} (8.84)	5.36^{BC} (2.23)
	T6	55.06^{A} (0.64)	0.78^{A} (0.10)	72.08^{A} (8.64)	18.88^{A} (7.52)	4.75^{BC} (1.78)
	T7	54.86^{A} (0.59)	0.80^{A} (0.09)	81.41^{A} (11.31)	12.05^{AB} (7.31)	5.32^{BC} (1.80)
	T8	55.20^{A} (0.87)	0.82^{A} (0.09)	80.00^{A} (11.39)	12.81^{AB} (7.39)	3.89^{C} (2.09)
	T9	55.66^{A} (0.87)	0.81^{A} (0.07)	80.72^{A} (12.91)	13.20^{AB} (7.85)	11.21^{A} (6.70)
	T10	55.49^{A} (0.57)	0.79^{A} (0.06)	74.45^{A} (12.65)	12.85^{AB} (5.72)	8.43^{AB} (6.20)
	T11	54.95^{A} (0.92)	0.74^{A} (0.09)	72.90^{A} (7.65)	12.85^{AB} (5.72)	11.59^{A} (7.02)
	T12	55.14^{A} (0.59)	0.79^{A} (0.07)	78.93^{A} (13.74)	11.04^{B} (5.07)	11.19^{A} (6.15)
Vochysia guatemalensis	T1	56.09^{A} (1.45)	0.64^{B} (0.07)	72.31^{BC} (8.01)	19.97^{A} (7.08)	1.74^{DE} (1.12)
	T2	55.87^{A} (0.73)	0.66^{AB} (0.06)	71.54^{BC} (14.16)	16.96^{AB} (5.80)	1.08^{E} (0.51)
	T3	56.11^{A} (1.18)	0.66^{AB} (0.06)	81.41^{A} (9.18)	12.14^{B} (4.88)	2.69^{CDE} (1.34)
	T4	55.04^{A} (3.57)	0.68^{AB} (0.09)	76.69^{ABC} (11.30)	12.02^{B} (6.98)	0.95^{E} (0.32)
	T5	55.78^{A} (2.12)	0.67^{AB} (0.06)	75.59^{BC} (8.68)	13.96^{AB} (6.89)	3.07^{CD} (1.83)
	T6	56.64^{A} (1.81)	0.64^{B} (0.07)	76.70^{ABC} (12.78)	16.67^{AB} (7.54)	2.15^{DE} (1.16)
	T7	54.02^{A} (4.95)	0.69^{AB} (0.06)	67.83^{C} (15.47)	15.28^{AB} (5.21)	3.25^{CD} (1.27)
	T8	56.38^{A} (0.88)	0.68^{AB} (0.04)	77.14^{ABC} (6.77)	13.94^{AB} (4.07)	1.97^{DE} (0.45)
	T9	57.72^{A} (2.96)	0.73^{A} (0.04)	87.76^{A} (10.31)	11.69^{B} (6.29)	7.67^{A} (3.41)
	T10	55.47^{A} (3.62)	0.70^{AB} (0.06)	76.06^{ABC} (13.66)	12.09^{B} (4.66)	4.44^{BC} (1.84)
	T11	56.10^{A} (1.85)	0.70^{AB} (0.08)	77.50^{ABC} (12.86)	12.08^{B} (5.74)	5.26^{B} (1.98)
	T12	55.02^{A} (4.29)	0.69^{AB} (0.08)	77.63^{ABC} (9.10)	12.61^{B} (4.93)	5.06^{B} (1.93)

Legend: the values in parentheses mean standard deviation. Different letters for each parameter represent statistical differences between different treatments (significances at 95%).

Evaluation of the maximum load applied to wood in stage 2 of the densification process showed that greater load was applied to *A. acuminata* (201438.92 N) followed by *V. ferruginea* (175665.90 N) and next by *V. guatemalensis* (138322.88 N). *A. acuminata* and *V. ferruginea* showed no differences between the treatments, while in *V. guatemalensis* treatment T4 presented the highest value and T2 and T6 the lowest values (Figure 1a, b and c).

Concerning heat 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, an effect of the treatments applied was observed, densified wood with the highest temperatures (180m°C in *A. acuminata* and *V. guatemalensis* and 160m°C in *V. ferruginea*) presented the highest heat rate. Also, the wood with 10 minutes compression time showed higher heat rate than the wood under 15 minutes compression, while the wood densified without steam also showed the highest values of heat rate compared to wood densified with steam (Figure 1d, e and f). As for *A. acuminata* wood, treatments T9 and T10 presented higher heat rate values, while T3 showed the lowest value (Figure 1d). For *V. ferruginea* and *V. guatemalensis*, T10 showed the highest heat rate and T3 the lowest (Figure 1e and f).

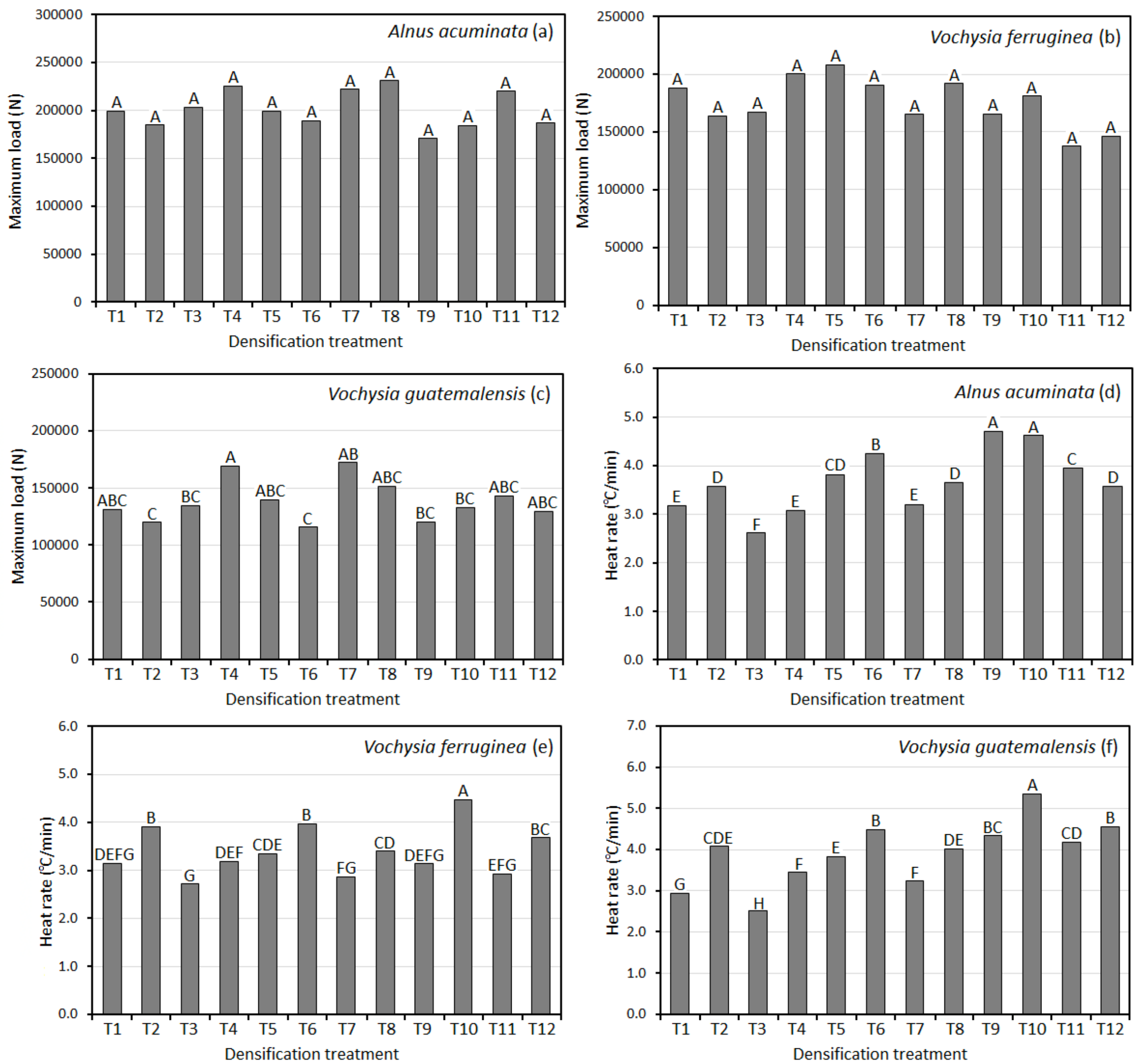

Fig. 1 Maximum load and heat rate during the THM densification process of wood of three forest species.

Legend: Different letters for each parameter represent statistical differences between different treatments (significance at 95%).

3.2. Properties of densified wood

Many statistical differences were observed between the treatments applied regarding the averages obtained for the properties evaluated in densified wood (Table 4). The thickness swelling for *A. acuminata* showed an average of 41.71%; treatment T7 presented the highest value, while treatment T1 was the lowest. For *V. ferruginea* the average thickness swelling was 23.42% for all the densification treatments, treatment T12 had the highest value and T2 the lowest. As for *V. guatemalensis*, treatment T3 had the highest thickness swelling average and treatments T6 and T11 the lowest, for an average between treatments of 20.73% (Table 4). For the three species the thickness swelling in the control treatment was less than 1%.

With respect to mechanical properties, no differences in the MOE and 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 T12 presented the highest value and treatment T6 the lowest. Treatments T1, T2 and T8 presented the highest averages of MOR in flexion, while treatment T12 obtained the lowest value. For densified wood of *V. guatemalensis*, averages were 9.15 GPa for MOE and 90.29 MPa for MOR. Treatment T5 showed the highest average MOE, while treatment T11 the lowest (Table 4). Treatments T1, T3, T4, T5 and T7 obtained the highest MOR in flexion (Table 4). The averages found for control wood (un-densified wood) were below the values obtained for densified wood in the 12 treatments used, except the average MOE in flexion for *V. guatemalensis*, where T11 obtained the lowest value (Table 4).

As for hardness, for *A. acuminata* wood no differences were found between the averages of the densification treatments applied (7520.41 N on average). The control was statistically lower than all densification treatments (Table 4). For *V. ferruginea*, there were few differences between the densification treatments (6263.30 N on average); T4 treatment had the highest value, T6 the lowest value among the treatments and the control again the statistically lower value (Table 4). In the wood of *V. guatemalensis* there were many differences between the treatments (7414.71 N on average); treatment T3 had the highest value, while treatments T9 and T12 showed the lowest averages of the densification treatments and the control also the statistically lowest average compared to densification treatments (Table 4).

Table 4 Physical and mechanical properties of densified wood of three forest species.

Species	Steam	Thickness swelling (%)	MOE in static bending (GPa)	MOR in static bending (MPa)	Hardness (N)
Alnus acuminata	T1	37.89^{C} (5.17)	12.56^{A} (1.81)	126.17^{A} (21.15)	7772.26^{A} (972.28)
	T2	39.06^{BC} (4.79)	13.78^{A} (1.87)	136.91^{A} (24.82)	8017.61^{A} (1099.41)
	T3	44.34^{AB} (4.57)	14.20^{A} (2.28)	147.60^{A} (25.75)	6924.15^{A} (1192.43)
	T4	43.42^{AB} (3.80)	12.52^{A} (2.45)	140.57^{A} (24.05)	7275.96^{A} (1034.31)
	T5	41.94^{ABC} (5.97)	12.65^{A} (2.10)	128.99^{A} (20.19)	7495.72^{A} (1269.98)
	T6	44.01^{AB} (4.51)	13.42^{A} (1.66)	140.89^{A} (22.69)	7432.95^{A} (1190.21)
	T7	44.54^{A} (4.82)	14.00^{A} (2.55)	142.15^{A} (37.22)	7427.59^{A} (947.17)
	T8	41.86^{ABC} (7.41)	12.15^{A} (1.98)	120.98^{A} (25.12)	7661.85^{A} (1428.34)
	T9	39.23^{BC} (4.38)	12.73^{A} (1.76)	128.05^{A} (17.65)	7384.06^{A} (1418.78)
	T10	40.72^{ABC} (4.29)	13.95^{A} (2.19)	145.16^{A} (26.56)	7046.68^{A} (1428.64)
	T11	41.26^{ABC} (4.62)	13.48^{A} (1.66)	130.42^{A} (23.78)	8181.47^{A} (1378.36)
	T12	42.26^{ABC} (4.73)	12.81^{A} (2.33)	125.06^{A} (34.11)	7731.08^{A} (1086.71)
	Testigo	0.89^{D} (0.41)	7.94^{B} (1.77)	62.18^{B} (13.26)	3042.75^{B} (813.65)

Vochysia ferruginea	T1	18.81^{CD} (6.01)	15.52^{AB} (2.73)	97.56^{A} (23.02)	6405.17^{AB} (1642.43)
	T2	17.04^{D} (6.41)	13.34^{BC} (2.01)	96.28^{A} (18.21)	6406.60^{AB} (1572.88)
	T3	19.50^{CD} (6.81)	14.30^{ABC} (2.08)	89.19^{ABC} (28.71)	6446.07^{AB} (1240.52)
	T4	25.79^{BC} (6.24)	14.30^{ABC} (2.07)	91.02^{AB} (28.84)	7392.78^{A} (1760.46)
	T5	21.96^{BCD} (5.63)	13.03^{BC} (4.23)	82.89^{ABCD} (19.99)	6780.70^{AB} (1730.74)
	T6	19.51^{CD} (6.00)	12.49^{C} (1.90)	89.07^{ABCD} (23.04)	5610.31^{B} (1320.49)
	T7	26.28^{BC} (8.76)	14.90^{ABC} (2.62)	71.31^{BCD} (14.01)	6348.66^{AB} (1527.10)
	T8	28.58^{B} (7.27)	13.10^{BC} (2.28)	98.03^{A} (26.07)	5816.61^{AB} (1320.49)
	T9	21.45^{CD} (5.41)	13.86^{ABC} (1.82)	65.47^{CD} (11.65)	6098.83^{AB} (1662.47)
	T10	19.99^{CD} (7.00)	14.25^{ABC} (3.14)	86.24^{ABCD} (16.72)	5824.01^{AB} (1648.31)
	T11	26.24^{BC} (4.74)	14.24^{ABC} (1.98)	78.74^{ABCD} (17.55)	6053.80^{AB} (1439.28)
	T12	35.88^{A} (8.05)	16.25^{A} (2.18)	64.91^{D} (24.45)	6145.32^{AB} (1818.43)
	Testigo	0.83^{E} (0.33)	9.63^{D} (0.52)	56.79^{E} (9.81)	2348.22^{C} (580.30)
Vochysia guatemalensis	T1	28.49^{AB} (5.69)	9.52^{BCD} (1.83)	99.69^{A} (21.04)	7186.55^{ABC} (1240.11)
	T2	20.98^{CD} (6.04)	9.03^{CDE} (0.97)	94.47^{AB} (14.65)	5731.70^{DE} (1203.27)
	T3	30.25^{A} (8.29)	9.99^{ABC} (0.94)	100.22^{A} (21.80)	8548.87^{A} (1454.36)
	T4	26.45^{AC} (5.43)	10.21^{ABC} (1.98)	107.25^{A} (27.65)	8317.37^{AB} (1278.51)
	T5	23.29^{BC} (5.40)	11.32^{A} (0.98)	110.81^{A} (19.71)	7053.08^{BCD} (1734.63)
	T6	13.71^{E} (4.85)	7.52^{EF} (1.58)	70.99^{DE} (13.96)	7052.22^{BCD} (1194.59)
	T7	29.05^{AB} (6.37)	10.88^{AB} (1.27)	108.81^{A} (24.73)	6646.33^{CDE} (1257.87)
	T8	16.60^{DE} (4.85)	8.97^{CDE} (1.48)	93.46^{AB} (18,57)	7419.09^{ABC} (1221.00)
	T9	15.57^{DE} (6.87)	8.82^{CDE} (1.23)	76.65^{BD} (12.98)	5610.94^{E} (1178.54)
	T10	15.21^{DE} (2.50)	7.62^{EF} (1.17)	75.05^{BD} (18.11)	5727.97^{DE} (1331.43)
	T11	12.75^{E} (2.68)	7.00^{F} (1.60)	66.77^{DE} (17.59)	5667.80^{DE} (1546.86)
	T12	16.42^{DE} (3.71)	8.96^{CDE} (1.26)	78.83^{BD} (15.41)	5344.20^{E} (1324.47)
	Testigo	0.67^{F} (0.25)	8.14^{DEF} (1.68)	52.15^{E} (11.66)	2008.17^{F} (555.52)

Legend: the values in parentheses mean standard deviation. Different letters for each parameter represent statistical differences between different treatments (significance at 95%).

3.3. Relationship between variables

Regarding the relationships between the initial characteristics of the wood and the variables obtained in the densification process, the initial density (density of the wood before the densification process) had the highest number of correlations with the variables obtained in the wood densification process of *A. acuminata*, *V. ferruginea* and *V. guatemalensis* (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 heat rate (Table 5). The initial density was related to almost all the variables of the densified process, except for colour change. A positive correlation was observed between the initial density and the final density, the maximum load and the stabilization recovery; and a negative correlation with the degree of compression, the % of densification and the heat 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 *V. ferruginea* densified wood, the initial thickness showed a positive correlation with the degree of compression. Initial density correlated to almost all the variables in the densification process, except for the heat rate. A positive correlation was observed with the final density, colour change, maximum load and stabilization recovery, and a negative correlation with the degree of compression and the % of densification. The initial moisture content correlated negatively with colour change and positively with the heat rate (Table 5).

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

Table 5 Correlation coefficients between the initial characteristics of the wood and the variables obtained in the THM densification process.

Species	Variable	Degree of compression	Final density	% of densification	Stabilization recovery	Color change	Maximum load	Heat rate
A. 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	0.08ns	-0.07 ns	-0.02 ns	0.06 ns	0.06 ns	-0.06 ns	-0.05 ns
V. 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**
V. 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**

Legend: ** =Statistically significant at 99%, *=statistically significant at 95% and "ns" not significant

As for the relationship between the variables obtained in the densification process and the wood properties, the final density (after densification) 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 degree of compression, the final density and the % of densification and a negative correlation with the stabilization recovery. As for the MOR in static bending and hardness, both correlated positively with the final density and the maximum load (Table 6).

For *V. ferruginea* wood, the thickness swelling had a negative correlation with the degree of compression, final density, maximum load and stabilization recovery. The MOE in static bending presented a positive correlation with the final density and the % of densification and a negative correlation with the stabilization recovery. The MOR in static bending had a positive correlation with the final density, maximum load and stabilization recovery and a negative correlation with the % of densification. The hardness had a positive correlation with the % of densification and the maximum load (Table 6).

In the densified wood of *V. guatemalensis* the thickness swelling showed a negative correlation with the heat rate only. The MOE in static bending had a positive correlation with the final density and the maximum load and a negative correlation with the heat rate. The MOR in static bending showed a positive correlation with the final density and maximum load, and a negative correlation with the degree of compression, the % of densification and the heat rate. In the case of hardness, a positive correlation with the final density and the maximum load was observed, and negative correlation with the heat rate (Table 6).

Table 6 Correlation coefficients between the variables obtained in the THM densification process and the densified wood properties.

Species	Variable	Degree of compression	Final density	% of densification	Maximum load	Heat rate	Stabilization recovery
A. 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**	-0.01v	0.22**	0.01 ns	-0.05 ns
	Hardness	0.02 ns	0.50**	-0.08 ns	0.47**	0.01 ns	-0.05
V. ferruginea	Thickness swelling	-0.15*	-0.23**	0.11	-0.18*	-0.07 ns	-0.21**
	MOE in static bending	0.01 ns	0.16*	0.35**	-0.03v	-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
V. guatemalensis	Thickness swelling	0.05 ns	-0.08	0.12	0.10	-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

Legend: ** =Statistically significant at 99%, *=statistically significant at 95% and "ns" not significant

4. Discussion

4.1. THM Densification process evaluation

The absence of statistical differences in the parameters evaluated in the densification process between treatments applied to *A. acuminata* and *V. ferruginea* suggests a stable densification process for these two species. This may be a consequence of the treatments used (Table 2) had no influence on the variables of the process, namely, degree of compression, final density, % of densification and maximum load applied during stage 2 (compression) (Table 3 and Figure 1). In the case of *V. guatemalensis* differences were noticed between treatments in the parameters evaluated in the densification process (Table 3). However, it was not possible to identify any behavioural pattern; for example, in the treatments where higher temperature was applied during densification (T9 a T12), final density, % of densification or maximum load values greater than in the other treatments were not observed (Table 3, Figure 1).

This means that other process variables or initial properties of wood influence the densification process, as is the case of the initial density of wood (Table 5). 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 and positive correlation with the final density and the maximum applied load (R> 0.73) and a negative correlation with the degree of compression and with the % of densification (Table 5).

The results presented above show that the initial density seems to be the most important factor in the densification process of the three species. Which was to be expected, since the moisture content and the initial thickness of the wood were controlled before the process. The moisture content was controlled by an adequate conditioning to the equilibrium moisture content to which densification was desired, and the thickness was controlled by using properly calibrated and sharp planers. On the other hand, the wood density before densifying is a more difficult to control variable because of its variability within the tree [5], especially when dealing with wood from tropical climate species, which feature high variability radially and throughout the trunk [8, 29].

As expected, colour change of the wood of the three species was higher in treatments T9 to T12, that is, in treatments with higher temperatures (Table 3). Temperature has a direct influence on wood colour; it affects its chemical composition [30] and causes wood darkening [31]. The colour changes obtained after wood densification were caused by the hydrolysis of the hemicelluloses [32], mainly on wood surface in contact with the metal plates that are responsible for transmitting heat. The heat of the metal plates causes a decrease in white hue (L*), attributed to the degradation or modification of components through reactions such as oxidation, dehydration, decarboxylation and hydrolysis [33], as well as lignin darkening, which is associated with the generation of chromophore groups [31], which causes darker colour at higher temperatures (Table 3, Figure 1).

As in the colour change, the heat rate was higher in treatments T9 to T12 and in treatments in which 10 minute-compression was used in stage 2 of the process, and when no steam was applied (Figure 1). This suggests that better conditions could be achieved to reach a uniformity in the densification of the cross section of the wood with these treatments. Likewise, the correlation analysis performed (Table 5) indicates that the heat 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 [34]. 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 [22].

With respect to the stabilization recovery, it 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 [12, 35]. During this process, the hemicelluloses hydrolysis, under the action of temperature and moisture, weakens the bond between the microfibers and lignin, freeing the internal stress [36]. In this case, it is possible that the wood of *A. acuminata* has a lower hydrolysis of the hemicelluloses so that the release of the elastic-strain is lower and therefore its stabilization recovery.

Other studies indicate that there is a significant reduction in the stabilization recovery in the THM process when a high degree of compression occurs, as a result of the rupture of cross-links responsible for memory effect in wood, plus lignin softening [23, 35]. However, in this case there were no differences between the degrees of compression between treatments, while between species the degrees of compression were very similar. However, the % of densification 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 also be the cause of a lower stabilization recovery (Table 3).

4.2. 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 though, none of the treatments reached 100% thickness recovery. The values of thickness swelling of densified wood of *A. acuminata* even 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 stabilization recovery than those of *V. ferruginea* and *V. guatemalensis* (Table 3).

The high values of thickness swelling obtained for *A. acuminata*, despite its high % of densification may be the result of the compression of its anatomical structures in the densification process. This species has vessels with diameter (75 µm) and length (150 µm), smaller than those of *V. ferruginea* (145 µm in diameter and 346 µm in length) and *V. guatemalensis* (169 µm in diameter and 339 µm in length) [10]. In stage 2 of the process (compression) the low-diameter vessels in *A. acuminata* become compressed until deformed but without collapsing (viscoelastic behaviour), unlike what can happen with the vessels of *V. ferruginea* and *V. guatemalensis*, which being larger tend to collapse. Thus, when the densified wood of *V. ferruginea* and *V. guatemalensis* was subjected to changes in temperature and humidity, these anatomical structures were unable to recover the thickness by absorbing moisture, contrary to *A. acuminata* that tended to recover its dimensions because its vessels did not present collapse due to their viscoelastic properties.

Regarding the mechanical properties, an improvement was observed in most of the properties analysed in relation to the wood without densification for the three species, with the exception of the MOE in static bending in some treatments of *V. guatemalensis* (Table 4). This means 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 properties [18]. In addition, Keckes et al. [37] point out, in relation to the change in the mechanical properties of densified wood, that deformation during densification does not deteriorate cell stiffness, since 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 the MOR in static bending and with the hardness in the three species (Table 6). Some authors point out that there is a relationship between the increase in MOE and MOR and the increasing degree of compression [22]. However, it should not be affirmed that the increase in the mechanical properties of densified wood was a product of the degree of compression obtained, since correlation analyses only relate the degree of compression with the MOE in static bending of *A. acuminata* (Table 6). In addition, the degree of compression was very similar between species as it derived from the target thickness of the densification process. The above statements indicate that in a densification process that started with wood with 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 degree of compression, % of densification, heat rate or stabilization recovery.

Finally, based on the previous analyses, it was not possible to determine which group of treatments (by temperature, time of compression and use or not of steam) improves the mechanical conditions of the densified wood. The correlation analysis showed that the mechanical properties were mostly influenced by the final density of the wood and the maximum load applied during stage 2 of the densification process, and these two parameters were related to the initial density of the wood in the three species (Table 5 and 6). Then, the initial density of the wood determined the final density of the densified wood and therefore its mechanical properties, so there was collinearity of the initial density and properties of the densified wood.

5. Conclusions

According to the results obtained, the densification process carried out allows densifying the wood of *A. acuminata* with a % of densification greater than 80% and the wood of *V. ferruginea* and *V. guatemalensis* with % of densification greater than 70%, where the densification process of the three species was influenced by the initial density of the wood. The treatments used influenced only variables such as heat rate and colour change, where the treatments T9 to T12 had the highest values.

Although the *A. acuminata* wood had a higher % of densification and a lower stabilization recovery, it obtained the highest percentage of thickness swelling, as 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 no influence, although a clear positive correlation of the properties with the final density and the maximum load was observed, being highly correlated with the initial density.

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Artículo 3: **Density profile and micromorphology variation of densified wood from three fast growth hardwood species in Costa Rica**

Roger Moya Roque

De:	Susan LeVan-Green <sue.levangreen@gmail.com>
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sue.levangreen@gmail.com
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Density profile and micromorphology variation of densified wood from three fast growth hardwood species in Costa Rica

Carolina Tenorio, Roger Moya, Mario Tomaziello Filho

Abstract

This study evaluates the effect of a thermo-hydro-mechanical densification process —using three temperatures, two compression times and the use or not of steam— and the effect of the initial micromorphology of wood on the density profile of densified wood of three low-density fast-growing hardwood species in Costa Rica (*Alnus acuminata*, *Vochysia ferruginea* and *Vochysia guatemalensis*). Four density profiles in the densified wood of the three species resulted from the densification temperature and the initial micromorphology of the wood. The initial diameter of the vessels affects the compression stage during the densification process and causes the formation of irregular density profiles. Therefore, this is the most important element in determining the type of density profile. The wood of *A. acuminata* (with small diameter vessels) densified at 180 °C tends to produce more uniform density profiles than the other two species that have larger diameter vessels. The time of compression and the use or not of steam as an initial stage in the densification process did not influence the types of density profile obtained. Similarly, the type of wood tissue and wood pattern, had no effect on the type of density profile. In conclusion, uniform and regular density profiles in densified wood are more likely to be achieved at high temperatures and with small diameter vessels.

Key words: THM densification, vessels, temperature, tropical species.

1. INTRODUCTION

There are various methods of wood densification. Among these, mechanical densification by transversal compression enhances the resistance and properties of low density woods (Wang and Cooper 2005; Fang et al. 2012). This compression is usually performed at temperatures above the glass transition temperature of the cell wall of the wood's various components, so that they deform instead of break when buckled (Laine et al. 2014). This action of the temperature during the densification process and its relationship with the glass transition of the cell wall of the wood during the time that the compression force is applied causes different effects on the anatomical elements of the wood, so that specific density profiles are produced through the thickness of the densified wood (Kutnar et al. 2009; Rautkari et al. 2011a).

The density profile represents the variation of the density through the thickness of the densified wood and it is a relevant attribute since it affects the physical and mechanical properties (Rautkari et al. 2013). The variation in density occurs because when transversely compressed, wood deformation (which is due to the collapse of the weaker, or less resistant to compression, anatomical elements) is not homogeneous (Blomberg and Persson 2004; Wang and Cooper 2005), producing an irregular density profile. Additionally, the density profiles that develop in compressed solid wood depend on other factors, such as the initial moisture content of wood, wood grain orientation, press temperature and time of compression (Rautkari et al. 2011b).

Appropriate densification processes allow to obtain uniform density profiles (Navi and Heger 2004; Sandberg et al. 2013). The thermo-hydro-mechanical densification process (THM) is a widely used densification method for achieving uniform density profiles as it prevents damage to wood structure when compressed (Navi and Heger 2004; Sandberg et al. 2013). A suitable wood densification process is achieved through the combined action of water steam, temperature and compression strength. The moisture induces a mechano-sorptive effect and further softens the wood, enabling mechanical compression of wood without cell wall fracture (Bao et al. 2017). 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).

Most research regarding THM densification has been conducted on wood from coniferous species and in close systems (Navi and Heger 2004), scarcely on wood from tropical hardwood species whose anatomical structures are more complex and have greater influence on the result of the process (Navi and Heger 2004). This is because the compression properties of the wood depend on its micromorphology (hierarchical structures): frequency, size and distribution of the anatomical structures (Darwis et al. 2017), which in the case of hardwood species are dominated by vessels, fibres and rays arranged in more complex matrixes (Gibson 2012) than the fibro-tracheids of conifers (Fratzl and Weinkamer 2007).

Alnus acuminata, *Vochysia ferruginea* and *Vochysia guatemalensis* are fast-growing hardwood species used in commercial reforestation programs in Costa Rica (Moya 2018). Lately, these species have been used in the development of THM densification processes and it has been observed that the densification process and the densified wood properties of these three species were influenced by the initial density of the wood (Tenorio and Moya 2021). Relevant studies have demonstrated the relationship between the density of the wood and the distribution, form and frequency of the different anatomical elements in hardwood species (Jacquin et al. 2017). Therefore, the initial morphology of these woods may have a clear relationship with the density profile and properties of the wood obtained after densification.

The objective of this study was to evaluate the effect of a thermo-hydro-mechanical densification process (employing three temperatures, two compression times and use or not of water steam) and the initial micromorphology of wood on the density profile of densified wood of three low-density fast-growing hardwood species in Costa Rica (*Alnus acuminata*, *Vochysia ferruginea* and *Vochysia guatemalensis*).

2. METHODOLOGY

2.1. Material and sample preparation

Wood from *Alnus acuminata*, *Vochysia ferruginea* and *Vochysia guatemalensis* coming from forest plantations in the provinces of Cartago and Alajuela in Costa Rica was used. The trees used were around 8 years old, therefore presented low heartwood content (Tenorio et al. 2016). Samples 300 mm long x 70 mm wide and 20 mm thick from each species were prepared. The type of sawing pattern of the wood —flat, quarter or rift sawn (Figure 1)— was determined and the sample tissue —sapwood, heartwood or a combination of both— was identified. The percentage of pieces presenting each characteristic was calculated (Table 1). Thickness, width, length, density and moisture content were determined for each one of the samples before densification (Table 1).

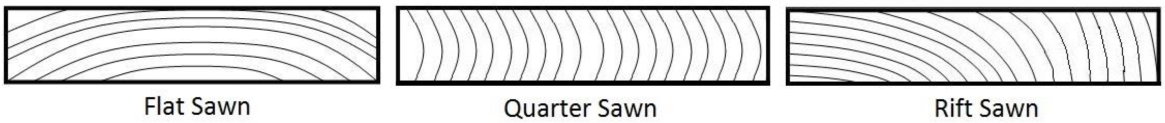

Figure 1. Types of wood patterns found for the three species.

The density was calculated as the ratio between weight and volume determined by measuring the initial thickness, initial width and length. The moisture content was calculated as the ratio of the initial weight to the oven-dry weight, expressed as a percentage according to ASTM D-4422 (ASTM 2016) (Table 1).

Table 1. General characteristics of the wood of the three species before the densification process.

<table>
<tr><th colspan="2">Species</th><th>Alnus acuminata</th><th>Vochysia ferruginea</th><th>Vochysia guatemalensis</th></tr>
<tr><td colspan="2">Initial thickness (mm)</td><td>19.7 (0.02)</td><td>20.0 (0.03)</td><td>20.4 (0.02)</td></tr>
<tr><td colspan="2">Initial wood density (g/cm^3)</td><td>0.43 (0.03)</td><td>0.45 (0.05)</td><td>0.39 (0.05)</td></tr>
<tr><td colspan="2">Initial moisture content (%)</td><td>9.97 (0.83)</td><td>10.68 (1.13)</td><td>12.56 (0.57)</td></tr>
<tr><td rowspan="3">Type of wood pattern (% of samples)</td><td>Flat sawn</td><td>60.00</td><td>30.83</td><td>63.33</td></tr>
<tr><td>Quarter sawn</td><td>10.83</td><td>25.83</td><td>2.50</td></tr>
<tr><td>Rift sawn</td><td>29.17</td><td>43.33</td><td>34.17</td></tr>
<tr><td rowspan="3">Type of wood tissue (% of samples)</td><td>Sapwood</td><td>78.33</td><td>5.83</td><td>30.00</td></tr>
<tr><td>Heartwood</td><td>20.83</td><td>45.83</td><td>39.17</td></tr>
<tr><td>Sapwood and heartwood</td><td>0.83</td><td>48.33</td><td>30.83</td></tr>
</table>

Legend: the values in parenthesis mean standard deviation.

2.2. Densification process

The densification process was tested under 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 (10 and 15 minutes) and with or without water steam, as the initial stage before compressing the wood. Thus, the work was performed with 12 treatments and 10 samples per treatment, for a total of 120 samples per species.

The densification process for the three species was the one described in (Tenorio and Moya 2021), consisting of three stages: 1) steaming or heating, where steam and heat was applied to half of the wood samples during 10 minutes, while the other half of the samples were heat applied only; 2) compression, where the wood samples were compressed perpendicular to the grain until reaching the target thickness of 9 mm (degree of compression of 55%) during 10 to 15 minutes, keeping the same temperature of stage 1 and 3) stabilization, where the samples were kept compressed and heated but without load application for 10 more minutes.

2.3. Evaluation of the densification process

The thickness of each piece was determined at the conclusion of stages 2 and 3 of the densification process (compression and stabilization thickness). After the process of densification, the width, length, weight and density of each wooden piece was determined. The final density (after densification) was calculated as a relation between weight and volume, determined by measuring the stabilization thickness, width and length.

The degree of compression was calculated as the ratio of the initial thickness to the compression thickness, expressed as a percentage. The % of densification was calculated as the relationship between the initial density (before the densification process) and the final density (after the densification process).

2.4. X-ray densitometry

Density profiles were determined for densified and undensified wood of each species. For each species 10 samples per densification treatment were used, measuring 50 mm x 50 mm x densified thickness obtained in the densification process, and 10 samples of undensified wood with dimensions 50 mm x 50 mm x 20 mm in thickness. Weight, width, height and thickness were determined for each sample. The density was measured at intervals of 0.1 mm through the thickness of the specimens using an X-ray densitometer (QMS, Model QDP-01). The density profile was measured with respect to the specimen thickness, through the specimen’s width in transversal orientation.

2.5. Microscopic examination

Sections 10 mm x 50 mm x densified thickness were taken from the samples employed to determine the density profiles. In the case of undensified samples, sections 10 mm x 50 mm x 20 mm were taken. All samples were polished in their transversal sections using a sander (Struers, model Tegramim 30). The samples were stuck on the support as shown in Figure 2a using a double-sided adhesive tape. For the three species the sanding and polishing program in Table 2 was used.

Table 2. Sanding characteristics used to polish the samples of the three species.

Sandpaper number	Sanding time (minutes)	Base speed (rpm)	Sample holder speed (rpm)
220	1.5	150	50
500	0.5	80	50
500	3.0	150	50
800	0.5	80	50
800	3.0	150	50
1200	0.5	80	50

After polishing, a microscope (Zeiss, model Axioscope) was used to take the pictures of the anatomical structures of each sample, using an Axicam 503 Color camera in reflexion mode using a 50x lens (Figure 2b). The image editing ZEN program was used. The undensified samples were also photographed in reflexion mode using a 10x lens. The undensified and the densified polished samples were photographed using an Epson scanner (model Expression 12000XL) to obtain a macro image and observe the density profiles.

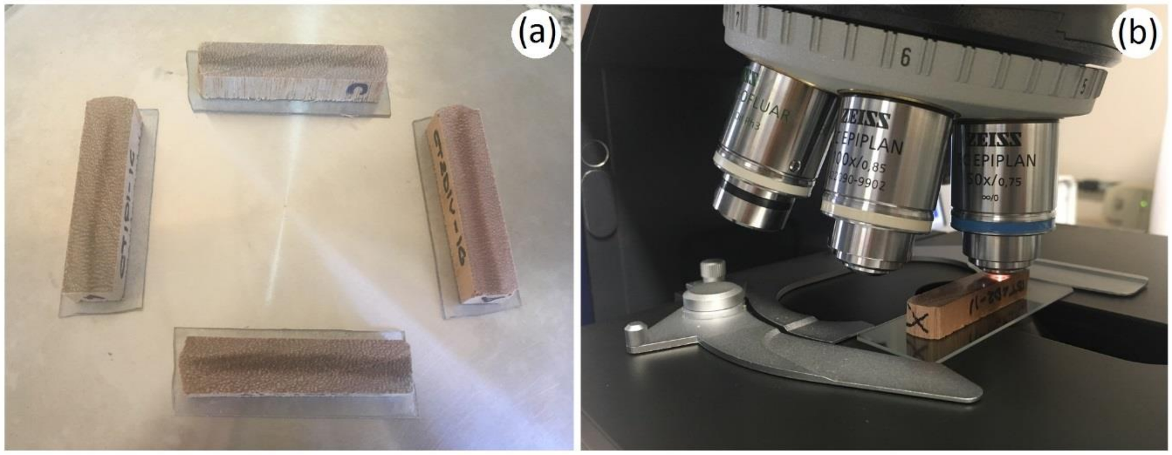

Figure 2. Densified wood samples placed in the sander holder (a); polished densified sample placed in the microscope (b).

2.6. Statistical analysis

Compliance of the measured variables with the assumptions of normal distribution, homogeneity of variances and the presence of outliers, was verified. An analysis of variance was applied to verify the effect of the densification treatments (temperature, compression time and use or not of steam) on each one of the variables obtained during the process of densification (degree of compression, % of densification and final density). Tukey's test was used to determine the statistical differences between the means of the variables previously indicated. An analysis of variance was applied to verify the effect of the densification treatments, the type of wood pattern and the type of wood tissue in the type of density profile. The analyses of variance and the Tukey test were performed using the SAS software (SAS Institute Inc., Cary, NC).

3. RESULTS

3.1. Characteristics of the process of densification

Table 3 presents the results obtained as part of the evaluation of the densification process. The three species showed approximately 55 % degree of compression and no statistical differences were observed between the treatments applied to *A. acuminata* and *V. ferruginea*. For *V. guatemalensis* the treatment of 160 °C with 15 minutes compression time and with steam application presented the lowest degree of compression at 51.66 %. The % of densification for *A. acuminata* was 84.86 % on average and for *V. ferruginea* 75.47 %. No differences were observed between the treatments applied. In the case of *V. guatemalensis* the lowest densification percentage was 58.92 %, shown by the treatment 160 °C, 15 minutes compression time and steam application, while the highest was 87.37 % with the treatment 180 °C, 10 minutes compression and steam application (Table 3).

No differences were observed between the averages of final density in the densification treatments for *A. acuminata* and *V. ferruginea*; both species presented a final density average of 0.79 g/cm^3 (Table 3). As for *V. guatemalensis* some differences between the treatments appeared; however, the treatments at higher temperature presented higher density averages. The treatment at 160 °C with 10 minutes compression without steam presented the lowest value of density average, at 0.59 g/cm^3, while the treatment at 180 °C, 10 minutes compression with steam presented the highest value at 0.74 g/cm^3 (Table 3).

Table 3. Wood characteristics obtained in the densification process of three forest species.

Species	Temperature (°C)	Time of compression (min)	Steam	Degree of compression (%)	% of densification	Final density (g/cm^3)
Alnus acuminata	140	10	Si	54.93^{A} (0.67)	88.16^{A} (5.61)	0.80^{A} (0.06)
		10	No	54.56^{A} (0.35)	84.22^{A} (6.29)	0.82^{A} (0.06)
		15	Si	55.01^{A} (0.66)	82.77^{A} (11.13)	0.77^{A} (0.05)
		15	No	54.94^{A} (0.77)	85.85^{A} (8.64)	0.82^{A} (0.05)
	160	10	Si	54.85^{A} (0.47)	88.45^{A} (8.94)	0.81^{A} (0.05)
		10	No	54.82^{A} (0.46)	86.22^{A} (6.63)	0.77^{A} (0.06)
		15	Si	54.85^{A} (0.55)	83.46^{A} (10.06)	0.82^{A} (0.05)
		15	No	54.81^{A} (0.50)	80.03^{A} (12.87)	0.76^{A} (0.06)
	180	10	Si	54.35^{A} (0.43)	81.59^{A} (6.89)	0.76^{A} (0.07)
		10	No	54.82^{A} (0.69)	88.12^{A} (12.53)	0.79^{A} (0.05)
		15	Si	54.88^{A} (0.44)	79.93^{A} (6.26)	0.78^{A} (0.05)
		15	No	55.10^{A} (0.59)	89.48^{A} (9.15)	0.76^{A} (0.05)
Vochysia ferruginea	140	10	Si	55.11^{A} (0.97)	70.48^{A} (8.52)	0.78^{A} (0.12)
		10	No	55.47^{A} (0.55)	72.95^{A} (11.22)	0.80^{A} (0.07)
		15	Si	55.57^{A} (0.58)	76.47^{A} (11.90)	0.81^{A} (0.08)
		15	No	55.41^{A} (0.44)	70.32^{A} (10.41)	0.76^{A} (0.11)
	150	10	Si	55.41^{A} (0.67)	80.10^{A} (11.65)	0.84^{A} (0.05)
		10	No	55.00^{A} (0.83)	69.18^{A} (5.87)	0.76^{A} (0.11)
		15	Si	54.63^{A} (0.55)	77.60^{A} (11.72)	0.79^{A} (0.06)
		15	No	54.98^{A} (0.99)	81.61^{A} (13.12)	0.81^{A} (0.09)
	160	10	Si	55.77^{A} (1.09)	77.34^{A} (8.82)	0.78^{A} (0.06)
		10	No	55.57^{A} (0.72)	71.50^{A} (8.98)	0.80^{A} (0.05)
		15	Si	55.22^{A} (0.62)	75.04^{A} (7.48)	0.77^{A} (0.07)
		15	No	55.35^{A} (0.69)	82.52^{A} (17.29)	0.77^{A} (0.08)
Vochysia guatemalensis	140	10	Si	56.49^{A} (1.78)	74.50^{ABC} (8.06)	0.66^{ABC} (0.04)
		10	No	56.24^{A} (0.47)	69.85^{BC} (11.94)	0.67^{ABC} (0.06)
		15	Si	56.38^{A} (1.61)	82.00^{AB} (10.38)	0.63^{BC} (0.06)
		15	No	56.08^{A} (0.54)	80.45^{AB} (7.85)	0.65^{BC} (0.05)
	160	10	Si	55.73^{AB} (2.21)	74.22^{ABC} (7.06)	0.67^{ABC} (0.04)
		10	No	56.43^{A} (1.66)	76.90^{AB} (15.33)	0.59^{C} (0.05)
		15	Si	51.66^{B} (6.20)	58.92^{C} (14.36)	0.70^{AB} (0.06)
		15	No	55.92^{AB} (0.79)	77.74^{AB} (5.47)	0.68^{AB} (0.04)
	180	10	Si	56.97^{A} (2.65)	87.37^{A} (7.58)	0.74^{A} (0.04)
		10	No	56.91^{A} (2.03)	78.54^{AB} (10.15)	0.67^{ABC} (0.06)
		15	Si	55.58^{AB} (1.83)	75.92^{AB} (10.92)	0.70^{AB} (0.09)
		15	No	54.65^{AB} (5.80)	75.78^{AB} (8.81)	0.70^{AB} (0.05)

Legend: the values in parenthesis mean standard deviation. Different letters for each parameter represent statistical differences between different treatments (significances at 95%).

3.2. Morphology of the undensified and densified wood

Cross-sections of the undensified wood micromorphology of the three species are presented in Figure 3. The three species presented solitaire and multiple diffuse vessels. 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 diameter (75 µm) and short in length (150 µm); *V. ferruginea* presents 2.84 pores/mm^2 of medium diameter (145 µm) and long (346µm) and *V. guatemalensis* presents 2.88 pores/mm^2, with regular size diameters (169 µm) and long, 339 µm on average.

Fiber are irregularly arranged in rows perpendicular to the growth rings or parallel to ray parenchyma, typical of non-stored fibers. The rows are crooked due to the presence of large vessels. *A. acuminata* formed more regular fiber rows than did the other two species. The shape and dimension of cross-sections of fibers are irregular (Moya et al. 2019). Round fibers, elliptical in tangential and radial direction, rectangular also in tangential and radial direction, square and rhomboidal fibers, among other shapes, can be observed. *A. acuminata* features finer rays than those of the other two species, which are multi-serial, showing 44-8 series per ray (Moya et al. 2019).

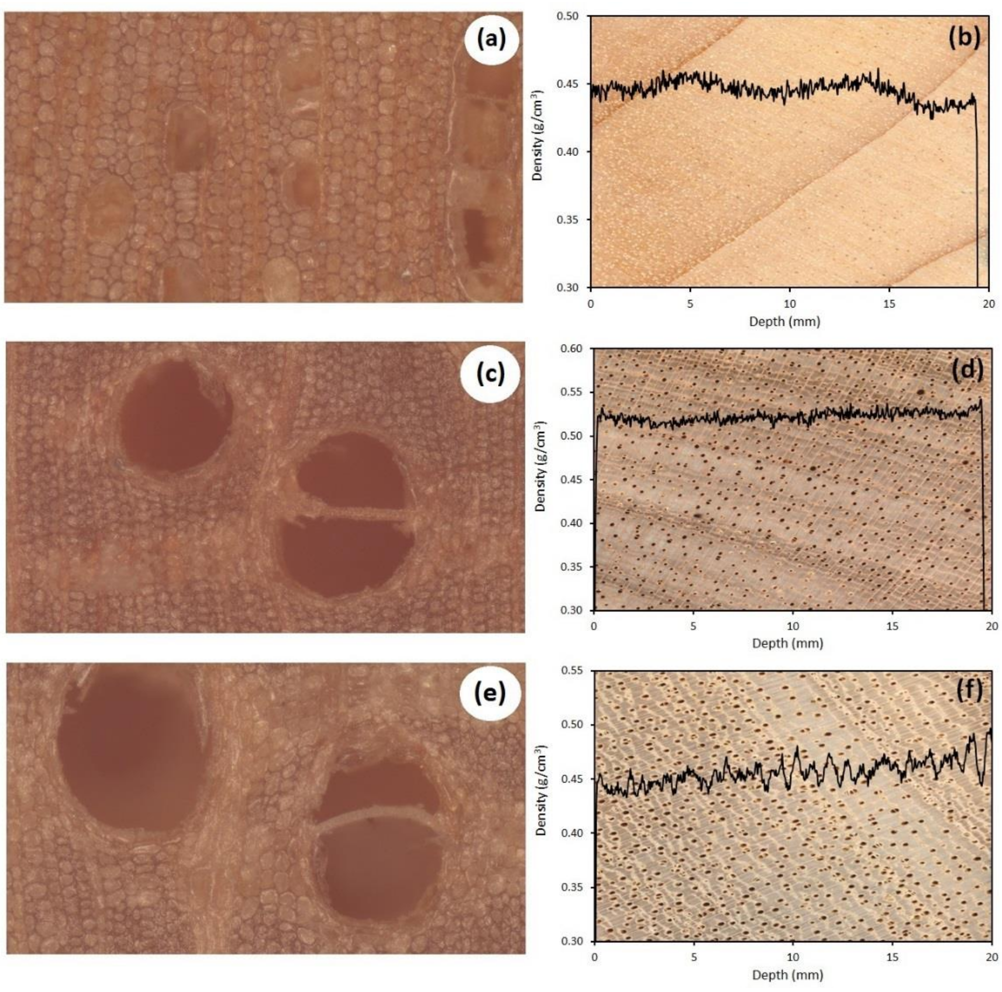

Figure 3. Anatomical features and density profiles of undensified wood of *Alnus acuminata* (a, b), *Vochysia ferruginea* (c, d) and *Vochysia guatemalensis* (e, f).

Figure 4 shows the anatomical structures in the cross section of densified wood of the three species. The effect of the densification process in the three species can be perceived in the size of their anatomical structures and the differences between species occur especially in the vessels. The fibers and rays are observed in areas of lower density of the profiles in figures 4a, 4d and 4g. Although the fibers maintain their original shape mostly, some deformations of the densification process are already evident (Figure 4a). The rays are wavy, especially in *A. acuminata* rays, which are thinner than those of the other two species.

Figures 4b, 4c, 4e, 4f, 4h and 4i presented cross-sections in areas of high density. The vessels tended to collapse completely, forming a row horizontal or perpendicular to the application of the compression strength (Figure 4c, 4f and 4i). The deformation occurring in the vessel caused the close rays to collapse and lose their original shape. Most fibers tended to take an “S” shape, especially those that were close to the collapsed vessels (Figure 4f). Importantly, in the case of the rays of *A. acuminada* the frequency of the waves was greater than those of *V. guatemalensis* and *V. ferruginea*.

3.3. Density profiles

The undensified wood of the three species presented almost uniform density throughout its thickness (Figure 3b, d and f). In the analysis for the densified wood four density profile patterns were observed throughout the sample thickness for the three species: 1) the thickness center of the sample having the highest density peak; it is possible to observe that the areas closest to the surface present lighter hue and more open pores compared to the central area of the sample, which is darker with almost closed pores (Figure 5a); 2) two high density areas standing at the center of the sample; the surface and the center of the sample feature open pores and light hue, while the areas close to the surface have close pores and darker hue (Figure 5b), 3) uniform density pattern and closed pores throughout the thickness of the sample; dark colored sample (Figure 5c) and 4) one side of sample thickness has the highest peak density; an area close to the sample surface presents closed pores and dark hue, while the opposite area shows open pores and light hue (Figure 5d).

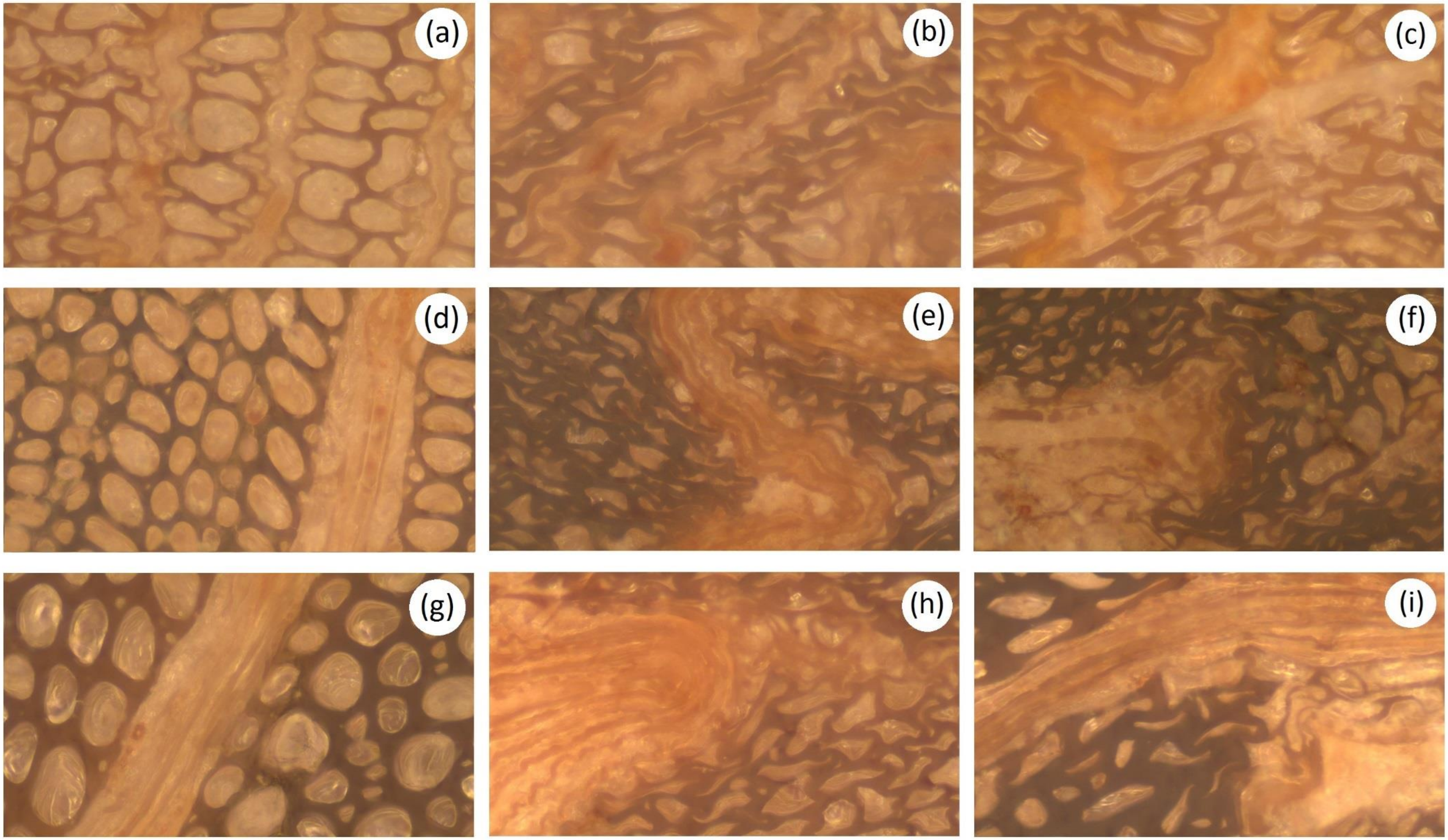

Figure 4. Anatomical features of densified wood of *Alnus acuminata* (a, b and c), *Vochysia ferruginea* (d, e and f) and *Vochysia guatemalensis* (g, h and i).

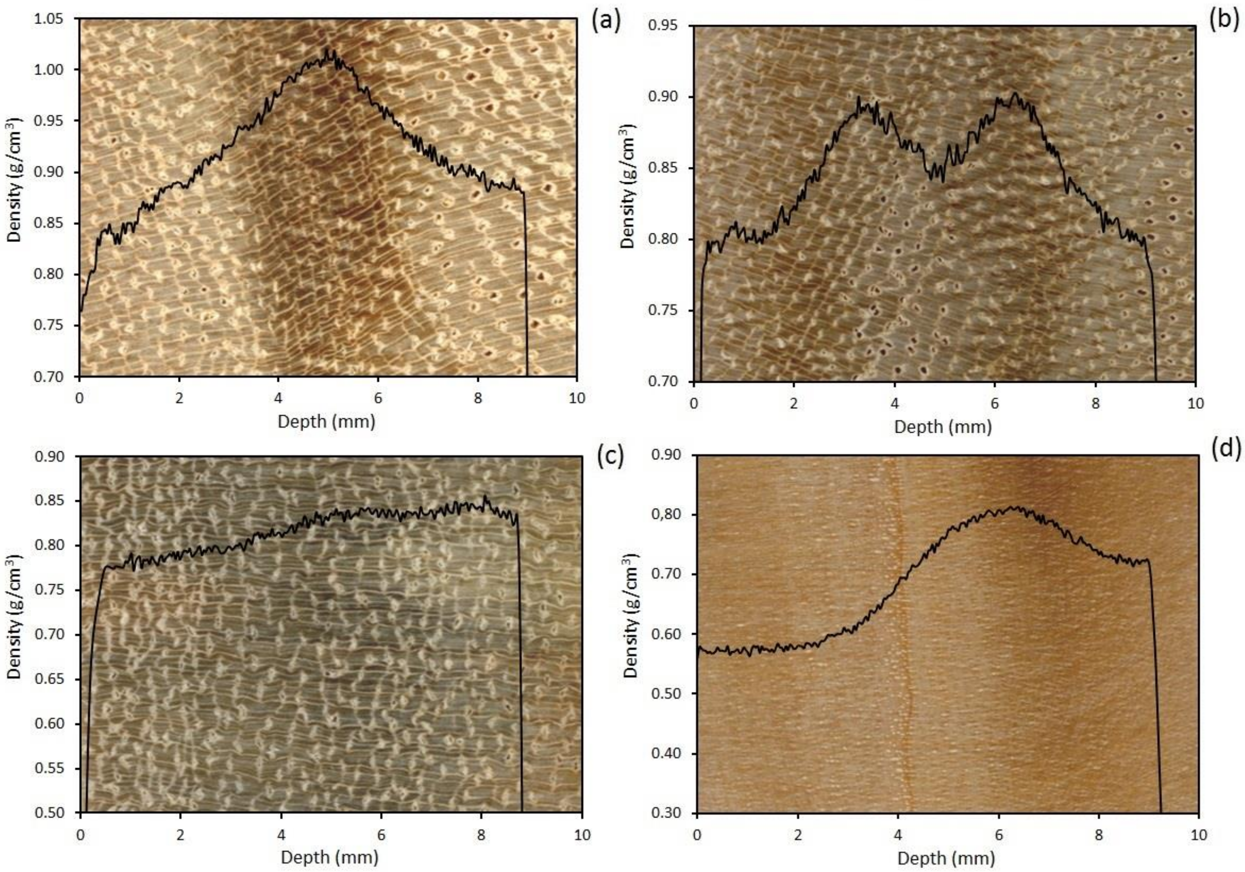

Figure 5. Profile density patterns of the densified wood of the 3 species, (a) profile type 1, (b) profile type 2, (c) profile type 3 and (d) profile type 4.

The variance analysis conducted to determine the influence of the densification treatments (temperature, compression time and use or not of steam), the type of wood pattern and wood tissue in the density profiles showed that only the temperature affected the type of density profile in the three species (Table 4).

Table 4. Effect of densification treatments, type of wood pattern and wood tissue in the density profiles of the densified wood of the three species.

Treatment	*Alnus acuminata*	*Vochysia ferruginea*	*Vochysia guatemalensis*
Temperature	13.96**	3.91*	5.65**
Time	1.89^{NS}	0.03^{NS}	0.02^{NS}
Steam	0.47^{NS}	1.38^{NS}	2.27^{NS}
Type of wood pattern	1.40^{NS}	2.74^{NS}	1.27^{NS}
Type of wood tissue	0.76^{NS}	0.18^{NS}	1.76^{NS}

Legend: * represents statistical significances at 90% and ** represents statistical significances at 95%.

Figure 6 shows the effect of temperature in the density profiles for each species. For *A. acuminata* (Figure 6a) the percentage of samples with density profile 1 decreased with increasing temperature. Density profile 2 presented samples only at the lowest temperature (140 °C). In density profile 3 the percentage of samples increased with increasing temperature and it is the profile with highest percentage of samples at the highest temperature (180 °C). Density profile 4 presented similar sample percentage at the three temperatures.

Regarding *V. ferruginea* (Figure 6b), density profile 1 showed the greatest percentage of samples at 150 °C and 160 °C. The percentage of samples with density profile 2 decreased with increasing temperature; as for density profile 3, sample percentage increased with temperature and with density profile 4 samples were only observed at the lowest temperature (140 °C).

As for *V. guatemalensis* (Figure 6c), density profile 1 presented the highest sample percentage at the three temperatures (140 °C, 160 °C and 180 °C). Sample percentage with density profile 2 decreased as the temperature increased. Samples with density profile 3 were only observed at the highest temperature (180 °C), being this profile in second place in this category of temperature, while density profile 4 was present in a very low percentage of samples at 140 °C and 160 °C.

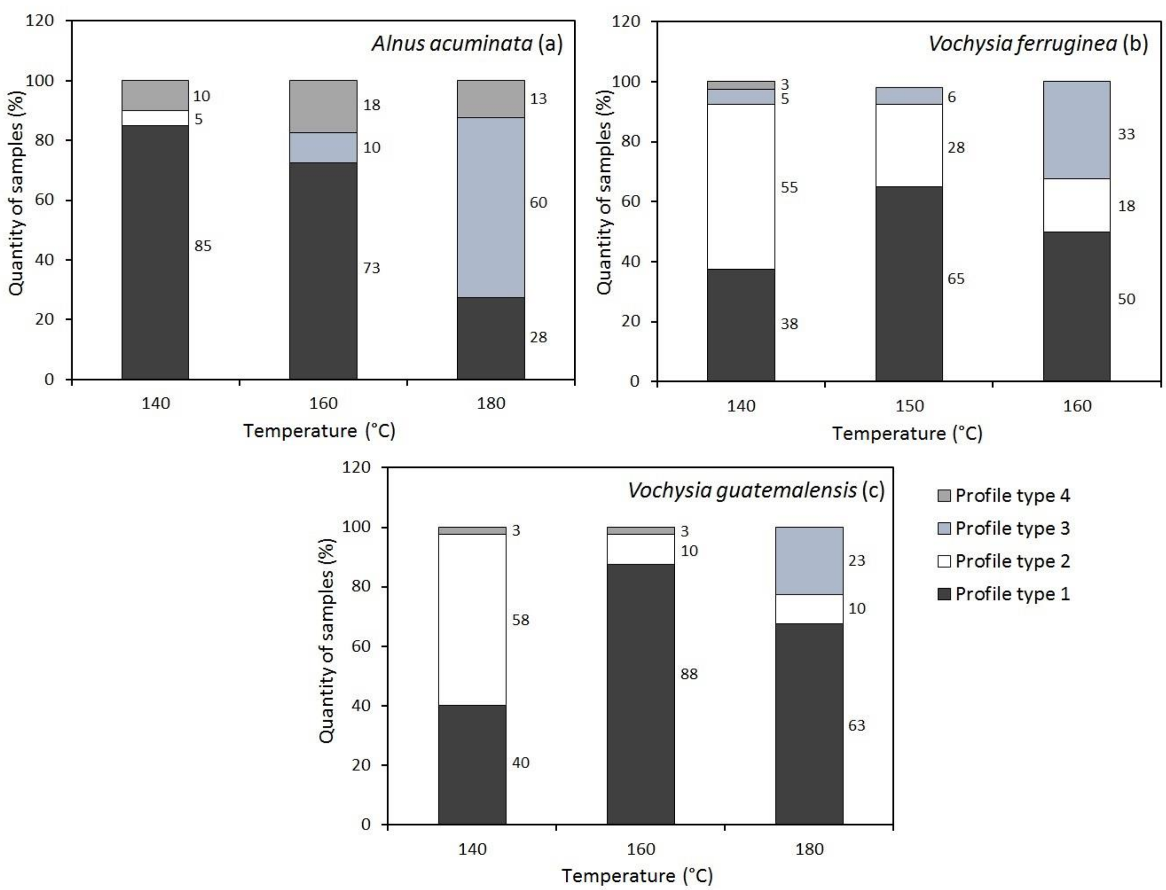

Figure 6. Effect of temperature in the density profiles of the densified wood of the three species.

4. DISCUSSION

The types of density profile reflect the variation in the compression of the anatomical elements in the cross-section of wood (Figure 5). Low density regions have areas with vessels that maintain their original shape, while in high density regions the vessels are completely deformed (Figure 4), meaning that the densification treatment significantly changed the micromorphology of the wood of the three species (Figure 4 and 5).

Profile variation (areas of high and low density), mainly density profiles types 1-3, shows that reduction of the empty spaces did not occur uniformly. In this regard, Wang and Cooper (Wang and Cooper 2005) point out that transversal compression of the wood is highly dependent on its anatomical structure, which means that the deformation is not homogeneous, resulting in different density profiles in the three species studied (Figure 5). Areas of higher density showed greater collapsing of their structures, making the micromorphology of this area different from the micromorphology of the areas of lower density within the same sample (Figure 4).

According to Kutnar et al. (Kutnar et al. 2009) the morphology of densified wood depends largely on the % of densification. High degree of densification causes a strong reduction of the empty spaces in the wood, evidenced in the high density regions of density profiles 1-3 (Figures 5a-b, 5d). During the THM densification process, in these types of density profiles the volume of the empty spaces is drastically reduced since the cells are deformed but their cell wall do not fracture. In these regions of high density profiles the vessels collapse and flatten in the direction in which the compression force was applied, while the rays appear to buckle and the fibers take an "S" shape in the areas high density, especially the regions near the vessels (Figure 4b-c, 4e-f, 4h-i).

Likely, in the case of *A. acuminata*, a species with vessels of smaller diameter (Figure 3a) in relation to the vessels of *V. ferruginea* and *V. guatemalensis* (Figure 3c and 3e), the compression transmission was performed more homogeneously, which led to greater number of densified wood with density profiles type 3 (Figure 6a), that is, uniform density profiles throughout the thickness of the samples (Figure 5c). This effect of vessel's size can also be observed when comparing the densified wood of the other two species, where *V. ferruginea* has a larger number of samples with density profile type 3 in relation to *V. guatemalensis*, which has the largest diameter of vessels (Figure 3c).

Likewise, the distribution and size of the fibers and the characteristics of the rays of each of the species also affect the formation of density profiles after compression. For example, in *A. acuminata* the shape and size of the fibers present a regular arrangement, as storied fiber; distribution that does not occur in the other two species. Its regular fiber arrangement and greater frequency of ray bending compared to the other two species, allows a more uniform the transmission of compression, so that less density variation or a greater number of density profiles type 3 are achieved (Figure 5c).

In the case of the densification treatments used in this study (three temperatures, two compression times and the use or not of steam), the type of wood pattern and wood tissue, only the temperature influences the type of density profile of densified wood of the three species (Table 4). Although most of the densified samples of the three species had a density profile type 1, there is a clear tendency to increase the percentage of samples with density profile 3 (Figure 3c) as the densification temperature increases, 180 °C for *A. acuminata* and V*. guatemalensis* and 160 °C for *V. ferruginea* (Figure 4).

This behavior is due to the fact that higher temperature enables relaxation of the inner stress and even minor thermal degradation of the cell-wall components takes place, leading to a more stable state after compression (Laine et al. 2014). More specifically, a higher temperature might lead to the breaking of the existing covalent and hydrogen bonds and the formation of new cross-linkages and hydrogen bonds between the cellulose and hemicellulose fixing the deformation (Dwianto et al. 1999; Navi and Heger 2004). In contrast, with lower temperatures there is very little stress relaxation and thus, the deformation is expected to be mainly elastic. The elastic energy is stored in the cell walls and as the load is removed, the stress is released causing immediate spring-back deformation (Dwianto et al. 1999; Navi and Heger 2004). This could lead to formation of density profiles types 1 and 2 (Figures 5a and b).

Density profiles type 4 (Figure 5d) were the least frequent in the densification process (Figure 6). Although both metal plates were monitored to maintain stability and equal temperature throughout the process, one of the plates must have presented higher temperature than the other at some time, causing higher heat transmission on one side of the thickness of the samples, thus creating a zone of higher density compared to the opposite zone.

5. CONCLUSIONS

The densified wood of the three species presented four density profiles as a result of the densification temperature and the initial wood micromorphology. The initial size of the anatomical structures, such as vessels, affects the compression stage during the densification process and causes the formation of less homogeneous or irregular density profiles. In the case of *A. acuminata*, the use of a temperature of 180 °C during the densification process and the small diameter of its vessels tend to produce more uniform density profiles (type 3) than the other two species that have larger vessels.

Of the densification treatments used, the time of compression and the use or not of steam as initial stage in the densification process had no influence on the types of density profile obtained. Similarly, the type of wood tissue and wood pattern had no effect on the type of density profile.

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Artículo 4: Flooring evaluation of a thermo-mechanical densified wood from three hardwood tropical species

Flooring evaluation of thermo-mechanical densified wood from three hardwood tropical species

Carolina Tenorio, Roger Moya

Abstract

Densification is one method used to modify low-density woods to make them achieve the hardness required for flooring application. The purpose of this study was to investigate the effect of thermo-mechanical densification with pre-heating in wood of *Alnus acuminata*, *Vochysia ferruginea* and *Vochysia guatemalensis* from fast growth plantations, seeking to stabilize and reduce spring-back of the densified wood and evaluate its performance in flooring applications. Parameters of the process of densification and characteristics of the densified wood flooring were analysed. The results showed that the wood of the three species turned dark. This is because brightness (L*) diminished and yellowness (b*) and redness (a*) increased. Weight loss due to pre-heating was statistically higher in *V. ferruginea* and *V. guatemalensis*. The final density and spring-back were statistically equivalent in the three species. The percentage of densification of *A. acuminata* and *V. guatemalensis* were statistically higher than for *V. ferruginea* and the degree of compression was statistically higher in *V. guatemalensis*. As for flooring evaluation, the percentage of densification, temperature and time of pre-heating affected the behaviour of densified wood flooring. The low percentage of densification and high weight loss in the pre-heating stage caused greater values of wear, wear index, residual deformation, residual indentation for the falling ball indentation test, and more damages in the surface indentation test in wood of *V. ferruginea*.

Keywords: tropical species, low density, spring-back, flooring properties.

1. Introduction

Wood as a constructive element is aesthetically agreeable, provides thermal isolation and mechanical resistance, in addition to being easy to maintain and long lasting if cared for appropriately (Blanco-Flórez et al. 2015). However, compared to other materials used for flooring, wood suffers damage caused by objects, wear from abrasive elements and people traffic, among others (Blanchet et al. 2003). The main factor when choosing wood for flooring is its density (Zhou et al. 2019). There is high correlation between the density of the wood and its mechanical properties; high density provides enhanced mechanical resistance, therefore improved hardness (Sadatnezhad et al. 2017). For this reason, solid wood from high density species has been widely used for flooring (Feifel et al. 2015).

In Costa Rica, wood with densities above 0.7 g/cm^3 has been used for flooring (United States Office of Inter-American Affairs 1943). Currently, however, the woods commonly used for flooring are banned, listed as endangered or threatened (Moya et al. 2013). As a result, prices of these woods are high, which promotes importation of woods or wood-based products to satisfy the demand of this sector (Serrano and Moya, 2011). This has led to the search for new options to replace high density woods with lower density woods produced in fast-growing forest plantations (Nölte et al. 2018).

Alnus acuminata, *Vochysia ferruginea* and *Vochysia guatemalensis* are among the forest plantations used in Costa Rica, the three of them fast-growing hardwood species (Moya 2018) with low density woods (specific gravity less than 0.45), considered in the market as easy-to-work softwoods (Tenorio et al. 2016a). Because of their low density, the mechanical properties of these woods are also low, therefore not convenient for structural uses, but for products with low structural demand in low commercial value markets (Serrano and Moya 2011).

On the other hand, the densification process can make replacing high density woods for low or moderate density woods possible, if these are modified to reach the hardness requirements needed for some uses, such as flooring (Fang et al. 2012). There are several processes of densification, generally consisting of the following stages: (i) soften the structure of the wood by using temperatures between 150 - 200 °C and convenient moisture contents; (ii) compress the wood by means of two metal plates and (iii) stabilize and keep the deformation obtained in the wood (Sandberg et al. 2013).

Thermo-mechanical densification is the simplest densification method. It reduces the empty spaces of the wood cells, called lumen, through the joint action of heat and radial compression (Navi and Heger 2004). Thermomechanical densification gives some enhanced mechanical properties to the compressed wood, but the compression set is unstable, and wood tends to recover its original shape even after large deformation, especially under conditions of high humidity and high temperature (Navi and Heger 2004; Sadatnezhad et al. 2017). This phenomenon is known as spring-back.

The permanent fixation of large deformation as a result of compression is very important for densified wood to be utilized as an engineering material (Inoue et al. 2008), especially for flooring applications. Many studies have been conducted regarding stabilization of compressed wood with techniques such as impregnation with resins and physical or chemical treatments (INOUE et al. 1993; Kutnar and Kamke 2012; Pelit et al. 2016). Among these techniques, physical treatments are preferred, such as steaming or heating, since they do not require any chemicals (Darwis et al. 2017). Heating and/or steaming before compression have been found to reduce densified wood spring-back significantly (Inoue et al. 2008; Darwis et al. 2017). However, heat treatments have the disadvantage of reducing the strength properties of the wood due to weight loss and chemical degradation of wood, when it exposed to high temperatures (Perçin et al. 2015).

Densification of wood from forest plantations in Costa Rica has been successfully tested in *A. acuminata*, *V. ferruginea* and *V. guatemalensis* wood (Tenorio and Moya 2020, 2021; Tenorio et al. 2020). In addition to adapting to the process of densification (Tenorio and Moya 2020), the characteristics of the densified wood of these species are appropriate and comparable to those of other high density tropical woods (Tenorio and Moya 2020, 2021). However, the inconvenient found in the treatment used in the initial stage of the process —heating and steaming for 10 minutes between two metal plates— is that it produces low dimensional stability and high spring-back in the densified wood obtained (Tenorio and Moya 2020), making it unsuitable for flooring application.

Thus, the objective of the present study was to investigate the effect of densification using pre-heating on stabilization and spring-back reduction of densified wood; then, evaluate the behaviour of this wood in flooring application. *A. acuminata*, *V. ferruginea* and *V. guatemalensis* woods were used and variables pertaining the process of densification (final density, degree of compression, % densification, spring-back and color change), as well as the characteristics of wood for flooring application (compression shear strength of the glue line, resistance to abrasion, resistance to indentation by falling ball, concentrated loading and surface indentation), were analysed. The results obtained will provide the necessary elements to make these species with low values in mechanical properties suitable for use in new high-performance wood products such as flooring, after being modified through a thermomechanical densification process.

2. Methodology

2.1. Origin and characteristics of the wood before densification

Alnus acuminata, *Vochysia ferruginea* and *Vochysia guatemalensis* woods coming from fast growing forest plantations in Cartago and Alajuela in Costa Rica were tested. The trees used were around 8 years old. The wood used was mostly sapwood, as trees this age generally present low heartwood content (Tenorio et al. 2016b). Wood samples 300 mm long x 70 mm wide and 20 mm thick were prepared from each species. The dimensions, density and moisture content of each sample were determined previous to densification (Table 1). The density was calculated by the ratio of weight and volume, determined by measuring the initial thickness, initial width and length. The moisture content was calculated as the ratio of the initial weight and dry oven weight, expressed as a percentage according to ASTM D-4422 standard (ASTM 2007).

The initial color of the wood before densification was also determined by means of a HunterLab Mini Scan XE Plus spectrophotometer, CIEL*a*b* system. The range of this measurement is 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 an illumination standard of D65 (corresponding to daylight in 6500 K). The mini Skan XE Plus generated three parameters for each measurement, namely: L* (luminosity), a* (tendency of color from red to green), and finally b* (tendency of color from yellow to blue).

Table 1. General characteristics of the wood of three forest species before densification.

Species	*Alnus acuminata*	*Vochysia ferruginea*	*Vochysia guatemalensis*
Initial thickness (mm)	19.88 (0.41)	19.88 (0.38)	20.05 (0.32)
Initial wood density (g/cm^3)	0.42 (0.03)	0.45 (0.05)	0.42 (0.06)
Initial moisture content (%)	10.75 (0.33)	10.99 (0.26)	10.87 (0.67)

Legend: the values in parenthesis mean standard deviation.

2.2. Process of densification

The process of densification for the three species was similar to the one described by Tenorio and Moya (2021), consisting of three stages: 1) heating, where heat was applied to the wooden samples inside a closed oven during 60 minutes, 180 °C for *A. acuminata* and *V. guatemalensis* and 160 °C for *V. ferruginea*; 2) compression, where the samples were compressed perpendicularly to the grain until reaching the target thickness of 9 mm (55% compression degree) during 15 minutes keeping the temperature used in stage 1 (heating) of the process; 3) stabilization, where the samples were kept pressed and heated but without load during 10 more minutes. After densification, the samples were placed into an oven at 103 °C for 24 hours. In total, 50 samples were densified per species.

2.3. Evaluation of the process of densification

At the end of stage 1 (heating) wood weight loss was calculated as the ratio between the initial weight of the wood and the weight after stage 1, expressed as a percentage of the initial weight. In addition, wood color was determined as described above.

During the process, thickness of each wood sample was determined at finalizing stages 2 and 3 (compression and stabilization thickness). After densification, the dimension, weight, color, density and percentage of densification of each sample were determined. For color measurement the same procedure used before densification was utilized. Two color changes (ΔE*) were observed, the first after stage 1 (heating), which was called heating ΔE*, and the second after finishing the process of densification (after placing the samples into the oven for 2 hours), called densification ΔE*. The density was calculated as the ratio between weight and volume of the wood sample after stage 3 (stabilization). For volume, the sample's dimensions (stabilization thickness, length and width) were determined. The densification percentage was calculated as the relation between the initial density and the density of the densified sample.

The thicknesses determined in stages 2 and 3 (compression and stabilization thickness), were used to determine the degree of compression and the spring-back of the sample thickness. The degree of compression was calculated as the ratio between 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 correlation between the compression thickness and the stabilization thickness, also expressed as a percentage.

2.4. Flooring evaluation

In the evaluation of the densified wood flooring, the shear strength of the glue line was determined for two densified pieces glued with an adhesive used to glue flooring, following the norm ASTM D905-08 (ASTM 2013). The adhesive was Advantage EP-950A from Franklin Adhesives Polymers (Franklin Division, Columbus, Ohio, USA). For this test, 20 samples per species were used.

Resistance to abrasion was determined following the ASTM D4060-14 standard (ASTM 2014). Three square samples of 100 mm densified thickness and 9 mm in thickness were used and the wear index was determined using equation:

$$I = \frac{(A-B)*1000}{C} \quad \text{(Equation 1)}$$

Where: I = wear index, A = weight of test specimen before abrasion (mg), B = weight of test specimen after abrasion (mg) and C = number of cycles of abrasion recorded.

Three indentation tests were performed with densified wood: falling ball indentation, concentrated loading and surface indentation, following the ASTM D2394-17 standard (ASTM 2017). As for falling ball indentation, 9 samples per species were tested from seven heights, starting at 122 cm up to 213 cm with 15 cm intervals. For the concentrated loading test, three boards 457 mm^2 were made using the densified wood of each species glued on a structural plywood 19 mm thick. The adhesive used was Advantage EP-950A. As support for the boards three pieces of *Gmelina arborea* were used, with dimensions of 5 cm x 7.5 cm x length of the lower side of the board, one at each side and one on the centre of the board. Two loading points were used for each board according to the ASTM D2394-17 standard (ASTM 2017). The residual deformation and the maximum deformation in a load vs. deformation plot for each species were determined. The surface indentation test was performed using a sample consisting of two densified pieces; one beside the other placed on structural plywood 19 mm thick, making 6 samples in total per species. For each test the percentage of samples completely, severe, or moderately damaged was determined, according to the ASTM D2394-17 standard.

2.5. 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 wood species on parameters of the densification process (wood color before and after heating, heating ΔE*, densification ΔE*, weight loss after heating, final density, percentage of densification, degree of compression and spring-back) and flooring parameters (shear strength of the glue line and residual deformation for concentrated loading test). The analysis of variance and Tukey tests were performed with the SAS software (SAS Institute Inc., Cary, NC). The category variables measured in flooring (wear index and surface indentation) were not applied any statistical test.

3. Results

3.1. Densification process

In this study, high temperatures (180 °C for *A. acuminata* and *V. guatemalensis* and 160 °C for *V. ferruginea*) produced changes in the color parameters during densification (Table 2). After stage 1 (heating) brightness (L*) and yellowness (b*) decreased in the three species and redness (a*) decreased in *A. acuminata* and *V. ferruginea* wood (Table 2). After densification, at the end of stage 3, the parameter brightness (L*) decreased in the three species, while yellowness (b*) and redness (a*) increased in the three species (Table 2). Heating ΔE* (color change after heating and before compression) was greater than densification ΔE* (color change after stage 3) for the three species (Table 2). *A. acuminata* and *V. ferruginea* woods presented heating ΔE* statistically higher compared to *V. guatemalensis* wood (Table 2), in contrast with densification ΔE*, where *V. guatemalensis* wood showed the statistically higher value (Table 2).

Weight loss after stage 1 (heating) was statistically higher in *V. ferruginea* and *V. guatemalensis* woods compared to *A. acuminata* wood (Table 2). With regard to the final density, no differences were observed among the woods of the three species (Table 2). The percentage of densification in *A. acuminata* and *V. guatemalensis* woods was statistically higher than in *V. ferruginea* wood (Table 2), while the degree of compression was statistically higher for *V. guatemalensis* wood (Table 2). Regarding wood spring-back after densification, no statistical differences among species were observed (Table 2).

Table 2. Characteristics of the wood of the three species after the densification process.

Species		*Alnus acuminata*	*Vochysia ferruginea*	*Vochysia guatemalensis*
Wood color parameter before heating	L*	67.66^{C} (2.47)	73.63^{A} (4.22)	70.82^{B} (2.48)
	a*	10.73^{A} (1.37)	8.78^{C} (1.90)	9.93^{B} (1.55)
	b*	22.38^{A} (2.40)	16.96^{C} (1.56)	21.40^{B} (1.51)
Wood color parameter after heating	L*	60.93^{B} (2.49)	65.06^{A} (4.63)	64.45^{A} (3.27)
	a*	6.49^{B} (0.75)	6.13^{B} (1.44)	10.03^{A} (1.52)

	b*	15.95^{B} (1.84)	16.03^{B} (1.18)	21.24^{A} (0.85)
Wood color parameter after densification	L*	60.01^{C} (2.08)	64.89^{A} (4.34)	62.14^{B} (3.38)
	a*	6.72^{A} (0.52)	6.84^{A} (1.66)	11.39^{B} (1.61)
	b*	16.00^{C} (1.52)	16.93^{B} (1.22)	21.68^{A} (0.71)
Heating ΔE*		10.36^{A} (3.01)	9.27^{A} (3.21)	6.33^{B} (2.18)
Densification ΔE*		1.69^{B} (1.45)	1.87^{B} (0.82)	2.99^{A} (1.63)
Weight loss after heating (%)		11.80^{B} (0.37)	12.16^{A} (0.43)	12.03^{A} (0.54)
Final density (g/cm^3)		0.71^{A} (0.06)	0.72^{A} (0.08)	0.69^{A} (0.08)
Percentage of densification (%)		41.61^{A} (2.57)	37.71^{B} (3.96)	40.10^{A} (3.33)
Degree of compression (%)		53.04^{B} (1.40)	52.33^{B} (1.68)	53.86^{A} (1.90)
Spring-back (%)		5.61^{A} (4.24)	3.62^{A} (4.06)	3.76^{A} (4.33)

Legend: the values in parentheses mean standard deviation. Different letters for each parameter represent statistical differences between different treatments (significance at 95%).

3.2. Flooring evaluation

In the flooring evaluation of the densified wood of the three species, the shear stress by compression loading was statistically higher in *A. acuminata* wood relative to *V. ferruginea* and *V. guatemalensis* woods (Table 3). In relation to resistance to abrasion, although no statistical analysis was performed, the wear index of densified wood of *V. ferruginea* presented the highest value, while *A. acuminata* and *V. guatemalensis* densified wood showed the lowest values, between 0.04 and 0.05 (Table 3).

For the indentation tests, in the concentrated loading test no differences were observed between densified wood of the three species in the residual deformation (Table 3). Figures 1a-1c present the load vs. deformation curves obtained for the wood of the three species in the test concentrated loading. In the case of *A. acuminata* densified wood the deformation observed when applying a load of 500 kg was of 8.34 mm on average (Figure 1a). For *V. ferruginea* the deformation was of 8.50 mm on average (Figure 1b), while for *V. guatemalensis* deformation of the densified wood was of 7.54 mm on average (Figure 1c).

As for the falling ball indentation test, the values of residual indentation vs. drop height for the three species were plotted in Figure 1d. The densified wood of the three species showed an increase in the residual indentation with increasing drop height. Such behaviour was more evident in densified wood of *V. guatemalensis*. As for densified wood of *A. acuminate*, with 122 cm drop height the residual indentation was 0.043 mm, while with 213 cm drop height it was of 0.077 mm. For *V. ferruginea* the residual indentation increased from 0.067 mm with 122 cm drop height to 0.085 mm with 213 cm drop height. On the other hand, in *V. guatemalensis* the residual indentation increased from 0.047 mm with 122 cm drop height to 0.089 mm with 213 cm drop height (Figure 1d). Moreover, *V. ferruginea* presented values of residual indentation higher than those of the other two species with most drop heights, except for drop heights 198 and 213 cm, where *V. guatemalensis* presented the greatest values.

In the surface indentation test, the three species presented moderate, severe and complete damages. As for *A. acuminata* and *V. guatemalensis*, 50% of the densified wood samples sowed moderate damage (Figures 2a and 2g), while 33.33% presented severe damage (Figure 2b and 2h) and only 16.66% showed complete damage (Figure 2c and 2i) (Table 3). As for *V. ferruginea*, 50% of the samples showed moderate damage (Figure 2d); 16.66% showed severe damage (Figure 2e) and 33.33% complete damage (Figure 2f) (Table 3).

Table 3. Values obtained in the flooring tests for the densified wood of the three species.

Parameter		*A. acuminata*	*V. ferruginea*	*V. guatemalensis*
Glue line shear strength (MPa)		6.20^{A} (1.98)	3.61^{B} (0.80)	2.93^{B} (0.83)
Wear index (g)		0.04	0.12	0.05
Residual deformation for concentrated loading test (mm)		1.14^{A} (0.39)	1.18^{A} (0.36)	1.26^{A} (0.70)
Surface indentation (% of samples)	Moderate	50.00	50.00	50.00
Surface indentation (% of samples)	Severe	33.33	16.66	33.33
Surface indentation (% of samples)	Complete	16.66	33.33	16.66

Legend: the values in parentheses mean standard deviation. Different letters for each parameter represent statistical differences between different treatments (significance at 95%)

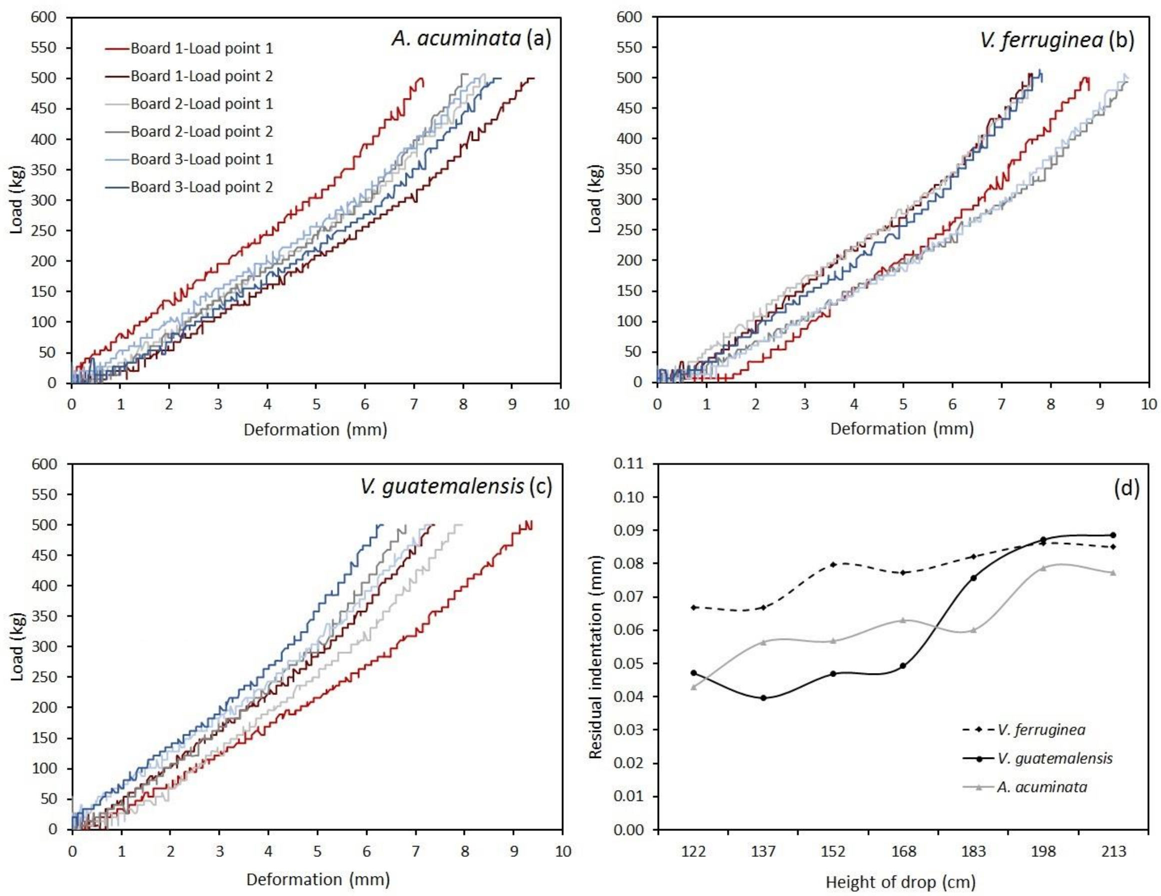

Figure 1. Load vs. deformation plotting for the concentrated loading test for *A. acuminata* (a), *V. ferruginea* (b) and *V. guatemalensis* (c) and residual indentation vs. drop height plotting for falling ball indentation test for the densified wood of the three species (d).

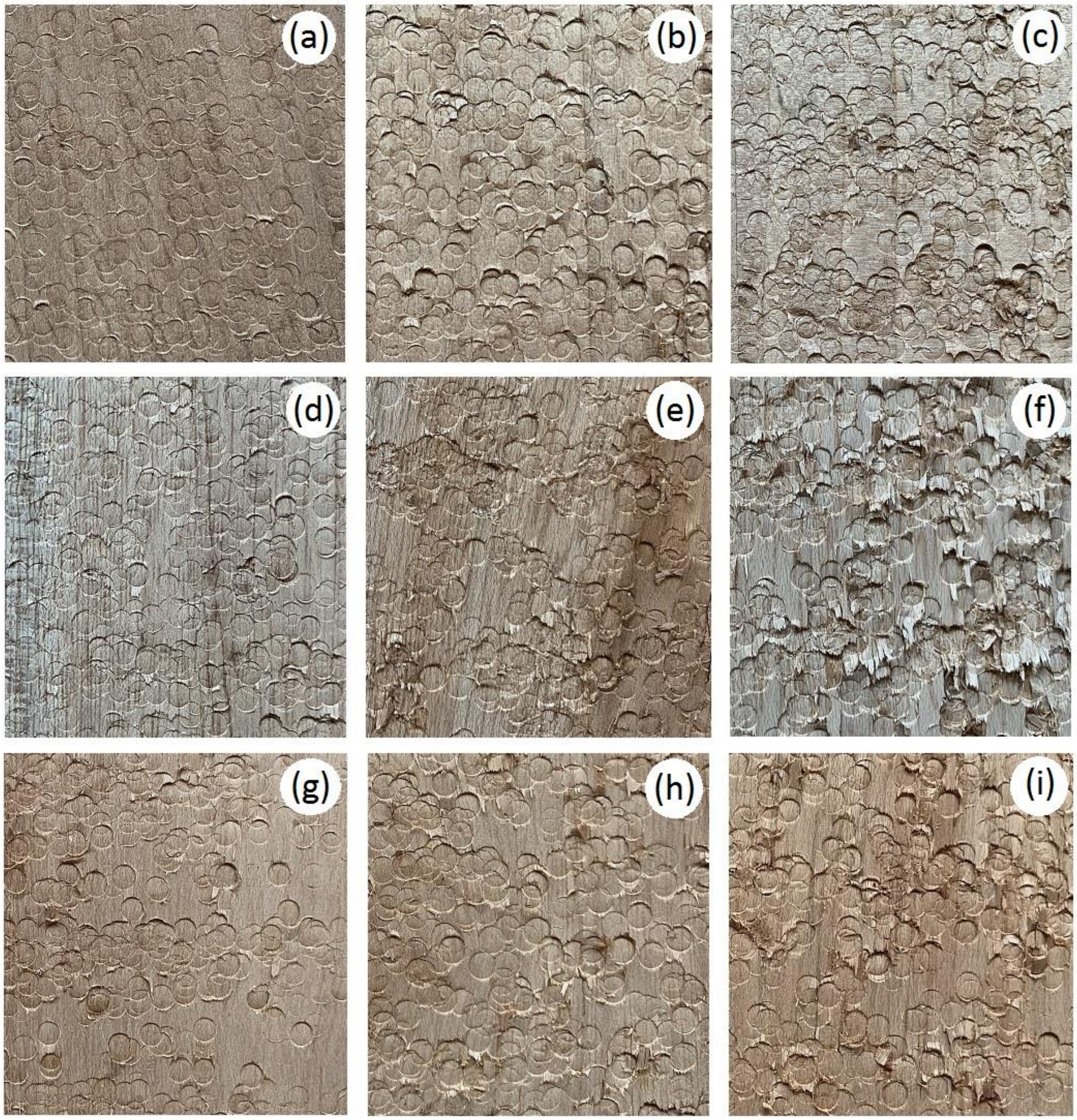

Figure 2. Moderate, severe and complete surface indentation in the densified wood of *A. acuminata* (a, b and c, respectively), *V. ferruginea* (d, e and f, respectively) and *V. guatemalensis* (g, h and i, respectively).

4. Discussion

4.1. Densification process

Color is an important aesthetic feature in wood derived products (Jalilzadehazhari and Johansson 2019), particularly if intended for flooring (Nordvik et al. 2009). Here, because high temperatures were applied (180 °C for *A. acuminata* and *V. guatemalensis* wood and 160 °C for *V. ferruginea* wood) during the densification process, changes were observed in the color parameters of the wood of the three species

(Table 2). Visually, these changes were perceived in the form of wood darkening caused by the decrease in the parameter L*. Darkening of the densified wood is attributed to the temperature (Salca et al. 2016), since it affects the woods' chemical composition (Pohleven et al. 2019). Color changes in the densified wood are caused by hydrolysis of the hemicelluloses (Candelier et al. 2016), and reduction of lignin, which is associated to production of chromophore groups (Salca et al. 2016), and change to darker color at high temperatures (Table 2). Hydrolysis of the hemicelluloses produces changes in parameter L*, while reduction of lignin produces changes mainly in parameter a* (Moya et al. 2012), which was reflected in parameters L* and a*, respectively, in the present study (Table 2). However, Matsuo et al. (2010) indicate that wood darkening resulting from a thermal treatment is due to reduction of the parameter luminosity L* instead of the change in parameter a*. Therefore, in this study the behaviour of parameter a* disagrees with what Matsuo et al. (2010) point out.

Regarding color change (ΔE*), it is worth noting that the ΔE* obtained after stage 1 (heating ΔE*) of the process were approximately 3 times greater than color changes after finalizing the densification process (densification ΔE*) for the three species (Table 2). The time of exposure to temperature in the various stages of the process explains this difference. The exposure time for heating ΔE* was 60 minutes, while for densification ΔE* it was only 25 minutes, corresponding to stages 2 and 3 of the process. According to Matsuo et al. (2010) the values of ΔE* increased logarithmically with the exposure time of the wood: the longer the exposure time, the greater the ΔE*. The same authors indicate that the changes are cumulative, which means that the total ΔE* of the wood is given by the sum of de heating ΔE* and densification ΔE*.

The thermo-mechanical densification process developed in this study rendered similar values among the three species in relation to the final density of the densified wood. Nevertheless, as expected, differences were observed regarding the percentage of densification. *V. ferruginea* wood presented the highest initial density (0.45 g/cm^3) but the lowest percentage of densification (Table 2) compared to *A. acuminata* and *V. guatemalensis* wood, which had lower initial density (Table 1) and higher percentages of densification (Table 2). To this respect, Tenorio and Moya (2020) point out that there is a high negative correlation between the initial density of the wood and the percentage of densification obtained, as confirmed in the present study´s analysis of the three species.

As for the spring-back values obtained in this study, from 3.62 to 5.61% (Table 2), they are much lower than those obtained for the same species using a thermo-hydro-mechanical densification process, where the spring-back obtained was greater than 10% for all three species (Tenorio and Moya 2020). The reduction in spring-back in this study is attributed to the heating time (stage 1) of 60 minutes, while in the previous study (Tenorio and Moya 2020) the heating time was 10 minutes. Spring-back reduction in this process results in greater stability of the densified wood (Inoue et al. 2008), which is ideal for flooring applications.

According to Sadatnezhad et al. (2017), time and temperature influence greatly wood fixation and can reduce spring-back. An increase in time during the heating stage, as occurred in the present study, increases the compressivity of wood and reduces the level of stress stored in the wood during densification. In addition, an increase in heating time affects the chemical composition of the wood (Inoue et al. 2008), because heating the wood to temperatures above 180 °C leads to degradation of components of hemicellulose and as a result, the stresses stored in the micro fibrils would be released (Norimoto et al. 1993; Dwianto et al. 1996) and the lignin will soften and flow to fill the space matrix that is in the wood fixing the wood dimensional stability (Darwis et al. 2017).

Inoue et al. (2008) also point out the importance of the adequate use of time and temperature as treatment to increase the stability of densified wood. These authors indicate that there is high correlation between the percentage of weight loss during the heating stage (stage 1 of the densification process) and wood spring-back, which is caused by loss of water and degradation of hemicelluloses during this stage. This behaviour was observed in the three species in this study. The determination of the weight loss of the samples (Table 2) demonstrated that the samples that presented more weight loss (*V. ferruginea* and *V. guatemalensis*) also showed less spring-back (Table 2). Hence, it can be affirmed that 60 minutes in stage 1 of the densification process increase the stability of densified wood of the three tropical species used by reducing wood spring-back.

4.2. Flooring evaluation

The values of shear strength in the glue line obtained for densified wood in the flooring evaluation, from 2.93 to 6.20 MPa (Table 3), are comparable to those obtained for solid wood glued with polyvinyl acetate (PVAc) wood adhesives, but inferior to values obtained with urea formaldehyde (UF) wood adhesives. For example, Moya et al. (2015b) in nine tropical woods, reported values in the range of 2.00-4.00 MPa for PVAc-adhesive, which contains the values obtained for densified wood (Table 3); while for UF-adhesive the range of values varied from 8.00 to 9.00 MPa, above the values found for densified wood (Table 3). Furthermore, another study conducted by Moya et al. (2015), confirms the behaviour described, with similar glue line shear strength when using solid wood with PVAc-wood adhesive and a lower value of glue line shear strength when using UF-adhesive. According to Fang et al. (2012) the densification treatment changes the wood's structure due to the temperature and pressure, which both reduce the interlocking surface or mechanical adhesion. Therefore, a reduction in the glue line shear strength in the densified wood should be expected. Additionally, changes in the properties of the wood, such as diffusion, absorption and in the wood's chemical structure can reduce the glue line shear strength.

A. acuminata densified wood obtained the highest average in shear strength (Table 3) of the three species. This is attributed to the fact that the densified wood of this species presented more compatibility with the adhesive utilized, resulting in reduced degradation of the structure of the wood during the process. *A. acuminata* densified wood presented less weight loss (11.80 %) during stage 1, with a glue line shear strength of 6.20 Mpa (Table 3), compared to the other two species, which showed greater weight loss, approximately 12.0 %, and less glue line shear strength between 2.93 and 3.61 Mpa (Table 3).

The wear index value obtained for the densified wood of *A. acuminata* and *V. guatemalensis* was lower in relation to that of *V. ferruginea* wood (Table 3), because the densified wood of *V. ferruginea* is more susceptible to surface wear (ASTM 2014). This wear index behavior can be explained based on the values of final density and percentage of wood densification. *V. ferruginea* presented lower percentage of densification, but final density equal to that of the other two species (Table 2). The wear index of *V. ferruginea* was higher than that of *A. acuminata* and *V. guatemalensis* (Table 3), suggesting that a low percentage of densification may be associated with greater wood susceptibility to wear. The cause of the decrease in flooring quality may be less formation of covalent and hydrogen bonds and of new cross-linkages and hydrogen bonds between the cellulose and hemicellulose during densification (Dwianto et al. 1999; Navi and Heger 2004) in wood with low densification percentages, as is the case with *V. ferruginea* in this investigation, because the fibers from one group detach more easily from the other group of fibers.

Regarding the values of residual deformation for the concentrated loading in the indentation test, although no differences were observed in the densified wood of the three species (Table 2), the average deformation obtained in the curves load vs. deformation was greater in *V. ferruginea* densified wood (8.50 mm) relative to the other two species (*A. acuminata* with 8.34 mm and *V. guatemalensis* with 7.54 mm) (Figures 1a, b and c). The result of this test indicates that *V. ferruginea* densified wood presents greater deformations for the same load than the other two species (less toughness), confirming the previous affirmation that the densified wood of *V. ferruginea* presents less quality than the other two species due to, as stated above, less formation of covalent and hydrogen bonds and of new cross-linkages of the wood components (Dwianto et al. 1999; Navi and Heger 2004).

Similarly, high values obtained for the residual indentation in the falling ball indentation test and the increased number of samples with moderate and complete damage in the surface indentation test in the densified wood of *V. ferruginea* (Table 3, Figure 2), demonstrate the lower quality of flooring made with this species with respect wood flooring from *A. acuminata* and *V. guatemalensis*.

As mentioned, higher wear index, increased residual deformation, high values of residual indentation for the falling ball indentation test and greater number of samples with moderate and complete damage for the surface indentation test in densified wood from *V. ferruginea* are associated to lower percentage of densification and high weight loss during stage 1 of the densification process. Thus, since the densified woods of *A. acuminata* and *V. guatemalensis* were better evaluated regarding parameters of quality of flooring and greater percentage of densification, they could present better performance in flooring applications.

5. Conclusions

The thermomechanical densification process, with a 60-minute pre-heating stage 1 and constant temperature during the entire densification process, reduces wood spring-back by 5%, but also tends to cause greater weight loss due to degradation of some of the wood's components, with consequent effects on its properties in the extent that the weight loss caused by the degradation of hemicelluloses during this stage increases.

The percentage of densification has a clear effect on the behaviour of the densified wood evaluated as flooring, even if the three species presented statistically similar values of final density. Low densification percentage and high weight loss of the wood due to temperature and time of exposure in stage 1, cause *V. ferruginea* wood to present greater wear index, higher residual deformation, high values of residual indentation for the falling ball indentation test and greater number of samples with moderate and complete damage in the surface indentation test than densified wood of *A. acuminata* and *V. guatemalensis*.

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