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Establecimiento de las condiciones de termotratamiento de madera de melina (*Gmelina arborea*) y teca (*Tectona grandis*) para mejorar sus propiedades

(DOCUMENTO I)

<u>INVESTIGADORES</u>:

Roger Moya R, Ph.D.
Carolina Tenorio M, M. Eng.
Luis Diego Mendez Alvarez, MSc.

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ÍNDICE GENERAL

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1. Autores:

1.1. Ing. Roger Moya R, Ph.D. (Coordinador).
1.2. Ing. Carolina Tenorio M, M. Eng.

2. RESUMEN

Este estudió evaluó el efecto del termotratamiento sobre las propiedades físicas (densidad, contracción, pérdida de masa, absorción de humedad), color medido por el sistema CieLab, durabilidad determinada por ensayos acelerados de resistencia a la pudrición, propiedades mecánicas como MOR y MOE en flexión, adhesión por tensión y propiedades químicas por FTIR en las maderas de *Tectona grandis* y *Gmelina arborea*. Las muestras de radial y tangencial de *G. arborea* fueron expuestas a cuatro niveles de temperatura (205, 210, 215 y 220º C) y las de *Tectona grandis* a 4 temperaturas también (185, 190, 195 y 200 ºC), además de muestras control (sin termotratamiento) para cada especie.

Los resultados por objetivos son mostrados a continuación:

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<tr><td>2. Determinar las propiedades sobre las propiedades mecánicas, pruebas de durabilidad en condiciones de laboratorio (acelerado), durabilidad en condiciones naturales por cementerio de estacas, cambio de color por intemperismo acelerado y natural, características químicas y características térmicas.</td><td>
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<tr><th></th><th><i>T. grandis</i></th><th><i>G. arborea</i></th></tr>
<tr><td>Densidad de madera (g cm^{-3})</td><td>0,61 a 0,49</td><td>0,43 a 0,47</td></tr>
<tr><td>porcentaje de contracción (%)</td><td>2,40 a 1,62</td><td>2,61 a 1,20</td></tr>
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</table>
</td></tr>
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Como conclusión general se puede afirmar que el tratamiento térmico influye en las propiedades físicas, mecánicas y químicas de la madera mejorando en algunos casos la calidad de las mismas y en otras afectándolas. Sin embargo, se deben tomar en cuenta las condiciones del proceso, así como las temperaturas y tiempo de exposición de la madera de acuerdo a la especie y el objetivo final que pueda tener la madera paa así poder obtener los mejores resultados.

Establishment of heat-treatment conditions of melina (*Gmelina arborea*) and teak (*Tectona grandis*) wood to improve its properties

Abstract

In recent years, new techniques and processes have emerged, such as heat treatment to modify and improve the properties of wood. This study evaluated the effect of thermo-treatment on physical properties (density, shrinking, mass loss, moisture absorption) color measured by the CieLab system, durability by accelerated testing of natural rot resistance, mechanics such as MOR and MOE in flexion, stress adhesion and chemicals by FTIR in wood of *Tectona grandis* and *Gmelina arborea*. Sapwood and heartwood samples, sawn under radial and tangential cutting patterns for *T. grandis* species and wood samples sawn under the radial and tangential cutting patterns for *G. arborea* were exposed to four temperature levels (205, 210, 215 and 220 ° C) and (185, 190, 195 and 200 ° C) respectively, plus control samples (without thermo-treatment) for each species. Results showed that density varied from 0.61 g cm-3 to 049 g cm-3 and from 0.43 g cm-3 to 0.47 g cm-3 fot *T. grandis* and *G. arborea* respectively, with a declining trend as temperature of the thermo-treatment increases in both species. Shrinking percentage fluctuated between 2.61 to 1.20% and from 2.40 to 1.62% for *T. grandis* and *G. arborea* respectively, where heartwood timber with tangential cutting pattern thermo-treated at 220°C for *T. grandis* species and *G. arborea* timber with tangential cutting pattern thermo-treated at 195 ºC showed the highest shrinking percentages in each species. Mass loss percentage ranged from 11.55 to 14.32 and from 7.75 to 14.09 for *T. grandis* and *G. arborea* respectively, reflecting an increase in the mass loss percentage with the increasing of the thermo-treatment temperature. Likewise, the absorption percentage varied from 1.05 to 2.66 % for *T. grandis* and from 1.39 to 2.37 % for *G. arborea* showing a decrease for *T. grandis* and an irregular behavior in *G. arborea*, when raising the heat treatment temperature Total color change (ΔE^*) of wood varied from 21.91 to 40.92 and from 24.95 to 30.24 % for *T. grandis* and *G. arborea* respectively, reporting an increase in total color change as the heat treatment temperature increases. The change in color showed the largest changes in the brightness parameter (ΔL^*), while redness (Δa^*) and yellowing (Δb^*) showed minor color changes. MOR varied from 44.18 to 76.63 MPa and from 49.70 to 60.68 Mpa for *T. grandis* and *G. arborea* respectively, affected mostly in heartwood timber with radial cut of *T. grandis* and *G. arborea* timber with tangential cut thermo-treated at temperatures of 220 and 200ºC respectively. Meanwhile, MOE values ranged from 3.07 to 3.78 GPa and from 6.94 to 8.52 GPa for *T. grandis* and *G. arborea*, respectively, reflecting the lowest values in sapwood timber with tangential cut at 205 ºC for *T. grandis* and in timber without heat treatment with tangential cut for *G. arborea*. Tension resistance showed a variation between 2.46 and 11.52 % and 4.13 to 8.69 % for *T. grandis* and *G. arborea* respectively, with a declining trend as the thermo-treatment temperature increases for both species. Moreover, it was found that failure percentage occurs mostly in wood with values higher than 26%. Durability of wood, showed that *L. acuta* degraded to a greater degree the wood in relation to *T. versicolor*. Likewise, it was obtained that *G. arborea* species is more susceptible to degradation compared to *T. grandis*. The bands of 1053 and 1108 cm-1 in thermo-treated wood had the highest intensity values regardless of the species, type of wood or cutting type, in relation to the different thermo-treatment temperatures, indicating chemical modification with the heat treatment. Finally, it was found that the heat treatment influences the physical, mechanical and chemical properties of the wood improving in some cases the quality of the same and in others affecting them, however, we must consider the conditions of the process, as well as the temperatures and time of exposure of wood per species and the objective of the same to obtain the best results.

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3. PALABRAS CLAVE

Madera, tratamiento térmico, estabilidad dimensional, durabilidad, contracción, color, densidad.

Key words: wood, heat treatment, dimensional stability, durability, shrinking, color, density.

4. MARCO TEÓRICO

La madera ha sido un material utilizado por la humanidad desde siglos pasados en diversas funciones como construcción, mueblería, energía, entre otros, y considerado aún como uno de los materiales más importantes como materia prima (Kránitz et al., 2015). Además, presenta excelentes propiedades mecánicas que permiten su uso en aplicaciones donde se requiere soporte de alta carga, o bien usos no estructurales como aislamiento termo-acústico, diseño, usos decorativos entre otros (Uribe y Ayala, 2015). No obstante, presenta como principal desventaja la absorción y desorción de agua lo que afecta directamente los cambios dimensionales que a su vez pueden provocar problemas como agrietamiento, alabeos entre otros que reducen la calidad y propiedades de la misma, lo que limita considerablemente su uso en algunas aplicaciones (Uribe y Ayala, 2015; Kocaefe et al., 2015), además de su susceptibilidad al daño por agentes bióticos y abióticos (Ortiz et al., 2014).

Debido a estos problemas, surgen nuevas técnicas y procesos para modificar y mejorar las propiedades de la madera e incentivar el uso de este material con la mejor calidad posible (Kocaefe et al., 2015). Uno de ellos es el tratamiento térmico utilizado como método de modificación de la madera que permite mejorar algunas características y propiedades como la estabilidad dimensional, permeabilidad, durabilidad natural entre otros (Korkut et al., 2008), obteniendo una mejor calidad natural de la madera (Noh et al., 2016), lo que permite su uso en exteriores debido a la reducción de su higroscopicidad (Olarescu et al., 2014). El tratamiento térmico, fue implementado en Europa a principios de 1990 y ha venido despertando un gran interés al permitir modificar la madera con un bajo impacto ambiental y de una manera financieramente viable (Kesik et al., 2014).

La modificación térmica o termotratamiento de la madera es un proceso mediante el cual la madera es tratada en una atmósfera de gas inerte de nitrógeno generalmente, a temperaturas de hasta 260 °C y tiempos de exposición variables (Ansell, 2012). Los parámetros utilizados en este proceso pueden diferir, por ejemplo, Esteves et al. (2014) y Kesik et al. (2014), indican temperaturas de termotratamiento entre un rango de 160-260 ºC, durante tiempos de exposición de entre 2 y 6 horas en un ambiente con baja presencia de oxígeno. Por otro lado, Priadi y Hiziroglu (2013) reporta temperaturas de termotratamiento que varían entre 130 a 200 °C, con un tiempo de exposición de entre 2 y 8 horas. Debido a estos factores, Kesik et al. (2014), menciona que la temperatura y los tiempos de exposición del material durante el proceso de termotratamiento difiere según la especie y las condiciones en que se ejecute el proceso, lo que permite ajustar el proceso en relación a las dimensiones de la madera, contenido de humedad y su posible uso.

Este proceso de modificación térmica de la madera es de gran importancia actualmente, sobre todo en algunos países nórdicos, debido a su bajo costo, en relación a tratamientos de impregnación química, que utilizan cantidades significativas de sustancias obteniendo como resultado un producto final más caro (Esteves et al., 2014). También, porque trae consigo una serie de mejoras en las propiedades de la madera como disminución de la contracción, mejor estabilidad dimensional y minimización de ataques por organismos fúngicos (Ansell, 2012 y Bakar et al., 2013). Estudios llevados a cabo por Esteves et al. (2014) en la especie *Pinus pinaster* confirman la mejora de las propiedades al termotratar la madera, en estos se reportó una mayor estabilidad dimensional y una disminución en la contracción volumétrica, la hinchazón y la densidad cuando la madera esta termotratada. Por su parte (Noh y Ahmad, 2016), también indican diminuciones de la densidad al termotratar madera de *Irvingia spp*. y *Dryobalanops spp.*

Sin embargo, uno de los inconvenientes que se le señala a la madera termotratada es la pérdida de resistencia mecánica. Kesik et al. (2014) termotratando madera de cuatro diferentes especies, a temperaturas de 130 ºC a 160 ºC por tiempos de 3 y 7 horas, encontró cambios en propiedades mecánicas como la dureza, con reducciones de entre 15 y 26%. Del mismo modo, Bekhta y Niemz (2003), en su investigación reportan disminuciones en resistencia a la flexión de 50% aproximadamente y de entre 4 y 7% en módulo de elasticidad para madera de *Picea abies* al termotratar a 200 ºC respecto a madera termotratada a100 ºC. Por su parte, Gunduz et al. (2009), indicó diminuciones en resistencia de flexión y compresión con valores de 7,42% y 7,55% respectivamente para madera de *Pyrus elaeagnifolia Pall*. Así mismo, Hidayat et al. (2016), obtuvo disminuciones significativas en valores de módulo de ruptura y módulo de elasticidad al termotratar madera de *Cylicodiscus gabunencis* a una temperatura de 180 ºC en tiempos de exposición de 3 y 4 horas.

Si bien es cierto, el termotratamiento provoca la disminución de algunas propiedades mecánicas de la madera limitando su uso, por lo que se debe tomar en cuenta la aplicación que va a tener el material (Baradit et al., 2013). Sin embargo, el termotratamiento produce un aumento en el valor estético y durabilidad natural de la madera (Olarescu et al., 2014), debido al cambio de color y cambios producidos en su estructura, ocasionados principalmente a la degradación de hemicelulosas (Salca et al., 2016) lo que mejora su apariencia y permite implementar su uso en diversas obras de ingeniería, construcción, arquitectura, entre otras.

Actualmente, este tipo de métodos no son utilizados en todos los países, a pesar de su excelente alternativa para mejorar las propiedades y características de la madera. Por ejemplo, en países pequeños como Costa Rica donde se cuenta con programas de reforestación que utilizan una gran variedad de especies para la producción de madera (Moya, 2004), esto podría convertirse en una alternativa para aumentar la competitividad de este tipo de madera en el mercado Costarricense.

Por otra parte, especies maderables como *Tectona grandis* y *Gmelina arborea*, que han adquirido importancia comercial en diversos proyectos de reforestación en Costa Rica (Serrano y Moya, 2013), debido a su rápido crecimiento, su capacidad de adaptación a las zonas abandonadas (Moya y Tenorio, 2013). Así mismo dichas maderas han tenido una buena aceptación en el mercado, por lo que las convierte en especies propicias para evaluar la posibilidad de aplicar un termotratamiento para aumentar aún más sus propiedades (Serrano y Moya, 2013).

Ante tales circunstancias, esta investigación tuvo como objetivo evaluar el efecto de cuatro temperaturas de termotratamiento para la madera *T. grandis* y *G. arborea* proveniente de árboles de plantaciones comerciales. Las propiedades de la madera en las que se evaluará el efecto del termotratamiento son las propiedades físicas, mecánicas y químicas mediante normas estandarizadas ASTM y técnicas espectrométricas para el caso de las propiedades químicas. Esta evaluación del termotratamiento pretende dar criterio técnico sobre un producto amigable con el ambiente, con mejores propiedades y características que permitan potencializar su uso y competir en el mercado nacional.

5. CONCLUSIONES

- El termotratamiento de la madera produce una tendencia a disminuir la densidad con el aumento de la temperatura del termotratamiento en ambas especies. No obstante, el porcentaje de contracción presentó un comportamiento irregular en relación a la temperatura. La madera de duramen con patrón de corte tangencial termotratada a 220°C en el *T. grandis* y la madera de *G. arborea* con patrón de corte tangencial termotratada a 195 ºC mostraron los mayores porcentajes de contracción. El porcentaje de pérdida de masa aumentó con el aumento de la temperatura de termotratamiento en ambas especies, mientras que el porcentaje de absorción disminuyo para *T. grandis* y la madera termotratada de *G. arborea* presentó un comportamiento irregular de la absorción con el aumento de la temperatura del termotratamiento.

- El cambio de color total (ΔE*) de la madera termotratada aumentó con el aumento la temperatura de termotratamiento. El cambio de color mostró los mayores cambios en el parámetro de luminosidad (ΔL*), en tanto el enrojecimiento (Δa*) y la amarillez (Δb*) mostraron menores cambios de coloración, por lo tanto, las tonalidades más oscuras se obtuvieron al termotratar madera de *T. grandis* y *G. arborea* a temperaturas de 220 y 200 ºC respectivamente. Los valores mayores de ΔE* se obtuvieron en madera de albura para *T. grandis* y *G. arborea* al ser maderas de tonalidades más claras, presentando un cambio de color más notorio en comparación a madera de duramen, no obstante, no se obtuvieron diferencias significativas entre algunas de las temperaturas de termotratamiento.

- El MOR fue afectado mayormente en madera de duramen con corte radial de *T. grandis* y madera de *G. arborea* con corte tangencial termotratada a temperaturas de 220 y 200ºC respectivamente. En tanto los valores más bajos de MOE se obtuvieron en madera de albura con corte tangencial termotratada a 205 ºC para *T. grandis* y en la madera sin termotratar con corte tangencial para *G. arborea*.

- La resistencia a la tensión por línea de cola presentó una tendencia a disminuir con el aumento de la temperatura del termotratamiento en ambas especies. En tanto se encontró que el porcentaje de falla ocurrió mayormente en madera con valores superiores a 26%.

- La durabilidad de la madera, medido por el ensayo acelerado de hongos, se encontró que *L. acuta* degrado en mayor grado la madera en relación a *T. versicolor*. No obstante, aunque presentó un comportamiento irregular en relación a las temperaturas de termotratamiento en ambas especies, se logró apreciar una disminución de pérdida de masa por ataque en las temperaturas de termotratamiento más altas. Del mismo modo se obtuvo que la especie *G. arborea* es más sus susceptible a la degradación en comparación con *T. grandis*.

- La relación de intensidad en las bandas a 1652 y 1733 cm^{-1} asociadas a la afectación de hemicelulosas que fue el polímero estructural relacionado en mayor medida a la afectación de las propiedades evaluadas, mostraron variación en las diferentes temperaturas de termotratamiento independientemente de la especie, tipo de madera o tipo de corte, en relación a las diferentes temperaturas de termotratamiento, indicando modificación química con el termotratamiento.

- El termotratar madera de las especies *G. arborea* y *T. grandis* a temperaturas de 200 y 220 ºC permite obtener las mejores condiciones para potencializar su uso, ya que mejora aspectos como apariencia, durabilidad, estabilidad dimensional, así como un material más ligero.

6. RECOMENDACIONES

- De acuerdo a los resultados obtenidos en este estudio, se logró determinar que el tratamiento térmico tanto en *T. grandis* como en *G. arborea* influye sobre las propiedades de la madera favoreciendo unas y afectando otras, por lo que para lograr las mejores características del material se debe hacer un balance entre lo deseado y lo óptimo. No obstante, lo antes mencionado depende del objetivo por el cual se está termotratando la madera ya que, por ejemplo, si requiere madera con un color más relevante y una mayor durabilidad para usos donde se requiere poco esfuerzo se recomienda utilizar altas temperaturas de termotratamiento. Por el contrario, si se requiere material con mejores propiedades mecánicas se recomienda utilizar temperaturas de termotratamiento más bajas, tomando en cuenta que esto a su vez me va a producir menores cambios de color, menor durabilidad, entre otros aspectos.

7. Artículos científicos:

Objetivo específico 1: Determinar las condiciones de calentamiento, temperatura y tiempo de termotratamiento para *T. grandis* y *G. arborea*

Objetivo específico 1: Determinar las propiedades sobre las propiedades mecánicas, pruebas de durabilidad en condiciones de laboratorio (acelerado), durabilidad en condiciones naturales por cementerio de estacas, cambio de color por intemperismo acelerado y natural, características químicas y características térmicas.

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Effect of Thermo-Treatment on the Physical and Mechanical, Color, Fungal Durability of Wood of *Tectona grandis* and *Gmelina arborea* from Forest Plantations

Luis Diego MÉNDEZ-MEJÍAS [1] *, Róger MOYA [2]

[1] *Maestría en Ciencias Forestales, Instituto Tecnológico de Costa Rica, P.O. Box: 159-7050 Cartago-Costa Rica*
[2] *Escuela de Ingeniería Forestal, Instituto Tecnológico de Costa Rica, P.O. Box: 159-7050 Cartago-Costa Rica*

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This study evaluated the effect of thermo-treatment (THT) at 4 temperatures on the density, shrinking, mass loss, moisture absorption, color, durability in terms of resistance to decay, flexural strength, tensile adhesion of glue line and the infrared spectrum of the wood of *Tectona grandis* and *Gmelina arborea*. Sapwood, heartwood and radial and tangential grain patterns were studied. The results showed that the THT temperature decreases the density, the percentage of moisture absorption, the modulus of elasticity and modulus of rupture in the flexure test and the tensile adhesion of glue line. The percentage of shrinking and durability presented irregular behavior relative to the THT temperature. The percentage of mass loss increased with increasing THT temperature in both species. The total color change (ΔE^*) of thermo-treated wood (THTwood) also increased with increasing THT temperature. Sapwood of *T. grandis* and *G. arborea*, having clearer shades, showed a more noticeable color change compared to hardwood; however, no significant differences were obtained between some of the THT temperatures.

Keywords: wood, thermo-treatment, dimensional stability, durability, shrinking, color, density.

1. INTRODUCTION *

Wood is a widely used material [1]; however, it presents some inconveniences, such as dimensional changes [1] and susceptibility to damage caused by biotic and abiotic agents [2]. Due to these problems, new techniques and processes have been developed to modify and improve the properties of the wood [3]. Thermal treatment is one of those methods, used to enhance some characteristics and properties of the wood, such as dimensional stability, permeability and natural durability, among others [4].

The thermal modification or thermo-treatment (THT) is a process in which the wood is treated in an inert gas atmosphere at temperatures generally as high as 260 °C and varying times of exposure [5]. The parameters used in this process differ according to the species and conditions in which the process is executed, in order to adjust to wood dimensions, moisture content (MC) and possible use of the wood [6, 7]. The cost of thermo-treated wood (THTwood) is lower than the cost of preservation treatments using chemical impregnation [6].

However, one of the inconveniences of THTwood is the loss of mechanical resistance. Kesik et al. [7] found changes in the mechanical properties of THTwood from four different species, i.e., reductions from 15 to 26 % in hardness. Similarly, Bekhta and Niemz [8] in their research report reductions in flexural strength of approximately 50 % and between 4 to 7 % in the modulus of elasticity (MOE) for wood of *Picea abies*. Meanwhile, Gunduz et al. [9] indicated diminutions in flexural strength and compression of 7.42 % and 7.55 % respectively for wood of *Pyrus elaeagnifolia*. Likewise, Hidayat et al. [10] obtained significant reductions in the MOR and MOE for THTwood of *Cylicodiscus gabunencis*.

Although THT causes changes in the mechanical properties of the wood, restricting somehow the use to be given to the material, it also enhances its aesthetics and natural durability [4] as a result from color and structural changes caused by hemicellulose degradation mainly [11], which improves wood appearance and allows to implement its use in engineering, construction and architectural works, among others.

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* Corresponding author. Tel.: +506 550-2433; fax: +506 591-3315.
E-mail address: *rmoya@itcr.ac.cr* (R. Moya)

Currently, this method is not used in all countries, despite being an excellent alternative to improve the properties and characteristics of the wood. For example, in small countries such as Costa Rica, where reforestation programs using a large variety of species for timber production are implemented [12], this could become an alternative to increase the competitiveness of this type of wood in the market. This applies to reforestation species such as *Tectona grandis* and *Gmelina arborea*, which have gained commercial importance in various reforestation projects in Costa Rica [13] due to their rapid growth and their ability to adapt to abandoned areas [14]. Likewise, these woods have been well accepted in the market, which makes it worthwhile to evaluate their suitability for THT application to further enhance their properties [13].

Given this context, the objective of this research was to evaluate the effect of four THT temperatures (205, 210, 215 and 220 °C) for *T. grandis* wood and 4 other temperatures (185, 190, 195 and 200 °C) for *G. arborea* wood from trees from commercial plantations. The THT effect on the wood of *T. grandis* and *G. arborea* were evaluated on the physical properties, like density, shrinking, weight loss, moisture absorption, color (measured by the CIEL*a*b* system), durability (by accelerated tests of resistance to natural degradation); mechanical properties, such MOR and MOE in the flexion and adhesion (by tension) test. THTwood was performed using industrial equipment seeking to obtain an environmentally friendly product, with enhanced properties and characteristics as to compete in the domestic market.

2. EXPERIMENTAL DETAILS

Provenance, characteristics of the material and sampling: The *T. grandis* and *G. arborea* wood was obtained from forest plantations for wood production located in the northern zone of Costa Rica, owned by the company Ethical Forestry S.A. The wood used was obtained from trees from second thinning from plantations of 11 and 8 years, with a spacing of 3 × 3 meters. It was selected this tree age because lumber is extracted from tree with those age. The logs were sawn into boards 7.5 cm wide × 2.5 cm thick × 250 cm long and radial and tangential wood samples were obtained. The wood was classified by sapwood or heartwood boards, which were then dried following the typical drying schedule at a target MC of 12 % [15]. In *T. grandis* the THT was evaluated in sapwood and heartwood, including different grain patterns (radial and tangential patterns). However, it was not possible to obtain radial sapwood due to its thickness and log dimensions. In *G. arborea*, the thermo-treatment was studied only for radial and tangential sapwood, since it is not possible to distinguish between sapwood and hardwood. 30 boards approximately 7.5 cm wide × 2.5 cm thick × 250 cm long were prepared for each species. Different grain pattern was studied because the wood properties are different [12, 15] and they have difference performance in the THT process [6]. Finally, the boards were divided in 5 samples with 7.5 cm wide × 2.5 cm thick × 50 cm.

Thermo-treatment process: Sawn wood in dry condition, at approximately 12 % MC, was THT at four different temperature levels: 205, 210, 215 and 220 °C for *T. grandis* and 185, 190, 195 and 200 °C for *G. arborea*. The precision temperature was kept during the heat treatment of wood was ± 2. In addition, each species included untreated (without thermo-treatment) samples. The THT temperatures were selected considering previous results [16] where suitable temperature ranges every 5 °C were found. Samples were introduced and stacked in a Volutec® brand oven and each THT process was performed independently. The process consisted of several drying stages during 17 hours at 130 °C, until reaching 0 % MC. Subsequently, an increase of 130 °C was applied to the temperature defined for the type of THT with a duration of 6 hours, and was held for two hours. The conditioning step was then applied for 7 hours, in which steam, water and temperature were applied to hydrate the wood and achieve about 6 % MC. Finally, a cooling process was applied for 3 hours. The THT process was performed under oxygen free conditions in the presence of nitrogen.

Determination of wood density, shrinking, mass loss and moisture absorption: Each THTwood samples was measured its wood density (WD), shrinking percentage (SP), mass loss per THT (MLT), and moisture absorption (M_{ABS}) after the THT process. The WD was determined by the relationship between the dimension (volume) measured in samples (approx. 7.5 cm wide × 2.5 cm thick × 50 cm) and the mass of each sample after the THT process. The SP was determined by measuring the width of each of the boards before and after the THT and then the difference between the two dimensions was determined and expressed in the initial measure as a percentage. For MLT, each sample was weighed before and after THT and the difference between the two masses was determined and expressed as a percentage of the initial weight. To determine MABS, 10 THTwood samples were used for each treatment conditioned to 12 % with dimensions of 2 cm × 2 cm × 2 cm. The samples were placed in a desiccator with a saturated solution of potassium nitrate at 22 °C (21 % equilibrium moisture content). The samples were weighed weekly until they reached a constant mass. The mass before and after this period was measured and MABS was calculated with Eq. 1.

$$\text{Moisture absorption (\%)} = \left(\frac{Mass\ at\ 21\%\ \text{(g)} - \text{Initial mass}\ (g)}{Initial\ mass\ (g)}\right) * 100. \quad (1)$$

Wood color change: A HunterLab miniSkan XE Plus spectrophotometer was used for color measurement, with standardized CIE L* a* b* chromatographic system. The measuring range was 400 to 700 nm, with an aperture at the measurement point of 11 mm. The observation of the reflection included the specular component (SCI mode), at an angle of 10°, which is the normal of the surface of the specimen (D65/10); a field of view of 2º (Standard observer, CIE 1931) and a level of illumination of D65 (corresponding to Daylight at 6500 Kelvin). The CIEL*a*b* color system allows three-dimensional color measurement. Color was measured in each of the boards before and after THT and the total color change (ΔE*) was then quantified according to ASTM D 2244 [17, 18].

Modulus of elasticity, modulus of rupture, tensile adhesion and percentage of failure of the glue line in wood: MOR and MOE were determined under the ASTM D143-14 [19]. The tensile adhesion of the glue line (Ts) was performed according to the European standard UNE EN-205 [20] and the percentage of failure (Fp) using the ASTM D 5266-99 standard [21]. The adhesive used was of the type Lanco®Vynil acetate Grip Bond 4™. The tests were carried out in a universal JBA test machine, model 855 and in all cases 30 specimens were used for each species according to each treatment.

Accelerated test of resistance to natural degradation: To perform this test, 30 samples of 2 cm × 2 cm × 2 cm of the THTwood were prepared, for each species/treatment, although the wood grain patterns were not taken into account, since it was not possible define the orientation because of the dimensions of the samples used. The accelerated test for resistance to natural degradation was carried out following the methodology of the ASTM standard designation D-2017-81 [22] and the resistance to attack of the fungi (M_{loss}) was measured by mass loss. Two types of fungi were used: *Trametes versicolor* and *Lenzites acuta*, of white and brown decay, respectively.

Statistical analysis: The mean and standard deviation of the data were estimated for each of the properties evaluated in the THTwood of *T. grandis* and *G. arborea*. Subsequently, an analysis of variance (ANOVA) was performed, for which the assumptions of normality and homoscedasticity of the data were verified. The data that did not comply with the assumptions of normality or homoscedasticity were transformed through the function "standardization" provided in the statistical program InfoStat, which allowed the fulfilment of the said assumptions. The statistical program Infostat was used to perform the statistical analysis of the data. The existence of significant differences between the means of the treatments was verified by a Tukey test, using a statistical significance of $p < 0.05$.

3. RESULTS AND ANALYSIS OF RESULTS

3.1. Determination of density, shrinking, mass loss and moisture absorption in thermo-treated wood

WD and SP variation for the different THT in relation to the type of wood (sapwood or hardwood) and type of grain pattern (radial or tangential) is presented in Table 1. WD diminished as the THT temperature increased, in both species, except in *T. grandis* radial hardwood THT at 210 °C and 220 °C, and tangential hardwood at 220 °C of the same species, where increased WD was observed relative to lower THT temperatures. In addition, higher values of WD observed correspond to the control treatment.

In relation to wood SP after THT, the results showed that *T. grandis* THTwood is statistically similar between the different temperatures for the two types of wood and the two grain patterns (Table 1). However, *G. arborea* THTwood is statistically different at some temperatures. The range of the values obtained in SP presented a variation of between 1.20 and 2.61 % in *T. grandis* and between 1.62 and 2.40 % in *G. arborea*. Similarly, it was observed that the SP values obtained at the different temperatures for *T. grandis* did not present a specific behavior. On the contrary, the values in SP obtained for *G. arborea* tended to increase as the THT temperature increased, except for tangential grain patterns of THTwood at a temperature of 200 °C, that showed a decrease in SP (Table 1). MLT of THTwood is shown in Fig. 1. In the case of *G. arborea* (Fig. 1 a), the MLT presented a similar behavior in both types of grain patterns, with a tendency to increase as the THT temperature increased. On the other hand, *T. grandis* THTwood (Fig. 1 b) showed similar behavior with both types of wood and grain patterns, where MLT diminished with temperatures from 205 °C to 210 °C. Subsequently, MLT increased progressively between temperatures from 210 °C to 220 °C (Fig. 1 b).

Fig. 1 shows the variation of the MABS for the different THT in the species *G. arborea* and according to the type of wood (sapwood or heartwood) in *T. grandis*. It can be observed that the M_{ABS} values for *G. arborea* (Fig. 1 c) presented an irregular behavior, where the THTwood at a temperature of 200 °C was the one that presented the highest MABS and the THTwood at a temperature of 185 °C presented the lowest MABS. Similarly, for *T. grandis* (Fig. 1 d), it was possible to observe an irregular behavior of the values between the different THT for the types of wood (sapwood and heartwood). However, it was observed that the sapwood showed higher M_{ABS} values for all treatments compared to heartwood, with values ranging from 1.99 to 2.66 % for sapwood and between 1.05 and 1.79 % for hardwood (Fig. 1 d). Firstly, it is important to mention that results showed that higher differences are found between grain pattern (radial or tangential) or type of wood (sapwood or heartwood than the difference between among temperature (Fig. 1, Fig. 2). This results show that the differences of used temperatures are very small and too small, therefore is invisible the influence of temperature of THT on the properties of wood. The decrease in WD of THTwood treated at different temperatures in the two wood types and grain patterns of *T. grandis* and *G. arborea* (Table 1) is congruent with reports of THTwood of *Pinus sylvestris* [23], *Pyrus elaeagnifolia* [9] and *T. grandis* [16]. This decrease in WD is associated to ML_T of the wood during the THT [24], as confirmed by the results obtained in the THTwood of *G. arborea* and *T. grandis* (Fig. 2 a, b).

Table 1. Wood density and shrinking percentage in wood of *T. grandis* and *G. arborea* for different types of grain pattern for sapwood and heartwood thermo-treated at four different temperatures

Type	Thermo-treated *Tectona grandis* wood	Thermo-treated *Gmelina arborea* wood

	Temperature, °C	Density, g cm^{-3}	Shrinking, %	Temperature, °C	Density, gcm^{-3}	Shrinking, %
Radial heartwood	Untreated	0.56^{A}	–	Untreated	0.47^{A}	–
	205	0.50^{B}	1.20^{A}	185	0.45^{AB}	1.62^{A}
	210	0.52^{B}	2.09^{a}	190	0.44^{B}	1.86^{A}
	215	0.49^{B}	2.24^{A}	195	0.44^{B}	2.20^{B}
	220	0.50^{B}	1.90^{A}	200	0.44^{B}	2.33^{B}
Tangential heartwood	Untreated	0.60^{A}	–	Untreated	0.46^{A}	-
	205	0.55^{B}	1.71^{A}	185	0.45A^{B}	1.89^{A}
	210	0.55^{B}	2.16^{A}	190	0.45A^{B}	2.25^{AB}
	215	0.54^{B}	1.94^{A}	195	0.45A^{B}	2.40^{B}
	220	0.55^{B}	2.61^{A}	200	0.43^{B}	2.36B
Tangential sapwood	Untreated	0.55^{B}	1.71^{A}	185	0.45A^{B}	1.89^{A}
	205	0.55^{B}	2.16^{A}	190	0.45A^{B}	2.25^{AB}
	210	0.54^{B}	1.94^{A}	195	0.45A^{B}	2.40^{B}
	215	0.55^{B}	2.61^{A}	200	0.43^{B}	2.36B
	220	0.53^{C}	2.07^{A}	–	–	–

Legend: Average values identified with different the letters are statistically different at α = 95 %.

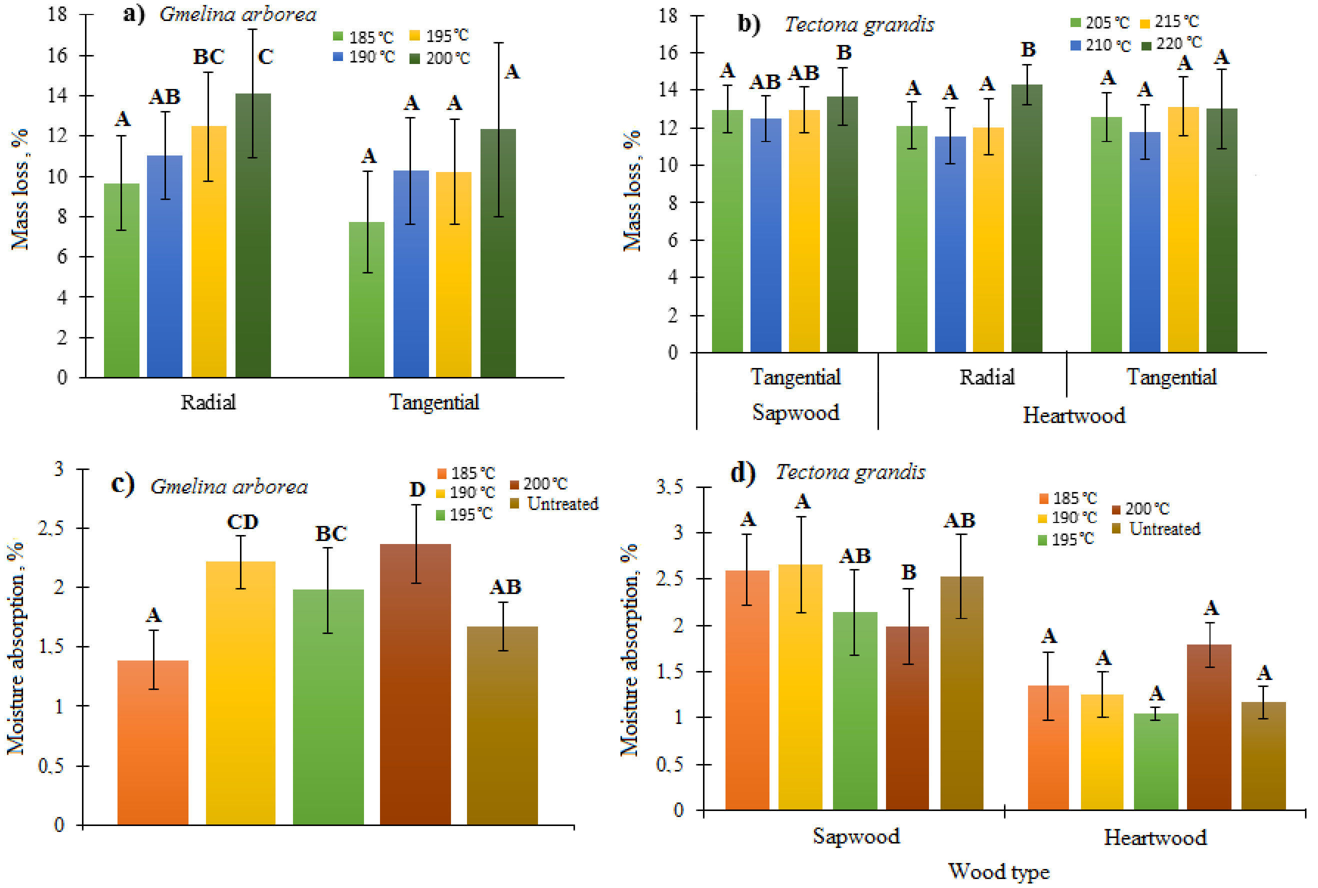

Fig. 1. a – mass loss in wood of *G. arborea;* b – *T. grandis* after thermo-treatment and moisture absorption in wood of *G. arborea*; c – *T. grandis*; d – for different types of grain pattern for sapwood and heartwood heat treated at four different temperatures. Legend: the error shows standard deviation and values identified with different letters are statistically different at α = 95 %

It was observed that ML$_T$ in the wood i4ncreased with increasing THT temperature (Fig. 2 a, b), which is congruent with reports on *Tilia cordata* Mill [4] and on THTwood of *Fagus orientalis* [25]. The ML$_T$ is attributed to the degradation of wood components (mainly hemicelluloses), produced by chemical reactions during the THT process, which causes the volatilization of wood extractives [26]. This thermal degradation causes a loss of weight in the wood, which is one of the most important indicators of the quality or the degree of the THT applied [27].

Not in all cases an increase in ML$_T$ with THT temperature occurs [1]. For example, there were no statistically significant differences by temperature in this study with regard to the THT tangential-grain wood of *G. arborea* (Fig. 1 a) and in THT tangential-grain heartwood of T. *grandis* (Fig. 1 b). In these cases, THT temperatures do not lead to significant changes in the loss of wood components and extractives. However, when ML$_T$

increases with temperature, for example, in *G. arborea* THT radial wood (Fig. 1 a) and in tangential and radial grain patterns of sapwood and hardwood of *T. grandis* (Fig. 1 b), it is due to high temperatures and prolonged times that produce a reduction of the mass [9].

WD reduction is one of the main indicators of the degree of THT [27], higher values of ML_T being obtained at higher temperatures. However, no significant differences were found between the THT temperatures of both species, except for tangential-grain sapwood at 205 °C, where significant differences with respect to the rest of THT temperatures were obtained (Table 1). This slight difference between WD of THTwood at the different temperatures can be explained by Gunduz et al. [9] and Uribe and Ayala [1], who point out that, although there is a decrease of the wood weight and volume with THT, such diminution is not significant as to cause a perceptible change in WD. Besides, our study, probably it was necessary to increase wood samples to increase results accuracy, especially statistical differences.

Regarding the SP of THTwood of both species, these values have the advantage of being lower than those of the International Thermowood Association [23], which indicates shrinking parameters for this type of wood between 5 and 8 %, while in this study the values range between 1.20 to 2.61 % (Table 1).

In general, with few exceptions, an increase in ML_T with increasing THT temperature was observed in both species (Table 1). This is because during THT process the wood is subjected to high temperatures that eliminate the water in the wood cell wall [28], therefore the shrinking. On the other hand, high SP values are expected in wood treated at high temperatures and in tangential grain pattern, for the reason that in this direction the strengths of the radiuses do not interfere due to the orientation of the parenchyma cells [29, 30]. This behavior was observed in *T. grandis* thermo-treated wood, where the highest shrinking percentages were obtained in tangential hardwood at a temperature of 220 ºC, whereas in *G. arborea* wood the greatest shrinking was obtained in tangential thermo-treated wood at a temperature of 200 °C (Table 1). However, some inconsistencies were found in the THTwood, for example, *T. grandis* heartwood, THT at 215 °C, showed greater shrinking in the radial direction with respect to shrinking in the tangential direction of sapwood and hardwood (Table 1). This behavior can be attributed to other factors such as the presence of juvenile wood [31], which presents high shrinking in the radial direction, or that the samples were obtained from central parts of the log, which is characterized by high presence of juvenile wood.

In THTwood, M_{ABS} tends to decrease with increasing THT temperature, due to its greater permeability caused by the chemical changes during the process [9], such as the removal of volatiles and organic compounds [1], mainly hydrophilic agents in wood [32]. Gunduz et al. [9] indicate that the THT significantly reduces radial and tangential stresses, resulting in lower M_{ABS}, changes that begin to occur at approximately 150 °C and intensify as the temperature increases. However, such behavior was not observed in the present study. The M_{ABS} in the THTwood increased in the different THT temperatures for the grain pattern types in *G. arborea* (Fig. 1 c). In the case of *T. grandis* THTwood (Fig. 1 d), no THT temperature produced a decrease of M_{ABS} in either sapwood or heartwood in relation to wood without THT.

Lack of improvement of M_{ABS} through THT in both species studied can be attributed to low M_{ABS} of the wood and good dimensional stability [12, 31]. Thus, thermo-treatment can cause few effects on water-related components and therefore few effects on M_{ABS} in THTwood, as found in the present study.

Fig. 2 shows the results obtained in the measurement of the total color change (ΔE*). In *G. arborea* THTwood (Fig. 2 a), the behavior of the ΔE* values is similar in both types of grain patterns, with a decrease of ΔE* in the THTwood from 185 °C to 190 °C. Subsequently, there was an increase of ΔE* as the THT temperature increased. Meanwhile, for *T. grandis* (Fig. 2 b), a similar behavior was again observed between different types of wood and grain patterns. THTwood at a temperature of 205 °C showed the highest ΔE* values. Furthermore, a decrease in ΔE* was obtained from 205 °C to 210 °C, and an increase later in ΔE* with the increase of the THT temperature (from 210 to 220 °C), (Fig. 2 b).

The change in perceptible color in the THTwood is related to the changes that the human eye perceives and this can be evaluated with the ΔE* [15]. It was found that, regardless of the species or grain pattern in the wood, there was a tendency to increase the ΔE* (high color change perceptible to the human eye) with the increase of the thermo-treatment temperature, coinciding with what was reported by the International Thermowood Association [23] for THTwood of *Pinus sylvestris*. Also, higher values of ΔE* were obtained in sapwood for *T. grandis* and *G. arborea* for being woods with whiter shades that when subjected to a THT process show a more noticeable color change compared to heartwood, where ΔE* is slightly less appreciable. However, in this type of white wood, the temperature range used has little ΔE* effect, since there were no statistically significant differences between some of the THT temperatures (Fig. 2). Again, our study, probably it was necessary to increase wood samples to increase results accuracy, especially statistical differences.

The color changes obtained after applying THT to the wood are mainly caused by the hydrolysis of the hemicelluloses [9, 33]. Hemicelluloses appear in the presence of essential components of wood, such as extractives, which not only contribute to color change [27] but also cause a decrease in the white tone (L* decrease). These changes are attributed to the degradation or modification of components through reactions such as oxidation, dehydration, decarboxylation and hydrolysis [26], as well as the darkening of lignin, linked to the generation of chromophore groups [11], which cause more noticeable color changes in woods such as *G. arborea* and in sapwood in *T. grandis*, since their shade is clearer.

In turn, Bourgois et al. [34] mention that diminution of the content of hemicelluloses, pentose in particular, in thermo-treated wood at 240 – 310 °C, causes an increase in ΔE*. This change is also associated with some functional groups as carbonyl, lignin structures, hemicelluloses or quinoid structures, linked to the generation of substances that produce color in THTwood [35].

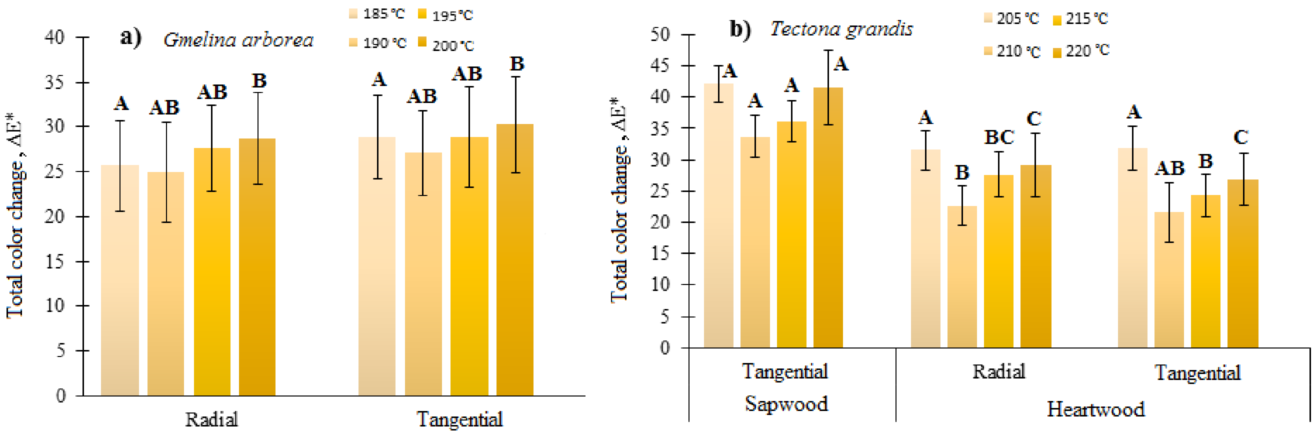

Fig. 2. a – total color change (ΔE*) in wood of *G. arborea*; b – *T. grandis* for different types of grain pattern for sapwood and heartwood thermo-treated at four different temperatures. Legend: the error shows standard deviation and values identified with different letters are statistically different at α = 95 %

Fig. 3. a – color changes in the wood of *Tectona grandis*; b – *Gmelina arborea*, according to the different thermo-treatment temperatures defined

Chen et al. [33] indicate that modification of wood components as hemicellulose and lignin degradation, as well as the presence of residual extractives due to wood exposure to high temperatures causes wood color to change. This change is more noticeable as the THT temperature increases. Thermal degradation is directly related to the degree of darkening of the properties of the color and the total color change (ΔE*), which depend on the conditions of the thermal treatment applied [11] and, as a result, the color changes are visually appreciable (Fig. 3).

3.2. Determination of the modulus of elasticity and modulus of rupture in flexion test in thermo-treated wood

Fig. 4 shows the mechanical properties evaluated in THTwood. The MOE for *G. arborea* presented similar irregular trend between the two types of grain patterns (Fig. 4 a). This property increased continuously from the control treatment to THTwood at 190 °C, diminished later in THTwood at 190 °C and 195 °C, increased again at 195 °C and 200 °C (Fig. 4 a).

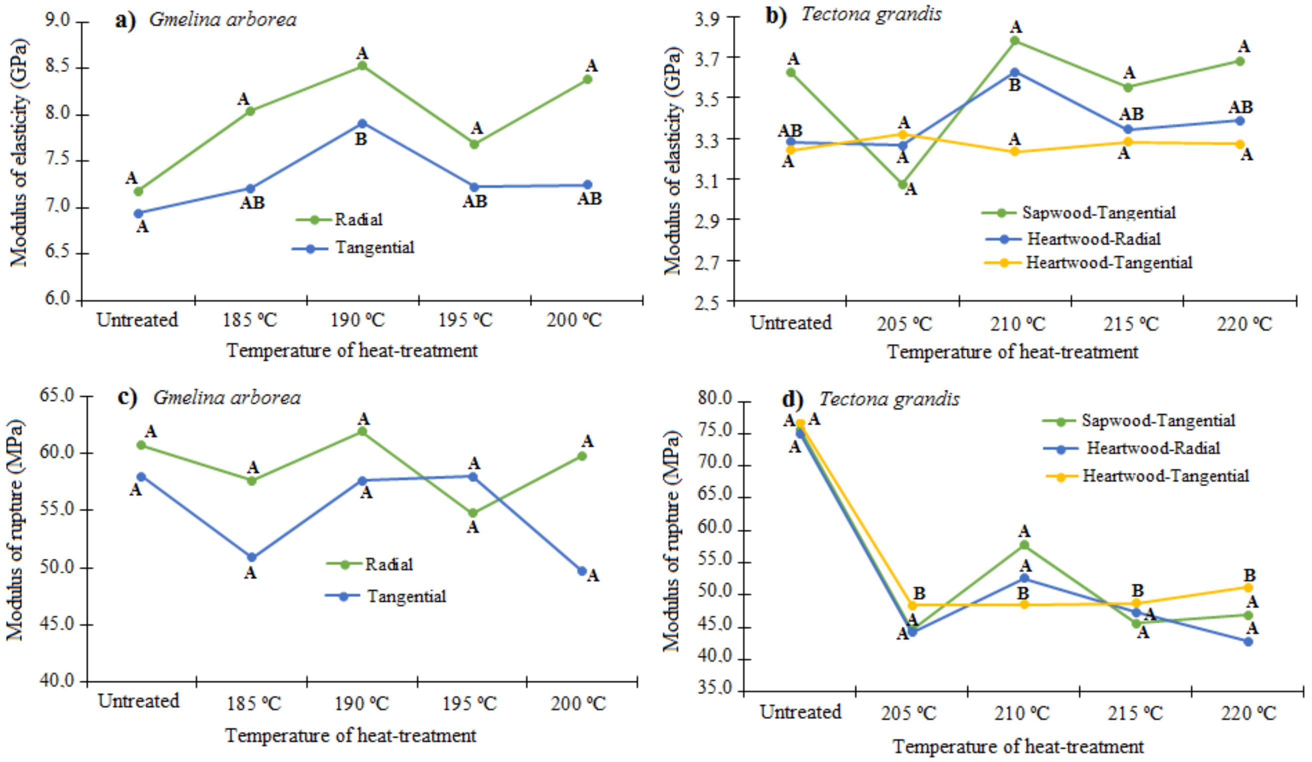

Fig. 4. MOE and MOR in wood for different types of grain pattern for sapwood and heartwood heat treated at four different temperatures: a – MOE in *G. arborea*; b – MOE in *T. grandis*; c – MOR in *G. arborea* and d – MOR in *T. grandis*. Legend: the error shows standard deviation and values identified with different letters are statistically different at α = 95

The values of MOE in *T. grandis* (Fig. 4 b) presented an irregular behavior between the different temperatures, with tangential sapwood and radial heartwood presenting the higher values of MOE, except for the thermo-treated wood at 205 °C.

In turn, the treatment at 210 °C presented the highest values in MOE for tangential sapwood and radial heartwood. As for tangential heartwood, the highest MOE values were obtained in the treatment at 205 °C (Fig. 4 b). The results obtained in the MOR in flexion test show that the variation of the data in *G. arborea* (Fig. 4 c) was irregular between the different THT temperatures for both types of grain patterns (radial and tangential). The MOR presented an increase-decrease behavior as the established THT temperatures changed, i.e., a decrease in the values of the THTwood at 185 °C in relation to the wood without THT. But there is an increase in the values of the THTwood at 190 °C in relation to the THTwood at 185 °C, followed by a decrease and increase of the MOR values in THTwood at 195 °C and 200 °C respectively (Fig. 4 c), except for the treatment at 195 °C for the tangential grain pattern, which presented values similar to the THT at 190 °C, without showing much variation. For *T. grandis* (Fig. 4 d), it was found that the MOR presented a similar behavior between the different types of wood and types of grain patterns, being the values of tangential heartwood those that presented the smaller variation between the different treatments applied. In the same way, a decrease of the MOR was observed between the values of the wood without THT in relation to the values obtained from the samples to which the THT was applied (Fig. 4 d).Although variation among the values of MOR obtained for THTwood of both *G. arborea* (Fig. 4 c) and of *T. grandis* (Fig. 4 d) in the different THT temperatures could be noticed, no significant differences could be established between the temperatures for the various types of wood and grain patterns analyzed (Fig. 4). The differences of used temperatures are very small and too small, therefore is invisible the influence of temperature of THT on the properties of wood. However, values of MOR and MOE for *T. grandis* (Fig. 4 d) decreased with the THT, coinciding with the International Thermowood Association [23], who indicates for *Pinus sylvestris* that MOR and MOE reductions and higher resistance losses were evident at temperatures above 220 °C, therefore THTwood for structural uses is not recommended. The weakening of the mechanical properties arises from the modification of the wood due to the chemical degradation of polymers [27]. According to Awoyemi and Jones [36], the reduction in MOR and MOE values is due to the degradation of tracheid walls – which in turn coincides with the degradation of the hemicelluloses, one of the main components of the cell wall – and because of the increasing content of crystalline cellulose and replacement of flexible hemicellulose-cellulose-hemicellulose bonds with more rigid cellulose-cellulose bonds [26].

Another important aspect indicated by Bal [37] regarding THTwood, is the decrease in the mechanical strength of the wood, which is higher as the THT temperature increases, due to hemicelluloses and celluloses becoming more rigid [26, 39]. The THT not always reduces the mechanical properties of the wood. Kocaefe et al. [39] found that both MOR and MOE increased up to a THT temperature of 160 °C, and then both decreased as the temperature increased.

3.3. Determination of the tensile adhesion and percentage of glue line failure in thermo-treated wood

The values of Ts presented a tendency to decrease with increasing thermo-treatment temperature, except for the tangential sapwood at 215 °C, which presented an increase of Ts in relation to the THTwood at 210 °C (Table 2). Also, wood without THT presented the highest Ts values for each of the wood types and grain patterns in both species.

Table 2. Tensile adhesion and failure percentage of the glue line in wood of *G. arborea* and *T. grandis* for different types of grain pattern for sapwood and heartwood heat treated at four different temperatures

Type	Thermo-treated *Tectona grandis* wood			Thermo-treated *Gmelina arborea* wood		
	Temperature, °C	Tensile adhesion, MPa	Failure of glue line, %	Temperature, °C	Tensile adhesion, MPa	Failure of glue line, %
Radial heartwood	Untreated	9.40^{A}	81.89^{A}	Untreated	8.01^{A}	84.39^{A}
	205	3.10^{A}	77.78^{A}	185	4.50^{A}	53.17^{A}
	210	2.85^{A}	61.78^{A}	190	4.95^{A}	70.17^{A}
	215	2.46^{A}	43.78^{A}	195	4.28^{A}	75.33^{A}
	220	2.58^{A}	63.72^{A}	200	4.13^{A}	80.81^{A}
Tangential heartwood	Untreated	11.52^{A}	55.06^{A}	Untreated	8.69^{A}	57.33^{A}
	205	5.54^{A}	51.50^{A}	185	6.35^{A}	64.56^{AB}
	210	4.54^{A}	48.06^{A}	190	5.34^{A}	81.89^{B}
	215	4.07^{A}	46.33^{A}	195	5.75^{A}	60.83^{AB}
	220	3.34^{A}	18.83^{A}	200	5.28^{A}	78.94^{AB}
Tangential sapwood	Untreated	11.52^{A}	69.33^{A}	–	–	–
	205	4.84^{A}	72.61^{A}	–	–	–
	210	4.24^{A}	60.94^{AB}	–	–	–
	215	5.45^{A}	41.33^{BC}	–	–	–
	220	3.13^{A}	26.39^{C}	–	–	–

Legend: Average values identified with different the letters are statistically different at α = 95 %

Regarding the parameter Fp, wood and grain patterns of the species did not present any specific behavior, except for tangential heartwood of *T. grandis*, which presented a tendency to diminish the Fp with increasing THT temperature (Table 2). The Ts by traction of overlapping joints is shown in Table 2, which shows that the resistance decreases as the THT temperature increases in both species, regardless of the type of wood and type of grain pattern defined. However, despite this decrease, no statistically significant differences were found between the different THT temperatures. Regarding the wood Fp, *T. grandis* presented a decrease with increasing THT temperature, indicating that THTwood at higher temperatures showed higher Fp. However, significant differences were only observed in the sapwood with tangential grain. On the other hand, the wood of *G. arborea* showed irregular behavior in relation to the Fp, as only the wood with tangential grain presented statistically significant differences.

In addition, it can be observed that the *G. arborea* THTwood had values of Ts slightly higher than those of *T. grandis*, which may be due to the fact that the THTwood of *T. grandis* used higher THT temperatures, resulting in greater wood fragility [25]. On the other hand, Kuzman et al. [40] indicate an increase in the tensile adhesion in bonded wood joints of *Fagus sylvatica* and *Picea abies* when heat treated at 190 °C, compared to non-thermo-treated samples. However, these authors did not obtain significant differences in the strength of the joints glued in THTwood at different temperatures, coinciding with results from this study where no statistically significant differences were found (Table 2). Ts decrease occurs mainly due to the degradation of hemicelluloses, which are less heat stable than cellulose and lignin [35, 40]. In addition, the increase in Fp values is due to the breakdown of OH bonds and volatilization of extractives due to heat, which interferes with the quality of the bond between the adhesive and the substrate [26].

3.4. Determination of durability in accelerated tests of resistance to natural degradation of thermo-treated wood

The M_{loss} values were particularly irregular for the two types of fungi (*L. acuta* and *T. versicolor*) used in the test and the different THT temperatures (Table 3). *T. grandis* untreated wood (control treatment) showed statistically higher values of M_{loss} for both types of wood (sapwood and heartwood) with both types of fungi. Meanwhile, the lower values of M_{loss} in *T. grandis* sapwood were obtained in the THTwood at 205 °C with *L. acuta*, and at 220 °C with T. versicolor, whereas in heartwood, the lowest M_{loss} was obtained in the THTwood at 210 °C for both types of fungi (Table 3). For *G. arborea* the highest values of M_{loss} were observed in the THTwood at 200 °C and 190 °C for *L. acuta* and *T. versicolor*, respectively, and the lower values corresponded to THTwood at 190 °C and 195 °C for *L. acuta* and *T. versicolor*, respectively (Table 3). The M_{loss} accelerated test of resistance to fungal decay (Table 3) classified the THTwood in the different temperatures and different types of fungi between "highly resistant" and "resistant" to the fungi *T. versicolor* and *L. acuta*. This according to the ASTM 2017 standard [22], since the M_{loss} values are between 0 and 24 %, contrasting with untreated sapwood of *T. grandis* species, which presents M_{loss} values above 25 % for *T. versicolor*, cataloging it as "moderately resistant" based on the aforementioned norm. An important aspect to be mentioned is that the THTwood at different temperatures showed no statistically significant differences between the different THT temperatures and the wood without THT (Table 3). This coincides with Kamdem et al. [41], who mention that M_{loss} obtained for THTwood of *Fagus sylvatica*, *Pinus sp.* and *Picea abies* exposed to two brown rot fungi (*Gloeophyllum trabeum* and *Poria placentra*) and a white rot (*Irpex lacteus*), did not present significant differences between the THT temperatures used (between 200 and 206 °C).

According to Venäläinen et al. [42], M_{loss} caused by fungi is due initially to polysacarid degradation (cellulose and hemicellulose) and later, at more advanced stages of brown rot fungus, to lignin degradation, while white rot fungi degrade lignin first and then hemicellulose, aspects that are reflected when applying the THT. According to Calonego et al. [43], the increase in resistance to disintegration caused by fungal attack is related to duration and THT temperature, being more evident at higher exposure times and temperatures.

Table 3. Weight loss by *Lenzites acuta* and *Trametes versicolor* in wood of *G. arborea* and *T. grandis* for different types of grain pattern for sapwood and heartwood heat treated at four different temperatures

Type	Thermo-treated *Tectona grandis* wood			Thermo-treated *Gmelina arborea* wood		
	Temperature, ºC	*Lenzites acuta*, %	*Trametes versicolor*, %	Temperature, ºC	*Lenzites acuta*, %	*Trametes ersicolor*, %
Sapwood	Untreated	14.76^{A}	29.32^{A}	Untreated	19.44^{A}	17.76^{A}
	205	8.31^{A}	12.14^{A}	185	19.48^{A}	11.95^{A}
	210	13.20^{A}	12.20^{A}	190	24.09^{A}	6.65^{A}
	215	9.46^{A}	6.82^{A}	195	19.05^{A}	22.44^{A}
	220	13.55^{A}	4.90^{A}	200	17.19^{A}	7.82^{A}
Heartwood	Untreated	13.91^{A}	6.33^{A}	-	-	-
	205	10.83^{A}	4.72^{A}	-	-	-
	210	9.65^{A}	1.97^{A}	-	-	-
	215	12.60^{A}	3.07^{A}	-	-	-
	220	11.85^{A}	4.53^{A}	-	-	-
Legend: Average values identified with different the letters are statistically different at α = 95 %.						

This is because the higher the THT temperatures, the higher the decrease in moisture, hemicelluloses, as well as lipids, starch and some fatty acids necessary for fungal growth and capacity of action [39].When comparing the results obtained in this study with the two wood species (Table 3), it can be observed that *G. arborea* presented M_{loss} per attack higher than those obtained in *T. grandis* with both types of fungus. Paul et al. [44] reported a slight increase in resistance to fungal degradation when the wood is THT at temperatures below 200 °C, as applied to the wood of *G. arborea*, which can explain the difference. In fact, authors such as Jämsä et al. [45] (1999) recommend using a minimum temperature of THT of 220 °C to achieve a significant increase in the resistance to the decomposition by fungi, aspects that were not applied in this study and could be influencing the results obtained.

5. CONCLUSIONS

According to the findings of this study, it was possible to determine that the thermal treatment, in both *T. grandis* and *G. arborea*, influences the properties of the wood, favoring some and affecting others. The differences of used temperatures are very small and too small, therefore is invisible the influence of temperature of THT on the properties of wood, but the higher change was observed in wood color. In order to achieve the best characteristics of the material, a balance between the desired and the optimal characteristic must be made. This depends on the purpose for which the wood is being heat treated. If the aim is obtain wood with an enhanced color and greater durability for uses where little effort is required, then high thermo-treatment temperatures are recommended. On the contrary, if what is needed is material with better mechanical properties, then lower thermo-treatment temperatures are advisable, taking into account that wood with less color changes and less durability will be produced, among other aspects.

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SCIENTIA
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Effects on density, shrinking, color changing and chemical surface analysis through FTIR of *Tectona grandis* thermo-treated

Density, shrinking, color changing and chemical analysis by FTIR of the thermally-treated teak wood

Luis Diego Méndez-Mejías[1] e Roger Moya[1]

Resumo

O tratamento térmico da madeira tem sido implementado, nos últimos anos, como uma opção para melhorar a sua durabilidade. Este estudo avaliou o efeito da temperatura do tratamento térmico na densidade, contração, alteração da cor (sistema L*a*b*) e composição química aplicando o FTIR na madeira de *Tectona grandis*. Amostras de madeira de cerne e de alburno com padrões de corte radial e tangencial foram expostas a três níveis de temperatura, 210 ºC, 215 ºC e 220 °C, durante 6 horas e as propriedades das madeiras tratadas foram comparadas com a madeira não tratada. Os resultados indicaram que a densidade da madeira decresceu de 0,61 para 0,49 g cm^{-3}, com uma tendência a diminuição com o aumento da temperatura. A contração da madeira variou de 2,61 a 1,58 % não sendo encontradas diferenças estatísticas entre as temperaturas. As análises do FTIR mostraram um leve aumento nas bandas de 1024, 1098, 1131, 1249, 2928 e 3340 cm^{-1}, indicando uma alteração da lignina com o aumento da temperatura usada no tratamento térmico. A alteração da cor da madeira foi maior na luminosidade (L*) seguido pelo seu avermelhamento (a*). A tonalidade amarela (b*) mostrou as menores alterações. Finalmente, o tratamento térmico a temperatura de 220 °C mostrou os melhores resultados em relação às propriedades da madeira avaliadas.

Palavras-chave: Tratamento térmico, madeira tropical, temperatura, estrutura da madeira, grupos funcionais, polímeros.

Abstract

Thermal-treatment wood is used as an option to improve durability of wood in recent years. This study evaluated the effect of temperature in thermally treated *Tectona grandis* wood in density, shrinking, color changing (L*a*b* system) and surface analysis through FTIR. Sapwood and heartwood boards sawn with radial and tangential patterns were exposed to three temperature-levels: 210 ºC, 215 ºC, 220 °C during 6 hours and thermally treated wood properties were compared with untreated wood. Results showed that density decreased from 0.61 to 0.49 g cm^{-3} with a tendency to decrease as temperature increases. Wood shrinking ranges from 2.61 to 1.58 % and no statistical differences between temperatures were found. The FTIR analysis showed a slight increase in the bands at 1024 cm^{-1}, 1098 cm^{-1}, 1131 cm^{-1}, 1249 cm^{-1} 2928 cm^{-1} and 3340 cm^{-1} with the temperature increases, indicating a lignin change. Color change showed major changes in luminosity (L*), followed by redness (a*); while yellowness (b*) showed minor changes. Finally, the thermal treatment at 220 °C had the best results regarding the evaluated properties.

Keywords: Heat-treatment, tropical wood, temperature, wood structure, functional groups, polymers.

INTRODUCTION

Wood is susceptible to dimensional changes due to the change in environmental conditions, which greatly limits its use in some applications (KOCAEFE et al., 2015). Nevertheless, these weaknesses can be improved through technical and engineering methods, where structural properties or durability were improved (PRIADI; HIZIROGLU, 2013).

Durability improved using chemical substances as wood preservatives or through nanotechnology (MOYA et al., 2014a). Use of these products becomes increasingly limited, calling for the need of research on new techniques to increase durability in wood with low toxicity methods or eliminating the use of chemicals (TREVISAN et al., 2014).

A technique to increase durability is thermal treatment. This process involves exposing wood to high temperatures (from 170 °C at 220 °C), in the absence of atmospheric oxygen, which leads

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[1] MsC in Forest Science. Instituto Tecnológico de Costa Rica / Escuela de Ingeniería Forestal. P.O. Box: 159-7050. Cartago, Costa Rica. E-mail: ldiegomm@gmail.com.

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to changes in its structure and chemical composition: it can improve dimensional stability of wood; increase resistance to several biologic agents and produce an increasing of color uniformity (ZIGON et al., 2015). Temperature and time used with thermal-treatment varies with species, wood dimensions, moisture content and final use of wood (KESIK et al., 2014).

Teak (*Tectona grandis*) is one of the most important tropical species due to its properties and versatility of uses (TEWARI; MARISWAMY, 2013). Wood from fast-growing plantations has a higher juvenile wood content, which usually has low physical, mechanical and durability properties (MOYA et al., 2014b).

Some studies in teak wood have emphasized behavior, processes and characteristics of thermal treatment. For example, Lopes et al. (2014a,b,c) showed changes in some properties of juvenile wood (density, moisture content, color change, color uniformity, and resistance to weathering) for thermo-treated teak wood in Brazil at temperatures of 180 °C and 200 °C during a time of 2 hours and 30 minutes. In another study carried out by KASHYAP et al. (2014), the effects of durability in laboratory conditions with white rot fungi of thermo-treated wood at three temperatures (160 °C, 180 °C and 210 °C) during exposition times of 0.5, 3, 5 and 25 hours were determined.

In spite of these studies, information about the process in thermally treated wood with industrial equipment is limited. In order to obtain an environmentally friendly product with better properties and characteristics enabling potential use and competition in the market this research was done. It aimed to determine the effect of three different temperature levels (210 °C, 215 °C and 220 °C) on thermally treated teak wood: in density, shrinking, color and surface analysis through Fourier Transform Infrared Spectroscopy (FTIR) with industrial equipment..

MATERIALS AND METHODS

Origin and characteristics of the material used

Wood used was wood from the second thinning from a *Tectona grandis* plantation, 11 years old, spacing of 3 x 3 m (1100 trees ha^{-1}). Density of the plantations was 475 trees ha^{-1}, with an average diameter at breast height of 23 cm and total height of 14 m. The plantation is located in the Northern Zone of Costa Rica and owned by the company Ethical Forestry S.A. 7-9 logs were felled from sample trees, logs were 1.25 m long. Logs were sawn into boards 7.5 cm wide x 2.5 cm thick x 1.25 m long and dried according to research conducted by Salas and Moya (2014).

Sampling

Thermal treatment was separate in sapwood and heartwood and within these types, wood with tangential and radial patterns. Then, 30 boards of about 7.5 cm wide x 2.5 cm thick x 1.25 m long were taken from each type of wood. Each of these samples was divided into 4 parts (boards of 7.5 cm wide x 2.5 cm thick x 30 cm long). Sapwood timber only had samples with tangential pattern, since it was not possible to get radial pattern.

Thermal-treatment process

Dried lumber, with approximately 12 % moisture content was thermally treated in three different temperatures levels: 210 °C, 215 °C and 220 °C. For each treatment, 30 boards were selected per type of wood (sapwood and heartwood) and cutting section (tangential and radial). Each thermal treatment process was made independently and performed in the absence of oxygen. Boards were introduced and stacked into a thermowood pilot plant with the trade-name Valutec® (http://www.valutec.se). The thermal treatment process started with the drying for approximately 17 hours at a temperature starting from 130 °C to obtain 0 % moisture content. Subsequently, temperature was increased from 130°C to the temperature defined for each type of thermal-treatment (210 °C, 215 °C or 220 °C) and maintained during 6 hours. Next, a conditioning stage was applied during 7 hours; with steam, water and temperature applied to moisturize the timber and achieve approximately 6 % moisture content. Finally, a cooling process was applied during 3 hours. The different treatments were defined as follows: untreated wood as control; thermal-treatments at 210 °C; 215 °C and 220 °C.

Determination of density and shrinking of wood

Density of wood from each treatment was determined using samples 5 cm wide x 2.5 cm thick x 2.5 cm taken from each thermally treated board. Volume and weight were measured and density was determined (mass/volume). For the shrinking percentage of wood, 5 samples from the 30 samples of each treatment were selected. Width before and after thermo-treatment was measured and equation 1 was used for shrinkage determination:

$$Shrinking\ (\%) = \left(\frac{Dimension\ before\ (\mathrm{mm}) - \mathrm{Dimension\ after}\ (mm)}{Dimension\ before\ (mm)}\right) * 100 \qquad (1)$$

Measurement and analysis of FTIR spectra on wood surface

For this analysis, 24 samples were prepared in total, 2 samples per type of thermal-treatment and per cutting section, with dimensions of 1.5 cm long x 1.5 cm wide and 0.4 cm thick, taken from the thermally treated boards. Subsequently, a Fourier Transformed Infrared Spectroscopy (FTIR) analysis was performed. Samples were measured on its surface with a Spectrometer Nicolet 380 FTIR (Thermo Scientific) using only a reflectance cell (equipped with a diamond crystal). All data were recorded at room temperature, in the spectral range of 4000-500 cm^{-1}, by accumulating 32 scans with a resolution of 1 cm^{-1}, with background correction every 10 minutes.

FTIR signals obtained from spectrum were subsequently processed with Microsoft Excel 2010 through scatter plots. An action range of 600 to 3600 cm^{-1} was defined to identify vibration of the bands and the main components in each of the samples tested, based on previous studies where action range for the different functional groups are defined (BERROCAL et al., 2016).

Color changes in wood

Color on wood surface was measured using the color system Lab. For measuring color, a spectrophotometer miniSkan XE Plus from HunterLab was used, with standardized chromatologic system CIE L*a*b. Measurement ranged from 400 to 700 nm, with an opening in the measuring point of 11 mm. Observing reflection includes the specular component (SCI mode), with a 10° angle, which is normal to the specimen surface (D65/10); a view field of 2° (Standard observer, CIE 1931) and an illumination level D65 (corresponding to Daylight in 6500 K). Color system CIELAB enables color measurement three-dimensionally.

Color was measured before and after the thermal treatment and then the total color change was quantified (ΔE^*), according the standard ASTM D 2244 (ASTM, 2005) through equation 2:

$$\Delta E *= \sqrt{(\Delta L *)^2 + (\Delta a *)^2 + (\Delta b *)^2} \qquad (2)$$

Where: $\Delta L^* = L^*_{\text{after thermal-treatment}} - L^*_{\text{before thermal-treatment}}$, $\Delta a^* = a^*_{\text{after thermal-treatment}} - a^*_{\text{before thermal-treatment}}$ and $\Delta b^* = b^*_{\text{after thermal-treatment}} - b^*_{\text{before thermal-treatment}}$

Selection of the best treatment

To select the treatment with the most favorable results with the least density variation, the least shrinking and the major color change, each variable was weighted with a score to calculate and index later. Weighting each variable evaluated was established according to data variation, considering those that bring a major change and thus are of greater importance as selection criteria. For density and shrinking, a contribution of 30 % was defined for each and color change represents 40 % on the selection of the treatment with the most desired results (Table 1). Categorizations defined for variables of density, shrinking and total color change can be seen in Table 1:

Table 1. Classification parameters for density, shrinking percentage and color change for thermally treated teak wood.

Tabela 1. Parâmetros de classificação para a densidade, porcentagem de contração e alteração de cor da madeira de teca tratada termicamente.

Density (g cm^{-3})	Shrinking (%)	ΔE^*	Classification
< 0.51	> 2.35	< 26.66	Bad (1 pt)
0.51 - 0.53	1.84 - 2.35	26.66 - 36.17	Regular (2 pts)
> 0.53	< 1.84	> 36.17	Good (3 pts)

Statistic analysis

A variance analysis (ANOVA) was applied to estimate data variation, where the assumptions of normality and homoscedasticity were verified. For color change data, parameters L*, a*, b* were transformed at Log_{10} to meet the assumptions. The existence of significant difference between treatments was verified through Tukey's Test ($P<0.05$). For FTIR spectra, a scatter plot of frequency values cm^{-1} of the bands (X axis) and absorbance (Y axis) was used, to try observing changes in the bands of the different functional groups.

RESULTS AND DISCUSSION

Density and shrinking of the wood

Variation of density and shrinking of different types of thermally treated wood (sapwood or heartwood) and cutting section (radial or tangential) is shown in Table 2. A slight decrease in wood density is observed when thermo-treatment temperature increases in the types of wood and cutting sections, however it fails to be statistically different; but there is a statistical difference with untreated wood or control (Table 2). Shrinking percentages obtained for samples from the different treatments tend to be more variable; however, it was not possible to establish statistical differences between temperature effects.

Table 2. Density and shrinking percentage of the thermally treated teak wood at three different temperature levels according to wood type and cutting section.

Tabela 2. Densidade e porcentagem de contração da madeira de teca tratada termicamente sob diferentes níveis de temperatura de acordo com o tipo de madeira e plano de corte.

Wood type	Cutting section	Treatment	Shrinking (%)	Density (g cm^{-3})
Sapwood	Tangential	Control	-----------	0.60 (±0.05)B
		210 °C	1.75 (±0.64)A*	0.54 (±0.04) A
		215 °C	1.58 (±0.80)A	0.53 (±0.04) A
		220 °C	2.07 (±0.57) A	0.53 (±0.04) A
Heartwood	Radial	Control	-----------	0.56 (±0.06)B
		210 °C	2.09 (±1.12) A	0.52 (±0.05) A
		215 °C	2.24 (±0.42) A	0.49 (±0.04) A
		220 °C	1.90 (±0.54) A	0.50 (±0.05) A
	Tangential	Control	-----------	0.61 (±0.06)B
		210 °C	2.16 (±0.94) A	0.55 (±0.05) A
		215 °C	1.94 (±1.66) A	0.54 (±0.05) A
		220 °C	2.61 (±0.79) B	0.55 (±0.05) A

*Value in parentheses indicates standard deviation and values with similar letters indicate no statistically significant difference between treatments for 95 % confidence.

Decreasing wood density according to the different thermal-treatment temperatures (Table 2) occurred due to degradation of wood components during the roasting process, specifically removing volatile contents (extractives, low molecular weight components) through chemical reactions occurring during the process (KOCAEFE et al., 2008).

Nevertheless, no significant differences were found between the different temperatures (Table 2), coinciding with the results by Kasemsiri et al. (2012) and Guller (2012) in *Juniperus virginiana* and *Pinus nigra* species, respectively. This lack of difference can be explained as indicated by Uribe and Ayala (2015), who mention that although there is variation by weight loss and decrease in the volume of the samples after applying the thermal treatment process, is not significant enough to cause a noticeable change between the different treatments. Besides, before 170 °C most components are eliminated (KOCAEFE et al., 2008), temperature that reaches biomass in any of the different thermal-treatment temperatures.

For the shrinking percentage in wood at different thermal treatment temperatures (Table 2), although no significant differences were found regardless the type of wood or cutting section, wood with tangential pattern had greater shrinking than radial cut for treatment at 220 °C. This difference appears due to tangential direction, which has greater dimensions instability due to vertical orientation of micro-fibrils in the S2 layer on the cell wall and the orientation of parenchyma cells in radial direction, (PRIADI; HIZIROGLU, 2013). However, such behavior is not met at the other temperatures, since tangential pattern in sapwood and heartwood showed lower shrinking values compared to values obtained in radial pattern wood (Table 2).

Analysis of FTIR spectra on the surface of wood

The FTIR spectra of the untreated and thermally treated teak wood are presented in figure 1. This shows that the greatest effects stem from the different functional groups in wood components in the range of 1800-800 cm^{-1}. From range 1800 cm^{-1} onwards, very little variation is observed, except for the bands between 3600 to 2750 cm^{-1} assigned to the stretching of hydroxyl groups (Figure 1).

In Figure 1a it can be observed that for sapwood timber with tangential pattern, bands for control and 220 °C treatments have similar behavior, but with a decrease in the different signals obtained compared to treatments at 210 °C and 215 °C, where 215 °C has the highest increase peak in the bands 1024 cm^{-1}, 1098 cm^{-1}, 1131 cm^{-1}, 1249 cm^{-1}, 1311 cm^{-1}, 1361 cm^{-1}, 2928 cm^{-1} and 3340 cm^{-1}, but with little difference regarding treatment at 210 °C. Then the great signal of FTIR were reflected in 1024 cm^{-1}, 1098 cm^{-1}, 1131 cm^{-1}, 1249 cm^{-1}, 1311 cm^{-1}, 1361 cm^{-1}, 2928 cm^{-1} and 3340 cm^{-1} for thermally-treated teak with temperatures from 210 to 220 °C.

Signals obtained for heartwood with tangential pattern by the different treatments (Figure 1b) show that treatments at 210 °C and 215 °C have a similar behavior with each other with a decrease in band intensity, compared to treatments at 220 °C and control that represent peaks with greater increase. Additionally, an increase in the bands at 1024 cm^{-1}, 1098 cm^{-1}, 1131 cm^{-1}, 1249 cm^{-1} 2928 cm^{-1} and 3340 cm^{-1} can be appreciated as thermal treatment temperature rises.

As for heartwood with radial pattern (Figure 1c), it was found that treatments at 210 °C, 220 °C and control have signals with similar magnitudes, while among them, 220 °C has the highest magnitude. Signals obtained for treatment at 215 °C have a decrease in all the signals compared to the other treatments.

Increase in the intensity of bands at 1508 cm^{-1} and 1428 cm^{-1} (Figure 1), assigned to the carboxyl group which may be due to stretching of aromatic skeleton components of lignin and band at 1740 cm^{-1}, assigned to the stretching vibration of carbonyl, carboxyl and acetyl groups (CHEN et al., 2014; LI et al., 2015). Therefore, there is a change in the chemical structure of lignin in wood that has been thermo-treated.

Changes obtained in the intensity of bands at 1131 cm^{-1}, 1098 cm^{-1}, 1024 cm^{-1}, and 669 cm^{-1}, may be related to the modification of cellulose and hemicellulose (YILDIZ et al., 2013). According to Lionetto et al. (2012), changes in bands at 1318 cm^{-1} and 1053 cm^{-1} are associated to increases in the stretching vibrations in glucose rings, which may be due to cleavage and dehydration of amorphous carbon hydrates or crystallization of the para-crystalline region of cellulose, so signals obtained in bands at 1311 cm^{-1} may be due to these changes. Signals obtained in 1024 cm^{-1} are assigned to stretching variation of C–O (LIONETTO et al., 2012).

In band at 1589 cm^{-1} a stretching of the aromatic rings of lignin can be detected (FACKLER et al., 2011). Signals obtained at 1311 cm^{-1} are assigned to C–O vibration in lignin and at 1361 cm^{-1} associated to deformation of C-H group in cellulose and hemicellulose. Moreover, Kocaefe et al. (2008) indicate that bands at 1260 cm^{-1} are due to cleavage of acetyl groups in hemicellulose, which form carboxylic acids and lignin degradation; in this case signals obtained in bands at 1249 cm^{-1} may be associated to these factors (Figure 1).

Signals obtained between 2928 cm^{-1} and 3340 cm^{-1} correspond to functional groups of hydroxyl, coinciding with that reported by Li et al. (2015) and Huang et al. (2012) who indicated that bands between the range 3800-2750 cm^{-1} are assigned to hydroxyl and methyl groups or stretching vibrations of methylene, in the components of hemicellulose, lignin and cellulose.

Otherwise, Yildiz et al. (2013) indicated that thermal treatment causes a reduction of hydroxyl groups that are replaced by hydrophobic acetyl groups resulting from the reticulation between wood fibers by the thermal treatment applied. This thermal process is related to an increase in the dimensional stability of wood (KASEMSIRI et al., 2012; LEKOUNOUGOU; KOCAEFE, 2014). Besides, changes produced in the chemical structure of lignin, cellulose and hemicellulose caused by temperature, prevents reabsorption of water molecules linked together or with wood polymers improving the dimensional stability of the material (PRIADI; HIZIROGLU, 2013).

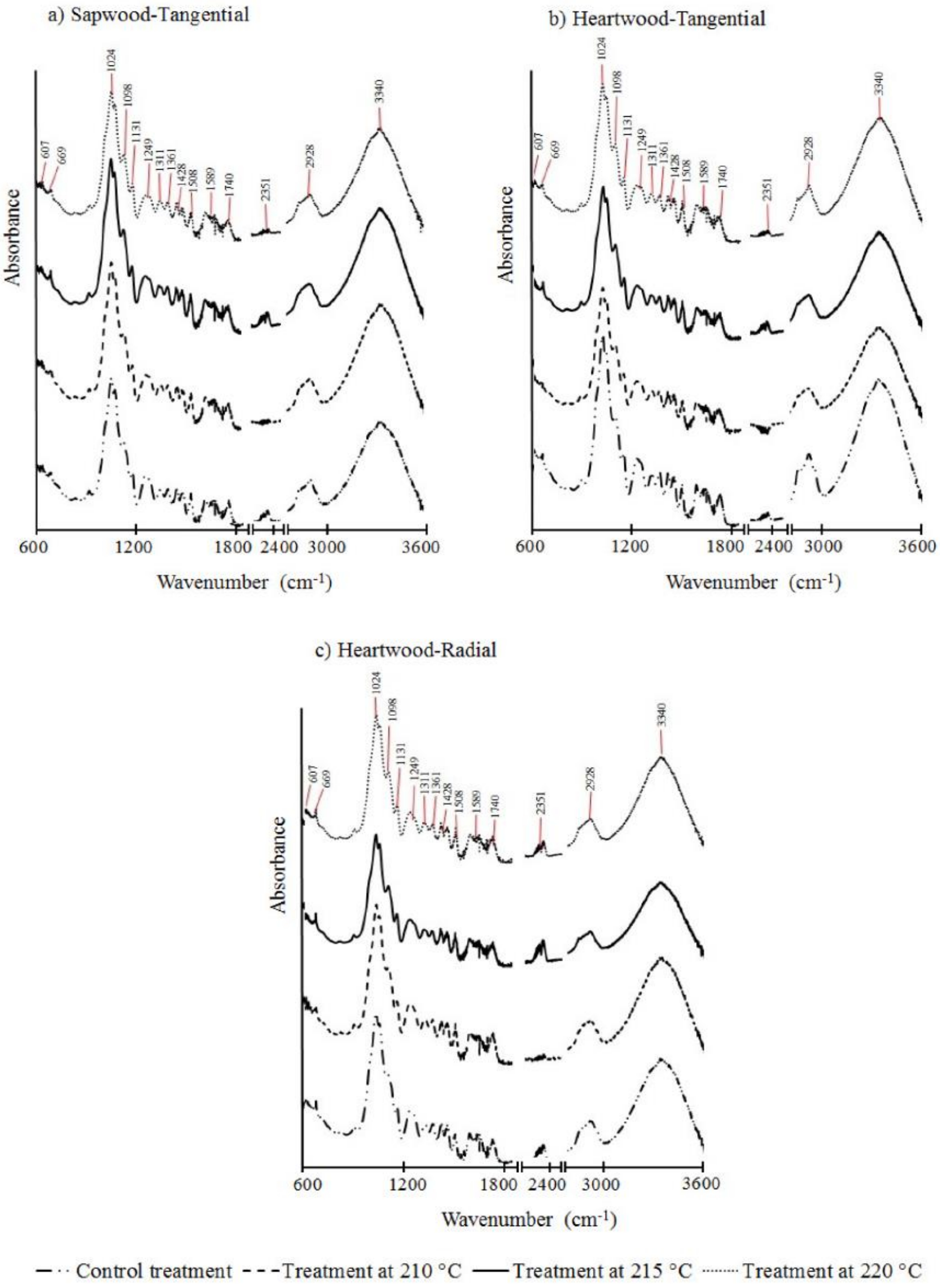

Figure 1. FTIR representation of the untreated and thermally treated teak wood at three temperature levels, for sapwood in tangential section (a), heartwood in tangential section (b) and heartwood in radial section pattern (c).

Figura 1. Representação FTIR da madeira de teca não tratada e tratada termicamente a três níveis de temperatura, para alburno na seção tangencial (a), cerne na seção tangencial (b) e cerne na seção radial (c).

Color change in wood

Color parameters of the surface of thermally treated teak wood is presented in Table 3. A growth trend can be appreciated in the total color change (ΔE^*) as the temperature of each thermal treatment process rises, where the samples of sapwood with tangential pattern changed the highest total color changes. Also, ΔE^* is greater in wood with radial pattern than in wood with tangential pattern in the different temperatures. Color changes are shown visually in figure 2, where it is observed that when temperature increases, teak wood tends to darken and wood color presented a tendency to color uniformity. Regarding the different parameters composing color, it was observed that ΔL^* and Δb^* follow a trend of increase in negative, the higher the treatment temperature is; whereas values for Δa^* had a different trend increasing from treatment at 210 °C to 215 °C and later with a decrease at 220 °C.

Table 3. Color change parameters (ΔL^*, Δa^*, Δb^*) and total color change (ΔE^*) of the thermally treated teak wood at three different temperatures according to wood type and cutting section.
Tabela 3. Parâmetros de alteração da color (ΔL^*, Δa^*, Δb^*) e alteração total da cor (ΔE^*) da madeira de teca tratada termicamente sob três diferentes temperaturas de acordo com o tipo de madeira e plano de corte.

Wood type	Cutting section	Treatment	Color parameters			
			ΔL^*	Δa^*	Δb^*	ΔE^*
Sapwood	Tangential	210 °C	-33.0 (±3.3)A*	5.3 (±1.2)A	-3.3 (±3.6)A	33.8 (±3.4)A
		215 °C	-35.3 (±3.2)B	5.3 (±1.5)A	-3.6 (±3.9)AB	36.1 (±3.3)B
		220 °C	-40.0 (±7.8)B	3.9 (±1.2)A	-6.7 (±4.2)B	40.9 (±7.8)B
Heartwood	Radial	210 °C	-21.4 (±3.0)A	0.9 (±1.9)A	-6.4 (±3.3)A	22.7 (±3.1)A
		215 °C	-26.0 (±3.2)B	1.1 (±1.5)A	-9.0 (±3.0)AB	27.6 (±3.5)B
		220 °C	-26.4 (±5.1)B	-0.7 (±1.7)A	-11.8 (±3.0)B	29.2 (±5.1)B
	Tangential	210 °C	-19.5 (±4.3)A	1.1 (±2.4)A	-7.3 (±7.0)A	21.9 (±5.2)A
		215 °C	-21.8 (±3.1)B	1.2 (±2.9)A	-9.7 (±4.1)AB	24.3 (±3.4)B
		220 °C	-24.9 (±3.6)B	-0.7 (±2.7)A	-9.0 (±4.7)B	26.9 (±4.2)B

* Value in parentheses indicates standard deviation and values with similar letters indicate no statistically significant difference between treatments for 95 % confidence.

Figure 2. Color changes in the thermally-treated teak wood, at three different temperatures.
Figura 2. Alteração da cor da madeira de teca tratada termicamente sob três diferentes temperaturas.

Values obtained for parameter ΔL^* were entirely negative (Table 3), which reduces the clarity of the thermally treated wood, situation that is visually checked with color change (Figure 2). In this case, values obtained indicate that there was a darkening of wood. However, decreasing of clarity occurs more in sapwood than in heartwood timber. Within this heartwood type, major change happens in the radial pattern and minor change in the tangential pattern (Figure 2).

Meanwhile, parameter Δa^* was not affected statistically by the treatment applied (Table 3), indicating that temperature applied to wood achieves a small variation of redness, but, color change in this hue is not significant. Variation occurred with a small increase in this parameter value after thermal treatment; showing an increase in the reddish hue of thermal-treated wood. The only exception occurred in heartwood with radial and tangential pattern for treatment at 220 °C that showed a trend to get a more greenish color (diminished value of a^*).

Also, a slight increase occurred in parameter Δb^* as temperature increases (Table 3), indicating a decrease in the yellow hue of wood, where the major change happens when temperature increases in sapwood with tangential pattern.

Finally, it was found that at higher temperature there is more color change (ΔE^*) regardless of the type of wood or cutting section (Table 3). However, samples of heartwood have higher values of

ΔE*. This behavior occurs due to the emergence of several chemical changes in wood when thermal-treatment is applied (CHEN et al., 2014). During thermal-treatment process, some components of wood are degraded or modified through reactions such as dehydration, hydrolysis, oxidation, decarboxylation and trans-glycosylation (KOCAEFE et al., 2008), which are more noticeable in sapwood that generally has a clearer tonality.

Presence of functional groups as carbonyl, quinoid structures, structures of lignin or hemicellulose, may play an important role forming colored substances during thermal treatment (YILDIZ et al., 2013), which can be seen in the signals obtained in bands at 1508 cm^{-1}, 1428 cm^{-1} and 1740 cm^{-1} (Figure 2) associated to carbonyl groups. Because of this, it is reasonable to assume the existence of chemical changes in lignin and hemicellulose produced by exposing the material to heat. Heat causes a change of color on the surface of wood due to residual extractives (CHEN et al., 2014). These chemical changes may be due to modification of lignin and hemicellulose in the structure of wood that correspond to the signals obtained in bands at 1131 cm^{-1}, 1098 cm^{-1}, 1024 cm^{-1}, 669 cm^{-1}, and 1260 cm^{-1} (Figure 2) causing color changes in wood. Thermal-treated wood produces, as consequence, degradation of lignin and hemicellulose and a darkening in the color of wood, which is greater as temperature increases (HUANG et al., 2012).

Selecting the best treatment

Input percentage for the variables of shrinking and density tend to remain and decrease as thermal treatment temperature rises. For ΔE* of the samples, an increasing trend is observed as thermal treatment temperature increases (Table 4). Regarding total contribution of the variables together, treatment at 220 °C yielded the highest percentage with the greatest percentage amount; however, samples subjected to this treatment showed greater shrinking and lower density represented with lower percentage values (Table 4).

Table 4. Percentage contribution of density, shrinkage and total color change of the thermal treatment of teak wood at three different temperature levels on quality parameter of best temperature.

Tabela 4. Percentagem de contribuição de densidade, contração e alteração da cor de madeira termo-tratada de *Tectona grandis* em relação a diferentes níveis de temperatura no parâmetro a qualidade da melhor temperatura.

Treatment	Shrinking (%)	Density (%)	ΔE* (%)	Total (%)
210 °C	11.05	12.00	10.00	33.05
215 °C	11.05	9.00	12.50	32.55
220 °C	7.89	9.00	17.50	34.39

CONCLUSIONS

Results obtained for the evaluated parameters showed that thermal treatment using pilot plant (Valutec®) at 220 °C had better results than at 210 and 215 ºC, reflecting the highest total percentage obtained from the selection variables defined. Although this is the treatment with highest color change percentage, it presents a higher shrinking and a lower density.

The evaluation of thermally treated wood properties showed that wood density decreased and total color change (ΔE*) increased with thermal treatment temperature increases; the greatest shrinking percentage was found in heartwood with tangential pattern and thermal-treated at 220 °C. Finally, FTIR spectra showed that chemical modifications of lignin, cellulose and hemicelluloses are produced in thermal treatment of teakwood, especially with temperature increasing.

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Durability of Thermally Modified Wood of *Gmelina arborea* and *Tectona grandis* Tested under Field and Accelerated Conditions

Róger Moya*, Lucia Fallas-Valverde, Alexander Berrocal and Dawa Méndez-Álvarez

Escuela de Ingeniería Forestal, Instituto Tecnológico de Costa Rica, Cartago 159-7050, Costa Rica

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ABSTRACT: This study evaluated the durability in terms of decay and mechanical resistance of thermally modified (TM) wood of *Tectona grandis* and *Gmelina arborea* treated at 160, 180, 200 and 220 °C. The TM wood of both species treated above 200 °C and 180 °C respectively presents lower weight loss (WL) after 300 days exposure in field and accelerated testing. It was also found that in field testing over 180 °C, the module of elasticity (MOE) and module of rupture (MOR) of the exposed and unexposed stakes of TM wood were not affected. Accelerated tests showed that the loss in flexural resistance was reflected more in the MOR than in the MOE. Finally, the accelerated and field tests showed that *G. arborea* and *T. grandis* TM wood treated at 180, 200 and 220 °C present statistically similar values of WL and flexural mechanical resistance.

KEYWORDS: Biodeterioration, tropical species, decay, thermal modification, teakwood

1 INTRODUCTION

Wood is a biomaterial susceptible to degradation by biotic and abiotic agents. A number of treatments have been applied to wood seeking to improve decay resistance and dimension stability; for example, thermal modification of wood. In this process, the wood is exposed to temperatures between 100 and 220 °C for several hours under nitrogen conditions. Thermal modification of wood essentially involves controlled degradation of the wood, primarily resulting in the destruction of hemicelluloses [1].

Thermal modification affects multiple properties of the wood [2]: reduces its mechanical resistance, increases its resistance to decay, and affects its moisture content, among other properties [3]. Esteves and Pereira [2] made an extensive review of the decay resistance of TM wood of various species. All studies about decay resistance in this review have used accelerated tests, focusing mainly on species from temperate climates.

The environmental conditions of tropical regions, such as high temperatures and rainfall throughout the year as occur in Costa Rica, enable the development of a large variety of timber species from forest plantations [4]. Two of these forest species, *Tectona grandis* and *Gmelina arborea*, have been successfully planted in forest plantations in Costa Rica [5, 6]. Nevertheless, the high amount of sapwood and heartwood of low durability [5, 6] makes both species susceptible to degradation. These woods are already being thermo-treated to improve their resistance to biodegradation [7].

Timber susceptibility to biodegradation is a disadvantage [2]. It has long been recognized that deterioration is more rapid in warm, moist climates than in cool or dry climates; thus, 3–5 years have generally been considered sufficient data for tropical regions [2]. On the other hand, knowledge about the loss of durability of TM wood in accelerated tests is also limited. Field test studies on durability of TM wood have been conducted under conditions other than those of tropical climate [8].

Therefore, the aim of the present study is to evaluate the durability of TM wood of *Tectona grandis* and *Gmelina arborea* from forest plantations in Costa Rica at 5 different temperatures. To this end, measurements were carried out on the loss of mechanical resistance of the TM wood after 300 days exposure in a field of stakes in two different sites; in addition, accelerated tests were conducted to evaluate resistance to fungi at various times, using brown- and white-rot fungi (*Lenzites acuta* and *Trametes versicolor*, respectively).

**Corresponding authors*: rmoya@itcr.ac.cr

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2 MATERIALS AND METHODS

2.1 Origin and Characteristics of the Material Used and Sampling

The wood of *Tectona grandis* and *Gmelina arborea* used was obtained from trees from the second thinning of fast-growth plantations of 11 and 8 years old, respectively. The plantations were located in the Northern Zone of Costa Rica (Latitude: 9° 50′ 59″ N and Longitude: 83° 54′ 37″ W), and belong to Ethical Forestry S.A. Thirty boards of about 7.5 cm wide × 2.5 cm thick × 2.50 m long were taken from the TM wood only. The trees were harvested in July and during the rainy season. Heartwood boards were divided into 5 parts (samples of 7.5 cm wide × 2.5 cm thick × 50 cm long). It is noteworthy that only samples of sapwood timber were available, since it was not possible to get the radial pattern.

2.2 Thermal Treatment Process

Dried lumber, with approximately 12% moisture content, was thermally treated at four different temperature levels: 160 °C, 180 °C, 200 °C and 220 °C. For each treatment, 30 boards were selected and 30 other samples were left untreated. Each thermal treatment process was made independently and performed under anoxic conditions. The boards were introduced and stacked into a Valutec® thermowood pilot plant [9]. The thermal treatment process started with drying for approximately 17 hours at a temperature ranging from 0 °C to 130 °C to obtain 0% moisture content (MC). Subsequently, the temperature was increased from 130 °C to the temperature defined for each type of thermal treatment (160 °C, 180 °C, 200 °C and 220 °C) and maintained during 6 hours. Next, a conditioning stage was applied during 7 hours; steam, water and temperature were applied to moisturize timber and achieve approximately 6% in MC. Finally, a cooling process was applied during 3 hours. The different treatments were defined as follows: control untreated wood and thermal treatments at 160 °C, 180 °C, 200 °C and 220 °C.

2.3 Evaluation of Durability of Thermally Modified Timber (TMT) Wood Stakes in Field Tests

From thermo-treated boards at 12% MC, 50 defect-free samples of 2 × 2 × 30 cm^3 from each temperature (Table 1) in each species were cut according to the ASTM D-1758-02 standard method A [10]. The samples were in contact with weed-free soil for 300 days. To this end, pots with fertile soil used in the nursery for plant reproduction were employed (Figure 1a). A total of ten stakes of 2 cm × 2 cm × 30 cm, 5 stakes of *T. grandis* and 5 stakes of *G. arborea*, corresponding to the treatments applied (4 temperatures: 160, 180, 200 and 220 °C; and untreated) were used per pot. In total, 20 different pots were filled. The stakes were buried 15 cm deep in the soil. The pots were separated into two groups of 10 pots each, each group exposed to different environmental conditions in two different sites. The first site was at 9° 50′ 59″ N and 83° 54′ 37″ W (Field tests weather condition 2) and the second site was at 10° 11′ 22″ N and 84° 31′ 23″ W (Field tests weather condition 2).

2.4 Evaluation of TMT Wood Stakes in Field Tests

After 300 days exposure, the mechanical resistance of TM wood was evaluated. Samples were extracted and placed under controlled conditions (temperature: 22 °C; relative humidity: 66%) to obtain 12% MC. Next, the stakes were weighed and their dimensions were measured. Static flexure was used to determine the mechanical resistance using the destructive method. For this, Tinius Olsen Horizon H10kT equipment was used, employing a span of 25 cm between supports and applying the load exactly on the ground line of the

Table 1 Experimental parameters determined and the number of samples for each test in TM wood of *Tectona grandis* and *Gmelina arborea*.

Test	Experimental parameters determined	Temperature of thermo-treatment (°C)					Total samples per specie
		Un-treated	160	180	200	220	
Resistance in flexural test	MOE and MOR in flexure and WL after 300 day of exposure	10	10	10	10	10	50
Accelerated test with stakes	MOE and MOR in flexure and WL each 7 days until 28 days	21	21	21	21	21	84
Accelerated soil block test	WL after 16 weeks of exposure	30	30	30	30	30	150

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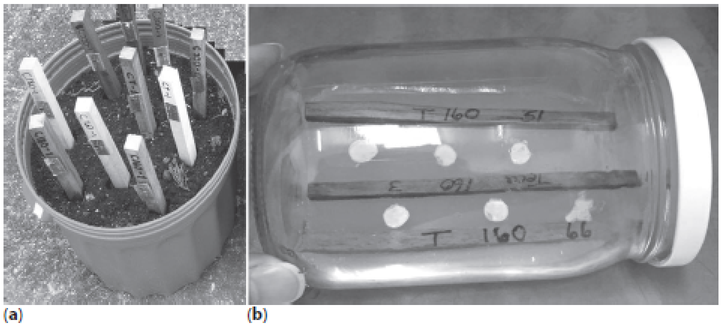

Figure 1 (**a**) Pots to test durability of TM wood of *Tectona grandis* and *Gmelina arborea* in field tests in two climatic conditions. (**b**) Accelerated decay test utilized in small TM wood samples of *Tectona grandis* and *Gmelina arborea* for flexural resistance determination.

stakes at 1 mm/min speed. The modulus of elasticity (MOE) and the modulus of rupture (MOR) commonly obtained in this type of test were determined.

2.5 Evaluation of Mechanical Resistance and Weight Loss for Accelerated Methods

This method was adapted from the method proposed by Silva *et al*. [11] to measure durability of wood-plastic composites by means of accelerated tests. In this test, small samples of 185 TM wood (6 mm × 6 mm × 100 mm) for each treatment (4 temperatures: 160, 180, 200, 220 °C; and untreated) and each one of the species were placed into a jar containing malt-agar culture (Figure 1b) previously inoculated with *Trametes versicolor* (white rot) and *Lenzites acuta* (brown rot). Then, 84 samples were selected for exposure to each fungus and 21 samples designated as control were separated and kept unexposed to the fungi. Three samples were placed into each jar with solidified malt-agar medium (Figure 1b). One week later, 21 wood samples per each species and fungus type were removed. Sample extraction (21 samples/species/fungus) continued in weeks 2, 3 and 4 of exposure. All of the samples extracted and the samples conditioned at 12% MC were then weighed again to determine WL due to exposure to the fungus (Equation 1). Next, the flexure test was performed using a Tinius Olsen H10kT universal testing machine with a span of 8 cm and a loading speed of 0.27 mm/min. Finally, all samples were placed at 105 °C for 24 hours and their dry weight was determined with an analytical balance.

$$Weightloss(\%) = \frac{Initial\ weight - final\ weight}{Initial\ weight} * 100 \tag{1}$$

2.6 Decay Resistance in Accelerated Soil-Block Test

Decay resistance specimens measuring 2.5 × 2.5 × 2.5 cm were cut from 5 different treatments: 4 temperatures (160, 180, 200 and 220 °C) and untreated. Three hundred blocks were extracted from TMT wood boards (5 treatments × 2 fungus × 30 samples). The white-rot fungus *T. versicolor* and brown-rot *L. acuta* were again used for testing natural decay resistance following the ASTM standard D2017-81 [12]. The relative decay resistance of each soil-block test was measured as the WL percentage (Equation 1) during a 16 week exposure to the fungi.

2.7 Statistical Analysis

Firstly, a regression analysis was performed between WL and thermo-treatment temperature for the TM wood stakes that were placed in field tests for 300 days. Then, a variance analysis (ANOVA) and Dunnett's average test ($P < 0.05$) was applied to the MOE and MOR of the flexural test of the same stakes. These analyses were performed in order to determine the differences between these parameters in the case of the untreated stakes (control stakes) relative to TMT wood stakes from different temperature treatments. Lastly, an ANOVA was applied to determine the WL differences between the two types of fungi (*L. acuta* and *T. versicolor*) obtained from the accelerated test. The thermo-treatment temperatures (160 °C, 180 °C, 200 °C and 220 °C) of the wood were the independent variables of the model and the WL was defined as the dependent variable; this analysis was applied to each fungus separately. The Tukey test ($P < 0.05$) was used to confirm the presence of significant differences between treatments. All statistical analyses were computed by SAS software.

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3 RESULTS

3.1 Evaluation of Stakes in Field Tests

The WL evaluation of the TM wood stakes at the end of the 300 days exposure showed that *G. arborea* TM wood (Figure 2a) presented greater WL than *T. grandis* TM wood (Figure 2b). Likewise, TM wood stakes exposed to weather condition 2 in the field tests presented greater WL than the stakes exposed to weather condition 1 in the field tests. A major aspect to observe is that WL at the end of the 300 days exposure decreases exponentially with the thermo-treatment temperature.

A small change was observed regarding WL in *G. arborea* stakes between stakes treated at 160 °C and 180 °C. However, TM wood stakes treated at 200 °C and 220 °C presented lower WL than for the previous temperatures (Figure 2a). As for *T. grandis* TM wood, a fall in the WL was observed in untreated stakes and TM wood stakes obtained at 160 °C. The differences between the other temperatures is small and relatively constant (Figure 2b).

The determination of the flexural resistance by means of the destructive method showed that the thermo-treatment reduces the values of MOR and MOE, compared to untreated wood (Table 2). In *G. arborea*, reduction of these values occurs mostly where wood was treated with temperatures of 200 and 220 °C (Table 2), whereas in *T. grandis*, MOR and MOE diminished progressively with increasing temperature.

As for mechanical resistance of stakes after exposure to the soil for 300 days, MOR and MOE diminished significantly in untreated stakes and in *G. arborea* TM wood stakes treated at 160 °C. On the other hand, no significant diminution was observed in MOR and MOE in TM wood treated with other temperatures (180, 200 and 220 °C) relative to TM wood at the same temperatures unexposed to the soil at both sites of exposure (Table 2). Meanwhile, *T. grandis* wood showed significant reduction of MOE in untreated stakes and stakes treated at 160 °C at both sites of exposure. In addition, significant diminution of MOR was observed in wood treated at 180 °C in field test 1 (Table 2).

After comparing the values of mechanical resistance of TM wood after exposure to soil contact for 300 days with the values of untreated wood unexposed to the soil, the highest loss of MOR and MOE evidently occurs in wood of untreated stakes of both species (Figure 3). Additionally, except for the MOR in *T. grandis* (Figure 3b), the least diminution of loss in MOE and MOR occurs in TM wood stakes treated at 160 °C. Finally, in the remaining temperatures, loss of resistance increases with increasing temperature. However, this reduction in mechanical resistance is due to thermo-treatment instead of the wood presenting higher degradation, since wood thermo-treated at over 180 °C presented no significant difference regarding resistance after 300 days exposure.

3.2 Evaluation of Mechanical Resistance and Weight Loss for Accelerated Methods

The WL increased in untreated and TM wood stakes of *T. grandis* and *G. arborea* with time of exposure to *T. versicolor* at 160, 180 and 200 °C, whereas at 220 °C, WL of TM wood remains constant with time (Figure 4a–c). As for TM wood exposed to *L. acuta* (Figure 4b–d), most WL occurs in weeks two and three for

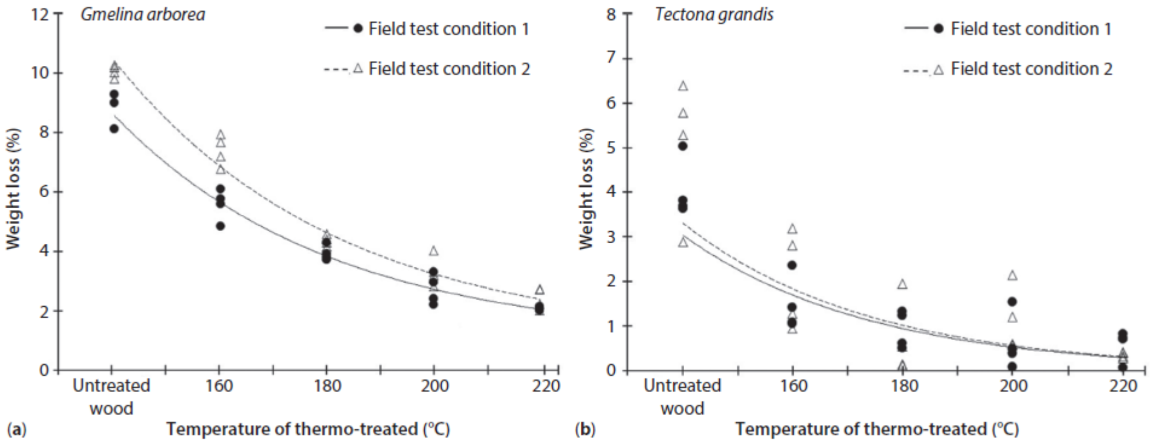

Figure 2 Weight loss percentage of TM wood stakes of *Gmelina arborea* (**a**) and *Tectona grandis* (**b**) exposed to the soil in two field tests weather conditions.

Table 2 Comparison of module of elasticity and module of rupture of TM wood stakes of *Gmelina arborea* and *Tectona grandis* exposed 300 days to the soil in two field test weather conditions relative to untreated and unexposed stakes.

Field test weather condition	**Treatment**	**TM wood of *Gmelina arborea***				**TM wood of *Tectona grandis***			
		MOE unexposed (GPa)	**MOE exposed (GPa)**	**MOR Unexposed (MPa)**	**MOR Exposed (MPa)**	**MOE Unexposed (GPa)**	**MOE Exposed (GPa)**	**MOR Unexposed (MPa)**	**MOR Exposed (MPa)**
Field 1	Untreated	6.92^{A}	4.96^{B}	68.9^{A}	58.9^{B}	9.62^{A}	7.78^{B}	107.5^{A}	90.5^{B}
	160 °C	6.22^{A}	5.64^{B}	61.9^{A}	60.2^{B}	9.18^{A}	8.86^{B}	106.9^{A}	93.8^{B}
	180 °C	6.32^{A}	5.59^{A}	56.4^{A}	57.9^{A}	8.74^{A}	8.76^{A}	102.7^{A}	101.5^{A}
	200 °C	5.67^{A}	5.55^{A}	53.9^{A}	52.8^{A}	8.58^{A}	8.51^{A}	98.6^{A}	98.9^{A}
	220 °C	5.68^{A}	5.65^{A}	54.7^{A}	54.1^{A}	8.38^{A}	8.33^{A}	97.8^{A}	98.5^{A}
Field 2	Untreated	6.91^{A}	4.32^{B}	67.4^{A}	54.4^{B}	9.15^{A}	7.92^{B}	105.5^{A}	90.6^{B}
	160 °C	6.38^{A}	5.55^{B}	64.0^{A}	59.7^{B}	8.94^{A}	8.34^{B}	103.0^{A}	93.9^{B}
	180 °C	6.06^{A}	5.76^{A}	59.3^{A}	59.1^{A}	8.86^{A}	8.68^{A}	100.0^{A}	98.9^{A}
	200 °C	5.75^{A}	5.76^{A}	56.9^{A}	55.9^{A}	8.18^{A}	8.31^{A}	96.1^{A}	95.6^{A}
	220 °C	5.62^{A}	5.53^{A}	55.6^{A}	57.0^{A}	8.03^{A}	8.07^{A}	95.9^{A}	97.8^{A}

Note: Average values identified with different the letters are statistically different at $\alpha = 99\%$.

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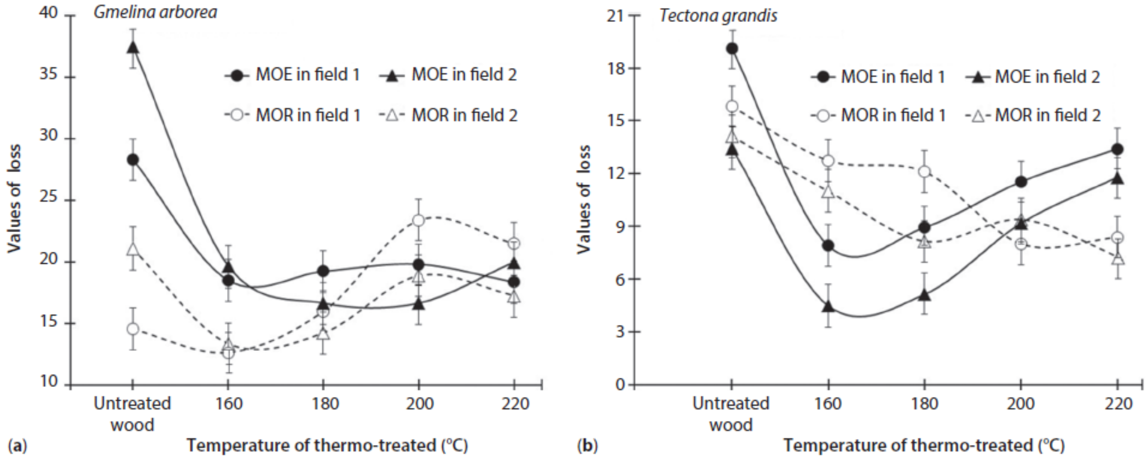

Figure 3 Loss of module of elasticity (**a**) and module of rupture (**b**) after 300 days exposed in soil for TM wood stakes of *Gmelina arborea* and *Tectona grandis* treated with different temperatures.

all treatments in two species; then, WL values remain constant for 21 and 28 days. Likewise, despite increasing their WL in the four evaluations, stakes thermo-treated at 220 °C present the lowest WL values.

The MOR variation showed the opposite effect, reducing its value with time of exposure to both *T. versicolor* and *L. acuta* in both species (Figure 5). As for the various temperatures, the TM wood samples at higher temperatures (200 and 220 °C) showed greater mechanical resistance to *T. versicolor* compared to untreated samples in both species. In addition, the lowest mechanical resistance was perceived in the thermo-untreated samples, where the decrease was high in relation to day of exposure (Figure 5a,c). Regarding *L. acuta*, the lowest values of MOR were observed at high temperatures (200 and 220 °C), while for the remaining temperatures few differences were observed between untreated and TM wood stakes (Figure 5b,d).

A different behavior was observed concerning the value of MOE, which was slightly affected by the time of exposure to the fungi, mainly in *T. grandis* (Figures 6a,b) and a higher change in *G. arborea* (Figures 6c,d). The lowest value of MOE regarding both types of fungi was observed in untreated stakes, whereas the highest values appeared for TM wood stakes at 200 and 220 °C. The samples thermo-treated at 160 and 180 °C present intermediate values between thermo-untreated stakes and TM wood stakes at 200 and 220 °C (Figure 6).

3.3 Decay Resistance in Accelerated Soil-Block Tests

According to the results obtained in the accelerated soil-block tests for *G. arborea*, the values of WL with *L. acuta* were lower than those obtained with *T. versicolor*. In addition, TM wood at 160 °C and 180 °C presented no significant differences relative to the control regarding *L. acuta* (Figure 7a). The WL values of TM wood at 200 and 220 °C were different among them, and statistically lower than in TM wood at 160 °C and 180 °C (Figure 7a). As for *G. arborea* TM wood colonized by *T. versicolor*, no differences were found in the WL between untreated wood and thermo-treated wood at 160 °C, nor were any differences observed between TM wood treated at 160 °C and 180 °C regarding WL, although differences did appear between untreated and TM wood treated at 180 °C. The lowest statistically compared WL in *T. versicolor* was for TM wood treated at 200 and 220 °C, showing no significant differences in wood at these two temperatures (Figure 7b).

The results from the accelerated soil-block test in *T. grandis* TM wood exposed to colonization by *L. acuta* and *T. versicolor* showed once again that most degradation occurred with *T. versicolor* (Figure 7c,d). The TM wood treated at 160 °C showed no significant differences in WL with both fungi compared to untreated wood (Figure 7c,d). Meanwhile, TM wood treated at 180 °C and 200 °C did not present significant differences in WL relative to *L. acuta*. The TM wood treated at 220 °C presented the lowest statistical value of WL (Figure 7c). On the other hand, TM wood treated at 180, 200 and 220 °C showed no significant differences in WL due to degradation by colonization of *T. versicolor* (Figure 7d).

4 DISCUSSION

The WL found for *G. arborea* and *T. grandis* subsequent to 300 days exposure to the field test varied within

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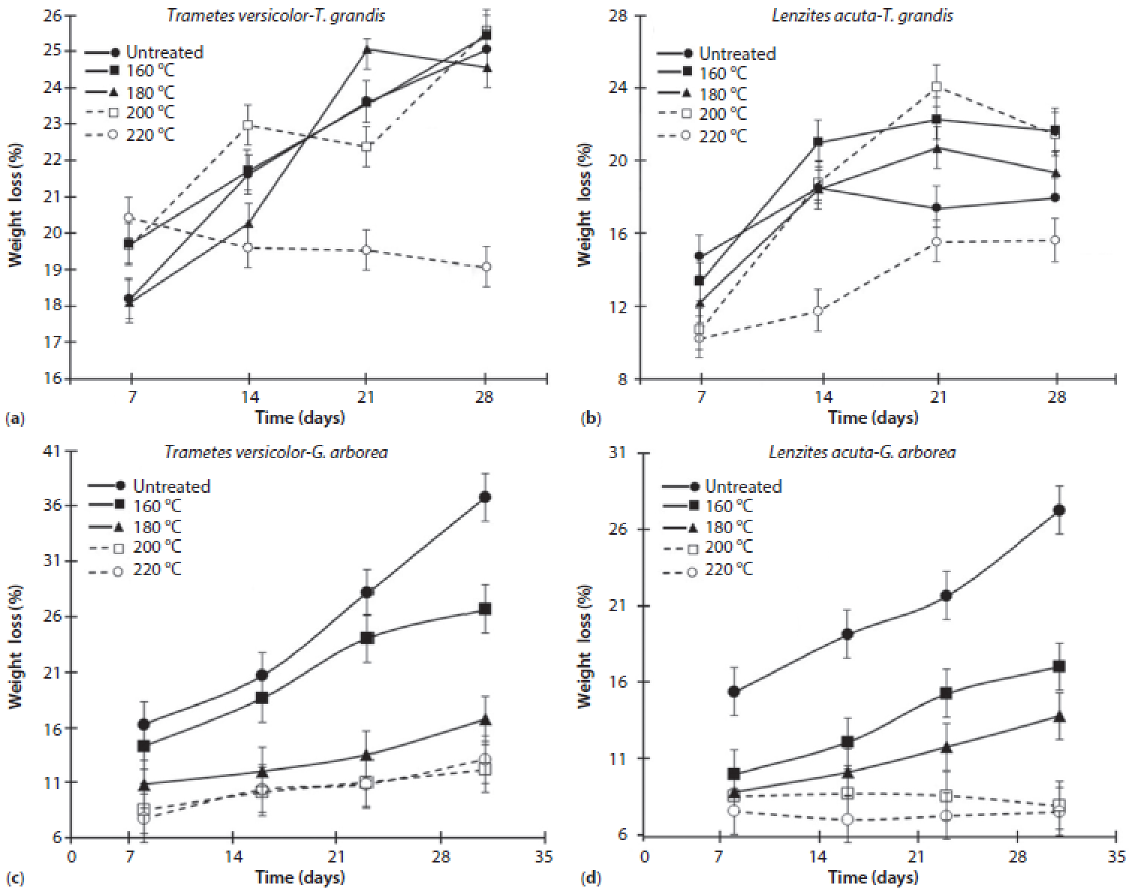

Figure 4 Variation of weight loss percentage of TM wood stakes of *T. grandis* (**a,b**) and *G. arborea* (**c,d**) exposed to accelerated test with *T. versicolor* and *L. acuta* during several days.

the range of 1.0 to 11.0% (Figure 2). For both species, WL of TM wood stakes at low temperature (160 °C) or stakes without thermo-treatment was higher compared to WL of TM wood stakes treated at higher temperatures. Differences were also observed between these two species concerning WL; thermo-treatment of *T. grandis* at higher temperatures increased its decay resistance against the deterioration agents in the field test compared to *G. arborea* treated under the same conditions (Figure 2).

The thermal modification of temperate species has been proven to enhance wood durability [7, 13]. The improvement of resistance to fungal attack is due to modifications of the chemical components (cellulose, lignin and hemicellulose) of the wood that reduce the vulnerability of the material to biological degradation [14]. In addition, degradation of these polymers by thermal modification also decreases water absorption, which limits shrinking and swelling, as well as absorption of water, which may also be conducive to diminished fungal growth [13].

There are some aspects that can explain the variations of decay resistance due to temperature. Firstly, water absorption creates conditions for the development of fungi, which in the end will affect the decay resistance. According to Boonstra *et al*. [15], with thermal treatment at low temperature, the low thermal and moisture conductivity of wood leads to high moisture variation close to the surface. As a result, conditions that favor fungi development appear [16], therefore the diminished durability of wood at low temperatures.

Another aspect that may explain the difference in durability of TM wood under multiple temperatures is the formation of several chemical components in the wood. High temperatures improve decay resistance,

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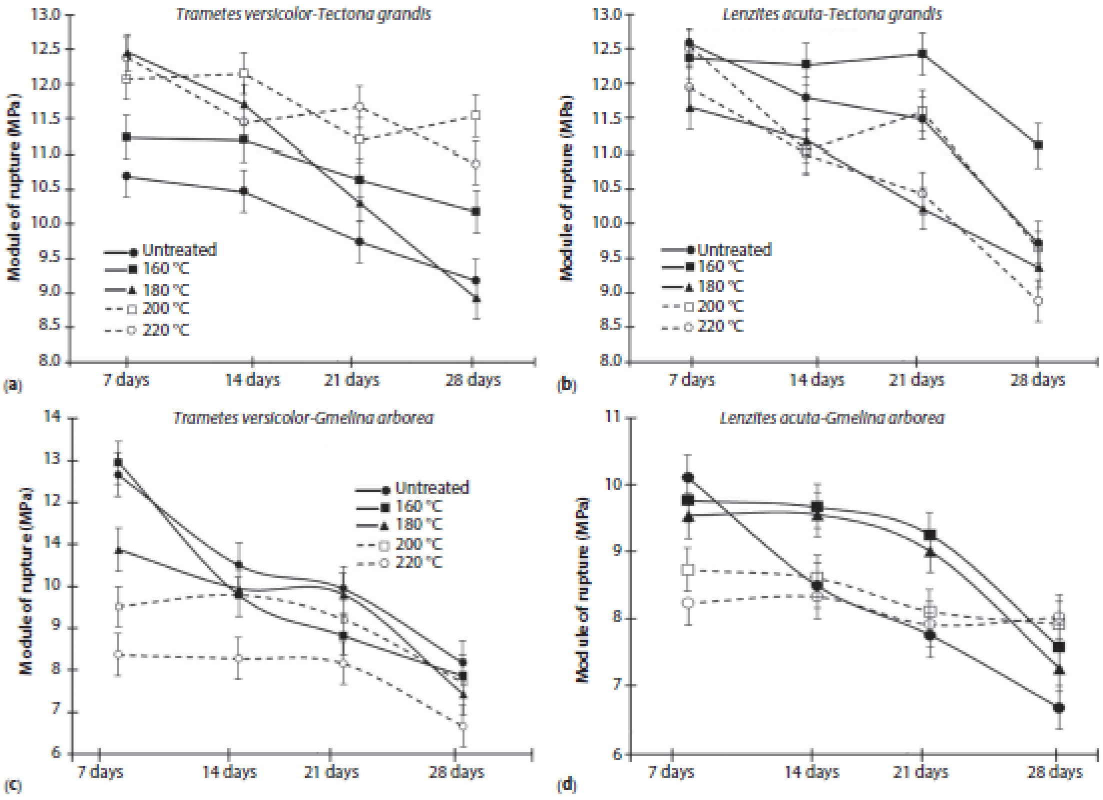

Figure 5 Variation of module of rupture of TM wood stakes of *Tectona grandis* (**a**,**b**) and *Gmelina arborea* (**c**,**d**) exposed to accelerated test with *T. versicolor* and *L. acuta* during several days.

as observed in this study (Figure 2) and in other studies on species from temperate climates [14]. Hakkou *et al*. [14], Trevisan *et al*. [17], and Del Menezzi *et al*. [18] stated that polymer degradation and chemical modification, well known to happen above 200 °C, are more plausible reasons to explain the durability improvement. Therefore, the adequate decay resistance obtained in both species in this study agrees with the findings of the above authors.

It was also observed that loss of decay resistance occurred in both thermo-untreated and TM wood. Both MOE and MOR of stakes thermo-treated at 160 °C and untreated exposed 300 days in the soil in two field test weather conditions (Table 2), and MOE and MOR of stakes of both species exposed to accelerated test with *T. versicolor* and *L. acuta* for several days (Figures 5 and 6), decreased with time. Loss of mechanical resistance in stakes is caused by the degradation of polymers, mainly cellulose and hemicellulose [19]. During wood decay, hemicellulose side chains such as arabinose and galactose are degraded first; afterwards, the main-chain hemicelluloses of mannose and xylose are mineralized [2]. Microscopic consequences of degradation of hemicellulose are produced by the development of cavities in the secondary walls of wood fibers, or the erosion of the wood cell wall outward from the cell lumen [19]. Consequently, decomposition of cellulose chains, the presence of cavities and the erosion in the cell wall produce structural weakening of the wood [20].

The WL with time (Figure 4), especially when the wood is exposed to accelerated testing with specific fungus, such as *T. versicolor* and *L. acuta* (Figure 4a and 4b), can be explained by the process of degradation of the wood. At the initial stages of exposure to the fungus, cavities appear on the cell wall [19] that produce early losses of resistance of low magnitude. These cavities enlarge as the process of degradation continues, allowing fungus hyphae to penetrate the cell wall until reaching the lumen and further extend the cavities [20]. The progressive increase of cell wall degradation caused by the hyphae produces this gradual

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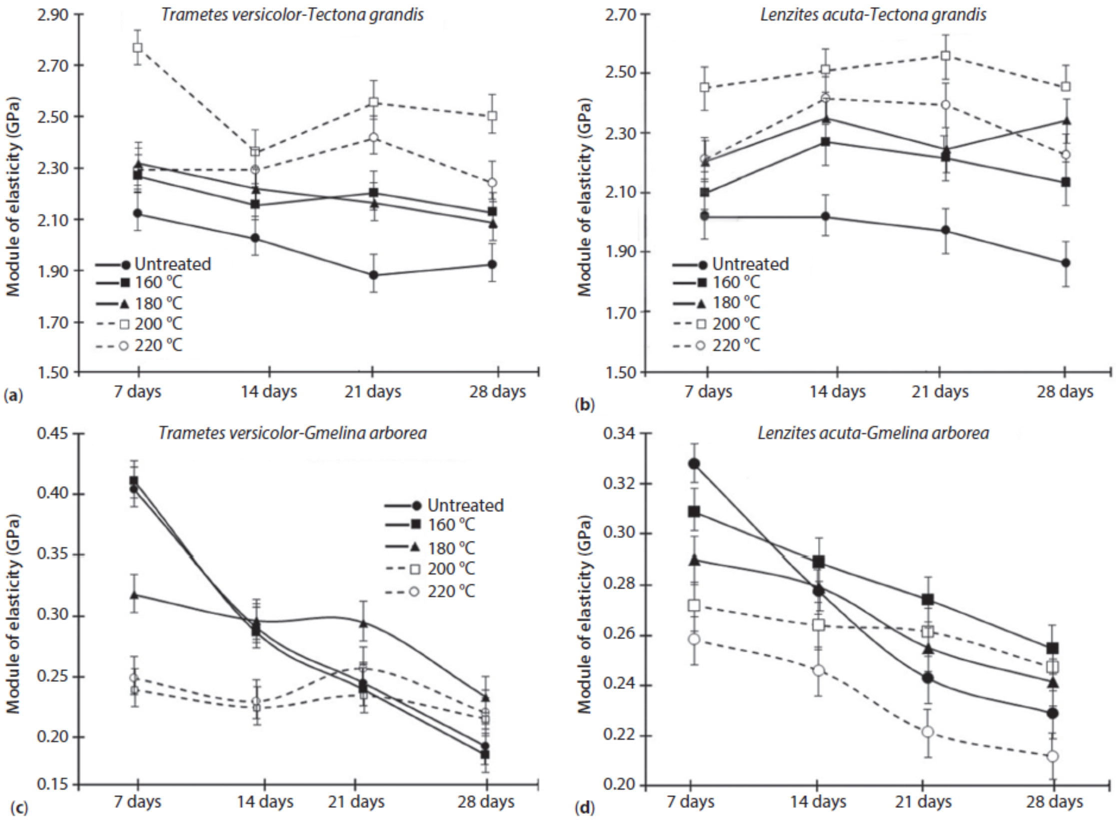

Figure 6 Variation of module of elasticity of TM wood stakes of *Tectona grandis* (**a,b**) and *Gmelina arborea* (**c,d**) exposed to accelerated test with *T. versicolor* and *L. acuta* during several days.

diminution of the mechanical resistance of the wood (Figures 5 and 6) due to weakened cell wall.

The TM wood showed loss of mechanical resistance after 300 days exposure to field test, compared to wood that is untreated and unexposed to field test. This should be taken with caution. First of all, it should be pointed out that thermo-treatment decreases the mechanical resistance of the wood, which was confirmed by the loss of mechanical resistance of the two species studied (Table 2); additionally, this loss is positively correlated to increasing thermo-treatment temperature [21]. This weakening is produced by the degradation of the polymers that compose the wood [3, 7, 22, 23]. Changes in the structure of the cell wall, such as the development of microcracks, also help weaken the mechanical resistance of the wood with thermo-treatment [24].

However, no statistical differences were observed in MOR and MOE of TM wood in the field test and stakes unexposed after 300 days of exposure (Table 2), mainly with respect to TM wood treated at temperatures above 180 °C in the two species evaluated (Figure 3). This indicates that loss of MOR and MOE in TM wood in relation to untreated wood and unexposed wood was due, not to biological degradation during soil exposure, but to natural loss of mechanical resistance caused by thermo-treatment. In both species evaluated, durability was achieved with TM wood at lower temperature (180 °C) and thermo-untreated wood. It was found that MOE and MOR of TM wood decreased after 300 days of exposure (Table 2; Figure 3). Low thermo-treatment temperatures, such as 160 °C, cannot completely modify the conditions of the cell wall nor the chemical components of the wood as to inhibit fungal attack [7, 21]; therefore, protection against biological degradation is not achieved as in wood treated at higher thermo-treatment temperatures.

One more important difference relative to loss of decay resistance of the two species evaluated in the test of TM wood exposed to accelerated test with specific fungi, such as *T. versicolor* and *L. acuta*, was that MOR presented greater loss (Figure 6a,b)

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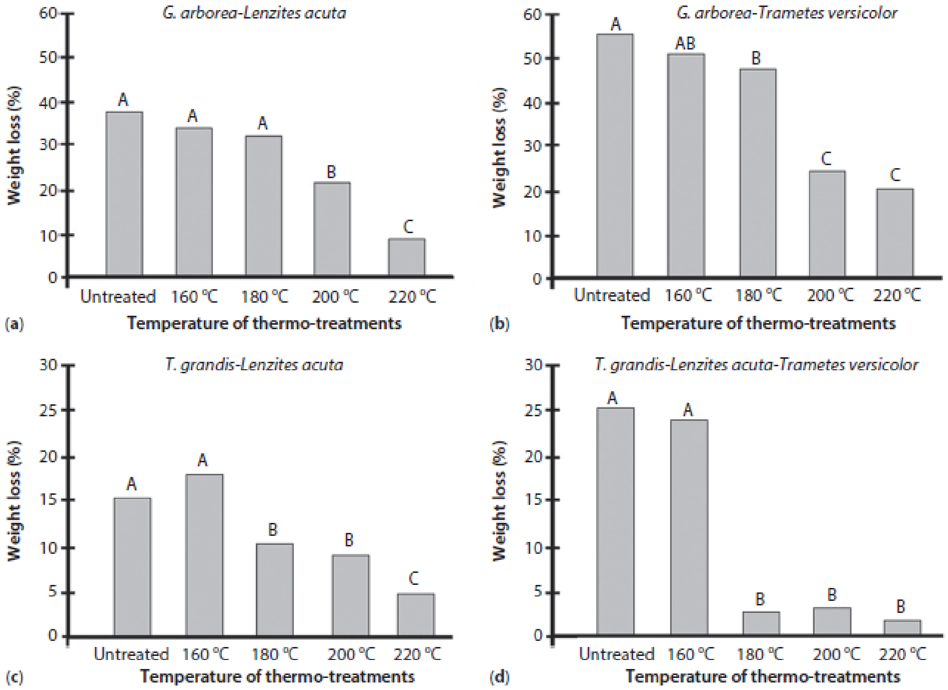

Figure 7 Weight loss percentage of TM wood of *Gmelina arborea* (**a**,**b**) and *Tectona grandis* (**c**,**d**) exposed to accelerated soil-block test with *T. versicolor* and *L. acuta*. Note: Average values identified with different letters are statistically different at α = 99%.

than MOE (Figure 6c,d; Figure 7c,d). According to Sandberg and Kutnar [1], the bending strength, which is a combination of tensile stress, compressive stress, and shear stress, is commonly used to compare mechanical properties of different processes. However, they mentioned that in general there is only a small change in MOE while a major decrease in MOR, independent of the process or the species. This same behavior was observed in the wood of *G. arborea* and *T. grandis*, also agreeing with tests carried out with Scots pine [25] and Norway spruce [26], which found that the MOR was more affected than the MOE.

Typical durability tests are accelerated tests under soil contact condition (Figure 7). According to the ASTM D2017 standard [12], the results of these tests can be classified into 4 different categories according to WL obtained after 16 weeks: highly resistant for WL under 10%; resistant for WL varying between 11% and 24%; moderately resistant if WL ranges from 25% and 44%; and slightly resistant to nonresistant if WL stands above 45% [12]. Consistent with the results obtained for WL, *G. arborea* TM wood presented higher values than TM wood of *T. grandis* (Figure 7a,b) and therefore *G. arborea* was generally classified as low durability. The TM wood of *G. arborea* treated at 160 °C and 180 °C and tested with two types of fungi was classified as moderately resistant, while *T. grandis* TM wood at the same temperatures and evaluated with the same types of fungi was classified as resistant. *G. arborea* TM wood treated at 200 and 220 °C was classified as resistant (WL between 11–24%), whereas TM wood of *T. grandis* at those same temperatures was classified as highly resistant (WL lower than 10%) with the two types of fungi.

An explanation of the improvement of resistance to fungi attack that results from the process of thermal modification of wood is that the formation of furfural might form toxic compounds and thermal treatment quickly degrades hemicelluloses [14, 17, 18].

The TM wood with the highest temperatures (200 and 220 °C) was classified as having the best durability compared to wood thermo-treated at lower temperature (Figure 7). Hermoso *et al.* [23] and Kocaefe *et al.* [21] explain that when higher temperature is applied during thermal treatment there is a reduction of hemicellulose content, humidity and other constituents, such as starch, fatty acids and lipids,

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which are essential for fungal growth. This chemical modification is explained by the fact that thermal treatments at above 200 °C modify the wood's structure, increasing its resistance to deterioration.

Lastly, TM wood showed greater WL with the white-rot *T. versicolor* than with the brown-rot *L. acuta* fungus (Figure 7), implying there was greater degradation of the structural polymers (lignin) of wood with the first fungus species. According to Kirk and Highley [27], this occurs because white-rot fungus has the capability to degrade wood cellulose and hemicellulose in greater proportion, after reducing lignin availability, while brown-rot fungus is more selective, degrading the cellulose of the cell wall only. The white- and brown-rot fungi removed the mannan, and usually xylan, faster than glucan, but the difference was not as pronounced for the white-rot as for the brown-rot organisms [27].

5 CONCLUSIONS

Thermal treatment in *G. arborea* and *T. grandis* wood from fast-growth plantations improves wood durability, noticeably increasing the decay resistance to accelerated tests with *T. versicolor* and *L. acuta* and in the field condition. However, the higher degradation occurred in *G. arborea* TM wood. The evaluation of the static flexural test showed that TM wood treated above 180 °C produced the lowest losses of MOE and MOR. But the accelerated tests showed more evidence of losses of flexural resistance than the field tests, the loss of resistance in the MOR being higher than in the MOE. According to results, the wood of *G. arborea* and *T. grandis* from fast-growth plantation thermo-treated at temperatures of 180, 200 and 220 °C, can be recommended. However, thermo-treatment at 160 °C in both species is not recommended.

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